Design and Performance of a Full-Scale Spray Calciner for Nonradioactive High-Level Waste Vitrification Studies

F. A. Miller

June 1981

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DESIGN AND PERFORMANCE OF A FULL-SCALE
SPRAY CALCINER FOR NONRADIOACTIVE HIGH-
LEVEL WASTE VITRIFICATION STUDIES

F. A. Miller

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Pacific Northwest Laboratory
Richland, Washington 99352

SUMMARY

Since 1977, the Pacific Northwest Laboratory has tested a large-scale spray calciner for the solidification of simulated high-level neutralized and acid radioactive wastes. This is a report of valuable operating experience gained during 1830 h of processing time in which ~390,000 L of simulated wastes were solidified.

Experience from spray calciner operations has demonstrated that atomizing-nozzle flow conditions that produce 100- μ median-volume-diameter or smaller droplets are required for large-scale spray calciners. Both internal- and external-mix nozzles have been tested. Due to its lower airflow requirements and the fewer large droplets produced, the internal-mix nozzle has been chosen for primary development in the spray calciner program at PNL. Atomizing nozzle air caps made of reaction-bonded or hot-pressed silicon nitride should provide satisfactory wear resistance and thermal shock resistance for long-term spray calciner operation.

While several alternative feed system components exist, spray calciner feed system experience at PNL has been primarily with a centrifugal pump, control valve, and magnetic flowmeter. This system has performed satisfactorily, and problems have been limited to the following:

- occasional plugging in the magnetic flowmeter by large foreign particles. (This problem was solved by reducing the size of the strainer-screen mesh to remove particles larger than the flowmeter orifice and by installing a larger flowmeter that had a 0.64-cm orifice instead of a 0.40-cm opening.)
- erosion of the pump impeller housing, pipe elbows and ball valve in the feed tank's 304L stainless steel recirculation line resulting from feeds containing a high percentage of solids. (Solutions to this problem include use of a more erosion-resistant piping material, long-radius elbows or piping bends, ball valves with port diameters at least as large as the pipe's inside diameter, and lower fluid velocities.)
- minor settling of solids around the outside edges on the bottom of the flat-bottomed tank. (Use of tank with a conical or dished bottom would be a solution to this problem.)

The spray calciner has been operated at chamber wall temperatures from 400^o to 900^oC, depending on the type of waste feed and the feedrate. Calcine sintering, corrosion, and thermal stresses limit the maximum temperature. Rates up to 400 L/h have been demonstrated.

Total furnace power requirements (not including heat losses) can be predicted by assuming that the waste feed is water and calculating the sensible and latent heat required to raise the inlet feed and atomizing air to the desired offgas outlet temperature. Furnace power consumption is not axially uniform. Data from actual calciner operation indicate that 60% of the power input is supplied by the lower half of the furnace. This is likely due to convective flow patterns inside the spray chamber.

Regression analysis of capacity data for various calciner spray chamber sizes at various temperatures has resulted in the following equation to predict drying capacity as a function

of spray chamber height (H), spray chamber diameter (D), and wall temperature (T) at an offgas temperature of 200°C:

$$\text{Capacity (L/h)} = 7.477 \times 10^{-11} (T, ^\circ\text{C})^{2.83} (D, \text{cm})^{1.41} (H, \text{cm})^{0.705}$$

Sintered metal filters provide high-efficiency filtration for thermally hot vapors from spray calciners. To determine the required filter surface area, a design face velocity (gas flowrate divided by filter area) of 0.91 m/min is normally used at PNL. The optimum filter material has not been established. One assembly of 316 stainless steel filters was severely corroded after ~1600 h of operating time, possibly because of corrosion from chloride, fluoride, and sulfate ions and/or high temperature oxidation. Inconel 625 or Hastelloy should provide improved performance.

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INTRODUCTION

The solidification of high-level liquid waste (HLLW) from nuclear-fuel reprocessing plants is a required element in current radioactive-waste management systems. An element in many process systems for the solidification of HLLW wastes is spray calcination. This solidification process (along with various vitrification and alternative processes) has been under development (under sponsorship of the Department of Energy [DOE] and previously by the Energy Research and Development Administration [ERDA]) at the Pacific Northwest Laboratory (PNL) for several years.

In the spray calcination process, liquid waste is spray-dried in a heated-wall spray dryer (termed a "spray calciner"), and then it may be combined in solid form with a glass-forming frit. This mixture is then melted in a continuous ceramic melter (Dierks 1980) or in an in-can melter (Blair 1979). Several sizes of spray calciners have been tested at PNL--laboratory scale (~1 to 3 L/h feedrate), pilot scale (~15 to 30 L/h feedrate) and full scale (~200 to 400 L/h feedrate).

Summarized here is the experience gained during the operation of PNL's full-scale spray calciner, which has solidified ~38,000 L of simulated acid wastes and ~352,000 L of simulated neutralized wastes in 1830 h of processing time. Operating principles, operating experience, design aspects, and system descriptions of a full-scale spray calciner are discussed. Individual test run summaries are given in Appendix A. Appendices B and C are studies made by Bechtel Inc., under contract by PNL. These studies concern, respectively, feed systems for the spray calciner process and a spray calciner vibration analysis. Appendix D is a detailed structural analysis made at PNL of the spray calciner. These appendices are included in the report to provide a complete description of the spray calciner and to include all major studies made concerning PNL's full-scale spray calciner.

CONCLUSIONS

Conclusions drawn from spray calciner design, operation, performance and materials experience during the development of PNL's Full-Scale Spray Calciner follow:

- Spray calciner feed system experience at PNL has been primarily with a centrifugal pump/control valve/magnetic flowmeter system. This system has performed satisfactorily, with problems experienced limited to:
 - occasional plugging in the magnetic flowmeter caused by large foreign particles. (This problem was solved by reducing the size of the strainer-screen mesh to remove particles larger than the flowmeter orifice and by installing a larger flowmeter that had a 0.64-cm orifice instead of a 0.40-cm opening.)
 - erosion of the pump impeller housing, pipe elbows and ball valve in the feed tank's 304L stainless steel recirculation line resulting from feeds containing a high percentage of solids. (Solutions to this problem include use of a more erosion-resistant piping material, long-radius elbows or piping bends, ball valves with port diameters at least as large as the pipe's inside diameter, and lower fluid velocities.)
 - minor settling of solids around the outside edges on the bottom of the flat-bottomed tank. (Use of tank with a conical or dished bottom would be a solution to this problem.)
- Compared to internal-mix nozzles, external-mix nozzles require a much higher air-to-liquid flow. Also, external-mix nozzles can produce a few large droplets at the center of the spray pattern at higher liquid flowrates that may cause an accumulation of a damp calcine on the calciner cone section. However, external-mix nozzles can be used with nonpressurized feed systems, such as air-lift feed systems, and do not experience the abrasive wear that occurs with internal-mix nozzles.
- Nozzle flow conditions that produce 70- μ median-volume-diameter spray droplets or smaller are required for small-scale calciners (drying capacity <80 L/h). For large-scale spray calciners (drying capacity >300 L/h), nozzle flow conditions that produce 100- μ median-volume-diameter spray droplets or smaller are required.
- The spray droplet size is inversely proportional to the gas-to-liquid flow ratio both on a mass basis, and volume basis.
- Increased solids loading decreases the spray droplet size.
- Maintenance for atomizing nozzles consists of occasional nozzle replacement due to gradual wear of the air-cap orifice. Based on extrapolated wear measurements, the frequency of nozzle replacement for a reaction-bonded silicon nitride nozzle is >25 d and up to several months, depending on whether a straight-line extrapolation or a parabolic extrapolation is made. Actual wear measurements indicate that the wear

rate occurs most rapidly in the first 30 h and then decreases; therefore, a parabolic wear rate curve is indicated. Longer-term testing (>25 d) is necessary to determine the actual required frequency of nozzle replacement.

- The spray calciner has been operated at chamber wall temperatures at 400 to 900°C, depending on the type of waste feed and desired feedrate. Corrosion, thermal stresses, and calcine sintering limit the maximum operating temperature.
- The spray calciner can be easily started up and shut down.
- With proper operating conditions, solids buildup on the spray chamber wall is minimal, and extended operation without the use of vibration is possible. However, to assure maximum heat transfer, the chamber wall is normally vibrated several times per hour with a 2-s pulse alternated between two vibrators at equal intervals.
- Total furnace power requirements (not including heat losses) can be predicted by assuming the waste feed is water and calculating the sensible and latent heat required to raise the inlet feed and atomizing air to the desired offgas outlet temperature.
- Drying rates up to 400 L/h have been demonstrated.
- Furnace power consumption is not axially uniform. Data from actual calciner operation indicates that 60% of the power input is supplied by the lower half of the furnace. This is likely due to convection patterns inside the spray chamber.
- Regression analysis of capacity data for various calciner spray chamber sizes at various temperatures has resulted in the following equation to predict drying capacity as a function of spray chamber height (H), diameter (D), and wall temperature (T) at an offgas temperature of 220°C:

$$\text{Capacity (L/h)} = 7.477 \times 10^{-11} (T, ^\circ\text{C})^{2.83} (D, \text{cm})^{1.41} (H, \text{cm})^{0.705}$$

- Sintered-metal filters provide high-efficiency filtration for thermally hot vapors from spray calciners.
- Sintered-metal filters can be effectively cleaned by a pulse of blowback air. A 0.25-s pulse of 40 psig air (static pressure) is normally used to clean 1/11 of the total filters simultaneously.
- The minimum offgas temperature established by experience from which a dry, flowable powder is produced (with proper atomization) is 200°C.
- To determine the required filter surface area, a design face velocity (gas flow divided by filter area) of 0.91 m/min is normally used at PNL.
- The optimum filter material has not been established. One assembly of 316 stainless steel filters was severely corroded after 1600 h of operating time, possibly due to corrosion from chloride, fluoride, and sulfate ions and/or high-temperature oxidation. Inconel 625 or Hastelloy should provide improved performance.

BACKGROUND OF FULL-SCALE SPRAY CALCINER

The Pacific Northwest Laboratory has developed under the sponsorship of the DOE (and formerly under ERDA) spray calciner processes to solidify liquid radioactive wastes. The spray calcination process is extremely versatile for drying waste compositions and concentrations of a wide variety (Bonner, Blair and Romero 1976). At PNL, the spray calciner has been operated as a unit process and coupled to in-can melters and ceramic melters for the production of a vitrified product. Calcine has also been produced for pelletization and associated alternate waste forms.

In conventional spray drying, a secondary stream of heated air or steam conveys heat to droplets to dry them. Since the large volume of secondary gas required by this process would necessitate additional effluent decontamination, a heated-wall spray dryer has been developed. The heated-wall spray-drying technique was developed for the solidification of radioactive wastes at Hanford in 1959 (Allemann and Johnson 1961). The spray solidifier was then tested extensively in a nonradioactive pilot plant and in design verification tests (Moore 1969; Schneider and Kelly 1969) before Waste Solidification Engineering Prototype (WSEP) runs using radioactive waste. In the cold pilot-plant studies, over 6000 L (1600 gal) of simulated waste were calcined in about 440 run hours. An element of the WSEP program was to yield information needed for technical, economic, and safety evaluations of the spray calciner. About 8600 L (2300 gal) of high-level aqueous radioactive waste containing 25 million curies were successfully solidified by spray calcination in 13 demonstration runs over a 3-yr period with a total run time of about 580 h (Bond et al. 1971). In the WSEP program, remote operation and maintenance of the calciner, feed system, and effluent treatment system were demonstrated by using an overhead crane and master-slave manipulators.

In the High-Level Waste Immobilization Program (formerly the Waste Fixation Program [WFP]), laboratory-scale, pilot-scale (Bonner, Blair and Romero 1976; Larson and Bonner 1976), and full-scale spray calciners (Mikols, Romero and Bonner 1978) have been constructed and operated using nonradioactive chemicals. Also, a laboratory-scale spray calciner/in-can melter has been used to calcine and vitrify radioactive HLLW from power-reactor fuels (Hanson, Knox and Berger 1980; Knox and Hanson 1980). The pilot-scale (about a 30-L/h nominal feed capacity) spray calciner was used to initiate nonradioactive development studies in 1974. In 1977, a full-scale (about a 350-L/h nominal feed capacity) spray calciner began operation to develop scale-up information and to obtain full-scale equipment operating experience with non-radioactive feed. This full-scale unit has solidified ~390,000 L of simulated wastes during 1830 h of processing time.

To complete spray calciner/melter process development for plant application, a process verification program (Nuclear Waste Vitrification Program) was implemented. The program included the processing of actual high-level waste from spent nuclear power-reactor fuels into borosilicate glass and filling two canisters with this glass in 1979 (Wheelwright et al. 1979; Bonner et al. 1980; Hanson and Bjorklund 1980). The pilot-scale unit (about a 15-L/h feed capacity) produced two canisters [20 cm (8 in.) in dia by 244 cm (8 ft)] long that contained

borosilicate glass. These two canisters represented the solidified borosilicate waste glass from about 1.1 MTU of spent pressurized-water-reactor fuel.

The following sections of this report summarize design and operating experience gained during testing of PNL's full-scale nonradioactive spray calciner using simulated wastes.

SPRAY CALCINER SYSTEM DESCRIPTION

The spray calciner consists of a spray chamber, cone section, furnace, vibrators, and off-gas filter assembly (see Figures 1 and 2). Waste feed is transferred to the top center of the spray chamber where it is broken into fine droplets by an air-atomizing nozzle and sprayed into the chamber. The spray chamber is a vertical cylinder externally heated in a wall temperature of 400 to 800°C, depending on feed composition, by a concentric cylindrical furnace. The atomized droplets are then dried and partially calcined (converted to oxides) by radiative and convective heat transfer as they fall through the heated chamber. Vibrators are periodically

FIGURE 1. Spray Calciner Schematic

FIGURE 2. Photograph of Spray Calciner

operated against the sides of the spray chamber at mid-height to minimize hold-up of dried powder that could reduce heat transfer.

After the calcined particles pass through the chamber, water vapor and atomizing air entrain a large percentage of the particles. More than 99.9% of the entrained particulates are removed from the offgas by sintered metal filters. A periodic pulse of blowback air counter-current to the offgas stream removes the layer of calcine that collects on the

filters. This calcine powder then falls into the cone section and is discharged to the melter along with the calcine that falls directly from the calcination chamber. All surfaces are sloped at least 60° from horizontal to avoid calcine accumulation, thus allowing the calcine to slide off the surfaces.

When the calciner is operated coupled to a melter to produce a vitrified product, glass-forming frit is added to the calciner cone section through an airlock valve. This airlock valve can be timed simultaneously with the filter blowbacks so that as much frit and calcine mixing as possible can be obtained as the frit and calciner fall through the calciner cone into the melter below.

The full-scale spray calciner (FSSC) at PNL has been operated coupled with both in-can and ceramic melters for the production of borosilicate glass, which contains the waste products. Schematics and photographs of these systems are shown in Figures 3 through 6. In addition, the spray calciner has been operated independently of a melter in the production of dry calcine powder. This powder has been used for various tests of the melter and for the production of alternative waste forms that use calcine pellets.

Several design features of PNL's FSSC are shown in Table 1. Design details can be found in the structural drawings in Figures 7 and 8.

In addition to sintered metal filters, the spray calciner offgas treatment system consists of an ejector venturi scrubber, a condenser, a packed tower and a high-efficiency particulate air (HEPA) filter followed by a vacuum-producing blower. The pressure on the system ranges from ~ 10 -in. H_2O vacuum at the calciner to ~ 80 -in. H_2O vacuum at the blower. Each liquid stream can be sampled, and gas samples can be taken at inlets and outlets and between each piece of equipment. Each piece of equipment can be valved-in separately to evaluate its performance. Typical flow through the system is 75 scfm at a 300 L/h calciner feedrate (after any steam has been condensed).

The ejector venturi scrubber is a 316 SS venturi with a 15.2-cm-dia inlet. The scrubber is designed to handle 500 acfm steam and 50 acfm NO_x at $400^{\circ}C$ and 25-in. H_2O vacuum. The scrub solution is 185 L/min at $30^{\circ}C$ and 60 psig. The gas is cooled to $50^{\circ}C$, and the scrubber is designed to remove 90% of the $1\text{-}\mu$ or larger particles.

The condenser, a 304 SS shell and tube heat exchanger with floating heads, is a single-pass tube-side process unit with 14.2-m^2 heat transfer area. Cooling water is fed to the shell side at $13^{\circ}C$ and 60 psig.

The packed tower is made of 14-in. Sch. 10 304L SS pipe. The bed height is 2.62 m of 2.45-cm x 2.54-cm x 0.080-cm Raschig rings. The bed volume is $\sim 0.25\text{ m}^3$. The tower has a 2.54-cm cooling coil with 1.1-m^2 heat transfer area.

The HEPA filter is a 30-cm x 30-cm x 15-cm filter rated at $5.25\text{ m}^3/\text{min}$ at a pressure drop of 0.19 cm Hg. The gas is heated above its dewpoint before passing through the filter.

The blower is a multistage, centrifugal, turbo compressor rated at 320 scfm at 80-in. H_2O vacuum.

FIGURE 3. Spray Calciner/In-Can Melter Schematic

FIGURE 4. Photograph of Spray Calciner/In-Can Melter System

FIGURE 5. Spray Calciner/Ceramic Melter System Schematic

FIGURE 6. Photograph of Spray Calciner/Ceramic Melter System

TABLE 1. Design Features of Full-Scale Spray Calciner

Calcination Chamber

Inside Diameter, m	0.91 (36 in.)
Height, m	3.20 (10.5 ft)
Heat Transfer Area, m ²	9.15 (99.0 ft ²)
Material	316 SS
Wall Thickness, cm	0.64 (0.25 in.)

Furnace

Type	Electric Resistance
Number of Zones	4
Length of Zones, m	0.76
Zone Power, kW	150
Heating Elements	Kanthal 1-A
Maximum Temperature	1000°C
Voltage	480
Phase	3
Power Control	Zero-Crossover, Silicon-Controlled Rectifier

<u>Filters</u>	<u>Pore Size</u>	<u>Filter Size</u>	<u>Filter Material Thickness</u>	<u>Filter Material</u>	<u>Internal Support</u>	<u>No. of Filters</u>	<u>Surface Area</u>
Before January 1980(c)	10 μ	2 in. dia x 36 in.	1/8 in.	316 SS	(a)	51	80.0 ft ²
	65 μ	2-3/4 in. dia x 36 in.	1/16 in.	316 SS	(b)	34	73.4 ft ²
							153.4 ft ²
After January 1980(c)	10 μ	2-3/4 in. dia x 36 in.	1/16 in.	316 SS	(b)	28	60.5 ft ²
	10 μ	2 in. dia x 36 in.	1/8 in.	Inconel	(a)	3	4.7 ft ²
	40 μ	2-3/4 in. dia x 36 in.	1/16 in.	316 SS	(b)	33	71.3 ft ²
							136.5 ft ²

Vibrators

Number	2
Type	Pneumatic, Dry Lubricant
Location	180° Apart at Midplane of Chamber Side
Model	Vibramight® 1300 AC
Motive Air Pressure	40 psig
Manufacturer	Cleveland Vibrator Company

(a) Internally supported by a solid 316 SS center rod.

(b) Internally supported by a perforated 316 SS cylinder.

(c) Because of corrosion (Dierks et al. 1980), the spray calciner filters were replaced in January 1980. Since funding was not available to purchase filters of a more corrosion-resistant material, stainless steel filters on hand were used as replacements. Three Inconel® filters were installed to obtain future corrosion data on this material.

PROCESS EXPERIENCE WITH FULL-SCALE SPRAY CALCINER

Process experience and technology developed during operation of PNL's FSSC during the period April 1977 to November 1980 are summarized in this section. The processing areas discussed include:

- calciner feed system
- atomizing nozzles
- spray calciner
- offgas filtration.

CALCINER FEED SYSTEM

The systems used to supply feed to the spray calciner are discussed in this section.

Operating Principles

The calciner feed system's major functions are to:

- maintain uniform waste feed composition;
- provide reliable transfer of waste feed to the calciner;
- deliver waste feed to the calciner at an accurately controlled rate.

The calciner system generally consists of four components:

- Feed tank--to homogenize, temperature-adjust, and hold the feed;
- Feed transfer--to move the feed from the feed tank to the spray calciner (i.e., air lift and pump);
- Feed flow control--to measure and regulate feed flow (i.e., magnetic flowmeter, control valve and associated instrumentation);
- Spray nozzle--to atomize the calcine feed.

Alternative components accomplish these major functions, depending upon the type of waste feed and whether the feed stream to the calciner nozzle is pressurized or nonpressurized (see page 23 for a description of internal- and external-mix nozzles). Based on a Bechtel study done for PNL (Bechtel, Inc. 1977, Appendix B), Table 2 lists alternative components for industrial acidic commercial wastes and defense neutralized wastes. The alternatives listed consider: a) anticipated cell radiation levels on the order of 10^6 R/h for acidic commercial waste feeds and 10^4 R/h for defense neutralized waste feeds; and b) anticipated undissolved solids of less than 2% for acidic waste feeds and of between 7% and 17% for basic waste feeds. The intense cell radiation levels with industrial (power reactors) acidic wastes require that any electric motors be shielded to provide a reasonable working lifetime; compressed air can be used instead of electric motors for feed motive energy. For basic wastes, an air-lift has not been satisfactorily demonstrated for wastes with very high percentages of solids in the feed. Unshielded electric motors have proven themselves satisfactory for radiation levels on the order of 10^4 R/h. These aspects are considered further in Appendix B.

TABLE 2. Feed System Component Alternatives

Feed and Nozzle Type		Alternatives		
Feed	Spray Nozzle	Feed Tank Agitation	Pump Type	Flow Measurement and Control
Industrial Acidic	External-Mix (requires nonpressurized feed)	1. Air (Pachuca Tank) 2. Mixing Eductor 3. Mechanical Agitator (Shielded)	1. Air-Lift 2. Centrifugal (shielded) 3. Positive Displacement	Magnetic or mass flow-meter and a control valve on the feed line
	Internal-Mix (requires pressurized feed)	1. Mixing Eductor 2. Air (Pachuca Tank) 3. Mechanical Agitator (shielded)	1. Centrifugal (shielded) 2. Positive Displacement (shielded)	
Defense Neutralized	External-Mix (requires nonpressurized feed)	1. Mixing Eductor 2. Mechanical Agitator 3. Air (Pachuca Tank)	1. Centrifugal 2. Positive Displacement 3. Air-Lift	
	Internal-Mix (requires pressurized feed)	1. Mixing Eductor 2. Mechanical Agitator 3. Air (Pachuca Tank)	1. Centrifugal 2. Positive Displacement	

Operating Experience

Feed system features and operations described here are those used at PNL for full-scale, nonradioactive immobilization equipment development. This experience would have to be selectively adapted for plant application since radioactive production plant systems have safety, operating and maintenance constraints not demonstrated for nonradioactive development situations. Simplified pressurized (as for internal-mix nozzle operation) and nonpressurized (as for external-mix nozzle operation) feed-system diagrams are shown in Figures 9 and 10, respectively. The pressurized feed system is that used for the full-scale, nonradioactive spray calciner operation. PNL has only had limited experience with the nonpressurized feed system, but it appears promising when using a feed with low solids content with an external-mix nozzle.

For the pressurized feed system in Figure 9, the waste is pumped from the feed tank to the calciner through a Y-strainer (to remove large solids), a flowmeter, and a control valve. The largest portion of the waste flow from the pump outlet is recycled directly back to the tank through a mixing eductor for tank agitation. The strainer is flushed when excessive pressure drop through the strainer is indicated by pressure gauges. Since only a few liters of waste are used to flush a strainer, the amount of waste generated in this manner would be small. For a nonpressurized external-mix nozzle, the waste can be air-lifted to a head tank, which overflows to the feed tank. The external-mix nozzle siphons waste from the constant-head tank through a control valve and flowmeter, then into the nozzle. Since an air-lift-type feed-transfer system has not yet been demonstrated for wastes containing a high percentage of

FIGURE 9. Pressurized Spray Calciner Feed System (Internal-Mix Nozzle)

solids and would most likely be used with waste feeds containing only a few small particles, a strainer is not shown in Figure 10.

The flush-water lines shown in Figures 9 and 10 are used for three purposes: 1) to cool the nozzle before beginning waste-feeding in order to prevent possible plugging in the nozzle from the drying of waste in the nozzle; 2) to provide high-pressure water that will remove possible blockages in the flowmeter or feed line by back-flushing to the feed tank (pressure gauges located as shown in Figures 9 and 10 indicate the location of the blockage); and 3) to flush the feed line after shutdown.

Experience at PNL has been primarily with the system shown in Figure 9. The system has performed satisfactorily, with problems limited to the following:

- occasional plugging in the magnetic flowmeter caused by large foreign particles.
[This problem was solved by reducing the size of the strainer-screen mesh to remove particles larger than the flowmeter orifice and by installing a large flowmeter that had a 0.64-cm (1/4-in.) orifice instead of a 0.40-cm (5/32-in.) opening.]

FIGURE 10. Nonpressurized Spray Calciner Feed System (External-Mix Nozzle)

- erosion of the pump impeller housing, pipe elbows and ball valve in the feed tank's 304L SS recirculation line resulting from feeds containing a high percentage of solids. (Solutions to this problem include use of a more erosion-resistant piping material, long-radius elbows or piping bends, ball valves with port diameters at least as large as the pipe's inside diameter, and lower fluid velocities.)
- minor settling of solids around the outside edges on the bottom of the flat-bottomed tank. (Use of tank with a conical or dished bottom would be a solution to this problem.)

Design Aspects

Design aspects for calciner feed systems are considered in detail in Appendix B, including shielded and unshielded centrifugal pump and chemical agitation, air agitation and airlift transfer, and feed-tank design.

ATOMIZING NOZZLES

Atomizing nozzles, which are used to disperse the waste feed into fine droplets, are discussed in this section.

Operating Principles

An important phase in spray-drying is waste feed atomization. The liquid must be dispersed into droplets small enough to dry before striking the calciner wall or the cone at the bottom of the chamber in order to prevent caking on these surfaces. Good atomization increases the heat-transfer area of the droplets and increases the drying rate.

Two types of atomizing nozzles have been tested for spray-drying waste feeds--internal-mix and external-mix nozzles. An internal-mix nozzle contacts pressurized liquid with pressurized air in a small internal chamber and then sprays the fluids through an orifice, as shown in Figure 11. The air velocity is very high and the initial liquid velocity is low. Atomization occurs as a result of the shearing action of the high-speed air upon the slow-moving liquid.

An external-mix nozzle exhausts pressurized air around the liquid feed outlet and siphons the liquid through the nozzle; Figure 12 illustrates this system. The air impacts upon the liquid outside the nozzle to break the liquid into droplets.

In addition to the nozzle design, spray-droplet size is influenced by the following:

- waste feed flowrate
- atomizing air flowrate
- calciner temperature
- percentage of solids in waste feed.

Kaser (1969) has shown that the mass median spray-droplet size is strongly dependent upon the ratio of liquid-to-gas flowrates, both on a mass and on a volume basis. The droplet size decreases with higher gas-to-liquid flow ratios. The calciner temperature may influence the droplet size by causing rapid boiling and shattering of the droplets at high temperatures. A

FIGURE 11. Internal-Mix Nozzle

FIGURE 12. External-Mix Nozzle

high percentage of solids in the waste feed was shown by Kaser (1969) to produce smaller droplet sizes, while filtering out solids caused the droplet sizes to approach those of water at similar flow ratios.

Operating Experience

Steam, hot air, and ambient-temperature air have been evaluated as atomizing gases. Both steam and preheated air cause nozzle plugging in small internal-mix nozzles by drying the waste in the nozzle (Bonner, Blair and Romero 1976). Conventional external-mix nozzles are also heated excessively by hot air or steam and can plug from waste dried in the nozzle. Therefore, ambient-temperature air has been the gas used for atomization at PNL. Hot air or steam could be used for atomization with an external-mix nozzle, since the steam or hot air contacts the liquid outside the nozzle; internal nozzle cooling could prevent the feed from drying in the nozzle. This has been demonstrated at Karlsruhe, Germany (Kanfmann et al. 1976).

Nozzle cleanout assemblies have been used for nozzles with air-cap orifices 0.32 cm or smaller. These assemblies have a plunger that is extended through the air-cap orifice when activated by compressed air. While these cleanout assemblies have proven useful for small-scale calciners, they have never been used during operation of the full-scale calciner, which uses air-cap orifices larger than 0.64 cm. Nozzle plugging in the full-scale calciner has occurred only twice in three years of intermittent testing. The cause of those pluggages was failure to pre-cool the nozzle with demineralized water before waste feed startup. Pre-cooling the nozzle with demineralized water prevents solids formation caused by flashing of the feed when the liquid reaches the hot feed line and nozzle during startup. Plugging in full-scale nozzles is not likely to occur if the nozzle is pre-cooled. In the event that a plug did occur, replacement of the nozzle would be simpler than would providing a mechanism for cleaning the nozzle.

Accurate pressure and flow measurements of the atomizing air are important. In-cell measurements of the atomizing air pressure and flow can be eliminated by sizing the air piping to minimize pressure drop and by measuring both the pressure and flow at appropriate points in the out-of-cell air piping.

For an internal-mix nozzle, a pressure switch should be interlocked with the feed pump power supply to turn off the pump if the atomizing air pressure drops below a minimum value. This will prevent waste feed from being forced into the atomizing air line if the nozzle air-cap orifice becomes plugged and the waste pressure exceeds the atomizing air pressure. In addition, an antibackflow device should be installed in the air line. This backflow risk does not present itself with external-mix nozzles.

When compared to internal-mix nozzles, external-mix nozzles have a disadvantage in that a much higher air-to-liquid flow is required for operation--requiring larger-capacity offgas treatment equipment. Also, external-mix nozzles tend to produce a large droplet at the center of the spray pattern at higher liquid flowrates, which may cause an accumulation of damp calcine on the calciner cone section. The number of large droplets can be reduced by maintaining a sufficient liquid siphon height, as shown in Table 3. The necessary siphon height must be determined by testing for individual calciner chamber heights and for varied flowrates. In addition, feeds with a high percentage of solids are more likely to cause plugging in feed systems with external-mix nozzles than with internal-mix nozzles, since an external-mix nozzle must siphon the waste feed.

Several possible advantages for the use of external-mix nozzles exist. A nonpressurized feed system can be used, such as air-lift-type feed system, for some waste feed types containing low solids content. This eliminates the need for a mechanical pump. Also, the risk that exists with an internal-mix nozzle of waste feed being forced into the atomizing air line is not present with the external-mix nozzle. Finally, the abrasive wear seen in external-mix

TABLE 3. Flow and Droplet Size Data for External-Mix Nozzles(a)

Nozzle Sizes		Air Pressure, psi	Air-Flow, scfm	Liquid Siphon Height, (b) cm	Liquid Flow, L/h	Estimated MVD Range, μ	Mass
Air-Cap Orifice, cm	Fluid Cap Orifice, cm						Air Flow Mass Liquid Flow
1.11	0.635	20	18.5	10.2	91	70-80	0.44
		20	18.5	20.3	55	50-60	0.73
		30	24.0	10.2	129	70-80	0.41
		30	24.0	20.3	98	60-70	0.54
		40	29.2	10.2	154	70-80	0.41
		40	29.2	20.3	124	60-70	0.51
2.86	1.59	20	105	10.2	452	200-250	0.51
		20	105	20.3	245	150-200	0.94
		25	120	20.3	336	150-200	0.78
		30	135	20.3	333	100-150	0.89
		40	165	20.3	429	100-150	0.84

(a) Data for water sprayed at room conditions.

(b) Liquid Siphon Height--Height of nozzle outlet above feed-tank liquid level.

nozzles is not nearly as severe with internal-mix nozzles, since the high-velocity air contacts the waste feed outside the nozzle.

Atomizing air and liquid flow data at various pressures and for various sizes of external- and internal-mix nozzles are listed in Tables 4 and 5. These data are supplied by a nozzle manufacturer for water sprayed at room temperature, but agree closely with flow measurements made for actual calciner operation with simulated waste feeds. These data serve only as a standardized reference since air and liquid pressure measurements for actual plant equipment will depend upon the location of the pressure sensors and upon additional system pressure drops.

The estimated median volume diameter (MVD)^(a) (Table 3) for spray droplets was also supplied by the nozzle manufacturer for water sprayed at room temperature. Since it is not

TABLE 4. Flow and Droplet Size Data for Internal-Mix Nozzles^(a)

Nozzle Sizes		Liquid Pressure, psi	Liquid Flow, L/h	Air Pressure, psi	Air-flow, scfm	Estimated MVD, ^(b) μ	Mass Air Flow Mass Liquid Flow
Air-Cap Orifice, cm	Fluid Cap Orifice, cm						
0.318	0.254	15 ^(c)		20		100	
		15 ^(c)		22		80	
		20	61	25	4.18	120	0.150
		20	32	30	5.3	50	0.362
		25 ^(c)		30		130	
		25 ^(c)		35		60	
0.793	0.638	15	309	20	17.6	200	
		15 ^(c)		25		70	
		20 ^(c)		25		200	
		20 ^(c)		30		70	
		20 ^(c)		35		50	
		25	436	30	22.5	250	0.113
		25	289	35	28.8	100	0.218
		25	173	40	35.3	50	0.446
1.39	1.11	10	546	14	51	200	0.20
		10	387	16	61	110	0.34
		10 ^(c)		20		30	
		15 ^(c)		25		70	
		20	750	26	72	255	0.210
		20	455	30	94	100	0.451
		20 ^(c)		35		35	

(a) Data for water sprayed at room conditions.

(b) MVD--Median Volume Diameter. Fifty percent of the total volume sprayed is made up of droplets with diameters less than the median value, and 50% are greater than the median diameter.

(c) Outside of the manufacturer's recommended operating range; higher air pressures can cause discontinuous liquid flow.

(a) Median Volume Diameter: 50% of the total volume of liquid sprayed is made up of droplets with diameters less than the median value and 50% with diameters greater than the median value.

TABLE 5. Required Mass-Flow Ratios

Nozzle Type	Nozzle Sizes		Droplet MVD, (a) μ	Air-to-Liquid Mass- Flow Ratios Required to Obtain Droplet MVD
	Air-Cap Orifice, cm	Fluid Cap Orifice, cm		
Internal Mix	0.318	0.254	70 100	0.3 0.2
	0.793	0.638	70 100	0.3 0.2
	1.39	1.11	100	0.4
External Mix	1.11	0.635	60 to 80	0.4 to 0.7
	2.86	1.59	100 to 150	0.85 to 0.95

(a) MVD--Mean Volume Diameter

practical to measure spray-droplet sizes inside an 800°C calciner chamber, the flow conditions required to produce a droplet small enough to dry before striking the spray chamber wall or cone section have been determined by experiments and correlated to the droplet sizes for water at room temperature atomized at similar flow conditions. For small-scale calciners (spray chambers less than 1.8 m in height), nozzle flow conditions are required that produce approximately 70- μ (MVD) or smaller droplets when spraying water at room temperature. For large-scale calciners (spray chambers >3.05 m high), nozzle flow conditions are required that produce approximately 100- μ (MVD) or smaller droplets when spraying water at room temperature. Table 5 indicates the air-to-liquid mass-flow ratios required to produce those droplet sizes for several atomizing nozzles.

Another factor that must be considered is that the nozzle spray pattern must be compatible with the calciner spray-chamber diameter. A wide spray pattern in a small-diameter spray chamber will cause droplets to contact the heated wall before they are dry, resulting in a scale buildup. While no rule has been developed to determine the smallest-diameter chamber compatible with a particular nozzle, the diameter of the spray chamber should at least be several inches larger than the width of the spray pattern in free air to prevent direct impingement of the spray on the chamber wall.

Design Aspects

Standard internal dimensions for the internal-mix nozzle fluid cap and air cap supplied by manufacturers have been used throughout spray calciner development. However, limited modifications have been made to 1) aerodynamically improve the spray pattern, which minimizes solids accumulation on the nozzle and on the top of the spray chamber, and 2) to prevent abrasive wear by use of more wear-resistant materials that are also thermal-shock-resistant. These internal-mix nozzle design modifications are discussed separately.

Aerodynamic Nozzle Design

Experience has shown that certain nozzle shapes cause excessive turbulence at the nozzle-exit orifice that results in dried solid accumulations (Bonner, Blair and Romero 1976). In the same manner, nozzles that do not protrude through the top of the spray chamber at least 1 in. for full-scale calciners can also cause turbulence that results in dried solid accumulations on the top inside surface of the spray chamber. Figure 13 illustrates examples of good and poor aerodynamic nozzle designs.

Nozzle-Wear and Thermal-Shock Testing

Abrasive wear experienced with stainless steel internal-mix nozzle air caps prompted a search for a more wear-resistant material. Some of the materials tested were: reaction-bonded silicon nitride (Si_3N_4), 99.5% alumina, Cer-vit (a machinable glass-ceramic), and 303 stainless steel. Since these materials (except for stainless steel) are susceptible to breakage by thermal shock, tests to determine resistance to thermal shock were also completed. Details of these tests are discussed by Miller and Stout (1981).

Abrasive wear occurs on the upstream edges of the air-cap orifices. Figure 14 illustrates the different stages of nozzle wear and locations of wear measurement. Two measurements for determining wear were taken before and after periods of simulated waste-feeding at room temperature: 1) the internal orifice diameter at the initial orifice tip length (at the initial upstream orifice edge) and 2) the orifice tip length.

FIGURE 13. Examples of Poor and Good Aerodynamically Designed Nozzles

FIGURE 14. Location of Internal-Mix Wear Measurements Taken

Nozzle air-cap wear does not significantly affect calciner performance until the diameter of the downstream edge of the air-cap orifice is enlarged. The downstream edge of the orifice does not change significantly until the orifice tip has worn through. Therefore, the most important parameter for determining the amount of wear on an air cap is the percent of the orifice tip that has worn through.

The results of the abrasive-wear testing for Spraying System nozzle setup No. 42 are shown in Table 6. Table 7 gives the composition of the feed slurry used for the test. The atomizing air was 25 psig and the feed pressure was 15 psig throughout the testing. A nozzle with a 0.32-cm air-cap orifice was used for testing because of its relatively small airflow requirements, which simplified the test equipment and minimized the size of offgas handling equipment. Full-size nozzles have greater orifice tip lengths and can be expected to perform longer than will small nozzles before the orifice tip is worn through and the atomization is significantly degraded.

TABLE 6. Results of the Abrasive Wear Testing

Air-Cap Composition	Hours Used	Internal Ori- fice Diameter (cm) Orientation		Average Diameter Increase, cm	Length of Orifice Tip, cm	% Orifice Tip Worn Through
		0°	90°			
Cer-vit	0	0.312	0.312	---	0.168	---
	15	0.379	0.376	0.064	0.114	32
	30	0.401	0.412	0.094	0.097	42
303 SS	0	0.315	0.312	---	0.157	---
	15	0.325	0.323	0.010	0.127	19
	30	0.353	0.366	0.046	0.102	36
96% Alumina	0	0.320	0.320	---	0.173	---
	15	0.434	0.467	0.132	0.157	9
	30	0.470	0.460	0.145	0.156	10
	45	0.457	0.457	0.137	0.155	10
	60	0.462	0.465	0.145	0.152	12
Reaction-Bonded Silicon Nitride	0	0.320	0.320	---	0.175	--
	15	0.353	0.345	0.028	0.163	7
	30	0.368	0.366	0.046	0.160	9
	45	0.373	0.381	0.056	0.157	10
	60	0.381	0.381	0.061	0.157	10

TABLE 7. Simulated Waste Feed Composition^(a)

Component	wt%	Chemical Form Simulated Feed
Fe(OH) ₃	53.4	colloidal
Al(OH) ₃	12.4	nonsoluble powder (-200 mesh)
MnO ₂	10.5	nonsoluble powder (-200 mesh)
Zeolite	6.7	nonsoluble grit (20-50 mesh)
Ni(OH) ₂	6.1	nonsoluble powder (-200 mesh)
CaCO ₃	4.4	nonsoluble powder (-200 mesh)
SiO ₂	3.6	-40, +60 mesh sand
NaNO ₃	1.9	soluble
Na ₂ SO ₄	1.0	soluble

(a) Feed concentration: 108 g/L of the above feed chemicals.

Alumina and reaction-bonded silicon nitride are the most wear-resistant, with only 10% to 12% of the orifice tip worn through after 60 h of operation. Cer-vit and 303 SS had 42% and 36%, respectively, of their orifice tips worn through after 30 h of operation. It is interesting that most of the wear to the alumina and reaction-bonded silicon-nitride air caps occurred within the first 30 h of operation. This may indicate a decrease in wear with time.

In addition to testing for wear resistance, all these materials, except 303 SS, were also tested for failure caused by thermal shock. Thermal-shock failure results from stress created by a large temperature gradient across the air cap. A large temperature gradient can occur when fluid flow is interrupted or started abruptly. For example, if the atomizing airflow is off during calciner heatup and then is turned on suddenly, a cracked nozzle air cap could result. Table 8 shows the results of the thermal-shock testing.

For the rapid-cooling tests, cooling rates were varied from 440°C/min to 1790°C/min in increments of about 270°C/min using a flow of compressed air. The nozzle was inspected after each incremental change, and the rate of cooling that caused damage to the air cap, if any, was noted. Temperature-change measurements were made with a thermocouple located in the mixing chamber portion of the air cap. This location approximates the temperature of the interior surface of the air cap. In the rapid-heating test, the nozzle assembly at room temperature was placed into a 925°C furnace. The maximum initial heating rate was ~200°C/min.

As can be seen from Table 8, the alumina air caps had the greatest variation between individual air caps for breakage conditions. Possible reasons for this are quality variations or stresses induced during air cap machining. Breakage of the Cer-vit air cap in the rapid heating test may have also been the result of a flaw since the other two Cer-vit air caps tested did not break under the more rigorous rapid-cooling test. The silicon nitride air caps tested did not break under any test conditions. Figure 15 is a photograph of the three air caps that broke and an alumina air cap that broke during use with a pilot-scale calciner.

TABLE 8. Results of the Thermal-Shock Testing

Air Cap Number	Material	Test Performed		Results
		Rapid Cooling	Rapid Heating	
1	96% Al ₂ O ₃	X		Broke at initial cooling rate of 440°C/min
2	96% Al ₂ O ₃	X		Broke at initial cooling rate of 1790°C/min
3	96% Al ₂ O ₃	X	X	Did not break
4	Cer-vit	X		Did not break
5	Cer-vit		X	Broke at initial heating rate of 200°C/min
6	Cer-vit	X	X	Did not break
7	Si ₃ N ₄	X		Did not break
8	Si ₃ N ₄	X	X	Did not break
9	Si ₃ N ₄	X	X	Did not break

These results indicate that alumina air caps are much more susceptible to thermal-shock breakage than are air caps of Cer-vit or silicon nitride. However, breakage occurs as axial cracks, as shown in Figure 15, and does not necessarily result in performance degradation. Extensive experience with pilot-scale and full-scale atomizing nozzles has shown that alumina air caps perform as well after thermal-shock breakage as before. The large air-cap pieces are held in place by the retaining cap and atomization continues normally.

Full-Scale-Calciner Nozzle Air-Cap Wear Measurements

Results of wear measurements of nozzle air caps (Spraying Systems Company Set-up No. 82) used during recent full-scale calciner tests are shown in Table 9. The feed-slurry compositions used during operation of these nozzles are given in Table 10. The feed-slurry composition used with the sintered silicon nitride air cap contained a large amount of glass frit for vitrification and thus was more erosive than feeds used for other nozzle-wear tests.

Comparison of full-scale nozzle-wear measurements with measurements made in the tests described previously indicate similar wear for full-scale nozzles of 99% alumina and pilot-scale nozzles of 96% alumina. Fourteen percent of the orifice tip was worn through after 100 h of operation of the full-scale nozzle compared to 12% of the tip worn through after 60 h of operation of the pilot-scale nozzle. However, wear measurements for full-scale nozzles of sintered silicon nitride were substantially higher than wear of pilot-scale reaction-bonded silicon nitride (29% of the orifice tip worn through after 71 h of operation for the full-scale nozzle compared to 10% of the nozzle tip worn through after 60 h for the pilot-scale nozzle). Two reasons for this difference are: 1) the full-scale nozzle was made of sintered silicon nitride, whereas the pilot-scale nozzle was made of reaction-bonded silicon nitride; the two different forming processes likely produced materials of different physical properties; 2) the

FIGURE 15. Failed Nozzles

TABLE 9. Full-Scale Nozzle(a) Wear Measurements

Nozzle Material	Hours Used	Average Feedrate, L/h	Average Internal Diameter, mm	Average Diameter Increase, mm	Length of Orifice Tip, mm	% of Orifice Tip Worn
99% Alumina	0	---	7.95	---	4.00	---
	100	190	9.05	1.10	3.44	14.0
Sintered Silicon Nitride	0	---	7.90	---	3.65	---
	71	93	9.29	1.39	2.61	28.6

(a) Spraying Systems Company Set-up No. 82 nozzle (air-cap orifice diameter = 0.793 cm).

feed used during operation of the full-scale nozzle contained more solids and was more likely erosive than the feed used during operation of the pilot-scale nozzle. However, the feed flow with the full-scale silicon nitride nozzle was much lower than normal, and the difference in the formation of the ceramic is expected to have been the major cause of the variation of the wear measurements. Therefore, the method of ceramic formation must be considered in addition to the type of ceramic used. Apparently, reaction-bonded silicon nitride has superior wear characteristics compared to sintered silicon nitride. Hot-pressed silicon nitride, formed at high temperatures and pressures (this material was not tested), may be superior to reaction-bonded silicon nitride in wear resistance based on the comparison of physical properties in Table 11.

TABLE 10. Feed Slurry Composition Used During Nozzle-Wear Measurement Tests

Feed Atomized by 99% Alumina Nozzle		Feed Atomized by Silicon Nitride Nozzle	
Makeup Chemical	g/L	Chemical	Chemical Form, g/L
Fe(OH) ₃	69.73	Fe(OH) ₃	52.37
Al ₂ O ₃ ·3H ₂ O	17.49	Al(OH) ₃	13.14
MnO ₂	13.92	MnO ₂	10.45
CaCO ₃	6.35	CaCO ₃	4.77
NiO·2H ₂ O	8.99	NiO·2H ₂ O	6.75
NaNO ₃	2.93	NaNO ₃	2.20
Na ₂ SO ₄	1.43	Na ₂ SO ₄	1.07
Zeolite	10.35	Zeolite	1.77
Na -EDTA	8.10	Cornstarch	0.43
Cornstarch	0.57	Coal	2.17
Anthracite Coal	2.89	Frit-211	192.9
NaCl ^(a)	1.6	TOTAL	294.0
NaF	0.178		
CsNO ₃	0.071		
Sr(NO ₃) ₂	0.071		
TOTAL	144.7		

TABLE 11. Comparison of Physical Properties for Reaction-Bonded and Hot-Pressed Silicon Nitride

Material	Property		Compressive Strength
	Density	Modulus of Rupture	
Reaction-Bonded(a) Silicon Nitride	Up to 2.7 g/cm ³	30,000 psi	77,000-112,000 psi
Hot-Pressed(b) Silicon Nitride	3.11 g/cm ³	70,000-120,000 psi	300,000-500,000 psi

(a) Product data from KBI-AME, Inc., bulletin file number 600 PDI-13.

(b) Product data from Ceradyne, Inc., Cerolloy 147A silicon nitride.

Operating-Life Estimates

Occasional replacement of the atomizing nozzle air cap on spray calciners will be necessary due to gradual wear of the air-cap orifice. Enlargement of the orifice can be detected by increased air and feed flowrates at particular pressures.

Since the performance of the atomizing nozzle does not change significantly until the downstream diameter of the air-cap orifice becomes enlarged, the expected working life of the nozzle can be estimated from extrapolations of the wear measurements presented in Tables 8 and 9. Figure 16 shows the actual wear measurements in percent of the nozzle air-cap tip worn

FIGURE 16. Nozzle Air-Cap Operating Life Extrapolation

through for the reaction-bonded silicon nitride versus hours of operation. The measured-wear data are then extrapolated by two different methods--a straight-line extrapolation and a parabolic extrapolation that follows the curvature of the measured data points. These two methods of extrapolation predict widely varying results--600 h (25 d) of operation for the straight-line extrapolation compared to several months of operation for the parabolic extrapolation. Obviously, longer-term testing (>25 d) is required to more accurately determine the actual frequency of nozzle replacement.

At this time, it can be concluded that a nozzle with an air cap of reaction-bonded silicon nitride will perform satisfactorily for at least 25 d and likely up to several months before replacement is required. In addition, hot-pressed silicon nitride is expected to have superior wear characteristics to reaction-bonded silicon nitride, based on the properties listed in Table 11, which would decrease even further the frequency of nozzle replacement required.

SPRAY CALCINER

The spray calciner, in which waste feed is dried and partially calcined, is discussed in this section.

Operating Principles

The spray calciner consists of a spray chamber, cone section, furnace, vibrators, and the offgas filter assembly (a more detailed discussion of the offgas filters is included in the next section). During operation, atomized waste feed is sprayed at the top-center of the spray chamber, which is a vertical cylinder externally heated by the concentric, cylindrical furnaces shown in Figure 1. The high velocity at which the atomizing air and waste feed droplets enter the spray chamber, in addition to the temperature difference between the chamber wall and the evaporating water droplets, creates a high degree of turbulence in the calciner.

The intensity of this turbulence was determined for a 20.3-cm-dia calciner using an eddy diffusivity measurement (Alleman and Johnson 1961). At a point 11 column diameters below the nozzle of the test apparatus, the Reynolds number was 80,000 to 90,000 for conditions at which the calculated Reynolds number based on the average volumetric flowrate and cross-sectional area was 260. The combination of this violent convective heat transfer with radiant heat transfer allows drying and partial calcination at a high rate.

After passing through the chamber, water vapor and atomizing air entrain a large percentage of the calcine particles. More than 99.9% of the particulates are removed from the offgas by sintered-metal filters. A periodic pulse of blowback air removes the layer of calcine that collects on the filters. This calcine then falls into the cone section and is discharged to the melter along with the calcine that falls directly from the calcination chamber. All surfaces are sloped at least 60° from horizontal to avoid calcine accumulation by allowing the calcine to slide off.

Frit is added to the calciner cone section through an airlock valve. This airlock valve can be timed simultaneously with the filter blowbacks to obtain as much frit and calcine mixing as possible as they flow through the calciner cone into the melter.

Two vibrators are mounted 180° apart at mid-height on the calciner chamber. These vibrators are supported independently of the furnace to minimize vibration of the furnace elements and insulation. During normal calciner operation with low-sodium wastes, frequent vibration is not necessary. However, to assure the calciner is free of excess calcine powder buildup the wall is usually vibrated several times per hour.

Operating Experience

Experience gained during operation of PNL's FSSC is discussed in the following subsections in these areas:

- calciner temperature selection
- calciner startup and shutdown
- steady-state calciner operation

- calcine product
- materials experience.

Calciner Temperature Selection

The spray calciner has been operated at chamber wall temperatures from 400⁰ through 900⁰C, depending on the type of waste feed and the feedrate. The required calciner chamber size for a desired waste feedrate can be minimized by operating at the highest temperature that will not cause the calcine to stick to the chamber wall by sintering or melting. Also, corrosion and thermal-stress concerns are more significant at higher temperatures, and must be considered in the mechanical design and temperature selection for the calciner.

Tests can be made to determine the sintering or melting temperature of a particular waste feed. Thermal-gradient feed analysis is used to determine the maximum operating temperature for several feed types. This test involves drying an aliquot of the proposed feed solution or slurry and placing it in a thermal-gradient furnace. Physical changes in the product characteristics can be examined and related to a temperature range. The maximum calciner wall temperature should be approximately 50⁰C less than the point at which the product begins to stick together. Additional calcine reaction temperature data can be obtained from differential thermal analysis and thermal gravimetric analysis (MacKenzie 1970; Erdey 1965) to determine the feed behavior in the calciner.

Spray Calciner Startup and Shutdown

The spray calciner is a simple piece of process equipment. The following general steps are involved in starting up a spray calciner (similar to the FSSC at PNL):

- Heat the calciner furnaces to the desired temperature. For an internal-mix nozzle, the atomizing airflow should be 20 to 30 scfm during the heatup cycle to cool the nozzle and minimize thermal shock to the nozzle when waste-feeding is begun (see the section "Atomizing Nozzles" for a discussion of thermal shock in the atomizing nozzle).
- Increase the atomizing nozzle air pressure to about 35 psig.
- Increase the calciner vacuum to 15 to 20 in. H₂O.
- Slowly increase the flow of demineralized water up to the desired processing rate, allowing the vacuum controller time to react to the increase in offgas flow.
- Quickly switch from demineralized water to the waste feed after the offgas system vacuum has stabilized. (Note: Extended periods of feeding demineralized water to the calciner without a layer of calcine on the sintered-metal offgas filters can cause plugging of the filter pores by submicron-size particles formed from impurities in the water.)
- Start flow of frit and filter blowback/vibrator timers or controllers.
- Make final adjustments to obtain desired atomizing air, waste feed flowrates and vacuum. A calciner vacuum of 10 in. H₂O is normally used to prevent pressurization during a filter blowback.

The following general steps are used for shutdown of the calciner:

- a. Stop flow of frit and blowback/vibrator timers or controllers.
- b. Switch from waste feed to demineralized water. Feed demineralized water for ~1 min to flush the feed line and prevent drying of the feed in the nozzle or piping.
- c. Shut off demineralized water.
- d. Turn off power to calciner furnace, if desired.
- e. Maintain atomizing airflow at 20 to 30 scfm for an internal-mix nozzle to prevent thermal shock to the nozzle until the calciner temperature has dropped to about 200°C, then shut off atomizing airflow. Airflow to the external-mix nozzle can be shut off before cooldown.

Emergency shutdown of the calciner can be done simply by stopping frit and waste feed flows to the calciner and, if desired, by turning off the atomizing airflow and power to the calciner furnace.

Steady-State Calciner Operation

After waste-feeding has been started, the calciner furnace power consumption and chamber wall temperatures should reach steady-state within about 5 min with good controller response time. The cone section and offgas temperatures require substantially more time to reach steady-state--up to several hours. The reason for this difference in time is that the furnace controllers maintain the chamber wall at a constant temperature before and after waste-feeding has been started, but the cone section and filters are cooled by the offgas stream. The thermal inertia of the metal cone section and filters causes this cooling to occur slowly.

Typical steady-state furnace-zone power consumption and calciner temperatures for the PNL full-scale calciner are shown in Table 12 for the indicated furnace temperatures and a feed-rate of 300 L/h. These values are representative for both acid and neutralized waste at the indicated furnace temperatures.

The temperatures and furnace power consumption should be monitored closely to ensure proper calciner operation. When power is varied to maintain constant furnace or chamber wall temperatures, a rapid or gradual drop in power consumption after the first 5 min of operation usually is an indication of a scale buildup on the inside surface of the calciner chamber. Scale buildup can be caused by moist calcine adhering to the wall or by calcine sintering to the wall if temperatures are too high. The sintering temperature of the calcine must be known to be sure which of the above possibilities is the actual cause. Moist calcine can occur when the calciner's drying capacity has been exceeded or if the atomizing nozzle is worn or damaged. Moist calcine caused by exceeding the calciner's capacity can be corrected by decreasing the feedrate of the waste, by increasing the furnace temperatures, or both. Calcine sintering to the wall can be remedied by lowering the chamber wall temperatures, and if the offgas temperatures are lower than desired, the waste feedrate should also be lowered. An increased rate of vibrator operation is also helpful in eliminating scale buildup for the above operating problems. If vibration does not remove the scale buildup, waste-feeding can be temporarily

TABLE 12. Typical Calciner Operating Parameters(a)

Parameters and Units	Value
Waste Feed Rate, L/h	300
Atomizing Airflow Rate, scfm	30
Furnace Temperature, °C(b)	
Zone 1 (bottom)	875
Zone 2	875
Zone 3	840
Zone 4 (top)	840
Furnace Power Consumption, kW	
Zone 1 (bottom)	90
Zone 2	92
Zone 3	55
Zone 4 (top)	53
Total	290
Calciner Chamber Wall Temperatures, °C	
Zone 1	750
Zone 2	755
Zone 3	775
Zone 4 (top)	800
Calciner Offgas Temperature, °C	250
Calciner Cone Temperature, °C(c)	230

(a) See Figure 1 for zone locations.

(b) Temperature set point maintained by controller

(c) Directly under the spray chamber centerline

stopped and the scale removed by washing or by thermal shock. Most scale buildup encountered to date has been encountered while processing high-sodium feed and has been readily removed by lowering a washing nozzle through the calciner spray nozzle port and spraying for a few minutes. The thermal-shock descaling technique requires that the chamber wall (and the scale) be heated up to maximum normal operating temperature and then water sprayed into the hot chamber through the atomizing nozzle for 2 to 5 min. This procedure can be repeated, if necessary. Even the most stubborn scale has been removed using this method.

Under proper operating conditions wall scale is minimal, and extended operation without use of vibration is possible. However, to assure the walls are free of powder buildup, the wall is normally vibrated several times per hour with a 2-s pulse alternated between two vibrators at equal intervals.

Calcine Product

The product of spray calcination in the heated-wall spray calciner is a very fine, dry powder that usually consists of metal oxides for the most part. Depending on the feed composition and the calcination temperature, the powder could consist of 1% to 42% alkali-metal nitrates, carbonates, sulfates, and/or waters of hydration.

Calcine particle size distribution is shown in Figure 17 (Bonner, Blair and Romero 1976) for acid-type waste and Figure 18 for neutralized waste. While 65% of the total number of particles for acid waste are in the diameter range of 1 to 5 μ , these particles account for only 15% of the mass. Only 17% of the total particles are 6 to 20 μ in dia, but they account for 75% of the mass. The large acid-waste particles (over 1 mm in dia) were analyzed and found to be of the same composition as the rest of the calcine, except for higher water and

FIGURE 17. Acid-Waste Particle Size Distribution

FIGURE 18. Defense Waste Calcine Particle Size Distribution

nitrate contents (Bonner, Blair and Romero 1976). For neutralized waste slurry, 88% of the total particles are less than $11\ \mu$, but account for only 2.8% of the mass. Since the neutralized waste is a slurry rather than a solution, the larger particles in the calcine represent larger particles in the slurry. These large particles are not the same composition as the smaller calciner particles from water-soluble compounds.

Typical calcine properties are presented in Table 13 (Bonner, Blair and Romero 1976). These properties are a function of waste composition and of calciner operating parameters. The calcine moisture content is normally less than 1% but can be as high as 4% without significantly decreasing the calcine's ability to flow. Residual nitrate content from acid wastes is a strong function of the alkali metal content of the waste. Most nonalkali metal nitrates are converted to oxides.

The sodium nitrate content of the calcine significantly affects the sintering temperature of the calcine. Studies have shown that PW-4b (low-sodium) simulated waste is still free-flowing after being heated at 900°C , whereas PW-6 (high-sodium) simulated waste agglomerates above 310°C . Simulated Savannah River neutralized waste was also found to be free-flowing after being subjected to 900°C temperatures.

Experience with calcine shows that it does not flow freely from bulk storage, that it dusts, that it is hygroscopic, and that it has a high surface activity. Bin-storage and conveyance of the calcines may be difficult. However, because the calcine is very active and presents large surface areas, it makes good feed for melting or pelletizing processes.

Calciner Materials Experience

Experience at PNL has primarily been with 310 SS spray chambers. Stainless steel 310 was selected for its high-temperature strength and oxidation resistance. Wall thickness

TABLE 13. Properties of Simulated HLLW Spray Calcines

Characteristic and Units	PW-4b	PW-6	PW-7a	Defense
Aerated Bulk Density, g/c^3	0.85	0.41	0.69 to 0.92	0.95
Working Bulk Density, g/c^3	0.97	0.48	0.87 to 1.04	1.03
Packed Bulk Density, g/c^3	1.24	0.61	1.15 to 1.33	1.27
Compressibility, %	32	33	40 to 31	25
Angle of Repose, degrees	42	39	43 to 42	27
Particle Density, g/c^3	4.4	3.8	5.1	Var. (a)
Surface Area, m^2/g	20	11	14	---
Nitrate Content, wt%	<1	19 to 42	2 to 22	---
Moisture Content, wt%	<1	<1	<1 to 4	1 to 3
pH of Equilibrium Aqueous Slurry	4.5	5.0	---	7.2

(a) The calcine particle density is variable (about $4.3\ \text{g}/\text{c}^3$ average) because particles from simulated defense waste slurries remain as separate and distinct compounds--i.e., the calcine is composed of MnO_2 particles, NiO particles, zeolite particles, particles containing water-soluble compounds, etc.

measurements have been made on calciners operated at PNL to determine the extent of corrosion. Measurements showed no detectable corrosion when a pilot-scale, 5.3-cm-dia spray chamber was tested after 522 h of acid-waste calcining at 700°C that included six heatup and cooldown cycles. The instrument used for the measurements had a precision of 0.00051 cm. A calciner corrosion/erosion rate of less than 0.0076 cm/yr was calculated based on this test (Bonner, Blair and Romero 1976).

Wall thickness measurements were made in March of 1980 on the full-scale 91.4-cm-dia spray chamber after 1650 h of actual operation. The operating period consisted of about 200 h with acid-type feed, 1450 h with neutralized feed, 37 heatup and cooldown cycles, and roughly 1000 h of idle time at temperatures between 700°C and 800°C. Hence, the spray calciner has been at 700°C to 800°C temperatures for about 2650 h. The differences between all thickness measurements made in April of 1977 and March of 1980 (Figure 19 and Table 14) indicate a maximum average loss in thickness of 0.033 cm. Based on the actual hours of equipment operation, a corrosion/erosion rate of 0.18 cm/yr is indicated. Based on the approximate hours at operating temperatures, a corrosion/erosion rate of 0.11 cm/yr is indicated. A corrosion allowance of 0.25 cm would be exceeded in 1.4 to 2.5 years at these corrosion rates.

The two sets of measurements for the pilot-scale and full-scale calciners indicate widely differing results. Additional operating time is needed to verify which of them more closely represents the actual case.

The cone section (304L SS) of the FSSC did not show any visual corrosion after 1600 h of operation. The cone temperature varies between 200°C and 350°C during calciner feeding and ranges from 400°C to 700°C during equipment idling while at operating temperature. Comparative wall thickness measurements are not available to determine measurable corrosion rates.

Inconel-601 appears to be superior to stainless steel 310 in oxidation resistance, scaling resistance, and mechanical strength based on the literature (Huntington Alloys, Inc. 1973). Consideration should also be given to Inconel-625 if added corrosion resistance is determined to be necessary. While it is not quite as resistant to high-temperature (about a 1200°C limit) oxidation as Inconel-601 is, Inconel-625 would be better suited for corrosive environments containing acids, chlorides, or fluorides, and would still most likely maintain an acceptable oxidation resistance.

FIGURE 19. Location of Wall Thickness Measurements

TABLE 14. Wall Thickness Measurements for Spray Calciner Chamber (see Figure 19)

Height From Bottom Flange, cm	Wall Thickness, cm							
	April 1977				March 1980			
	1	2	3	4	1	2	3	4
30.48	0.645	0.633	0.638	0.633	(a)	(a)	(a)	(a)
60.96	0.648	0.633	0.645	0.643	(a)	(a)	(a)	(a)
91.44	0.650	0.653	0.650	0.650	0.630	0.625	(a)	0.638
121.92	0.650	0.653	0.655	0.655	0.627	0.627	0.640	0.627
152.40	0.660	0.658	0.658	0.658	0.635	0.625	0.630	0.633
182.88	0.660	0.653	0.658	0.655	0.627	0.630	0.630	0.627
213.36	0.650	0.653	0.655	0.653	0.620	0.615	0.617	0.620
243.84	0.650	0.645	0.650	0.650	0.617	0.627	0.612	0.617
274.32	0.645	0.650	0.655	0.648	0.620	0.633	0.607	0.605
304.80	<u>0.650</u>	<u>0.650</u>	<u>0.660</u>	<u>0.655</u>	<u>0.622</u>	<u>0.630</u>	<u>0.612</u>	<u>0.635</u>
Avg: 30.48 to 304.80	0.650	0.648	0.653	0.650				
Avg: 91.44 to 304.80	0.653	0.653		0.653	0.625	0.627		0.625
Avg: 121.92 to 304.80			0.655				0.622	

(a) Lower furnace zone not removed at this time; making measurements at these locations is impossible.

Average Loss in Wall Thickness, cm

Location	April 1977	March 1980	Difference
1 (91.44 to 304.80 cm)	0.653	0.625	0.028
2 (91.44 to 304.80 cm)	0.653	0.627	0.026
3 (121.92 to 304.80 cm)	0.655	0.622	0.033
4 (91.44 to 304.80 cm)	0.653	0.625	0.028

Yearly Corrosion Rate Based on Hours of Actual Operation

$$\frac{0.033 \text{ cm}}{1650 \text{ h operation}} \times \frac{8760 \text{ h}}{\text{yr}} = 0.18 \text{ cm/yr}$$

Yearly Corrosion Rate Based on Hours at Operating Temperature

$$\frac{0.033 \text{ cm}}{2650 \text{ h}} \times \frac{8760 \text{ h}}{\text{yr}} = 0.11 \text{ cm/yr}$$

Vibrators

Under normal operating conditions with low-sodium feeds, calciner chamber wall scale is minimal, and extended operation without vibration is possible. But to extend the flexibility and operability of the unit, vibrators have been introduced to keep wall scale to an inconsequential level.

Successful operation has been experienced with a single vibrator mounted on top of a small-scale calciner. But vibration was quite inefficient, and vibrators had to be replaced frequently because of lubrication problems caused by the high-temperature environment (Bonner, Blair and Romero 1976). Further investigation led to use of two specially modified vibrators of the kind shown in Figure 20 mounted at mid-height on the spray chamber, 180° apart. The vibrator has an extended piston that passes through holes in the furnace and strikes on the chamber wall. This arrangement not only limits wall scaling more effectively, but also locates the vibrators where they are less likely to overheat.

To help determine the effect of vibration on the spray chamber, a 100-h test was conducted with a 53.3-cm-ID, 0.95-cm-wall 310 SS chamber to simulate up to 4000 h of operation (Bonner, Blair and Romero 1976; Blair 1976). The diameter of the spray chamber across the impact pads and at 90° to the pads was measured before and after the 700°C calciner walls experienced 4000 vibrator cycles. No measurable difference in the calciner shape was found when measuring to 25 μ .

Use of the impact sound intensity is the easiest way to determine whether the vibrator is hitting correctly. If the vibrator is too far from the wall, the noise of impact will of course be absent; if the vibrator is too close to the wall, the sound of impact will be muffled since the piston is then almost always in contact with the chamber. A method of adjustment is needed to assure proper spacing between the vibrator shaft and the spray chamber.

Originally, the full-scale calciner was equipped with 1.91-cm-thick vibrator impact pads welded onto the outside of the chamber wall to add strength. The weld on one of these impact

FIGURE 20. Side-Mounted Calciner Vibrator

pads failed during a test, causing the pad to fall to the bottom of the furnace and short out a furnace element. The probable cause of the weld failure was the difference in thermal expansion and heat retention between the 1.91-cm-thick pad and the 0.64-cm-thick wall. The failed impact pads were replaced by cutting 38.1-cm-dia holes around the pads and replacing them with circular 1.91-cm-thick plates rolled to match the diameter of the chamber (Figure 21). This new impact design was tested, but poor weld penetration made the weld insufficient. It was ground out and rewelded. After the pads were rewelded (which required several weld passes to refill the groove that had been ground out) the spray chamber surfaces around the circular impact pad became deformed. The 1.91-cm impact pad retained welding heat longer than did the 0.64-cm chamber wall, which caused the chamber to buckle slightly while cooling. Other than being slightly out-of-round, no structural damage to the spray chamber is evident, and calciner operation has been satisfactory. If the vibratory pad installation welding had been satisfactory on the first attempt, it is doubtful that the chamber distortion would have been encountered.

Originally, vibrators on PNL's FSSC were mounted on the side of the furnace zones. Forces from operation of the vibrators caused damage to the furnace fire brick. Supporting edges for the electrical-resistance furnace elements cracked and broke away, allowing furnace elements to fall out of place. Vibrator supports were modified after ~600 h of operation to support the vibrators independently of the furnace. Attempted repair of the furnace brick at this time succeeded only temporarily in holding the heating elements in place. The low-cost resistance furnace required replacement of coiled-wire heating elements on four occasions during the first 1600 h of operating time and during 35 heatup and cooldown cycles. Furnace brick in two furnace zones were replaced after 1600 h of operation due to the initial damage resulting from mounting the vibrator onto the furnace zones. It is expected that appropriate furnace design and independent mounting of the vibrators would lead to an acceptable resistance-heated furnace life for plant application.

A vibrator analysis design study is attached in Appendix C. Also, a spray calciner structural analysis is attached in Appendix D.

FIGURE 21. Modified Vibrator Impact Pad

Design Aspects

The design of a spray calciner is discussed in the following two areas:

- furnace requirements
- spray-chamber size requirements.

Calciner Furnace Requirements

Energy needed to dry and calcine liquid waste is supplied by the calciner furnace. Also, a significant portion of the total energy supplied by the furnace is lost by conduction through the furnace walls and through the insulation covering the top of the furnace. If induction heating is used, additional heat will be lost to the induction-coil cooling water. Therefore, both the energy used to dry the waste and the energy lost to the surrounding environment must be included for a determination of furnace power requirements.

Furnace power consumption measurements for the FSSC have been made for various calciner feedrates at chamber wall temperatures of 750° to 800°C. These measurements are shown in Figure 22 for total power consumed and in Figure 23 for power consumption per zone. As can be seen from the figures, power consumption varies linearly with feedrate.

An excellent design method for determining the furnace power requirements for drying and heating the waste feed (calcination can be neglected) is to calculate the sensible and latent heat required to raise the inlet waste feed and atomizing air to the desired spray calciner outlet temperature. For most waste feed compositions this power requirement can be closely estimated by assuming that a liter of waste feed is a liter of water. Calculated and measured power consumption for various flowrates are shown in Table 15. Measured values compare very closely with the calculated values for the range of data available.

Furnace power consumption is not axially uniform. Data from actual calciner operation, as shown in Figure 23, indicates that the lower half of the furnace (zones 1 and 2) supplies the greatest proportion of the power. Possible explanations for this occurrence are: 1) the

FIGURE 22. Calciner Furnace Total Power Consumption

FIGURE 23. Calciner Furnace Zone Power Consumption

TABLE 15. Calculated and Measured Heating Requirements

Flowrate, L/h	Total kW ^(a) Used (measured)	kW Lost ^(b)	kW Absorbed ^(c) (measured)	kW Absorbed ^(d) (calculated)
0	60	60	0	---
200	212	60	152	158
300	290	60	230	234
400	365	60	305	310
500	---	---	---	386

- (a) Measured kW at 750° to 800°C chamber wall temperatures for a PNL full-sized spray calciner.
- (b) Kilowatts lost through the top of the furnace and the furnace walls to the cell. The kilowatts lost could vary significantly between individual calciners. Induction-heated calciners will lose additional heat through the induction-coil cooling water.
- (c) Kilowatts absorbed by the waste feed and atomizing air (total kW used minus kW lost).
- (d) Basis for calculated kW absorbed: i) 89 kg/h of air (atomizing air, in-leakage) heated from 25° to 250°C, and ii) the indicated feedrate (assumed to be water) heated from 50° to 250°C.

atomizing nozzle spray pattern is wider in the lower portion of the calciner, which increases the radiative heat transfer coefficient since more particles are exposed directly to the calciner wall; 2) the convective heat transfer coefficient is greater in the lower portion of the calciner because of the high turbulence caused by the large volume of steam generated as the droplets fall through the calciner; 3) the general direction of the convection currents is upward next to the heated calciner wall and downward near the cooler center region of the calciner, which transfers heat from the lower zones to the upper zones and reduces the power

required to maintain the calciner temperature set point in the upper zones. The actual explanation for the power consumption differences between the zones is most likely a combination of the above effects. These effects must be considered when calculating the calciner furnace power capacity per zone. For example, the total measured power consumption for 300 L/h is 290 kW, but the minimum capacity for a four-zone furnace should not be evenly divided into 72.5 kW/zone. The lower two zones each supply about 30% of the power and the upper two zones each supply about 20%. Therefore, the minimum rated capacity of the lower two zones should be 87 kW and that of the upper two should be 58 kW. Calciner designs at PNL have all the zones at equal power, which for 300 L/h should be an 87-kW minimum per zone for a four-zone furnace. In addition, a safety factor should be added to the minimum calculated power requirement.

The number of furnace zones required depends upon the desired uniformity of longitudinal chamber temperatures and the cell height available for removing and installing the furnaces if annular furnaces are used. As discussed previously, the power required to maintain a uniform chamber temperature varies axially. For maximum heat transfer into the calciner, a uniform chamber temperature at the highest operating temperature possible is desirable. Multiple furnace zones provide the most flexibility in attaining the uniform maximum temperature. Also, if the furnace is designed for removal of individual zones, the more furnace zones for a given chamber height, the less the cell height required to lift each annular furnace zone over the spray chamber. A disadvantage of more zones is that more electrical connections and control thermocouples are required. The PNL full-scale calciner is equipped with four furnace zones and provides excellent temperature control with a manageable number of electrical connections. As few as two furnace zones could be used to provide adequate temperature control, since the power consumed by zones 1 and 2 is similar and that consumed by zones 3 and 4 is similar (Figure 23). Use of "clam shell" type furnace zones would not require lifting the furnace zones over the spray chamber for placement.

The electrical power to each furnace zone may be controlled with a zero-crossover, silicon-controlled-rectifier (SCR) power supply, using as a reference temperature a thermocouple penetrating through the side of the furnace wall at the middle of each zone. The reference temperature should be recorded, and an alarm activated outside the control range to detect malfunctions of the control system. At least one redundant thermocouple for each zone that could be used for electrical power control should also be recorded and alarmed. Additional thermocouples to monitor the calciner operation could be positioned on the calciner spray chamber, on the cone section directly below the centerline of the spray chamber, and in the offgas filter zone. The temperatures should be recorded to detect long-term changes that would signal buildup of scale or other operating problems.

A calciner stress analysis study for a remotely operated furnace is attached in Appendix D.

Spray Chamber Size Determination

The calciner spray chamber heat-transfer area required to produce a dry powder depends on the following factors: 1) calciner feedrate, 2) feed atomization (drop diameter), and

3) calciner chamber temperature. The calciner feedrate is selected for process design. Feed atomization is achieved by proper nozzle operation, as discussed in the atomizing nozzle section. Determination of the proper chamber wall temperature is discussed in the calciner operation section. On definition of these parameters, the calciner chamber heat-transfer area can be estimated.

Correlations relating capacity to spray chamber diameter, height, and wall temperature have been developed from PNL calciner operating data. Table 16 lists data that was obtained from pilot-scale and full-scale calciners. Computer regression analysis of this data yields the correlations shown in Table 17. Using the data points indicated, equations were developed to 1) relate capacity to wall temperature, chamber diameter, and chamber height; 2) relate capacity to chamber height and diameter at 700°C wall temperature; 3) relate capacity to chamber height and diameter at 550°C wall temperature; and 4) relate capacity to temperature at a chamber diameter of 91.4 cm and height of 320 cm. Capacity was defined in these tests as the maximum feedrate that produced a powder that would flow freely through the calciner cone section and melter connecting section. Based on experience, an offgas temperature of 200°C from the full-scale calciner was used as the reference process variable that would indicate a dry, "flowable" powder was being produced. Therefore, the calciner feedrate that produced an offgas temperature of 200°C was defined as the maximum capacity at the chamber wall temperature. At maximum pilot-scale calciner capacity, the measured offgas temperature was normally slightly less than 200°C (about 180°C). Therefore, the offgas temperature for which a dry, flowable powder is produced will vary among calciners.

Other considerations, such as condensation of volatile materials from the melter (Ru, Cs, etc.), may require that an offgas temperature higher than the minimum required to produce a

TABLE 16. Calciner Capacity Data

Data Point	Wall Temp., °C	Dia, cm	Height, cm	Capacity, L/h
1(a)	700	91.4	320	315
2(a)	700	91.4	240	205
3(b)	700	53.3	198	95 ^(c)
4(b)	700	33.5	198	40
5(a)	550	91.4	320	160
6(a)	550	91.4	240	105
7(b)	550	53.3	198	62
8(b)	550	33.5	198	26
9(a)	400	91.4	320	50

(a) Data from PNL test No. FSSC 38.

(b) Data from Bonner, Blair, and Romero (1976).

(c) This capacity was estimated because furnace power capacity was not sufficient to maintain wall temperature (Bonner, Blair and Romero 1976). Deletion of this data point would not significantly affect Equation 1 in Table 17, but may affect Equation 2.

TABLE 17. Calciner Capacity Correlations

Data Points Used From Table 16	Equation	Ave. % Error in Predicting Data Points	Standard ^(a) Deviation From Ave. % Error
1 through 9	Capacity L/h = $7.477 \times 10^{-11} (T, ^\circ\text{C})^{2.83} (\text{Dia, cm})^{1.41} (\text{H, cm})^{0.705}$ (1)	12.4	9.1
1 through 4	Capacity (L/h) @ 700°C Wall Temp. = $0.0011 (\text{Dia, cm})^{1.46} (\text{H, cm})^{1.03}$ (2)	8.0	6.0
5 through 8	Capacity (L/h) @ 550°C Wall Temp. = $0.0048 (\text{Dia, cm})^{1.28} (\text{H, cm})^{0.793}$ (3)	11.6	8.6
1, 5, 9	Capacity L/h @ 91.4-cm dia, 320-cm H = $1.277 \times 10^{-7} (T, ^\circ\text{C})^{3.31}$ (4)	5.1	2.9

$$(a) \text{ Standard Deviation} = \sqrt{\frac{(\text{Ave. \% error} - \% \text{ error})^2}{\text{Degrees of Freedom}}}$$

flowable powder be used to prevent accumulation of volatile materials within the calciner. Each 50°C increase above the minimum offgas temperature required to produce a flowable powder (about 200°C) will decrease the capacity indicated in the equations in Table 17 by ~10%. Additional long-term testing with full loadings of feed constituents (such as ruthenium) is required to determine if accumulation of volatile materials in the calciner or melter is a problem.

The correlations in Table 17 are reasonably accurate in predicting the data from Table 16. Equations 2 through 4 are more accurate in predicting the data points used (as indicated by the average percent error) than is the more general Equation 1.

The correlations indicate capacity is most dependent on wall temperature, with chamber diameter being the second most important, and chamber height having the least effect on capacity. While developing an accurate capacity equation based on heat-transfer theory for spray calcination would be very difficult and costly, brief consideration of simplified heat-transfer considerations shown below give some understanding of the relative importance of the variables in Table 17:

$$q_T = q_c + q_r, \quad (5)$$

$$q_c = UA\Delta T, \quad (6)$$

$$q_r = A\sigma F_{12} T_1^4 - A_2\sigma F_{21} T_2^4, \quad (7)$$

where

q_T = total heat flux, q_c = heat flux transferred by convection, q_r = heat flux transferred by radiation

U = overall heat-transfer coefficient

A = heat-transfer area, A_1 = heat-transfer area of hot surface, A_2 = heat-transfer area of cold surface

ΔT = temperature difference, T_1 = temperature of hot surface, T_2 = temperature of cold surface

F = view factor

σ = constant.

The drying capacity of a spray calciner is proportional to the amount of heat transferred into the spray chamber (assuming evaporation of water from the surface of the droplet is not rate-limiting, which appears to be the case for the simulated wastes tested). This heat is transferred from the chamber wall to the spray droplets by two mechanisms: 1) convective heat transfer, and 2) radiative heat transfer. Convective heat transfer, as simplistically represented by Equation 6, is roughly proportional to the first power of the temperature difference--whereas radiative heat transfer is proportional to the difference of the fourth powers of the temperatures. Therefore, one would expect that a combined temperature term for convective and radiative heat transfer would be between the first and fourth powers of the temperature. This is the case for Equations 1 and 4 of Table 17. Since the exponents of temperature in Equation 4 and Equation 1 are closer to four than one, radiative heat transfer appears to be the primary mode of heat transfer at these temperatures.

The diameter of the spray chamber affects the heat-transfer area and the residence time of a particle within the spray chamber. Increased residence time allows a particle to receive more of the heat flux represented in Equations 6 and 7. The heat-transfer area is proportional to the first power of the diameter ($\pi \text{ Dia} \times H$), whereas the residence time is proportional to the volume of the spray chamber ($\pi/4 \text{ Dia}^2 \times H$) and the square of the diameter. Therefore, a single term representing the effect of diameter on capacity would be expected to be between the first and second power of the diameter. This proves to be the case in Equations 1 through 3 of Table 17.

The height of the spray chamber also affects the heat-transfer area and the residence time of a particle. Both the heat-transfer area ($\pi \text{ Dia} \times H$) and volume ($\pi/4 \text{ Dia}^2 \times H$) of the spray chamber are proportional to the first power of the height. Therefore, a single term representing the effect of chamber height on drying capacity would be expected to be approximately the first power of the height. Equations 1 and 3 in Table 17 are slightly less than the first power of the height, while Equation 2 is very close to the first power of the height. At lower temperatures the drying capacity appears to be less dependent on height and diameter of the spray chamber than at higher temperatures (see exponents of Equations 2 and 3 of Table 17).

OFFGAS FILTRATION

Sintered-metal filters, used to filter particulates from the spray calciner offgas stream, are discussed in this section.

Operating Principles

Sintered-steel filters have found use in the nuclear industry in applications requiring high-efficiency filtration of thermally hot and/or highly radioactive vapors. Experience in spray calciner development during the last 20 yr has demonstrated high reliability and high efficiency of the filters over a wide range of operation conditions. Due to the small particle size of spray-dried calcine, much of the solid particles are entrained by the calciner offgas. Sintered-metal filters have proven to be very efficient for removing these particles from the calciner offgas.

During calciner operation, a layer of powder filtered from the offgas builds on the filter's surface and forms an additional filter media. As this layer of powder becomes thicker, the filter pressure drop increases. Eventually, the pressure drop will increase until the calciner has insufficient vacuum or the desired filter pressure drop is exceeded. Before this occurs, a 0.25-s pulse of high-pressure air is directed counter-currently to the offgas stream from above the filters. This reverse airflow, or filter blowback, dislodges the layer of powder from the filter surface and allows it to fall to the melter below. To prevent pressurization of the calciner during filter blowback, a short air pulse is used and only a few of the filters are blown back at one time. This allows the offgas to be drawn through the remaining filters to maintain a vacuum in the calciner. This pulsed blowback is alternated between the remaining small groups of filters until all filters have been cleaned.

The blowback air is routed to each group of filters that are blown back simultaneously through a separate line controlled by a solenoid-operated valve. A small venturi is mounted at the top of each filter to draw calciner offgas back into the filters to provide additional blowback air volume.

Operating Experience

Filter efficiency improves as a layer of calcine forms on the filter. The calcine coat acts as the filter media to prevent very small particles from reaching the sintered-metal pores, which may otherwise result in gradual plugging of the filters. Therefore, fewer filter blowbacks should increase the operating life of the filters by minimizing the time the filter surface is exposed to moving particles and very small particles.

During the first five WFP spray calcination runs at PNL (Bonner, Blair and Romero 1976), the filters were left undisturbed and the effect of calcine buildup was observed. During the first run, the average filter particulate DF was about 700. During each successive run, the DF steadily improved until during run 5 the DF had improved to 13,000. Following this run, tests with filter-washing were initiated that reduced the DF to near the clean-filter efficiency. Since filters will normally not be washed, it is anticipated that in an operating plant the calciner filter DF for particulates will be well above 10^3 . A filter decontamination

factor of 2.7×10^4 was measured for particulates during extended full-scale tests. Actual spray calciner permeability data is presented in Figure 24 (Bjorklund 1976).

Two basic types of blowback patterns have been used with PNL's FSSC--equal and unequal intervals. These patterns are illustrated in Figures 25 and 26. The filters are divided into several groups. The filters in each group are blown back simultaneously from the air supply. In the unequal filter-blowback pattern, each filter group is sequentially blown back in a short time with 1 s between group blowbacks. Then a long interval (up to an hour) is allowed to pass before the blowback cycle is repeated. The equal-interval blowback pattern sequentially blows back each filter group with the same time period (e.g., 1 min) between each blowback.

The equal-interval blowback pattern provides a more continuous flow of calcine to the melter than does the unequal pattern because much of the calcine is entrained to the filters and remains there until blowback. Also, the equal-pattern blowbacks can be timed with the frit airlock valve operation to allow batches of calcine and frit to fall simultaneously to the melter, thereby better mixing the calcine and frit. For the unequal-interval blowback pattern, it is expected that calcine-frit mixing then can be accomplished by using a very fine frit that is entrained with the offgas, or the frit components can be added to the waste feed.

For the equal-interval blowback pattern, the gas velocity through the clean filters is much greater than that through the filters that are still coated after a group of filters is blown back. The filter manufacturer's maximum recommended filter-face velocity (offgas flow-rate divided by filter area) is 1.8 m/min. But for a group of filters just cleaned in a

FIGURE 24. Spray Calciner Permeability Data

FIGURE 25. Unequal-Interval Filter Blowback Pattern

FIGURE 26. Equal-Interval Filter Blowback Pattern

filter system, the actual face velocity can approach several times the maximum recommended value due to the reduced pressure drop relative to the remaining filter groups. Excessive gas velocity may drive small particles deep into the filter pores, where they could be difficult to remove by filter blowbacks--eventually causing an increased resistance to gas flow. Of course, this high velocity drops off quickly as a layer of powder redeposits on the filter surface, but the face velocity for the recently "cleaned" filter remains higher than the average velocity for the entire filter system for a significant period. For the unequal-interval blowback pattern, all filter groups are cleaned in the shortest possible time that will not cause the calciner to lose vacuum from the air pulses. This minimizes the time that a group of clean filters will have greater-than-normal face velocities.

To summarize the advantages and disadvantages of the two filter blowback patterns, the equal-interval pattern provides a more continuous flow of calcine to the melter than does the unequal-interval pattern, whereas the unequal-interval pattern may allow longer filter life by minimizing the filter-face velocity after blowbacks.

For the unequal-interval blowback pattern, the frequency of filter blowbacks should be maintained to allow the filter pressure drop to approach the maximum desired value (about 10 to 20 in. H_2O) before blowbacks occur. For the equal-blowback pattern, a more moderate pressure drop should be maintained (about 6 to 10 in. H_2O); this will reduce the gas velocity through a group of filters after blowback.

It is desirable to minimize the calciner pressure surges caused by the pulses of blowback air. Variables that affect the magnitude of these surges are blowback pulse duration, blowback pressure, offgas line flow resistance, and the fraction of filters pulsed simultaneously. Experience has shown that short, 0.25-s pulses of ambient-temperature air are as effective as longer pulses for cleaning the filters. Static blowback pressures of 40 psig to 100 psig have been successfully used. One-fifth of the filters are blown back without pressurizing a pilot-scale calciner (30 to 70 L/h feed), while 1/11 of the filters in a large-scale calciner (300 to 400 L/h feed) are blown back simultaneously.

Tests have been conducted with a combination of cyclone and filters. When a cyclone was used upstream of the filters, the filter cake was not easily removed by the pulsed blowback (Kaser and Moore 1966). With the cyclone, only the finest particles reached the filter, and these particles formed an especially adherent filter cake. Preliminary tests using a cyclone alone for offgas filtration resulted in a particulate DF of 20.

Coupling the calciner to a melter has not been noted to affect the operation of the spray calciner filters. However, during periods of extended calciner shutdown the amount of volatiles from the melter should be minimized by lowering the melter temperature to just above the melting temperature of the glass. Also, the filters should not be blown back just prior to shutdown; a layer of calcine should be left on the filter surfaces. This layer of calcine will allow volatiles from the melter that condense on the filters to be blown off. Extended periods of shutdown with a hot melter connected to the calciner should be avoided.

The minimum offgas temperature established by experience from which a dry, flowable powder is produced (with proper atomization) is 200°C. Other considerations, such as volatiles condensing on the filters and causing pluggage, may increase this minimum temperature. Long-term testing with components such as mercury, ruthenium, and cesium has not been done, so it has not been determined if an offgas temperature higher than 200°C is necessary to allow these volatile components to pass through the filters (which prevents pluggage).

The maximum allowable filter operating temperature is determined by the limitations of the sintered-metal filter material or the powder-layer melting temperature where the powder layer begins to consolidate. After discontinuing liquid feeding, filter temperatures will increase if atomizing air flow is continued and furnace temperatures are not reduced.

Therefore, it is important to monitor filter temperatures after waste-feeding to the calciner has been stopped in order to avoid exceeding the maximum recommended filter material operating temperature or the sintering powder temperature.

Design Aspects

The sintered-metal filters in use are fabricated from metal powder that is formed into a sheet and sintered. The plate formed is then rolled into a tube and welded. Another technique involves extruding the metal powder as the tube before sintering to form a seamless filter. One company grinds the external surface of the sintered filter to provide a smoother surface and to make release of the cake easier. Varying the metal particle size in fabrication alters the filter pore size. Small pores provide the best filtration efficiency but also exhibit the largest resistance to vapor flow. This layer acts as a filtering media and produces a larger differential pressure. The sintered-metal tube is welded to a venturi as shown in Figure 27. Each venturi is welded into a plate as shown to form a filter bank.

To minimize the effect of vibration or mechanical shock during installation and operation, a support rod of the type shown in Figure 28 is installed in each filter and the ends of the support rods are also connected together by reinforcing rods, as shown in Figure 29. An

FIGURE 27. Filter Element with Venturi and Blowback Nozzle

FIGURE 28. Filter Element with Support Rod

alternate method for reinforcing the sintered filter media is to support it with a perforated internal shell, as shown in Figure 30 (Bond et al. 1970).

Filter-face velocity (gas flowrate divided by filter area) is an important filter-design parameter. The manufacturer's maximum recommended face velocity is 1.83 m/min. A conservative design face velocity of 0.91 m/min is normally used at PNL.

Filters with nominal pore sizes of 10 and 65 μ have been tested. No conclusive results have been established as to the optimum filter pore size. A sintered-metal filter manufacturer has recommended that 10- μ filters be used instead of 65- μ filters to prevent more of the very small calcine particles from penetrating the filter pores immediately after a filter blowback when the filter surfaces are new. After a layer of calcine has formed on the filter surfaces, the filtering efficiencies of the various pore sizes would be essentially identical, since the layer of calcine is then the primary filtering media.

A typical filter and blowback system is shown in Figure 31. The use of a venturi is intended to increase the entrainment of filtered gas by the blowback gas jet, thus decreasing the amount of external air required to clean the filters.

FIGURE 29. Filter Element Reinforcing Rods

FIGURE 30. Spray Calciner Filter Assembly

A rapid filter pressurization is believed desirable for uniformly removing calcine cake from the filter. Consequently, during the WSEP Program at PNL (Bonner, Blair and Romero 1976), blowback air valves were located adjacent to the filters. Some problems were encountered,

FIGURE 31. Typical Filter and Blowback System

however, due to radiation-induced failure of the solenoid valves used. Valves that will withstand the radiation levels expected during HLLW processing are expensive and a nuisance to maintain in-cell. Therefore, out-of-cell placement of blowback valves is desirable. No significant detrimental effect on blowback efficiency is found when the valves are moved 4.9 m from the filters.

The low filter differential pressure encountered in the spray calciner is only a few percent of the allowable differential for unreinforced sintered-metal filters. Therefore, it is highly unlikely that failure due to over-pressure would occur. The primary design parameter concerning the filter pressure drop is the size of the offgas vacuum blower necessary to maintain the required calciner vacuum (10 in. H_2O). Operating parameters (see "Operating Experience" section) to minimize the filter-face velocity may limit the filter pressure drop to between 7 in. and 20 in. H_2O .

The optimum filter material has not been established. Experience has primarily been with 316 SS. One assembly of 316 SS filters was severely corroded after ~1600 h of run time and 34 heatup and cooldown cycles. Analysis of the metal showed that corrosion products existed not only on the filter surface, but penetrated the depth of the filter material. Metallurgical scanning electron microscope (SEM) analysis of a section of the filter material did not indicate any residual chloride or fluoride within the filter pores, or any other residual compounds. Since the corrosion existed throughout the thickness of the metal (though no residual compounds could be found within the filter pores), the corrosive species must have been volatile or soluble in the flush water. Chloride, fluoride and sulfate ions are likely the major cause (Dierks et al. 1980). Another contributing factor to the filter failure is that the filter temperatures exceeded 500°C during a 25-d test. The filters were at 475°C to 525°C for 12 h, 525°C to 575°C for 5 h and 575°C to 590°C for 1 h. While these temperatures do not usually cause any problems for 316 SS, the extremely fine metal-to-metal bonds in the sintered metal are very susceptible to the slightest oxidation. A small amount of corrosion that is hardly detectable in vessels or piping can severely damage filters. Inconel-625 or Hastelloy should provide improved performance, but long-term experience with these materials has not been established for a spray calciner.

Steps can be taken to partially remove particle buildup in the filter pores. With calciner feeding and melting stopped, the filters can be repeatedly blown back for approximately 24 h while the offgas flow and filter-face velocity are at a minimum. This can remove some of the particles trapped in the filter pores and temporarily reduce the filter pressure drop. Also, washing the filters with water or nitric acid when the calciner furnaces are off has effectively cleaned the filters (Bonner, Blair and Romero 1976). The washing was done by trickling water or nitric acid into the filter from above the venturi while activating the blowback cycle. The liquid can be introduced through the blowback line. This procedure is not expected to be required routinely, but the capability should be available if needed. The wash solution can be collected in an empty canister and evaporated during the melter heat-up cycle.

PROCESS FLOWSHEET AND HEAT BALANCE

The spray calciner flowsheet and heat balance for typical operating conditions are discussed in this section.

PROCESS FLOWSHEET

A typical spray calciner flowsheet with material balance is shown in Figure 32 for a neutralized Savannah River Waste composition (simulated). Approximately 352,000 L of simulated waste of similar composition have been processed in PNL's FSSC. The flowsheet indicates a typical feedrate of 300 L/h to the spray calciner atomized by 30 scfm air in a Spraying Systems Co. Set-Up No. 82 nozzle. The calcine produced from this feed concentration is combined with 50.4 kg frit/h to produce 70 kg/h of glass product.

PROCESS HEAT BALANCE

The spray calciner heat balance for neutralized and acidic simulated wastes is shown in Figure 33. The following bases and assumptions are used to develop the heat balance:

- liquid feed and atomizing air temperatures are 50⁰ and 25⁰C, respectively;
- outlet temperature for offgas and calcine is 250⁰C;
- enthalpy changes in chemical decompositions are assumed to be negligible, except for HNO₃;
- calcine heat capacity is assumed to be 0.20 cal/g⁰C (representative of most oxides);
- frit is assumed to collect a negligible amount of heat in the calciner;
- half of the HNO₃ is assumed to go to NO and half to NO₂ for acid wastes.

The balance is at typical operating conditions of 300 L/h feedrate and 30 scfm atomizing airflow.

SPRAY CALCINER NORMAL OPERATING PARAMETERS @ FLOW CONDITIONS BELOW

FILTER PRESSURE DROP	3-10 IN H ₂ O
CALCINER FURNACE POWER CONSUMPTION	(kW)
ZONE 1	90
ZONE 2	92
ZONE 3	55
ZONE 4	53
TOTAL	290
CALCINER FURNACE TEMPERATURE	(°C)
ZONE 1	875
ZONE 2	875
ZONE 3	840
ZONE 4	840
CALCINER SPRAY CHAMBER WALL TEMPERATURE	(°C)
ZONE 1	750
ZONE 2	755
ZONE 3	755
ZONE 4	800
CALCINER FILTER HOUSING TEMPERATURE	(°C)
UPSTREAM OF FILTERS	250
DOWNSTREAM OF FILTERS	240
CALCINER CONE TEMPERATURE	(°C)
DIRECTLY UNDER BARREL	230
CALCINER HEAT LOSS AT ABOVE FURNACE TEMPERATURES AND G/LITER/HR FEEDRATE (MEASURED) 60 kW	

PARAMETERS		STREAM #									
		1	2	3	4	5	6	7	8	9	10
MASS FLOW kg/hr		174.8	323.7	50.4	21.9	1.97	19.6	502.8	2		70
VOLUMETRIC FLOW	SCFM	30			10	0.90		203	1.3		
	STANDARD liters/hr	5.1x10 ⁴	300		1.70x10 ⁴	1.53x10 ³		4.48x10 ⁵	2260	85	
TEMPERATURE °C		AMBIENT 25	<50	AMB 25	AMB 25	AMB 25	250	250	~500	20	1050
PRESSURE	psig	35	25		0	40					
	inches H ₂ O VACUUM			5			5	5	5		5
SPECIFIC GRAVITY @ 25 °C			1.08							1.0	2.8
VOLUMETRIC FLOW AT LISTED CONDITIONS	CFM	9.69			10.9	0.26		511	3.8		
	liters/hr	1.65x10 ⁴	300		1.86x10 ⁴	448		8.68x10 ⁵	6480		

- (A) AIR FLOW AND PRESSURE REQUIRED FOR SPRAYING SYSTEMS CO. SET-UP #82 NOZZLE
- (B) AIR ASSUMED TO BE 79% N₂ AND 21% O₂ (BY MOLE OR VOLUME)
- (C) FEED CHEMICAL COMPOSITION (WT. %)
Fe(OH) ₃	53.16
Al ₂ O ₃ - 3H ₂ O	13.33
MNO ₂	10.61
CaCO ₃	4.84
NiO - 2H ₂ O	6.86
NaNO ₃	2.23
Na ₂ - SO ₄	1.09
ZEOLITE	7.89

4286 LITERS OF SLURRY IS COMPOSED OF 367.6 kg OF THE ABOVE COMPOSITION AND 4261 LITERS OF WATER AND WILL YIELD 1000 kg OF GLASS WHEN COMBINED WITH 720 kg OF FRIT.

(D) FRIT 211 COMPOSITION	(WT. %)
SiO ₂	58.3
Na ₂ O	20.6
Li ₂ O	4.4
B ₂ O ₃	11.1
CaO	5.6

(E) CALCINE COMPOSITION (ASSUMING FEED CHEMICALS ARE CONVERTED 100% OXIDE FORMS)	(WT. %)
Fe ₂ O ₃	52.14
Al ₂ O ₃	11.43
MNO ₂	13.93
CaO	3.56
NiO	~.37
Na ₂ O	1.07
Na ₂ SO ₄	1.43
ZEOLITE	10.36

(F) OFF-GAS COMPOSITION - (VOLUME OR MOLE %)	
H ₂ O	84.37
O ₂	3.26
N ₂	12.27
CO ₂	.06
NO _x	.04

(G) GLASS COMPOSITION	(WT. %)
Fe ₂ O ₃	14.6
Al ₂ O ₃	3.8
MNO ₂	3.9
CaO	5.3
NiO	1.7
Na ₂ O	15.4
Na ₂ SO ₄	.4
B ₂ O ₃	8.0
Li ₂ O	3.2
SiO ₂	43.7

FIGURE 32. Spray Calciner Flowsheet

	HLW FEED (IN)	ATOMIZING AIR (IN)	FURNACE HEAT LOSSES	CALCINE (OUT)	OFF GAS (OUT)	SC FURNACE POWER INPUT	FILTER BLOWBACK-AIR (IN)	AIR INLEAKAGE (IN)	FRIT (IN)	FRIT (OUT)
	①	②	③	④	⑤	⑥	⑦	⑧	⑨	⑩
NEUTRALIZED WASTE										
Kg/h	325	65.6	-	19.6	390	-	1.90	21.9	50.4	50.4
kW	0 ^(a)	0 ^(a)	60	1.0	230	291	0 ^(a)	0 ^(a)	0	0
°C	50	25		250	250		25	25	25	100
ACID WASTE										
Kg/h	373	65.6	-	27.8	432.8	-	1.90	21.9	55.7	55.7
kW	0 ^(a)	0 ^(a)	60	1.3	228	289	0 ^(a)	0 ^(a)	0	0
°C	50	25		250	250		25	25	25	100

NOTES:

ASSUMPTIONS LISTED IN TEXT

^(a) INLET FEED AND AIR CONDITIONS ARE USED
AS A ZERO REFERENCE

FIGURE 33. Spray Calciner Material and Heat Balance

PROCESS CONTROL PARAMETERS

Typical process control parameters that provide on-line process quality guidelines for the production of a satisfactory product are discussed in this section.

SPRAY CALCINER FEED SYSTEM OPERATING PARAMETERS

The feed system should provide a controlled flow of waste (at the proper pressure and of uniform composition) to the spray calciner. The waste feed flowrate should be maintained within 3% to 5% of the flow set point to provide the desired processing rate and frit-to-calcine ratio. The proper waste-feed pressure will depend on the size and type of nozzle. For the reference system with an internal-mix nozzle air-cap orifice of 0.793 cm, 20 to 25 psig feed pressure is needed at the nozzle for the required flowrate and atomization. See Table 18 for processing parameters of the feed system.

To prevent solids accumulation in the bottom of the feed tanks and to provide a uniform glass composition, sufficient tank agitation should be available to maintain less than a 5% variation in any feed component from the top to the bottom of a tank. The waste temperature should be maintained at $<50^{\circ}\text{C}$ to prevent excessive vaporization of the feed as it approaches the hot nozzle, which can result in a plug in the feed line or nozzle.

ATOMIZING NOZZLE OPERATING PARAMETERS

The degree of waste feed atomization for a given nozzle depends on the air-to-feed mass-flow ratio. The required mass ratio has been determined, by operating experience, to be >0.2 for an internal-mix nozzle with a 0.793-cm air-cap orifice. This required ratio will vary with type and size of the nozzle. A general rule is to refer to the nozzle manufacturer's specifications for the required flow conditions that will produce spray-droplet mean volume diameters of 70 to 100 μ when spraying water at room temperature.

TABLE 18. Spray Calciner Feed System Process Parameters

<u>Variable</u>	<u>Requirement</u>	<u>Discussion</u>
Feed Flow Control	Maintain waste feed flowrate within 3% to 5% of set point.	Provides desired glass composition and processing rate.
Feed Pressure	Provide necessary feed pressure to atomizing nozzle to allow proper atomization: 20 to 25 psig for an internal-mix nozzle with a 0.793 cm air-cap orifice.	Required feed pressure varies with type and size of nozzle. Refer to nozzle manufacturer's specifications for required feed pressure.
Feed Composition Uniformity	Less than 5% variation in any feed component from the top to the bottom of the tank.	Provides uniform glass composition. Prevents solids from settling and possible feed line pluggage.
Feed Temperature	Less than 50°C .	Required to prevent drying in calciner nozzle or feed line, which can result in a plug.

The diameter of the nozzle spray pattern must not cause direct spray impingement onto the spray chamber wall. The direct impingement could cause buildup of wet calcine on the spray calciner chamber walls. The spray chamber diameter must be larger than the diameter of the turbulent region of the spray pattern specified by the nozzle manufacturer. At PNL, nozzles that produce a spray pattern of 33 cm to 38 cm in dia when spraying water at room temperature have been successfully used in a 91.4-cm-dia spray chamber. An optimum spray-pattern diameter, if one exists, has not been determined. These operating parameters are summarized in Table 19.

SPRAY CALCINER FURNACE OPERATING PARAMETERS

Operating parameters for the spray calciner furnace are summarized in Table 20. Control of the spray chamber wall temperature by the furnace is needed to assure that the calcine feed slurry is converted to a powder that will freely flow into the melter. The powder must not build up on the calciner walls or cone section, which are sloped 90° and 60°, respectively, from the horizon. The extent of feed-component conversion to oxides (or calcination) is of secondary importance, since oxidation is completed in the melter.

To obtain a calcine that will readily flow into the melter, two conditions must be met:
1) the calcine must be maintained below its sintering temperature (the temperature at which

TABLE 19. Atomizing Nozzle Operating Parameters

Variable	Requirement	Discussion
Atomizing air-to-waste feed mass flow ratio.	Greater than 0.2 air-to-feed flow ratio for an internal-mix nozzle air-cap orifice of 0.793 cm.	Required flow ratio will vary with different types and sizes of nozzles. Droplets with mean volume diameters of 70 to 100 μ or less are desired.
Spray Pattern	Maximum diameter of the spray pattern must be less than the diameter of the spray chamber.	Required to prevent direct impingement of the spray onto the spray chamber wall that may result in caking.

TABLE 20. Calciner Furnace Operating Parameters

Variable	Requirement	Discussion
Wall Temperature	Greater than 250°C below calcine sintering temperature	Prevents calcine from adhering to the chamber wall.
Offgas Temperature	Feedrate and wall temperature selected must produce an offgas temperature of greater than 2000°C(a) for a reference-sized spray calciner (91.4-cm.-dia x 3.2-m-high spray chamber).	Temperature required to ensure calcine is sufficiently dry to produce free-flowing calcine. Required temperature may vary with size of chamber.

(a) Other considerations, such as volatile species condensing on the filters, may require higher offgas temperatures. The effects of volatile species require further study.

the particles begin to stick or melt together), and 2) the calcine particles must be sufficiently dry. The processing guideline used at PNL is to maintain the spray chamber wall temperature at least 50°C below the experimentally determined calcine sintering temperature. This prevents the calcine from sticking to the hot wall and consolidating. Feed compositions with sintering temperatures from 450°C to over 900°C have been successfully processed by maintaining the wall temperature at least 25°C below the sintering temperature. Special techniques have been developed at PNL by which a frit component such as powdered silica is added to the feed to increase the effective sintering temperature of high-sodium feeds. This method has been used to increase the calciner operating temperature and processing rates for high-sodium feeds.

Experience has shown that the calcine moisture content should be below about 4% to assure that the calcine flows freely through the calciner cone section. Operating experience has demonstrated that an offgas temperature (measured at the sintered-metal filters) of greater than 200°C will produce a dry calcine of <4% moisture for the PNL full-scale calciner. The offgas temperature is affected by both the spray chamber wall temperature and the feedrate of the waste. An increase in the wall temperature will increase the offgas temperature, whereas an increase in feedrate will decrease the offgas temperature. Wall temperatures ranging from 400° to 800°C and feedrates of up to 400 L/h have been successfully demonstrated. The only processing requirement is that the feedrate and spray chamber wall temperature selected produce an offgas temperature of greater than 200°C while still not causing the calcine to sinter.

VIBRATORY OPERATING PARAMETERS

The calciner is equipped with two vibrators (Model 1300AC, Cleveland Vibratory Company) or the equivalent mounted 180° apart at mid-height of the spray chamber. Wall vibration in the spray calciner chamber prevents calcine buildup on the walls by shaking off loose calcine. If the spray calciner is operated within the control parameters discussed, extended operation can be achieved without vibration. While the longest period of operation without vibration has not been determined, up to 2-1/2 h of operation without vibration have been successfully completed. Normally, to maximize heat transfer by assuring minimal calcine buildup on the walls, each vibrator is operated 1 to 3 times/h with 2-s pulses at equally spaced intervals. The frequency of vibration required to maximize heat transfer must be determined for different waste compositions. See Table 21 for operating parameters of the vibrator.

TABLE 21. Vibrator Operating Parameters

<u>Variable</u>	<u>Requirement</u>	<u>Discussion</u>
Frequency of Operation	Sufficient to prevent scale on the spray chamber wall that may reduce heat transfer. For low-sodium feeds, 1 to 3 two-s pulses/vibrator/h can be used.	Frequency must be determined for different feed types.

In the event that operating problems such as calcine sintering or poor atomization cause a scale that is not easily removed by vibration only, a thermal-shock technique can be used. This method requires that the chamber wall (and the scale) be heated up to the normal maximum operating temperature and then water-sprayed into the hot chamber through the atomizing nozzle for 2 to 5 min. Even the most stubborn scale has been removed using this method.

SINTERED-METAL FILTERS OPERATING PARAMETERS

Filter efficiency improves as a layer of calcine forms on the filters. This calcine layer acts as the filter media and prevents very small particles from penetrating the sintered-metal pores, which can result in gradual plugging of the filters. Therefore, fewer filter blowbacks will increase the operating life of the filters by minimizing the time the filter surface is exposed to moving particles and calcine fines that may become lodged in the filter inner structure.

Two basic types of blowback patterns have been used--equal-interval and unequal-interval patterns. The filters are divided into several groups, with the filters in each group being blown back simultaneously from the same air supply line. For the unequal filter blowback patterns all filter groups are blown back within a few seconds, then a longer interval passes before the next set of blowbacks. The equal-interval blowback pattern sequentially blows back filter groups with equal time intervals (e.g., 1-min intervals) between blowbacks.

It is desirable to minimize the calciner pressure surges caused by pulses of blowback air. Variables that affect the magnitude of these surges are blowback pulse duration, blowback pressure, offgas line flow resistance and the fraction of filters pulse, simultaneously. Experience has shown that short, 1/4-s pulses of ambient-temperature air are as effective as longer pulses for cleaning the filters. Static blowback pressures of 40 to 100 psig have been successfully used. One-fifth of the filters are blown back without pressurizing a small-scale calciner (to 70 L/h), while 1/11 of the filters in a large-scale calciner (300 to 400 L/h) are blown back simultaneously. See Table 22 for operating parameters of the sintered-metal filters.

TABLE 22. Sintered-Metal Filters Operating Parameters

<u>Variable</u>	<u>Requirement</u>	<u>Discussion</u>
Blowback Frequency	Sufficient to maintain filter pressure drop less than 10 to 20 in. H ₂ O for unequal-interval blowback pattern and 6 to 10 in. H ₂ O for equal-interval blowback pattern.	See text for discussion.
Blowback Pulse Duration	0.25-s pulse of ambient temperature air.	Experience has shown that 0.25-s pulses of compressed air appear to be as effective as longer pulses for cleaning filters.
Blowback Air Pressure	Greater than 40 psig.	Air pressures of 40 to 100 psig have been successfully used.

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APPENDICES

APPENDIX A

RUN SUMMARIES

RUN SUMMARIES

At the time of this writing, approximately 38,000 L of simulated acid wastes and 352,000 L of simulated neutralized wastes have been solidified by the FSSC in 1830 hours of processing time. This appendix contains details of the individual tests. Many of these tests were made largely to gain information about melters coupled to the spray calciner; however, important spray calciner information was also gained. Much of the information in this appendix is interpreted in the body of the report. A table summarizing processing information for each of the tests (where available) is presented at the end of the appendix.

PRELIMINARY TESTS

Preliminary FSSC check-out tests were made on April 29, May 25, and June 23, 1977. On April 29, the FSSC was operated in conjunction with the Full-Scale In-Can Melter (FSICM). Liquid feedrates up to 120 L/h were demonstrated with 150 kg of glass produced in about 2.5 h.

On May 25, 1977, the FSSC was successfully operated at 210 L/h for 2 h using simulated PW-7a feed. The entire run, lasting about 5 h, consumed 700 L of feed and produced 83 kg of calcine. The maximum throughput was thought to be limited by the high pressure drop across the feed control valve. After operating at 210 L/h for 2 h, all process temperatures had reached equilibrium values. No unexpected problems were encountered during the run.

FSSC preliminary Run 2 operating parameters are given in Table A.1.

The coupled Full-Scale Spray Calciner/In-Can Melter (FSSC/ICM) system was run up to 160 L/h on June 23, 1977. This feedrate gave a combined calcine-frit melting rate of about 70 kg/h in the ICM. During the run, 320 kg of glass were formed.

A Deanline® feed pump was used in place of the cantilever pump, which was down for repairs during June. The Deanline pump did not generate enough feed pressure to produce feed flow greater than 160 L/h; thus, the atomizing air pressure was reduced from 60 to 55 psi to permit increased feedrates with Spraying Systems Nozzle Set-up No. 72. Examination of the inside of the spray chamber and cone revealed a dried mud-like deposit on the cone wall directly below the spray nozzle. This apparently resulted from incomplete atomization of the feed solution due to low air pressure.

In early June, the cantilever feed pump was taken out of operation because of damage to the lower main-shaft bearing. Disassembly of the pump bearing housing revealed that acidic PW-7a feed solution had splashed on the bearing seal and caused the seal to fail, exposing the bearing. The bearing has been replaced using a new all stainless steel housing assembly and seal guard plus acid-resistant sealing material.

FULL-SCALE SPRAY CALCINER TEST 1 (FSSC-1)

The FSSC/ICM was run on a semi-continuous basis for 3 d. One 30.48-cm-dia and one 50.8-cm-dia canister, both 2.44 m long, were filled with simulated PW-7a waste glass. This run

TABLE A.1. Full-Scale Spray Calciner Operating Parameters (Run 2)

Parameter	Value
Operating Temperature (barrel outside wall)	800°C
Offgas Temperature at Filters	
100 L/h	450°C
160 L/h	390°C
210 L/h	340°C
Atomizing Air	
Pressure	60 psi
scfm	21 scfm
Temperature	24°C
Filter Blowback Cycle	
Interval	11 min
Pulse Duration	0.2 s
Pressure	70 psi
Maximum Pressure Drop Across Filters	<5 in. H ₂ O
Vibrator Cycle	
Interval	3 min
Duration	5 s

successfully demonstrated a new bottom support design for canisters in the ICM and established a maximum melting rate of ~90 kg/h for 20-in.-dia canisters. During 15.7 h of feeding time, 2068 L were processed. Data attained during the 3-d run indicated that the Set-up No. 72 spray nozzle was limiting the maximum calciner feedrate.

The calciner's ruggedness and reliability under adverse conditions were demonstrated when the furnace temperature was accidentally raised to 1050°C for about 3 h at the beginning of the 3-d run. Preliminary checking revealed no serious damage to the barrel or furnace.

FSSC-2

This test was run to evaluate the performance of Spraying Systems Company's external-mix air-atomizing nozzle Set-up No. 70, 1/2J (air cap #437, fluid cap #250375). This nozzle differs from the internal-mix nozzles in that no mixing chamber is used to mix the liquid and air streams. Since liquid feed does not pass through an internal mixing chamber, the feed flow control is not affected by the liquid-to-air pressure ratio; rather, a feed control valve adjusts the flow rate independently of the atomizing air pressure.

No operating problems occurred during this test. In Figure A.1, results of particle-size analysis made on calcine collected during this test at various calciner feedrates are compared with particle-size analysis of calcine produced using internal-mix nozzles during other tests.

FSSC-3

This brief test was made to determine if material would build up on heat transfer fins in the ICM canister at a 90 kg/h glass-batch feedrate. No build-up occurred.

FIGURE A.1. Calcine Particle Size Versus Spray Calciner Flowrate

FSSC-4

This test was run to evaluate the performance of Spraying Systems Company's internal-mix air atomizing nozzle Set-up No. 82, 1/2J (air cap #4691312, fluid cap #250375). This nozzle differed from the Spraying Systems Set-up No. 72 used previously in that the air/liquid mixing chamber was slightly larger. This permitted greater liquid flow capacities under similar air and nozzle pressure conditions. The calcine particle sizes produced are compared to those of other nozzles in Figure A.1. The calcine particle size is a function of the air-to-liquid pressure ratio. However, this nozzle could be operated at a relatively low atomizing-air-to-liquid-feed pressure ratio (i.e., 1.5) and still maintained a fine particle sized calcine product. It appeared that this nozzle could be used to successfully produce a fine-sized calcine at flowrates much greater than 200 L/h. This is the only one of the three nozzles tested that appears to be capable of this feat.

Additional tests were completed on calcine samples taken from FSSC Runs 1 through 4. Calcines from these tests were produced at various feedrates between 85 and 200 L/h. These samples showed a wide variation in nitrate content. It was noted that various nitrates from these PW-7a calcine samples were released at specific process temperatures. Consequently, it was possible to assemble the following graph (Figure A.2) using thermal gravimetric analysis.

FSSC-5

The FSSC/ICM equipment operation was demonstrated over a 10-d period during this test, which produced over 5,000 kg of simulated vitrified power-reactor wastes. The run lasted

FIGURE A.2. PW-7a Calcine Exit Temperature Versus Wt% NO₃

252.4 h and consumed 19,647 L of simulated liquid feed. The average feedrate was 77.8 L/h. No problems were encountered during operation of the calciner. The nozzle showed no significant signs of wear or cracking after this extended period of operation.

While the last two canisters were being filled, the gravimetric feeder used to meter frit into the melter began to drift from the set rate. This occurred because frit entered between the belt and rollers and distorted the belt until it slipped. The feeder was cleaned on about the the fifth day of the run. It is recommended that the feeder be cleaned and checked every 2 d when it is operated continuously.

The airlock was partially disassembled for inspection after the test. Both seats and mating surfaces were in good condition. One pin was missing from the shaft on the lower valve, and the other pin was loose. The only problem experienced with the airlock during the 10-d run was that on the last night, a set screw on a lever that trips a limit switch became loose, and the lever shifted so that it no longer tripped the switch.

In addition, after the test, gaskets on the top and bottom flanges of the spray chamber were replaced with Inconel-wound blue African asbestos-type gaskets. Gaskets on the filter housing were also replaced. A leak check at room temperature indicated less than 2.5 scfm inleakage at 12.5 in. H₂O vacuum. Damaged furnace elements were also replaced at this time.

FSSC-6

FSSC No. 6 was performed primarily to check out operation of the large-scale spray calciner after replacement of gaskets during November 1977. During this run, a stainless steel nozzle having a 0.79-cm air-cap orifice (Spraying Systems Set-up No. 82) was used to test the spray calciner at feedrates between 40 and 300 L/h. Approximately 400 L of PW-7a feed were put through the calciner during the 1.92-h test. The calciner proved to be in excellent shape after rebuild operations. No problems were encountered.

FSSC-7

FSSC-7 was made with the calciner coupled to the in-can melter. The main purpose of the calciner test was to evaluate its operation at process temperatures greater than 800°C. During this test the calciner zone temperatures were set as follows:

Zone No. 4 (top)	900°C
Zone No. 3	900°C
Zone No. 2	850°C
Zone No. 1 (bottom)	800°C

This selection of temperatures caused the calciner furnace to operate with relatively uniform power consumption in all four zones. In previous tests, temperatures had been maintained relatively uniformly. No significant difference in calciner performance was noted. During the first part of this test, the calciner processed 464 L of feed in ~4.20 hours during ICM filling of a 30.48-cm-dia canister. During the second portion of the test, the calciner ran at 232 L/h for 6.7 h and 211 L/h for 5.3 h to deliver 2673.1 L of feed to a 61-cm canister in the ICM. The same stainless nozzle used in FSSC-6 (Spray Systems Set-up No. 82) exhibited no wear after this test.

FSSC-8

The objective of FSSC-8 was to evaluate operation of the calciner at feedrates greater than 200 L/h and temperatures greater than 800°C. During this test, the calciner was operated at feedrates between 17 and 325 L/h over a 4.350-h period. A total of 928.8 L of PW-7a feed were consumed. The calciner operating temperatures were set the same as in FSSC-7. Data from FSSC-7 and -8 suggest that between 0.72 and 0.78 kW are used by the calciner to effectively dry 1 L/h of PW-7a feed. No problems were encountered when feeding at 300 L/h using an atomizing-air-to-liquid-feed pressure ratio of 1.35. However, feed capacities higher than this were not adequately accessed due to feed system limitations. The same stainless steel nozzle used in FSSC-6 and -7 was employed during this test. With over 4065 L of PW-7a throughput and nearly 20.6 h of operation, this nozzle exhibited no noticeable signs of wear.

FSSC-9

Simulated defense waste from the SRL-TNX process was used in this test. The material was shipped from Savannah River Laboratory (SRL). Feed analysis before the test indicated an

TABLE A.4. Feed Composition for Spray Calciner Test FSSC-10 and FSSC-11

Component	kg/100 L	
	FSSC-10	FSSC-11
Na_2CO_3	3.04	---
NaHSO_4	0.030	0.030
CaCO_3	0.291	0.291
$\text{Fe}(\text{OH})_3$	2.03	2.03
$\text{Al}(\text{OH})_3$	3.40	3.40
MnO_2	0.505	0.505
$\text{Ni}(\text{OH})_2$	0.127	0.127

Add H_2O and HNO_3 to bring batch to
pH = 9.5.

order to speed up the settling, a study was made to identify an effective flocculent. Test results indicated that an industrially available agent, Magnafloc® #836A, effectively reduced tank settling times to one quarter. The amount of agent required was only 2 mg/L. Greater amounts (3 and 4 mg/L) showed no significant improvement in settling rates. Successive washings did not require additional flocculent additions.

Another significant test made during this run involved the variation of filter blowback cycles. Four different cycles were compared for steady-state calciner operation at 200 L/h. They were as follows:

- Cycle 1: 0.2-s blowback pulse to each of 11 filter banks on 1-min intervals;
- Cycle 2: 0.2-s blowback pulse to each of 11 filter banks on 3-min intervals;
- Cycle 3: 0.3-s blowback pulse to each of 11 filter banks on 5-s intervals with 15 min between pulse cycles;
- Cycle 4: 0.3-s blowback pulse to each of 11 filter banks on 5-s intervals with 30 min between pulse cycles.

The test indicated that Cycles 3 and 4 provide much better filter operation and usage. Cycles 1 and 2 clean only one filter bank at a time with a long waiting period between subsequent cleanings. The filter bank that is cleaned during a blowback pulse acts as a point of low resistance. Consequently, a large portion of the offgases are drawn through the filters that have just been cleaned. Since one bank of filters only accounts for a small percentage of the total available filter surface area, the greater flux through this one bank causes rapid calcine buildup on these filters. This results in rapidly occurring high filter-pressure drops. Cycles 3 and 4 clean all the filters at approximately the same time. Hence, subsequent calcine buildup on (and pressure drop across) the filters occur uniformly on all filters over a longer period of time. Calciner tests at 200 L/h indicated that the rate of change of filter pressure drop resulting from Cycles 3 and 4 was about 2 to 4 in. H_2O per hour. Based on this data, the time between filter blowbacks could be extended to an hour or more durations. Further tests are needed on the large-scale calciner to verify this.

aluminum anion deficiency. Only 3.72 g Al/L were obtained compared with a desired value of nearly 20 g/L. Consequently, a sufficient quantity of $\text{Al}(\text{OH})_3$ was added to the feed solution in order to avoid adversely affecting planned melter runs for the produced calcine. The feed solution composition is given in Table A.2. A summary of some process data is included in Table A.3. It is of interest to note that the atomizing air and liquid-feed pressure ratio was successfully used to control feed to the calciner. These data are also presented in Table A.3. Completion of this 11.3-h test brought the total operating experience on the large-scale spray calciner to 311.3 h with a total feed consumption of 30,282 L.

FSSC-10

This test was initiated by SRL to obtain more operating data and information about large-scale spray calciner operation. The run was made using the "composite" composition given in Table A.4. About 848 kg of calcine were produced during this run. The calcine will be used in subsequent in-can and ceramic-melter experiments.

During feed preparation, an inadvertent addition of NaOH to each of two feed tanks required that excess Na be removed. Tank washings were initiated immediately. However, it was not long before it was realized that the tank settling times were quite excessive. Between 18 and 36 h were needed for solution solids to settle before half the tank could be decanted. In

TABLE A.2. SRL-TNX Feed Composition for FSSC-9

<u>Ion</u>	<u>g/L</u>	<u>Molarity</u>
Na	13.7	0.596
Fe	18.4	0.329
Al	20.4 ^(a)	0.138
Mn	8.3	0.151
Ca	2.3	0.057

(a) 16.7 g/L of Al were added here at PNL to adjust the weight ratio of Al/Fe to 1.108.

TABLE A.3. Selected FSSC-9 Process Data

<u>Feed Rate, L/h</u>	<u>Time Held, h</u>	<u>Liters Consumed</u>	<u>kW/L/h</u>	<u>Nozzle Pressure Data</u>		
				<u>Atomizing Air, psig</u>	<u>Liquid Feed, psig</u>	<u>Pressure Ratio</u>
100	0.083	8.3				
175	3.050	533.7	0.746	63.0	38.5	1.636
238	0.100	23.8				
250	3.733	933.3	0.726	59.0	40.0	1.475
280	1.400	392.0		56.0	40.0	1.400
292	0.317	92.5		53.0	38.0	1.395
300	2.617	785.0	0.680	51.5	37.0	1.392

FSSC-11

This test was similar to FSSC-10. The major difference was that SRL's "low-sodium" feed composition was used (see Table A.4). No process differences were noted between FSSC-10 and FSSC-11. About 3000 L of feed were consumed in 14.6 h during FSSC-11.

During the last 2 h of this run, a test was conducted on the feed system to demonstrate process control without the use of a feed control valve. For this test, the existing feed control valve was manually opened 100%, and the head from the feed pump was arbitrarily throttled. Flow to the calciner was controlled by adjusting the atomizing air pressure. The results of this test are displayed in Figure A.3. This test demonstrated that it may be possible to eliminate a feedrate control valve for hot-cell applications that require only approximate flow control.

FSSC-12

This large-scale spray calciner test was made with the calciner coupled to a 30.5-cm-dia high-efficiency Stairmand®-type cyclone separator. Approximately 500 L of liquid waste were run through the process equipment at rates between 150 to 200 L/h. The cyclone separator was determined to be not efficient enough for use by itself with spray calciners due to the large percentage of fine particles formed.

FSSC-13

This experiment was performed to verify compatibility of the SRL high-iron feed composition with the spray calcination process. The feed composition used is given in Table A.5. The calciner was operated at a nominal 200 L/h feedrate until ~500 L of feed were processed. No problems were encountered during the run. The calcine will be used in future ICM experiments. Filter blowback data were collected during this run for comparison with data from FSSC-14. One-half-hour blowback cycles were employed during this test. The rate of Δp was essentially constant during the half an hour between blowbacks. This value was determined to be 3.2 in. H₂O per hour, which is comparable to the 3.0 in. H₂O per hour noted for 15-min blowback cycles during FSSC-10. Filter face velocities during both of these runs were estimated to be about 0.91 m/s.

FSSC-14

This experiment was conducted to verify compatibility of the spray calcination process with the SRL high-alumina feed given in Table A.5. As with the previous run, the calciner was operated at a nominal 200 L/h. Approximately 1000 L of feed were processed. This calcine will be used in future ICM experiments. No problems were encountered during the run. Filter blowback times were increased to 1-h durations for this experiment. No system differences were noted between the half- and one-hour filter blowback cycles operating conditions.

FSSC-15 and -16

The purpose of these two experiments was to verify successful operation of a coupled spray calciner and ceramic melter system. Over 12 h of coupled system operation were completed. Over 1000 L of PW-7a liquid feed were processed. Results indicated that operation of the calciner system at low vacuums (3 to 7 in. H₂O) leads to better passage of fine

FIGURE A.3. Liquid-Feedrate Control Using Nozzle Pressure Ratio

TABLE A.5. FSSC-13 and FSSC-14 Feed Compositions

Feed Additive	Molarity	
	FSSC-13 High-Iron	FSSC-14 High-Aluminum
Al(OH) ₃	0.128	0.779
CaCO ₃	0.029	0.004
Fe(OH) ₃	0.362	0.034
MnO ₂	0.021	0.027
Na ₂ CO ₃	0.286	0.286
Na ₂ SO ₄	0.003	0.003
Ni(OH) ₂	0.083	0.007

particulates into the melter. High vacuums seem to cause fine particles to become entrained in inleakage flow from the ceramic melter to the spray-calcliner filters. Consequently, when filter blowback occurs, a large volume of calcine is released from the filters, which results in poor mixing of calcine and frit. To minimize this effect, the system vacuum was reduced to ~7 in. H₂O (from 10 to 15 in. H₂O) and filter blowback cycles maintained at 15-min maximums. No major process or equipment problems surfaced during this coupled system operation.

FSSC-17

FSSC-17 was a coupled spray calciner/in-can melter test. A total of 3133 L of PW-7a feed was calcined. The average feedrate was 206 L/h and peak feedrate was 221 L/h. The blowback

interval was 1 h and the blowback duration was changed from 0.3 s to 0.2 s to reduce the effect of blowback on calciner pressure. Filter Δp indicates that even longer periods between blowback would be possible. A No. 82 nozzle set-up was used and the atomizing air-to-feed-pressure ratio was varied in the range of 1.54 to 1.82. A fine, dry calcine was produced at all operating conditions encountered during the run.

FSSC-18

This run was made to provide calcine for an ICM test. Due to the presence of zeolite particles in the feed, the strainer and the control valve were removed from the feed system to minimize plugging. Feed control was maintained by adjusting the relative pressures of the feed and atomizing air streams. A feedrate of ~ 150 L/h was obtained at a feed pressure of 27.7 psig and an atomizing air pressure of 58.1 psig. A total of 320 L were calcined in a period of 3 h.

FSSC-19

The object of this test was to ensure equipment operability in preparation for a 10-d spray calciner/in-can melter test (FSSC-20). A calciner furnace heating element appeared to have burnt-out. Some problems were encountered with maintaining a constant feed flowrate with the SRL-type feed.

FSSC-20

The coupled spray calciner and ceramic melter were operated during a 10-d period. Approximately 8,660 kg of glass were produced and 28,900 L of feed were processed. The feed for the calciner was an SRL slurry designated "Composite-Without-Sodium" that initially contained 0.0617 kg of oxide/L. The glass former was frit 411, which was added in a ratio of 3:1 (frit:oxide) by weight. Before the experiment began, the melter tank contained a glass formed from a calcined PW-7a slurry and 76-199 frit at a 2:1 ratio.

The experiment started on September 19, 1978; however, due to the extensive foaming experienced, the feed to the calciner was shut down and additional frit was subsequently added to the melter to decrease the foaming tendencies in the melter tank.

The experiment was restarted on September 20, 1978 and continued for 227 h. During these 227 h, the feed to the calciner was turned off 20 times for minor maintenance problems. The total downtime was ~ 13.4 h and is detailed below:

<u>Number of Occurrences</u>	<u>Problem</u>	<u>Total Down-Time, h</u>
12	Plug in the melter-to-calciner flexible pipe section	2.9
5	Plug in the feed system to the calciner	2.5
2	Calciner nozzle change	2.0
1	Offgas train clean-up	6.0

The experiment was started at a calciner feedrate of 81 L/h and a frit feedrate of 15 kg/h, which resulted in a glass formation rate of 20 kg/h. Over the next 50 h, the feed-rates were increased to a glass formation rate of 39 kg/h. During this period, 8 of the 12 plugs that formed in the calciner-to-melter connecting section were experienced. The primary reason for the formation of these plugs was a lack of cold cap on the molten glass surface in the melter, which caused temperatures to rise above the stick point of the frit in the frit-calcine melter inlet nozzle.

For the next 50 h, the production rate was held at 39 kg of glass per hour, and only three plugs occurred in the frit-calcine inlet pipe as the cold cap grew and the tank vapor space temperatures decreased. The failure of the rupture disc that protected the melter tank from over-pressurization caused the melter offgases and calcine fines to by-pass the offgas filters and to discharge directly into the offgas condenser. Six hours were required to clear the condenser and the condensate line of the compacted calcine.

During this experiment the calciner was not operating with its full complement of heating elements, and the maximum evaporation rate was limited to ~150 L/h. A summary of the processing conditions follows:

<u>Total Hours</u>	<u>Downtime, h</u>	<u>Feed Oxide, kg/L</u>	<u>Feed-rate, L/h</u>	<u>Frit Rate, kg/h</u>	<u>Glass Rate, kg/h</u>
100	11	0.0617	81 to 150	15 to 30	20 to 39
85	2	0.0740	150	32	43
33	0.8	0.0802	150	35	47
9	0	0.0864	150	37.5	50.5

As the production rates increased, the melter tank conditions improved and the plugging problem with the frit-calcine inlet pipe disappeared; however, the calciner feed system plugging problems increased as the solids content of the slurry increased.

FSSC-21

This test was to determine that the equipment was operating properly in preparation for a spray calciner/in-can melter test. No problems were encountered other than a reduction in the calciner furnace heating capacity due to several burnt-out heating elements.

FSSC-22

The FSSC/ICM was operated so that a 61-cm-dia canister could be filled with simulated SRL waste glass. A melting rate of 50 kg/h was achieved during the processing of 3300 L of simulated SRL waste. The processing rate was limited because the spray calciner was partially disabled when several furnace elements burned out.

After FSSC-22, the calciner furnaces were rewired for three-phase power. New higher-power density heating elements (Kanthol A-1®) were installed to increase the furnace output from 80 kW per zone to 150 kW per zone (600 kW total). The refractory brick of the second zone was severely damaged and that of the third zone was moderately damaged around the heating

element supporting edges. The probable reason for this damage is that the vibrators, located between the second and third zones, were supported by the second zone. As a result, replacement refractory was ordered, and the vibrator supports were moved to support the vibrator independently of the furnace zones. Since the lead time of the new refractory brick was excessive, the existing bricks were repaired with a castable refractory to allow the spray calciner to be put back in operation.

FSSC-23

This was the first run made with the full-scale calciner after furnace element replacement and the conversion to three-phase power supply. The rebuilt furnace was rated at 150 kW per zone, or 600 kW total.

The run was made using PW-7a feed since the chemicals were readily available. Two types of nozzles were tested during the run--Spraying System's external mix nozzle No. 170 and Spraying System's internal-mix nozzle No. 82 with an alumina air cap modified to a 0.95-cm orifice. External-mix nozzles were designed to operate with liquid pressures near or below atmospheric pressure. Since the liquid-feed recirculation line could not be throttled down sufficiently without plugging to obtain liquid pressure near atmospheric, this was not a good test of the external-mix nozzle. The external-mix nozzle was used for about an hour with flowrates from 150 L/h to 190 L/h. When using this nozzle, calcine samples seemed to be dry, but calcine particle sizes were much larger than normal.

For the test, the orifice size of internal-mix nozzle No. 82 was enlarged from 0.79 cm to 0.95 cm. The reason for doing this was that for high feedrates (>300 L/h), the TK-307 pump was not capable of supplying feed at pressures high enough to allow sufficient atomizing air-flow. In other words, to obtain 300 L/h feedrate with the maximum feed pressure from the TK-307 pump, the atomizing air pressure had to be reduced below what was needed for good atomization. The 0.95-cm orifice allowed high liquid flowrate while high atomizing air pressure was maintained.

Using the internal-mix nozzle, calcine samples were very fine and dry for feedrates as high as 335 L/h. The feedrate was then increased to 360 L/h, and 40 min later the calcine samples became moist. Since the feed tank was nearly empty at this time, the run was stopped completely.

During the run, the furnace temperature controllers were set at 800°C. At this set point, the outside of the calciner barrel was actually 700 to 725°C throughout the run. By increasing the set point to maintain the barrel temperatures at 800°C, the calciner was expected to process at least 360 L/h for extended periods of time.

For this run, 900 L of waste was calcined, and 70 kg of calcine were produced in 4 h.

FSSC-24

The purpose of this run was to demonstrate that the feed composition defined by SRL could be calcined at feedrates of 300 to 325 L/h for extended time periods in preparation for a 5-d run. The feed composition is shown in Table A.6.

TABLE A.6. Composition of SRL Feed for FSSC-24

<u>Chemical</u>	<u>wt% of Each Chemical</u>
Fe(OH) ₃	53.7
Al(OH) ₃	13.5
MnO ₂	10.7
CaCO ₃	4.9
Ni(OH) ₂	5.8
NaNO ₃	2.25
NaSO ₄	1.12
Zeolite	8.0
	<u>99.97</u>

Concentration: 9.09 kg of the
above chemical mixture are used
to make 100 L of solution.

No problems were experienced during the run, although the strainer had to be flushed twice because of excess solids build-up. During shutdown of the run, the 0.95-cm feed line became plugged with solids while the feedrate was slowly being decreased, which emphasizes that turbulent flow must be maintained at all times to keep solids in the feed from settling out and causing plugs.

During the run, 1550 L of simulated SRL waste was calcined and 112 kg of calcine was produced in 5 h. The calciner was operated at 325 L/h for 3 h. The Spraying System's nozzle No. 82 was used with the 0.95-cm orifice.

Calcine Cone Modification

To allow clearance for a new ICM connecting section to fit under the FSSC, the bottom flange of the calcine cone was raised 7.62 cm. The flange was also enlarged from 10.16 cm to 15.24 cm to minimize the chance of plugging in the bottom of the calciner cone. These modifications were completed just before run FSSC-24.

FSSC-25

This 5-d test demonstrated that the FSSC/ICM is a viable system for high-capacity immobilization of SRL radioactive wastes in glass. Production rates of >80 kg/h of glass were achieved. The major problem that occurred during the run was not with the spray calciner or ICM, but with the liquid-feed system. Large particles in the feed slurry lodged in the magnetic flowmeter (0.40-cm orifice) and in the 0.95-cm tubing line just upstream of the flowmeter. Once a large particle became lodged, smaller particles, such as zeolite or nickel hydroxide, built up around the large particle and completely plugged-off flow to the spray calciner. These plugs could usually be dislodged by quickly closing and opening a valve on the 2.54-cm recirculation loop, which would produce a sudden surge of pressure and dislodge the plug. At times a plug was so tight that this sudden burst of pressure was not sufficient

to remove it. When this happened, it was necessary to use a hydraulic hand pump to break the plug loose. The first few times the hydraulic pump was used, the plug was pressurized towards the spray calciner nozzle. As a result of the sudden burst of pressure when the plug broke free, the orifice of the ceramic air cap broke. The hydraulic pump was then repiped to pressurize the plug upstream through the flowmeter and into a bucket.

Examination of the material in the bucket showed several large pieces of glass frit and a few small pebbles. Occasionally, the Liquid-Fed Ceramic Melter (LFCM) is tested with glass frit in feed slurry from feed makeup tanks used for the spray calciner. A week before the 5-d run, the LFCM was to be tested with frit in the feed slurry, but the wrong frit was added, which caused the feed transfer lines to plug up with large frit particles. These transfer lines were unplugged and flushed out, and the feed makeup tank was drained and vacuumed out before the start of the 5-d run. It is apparent that a significant amount of glass frit particles remained in the feed system after flushing, which caused plugging problems during the 5-d run.

Two nozzle air-cap orifices of different sizes were used during the run. One was a standard Spray System's Set-up No. 82 with a 0.79-cm air-cap orifice, and the other was a Spraying System's Set-up No. 82 with the air-cap orifice enlarged to 0.95 cm. Typical operating parameters for these two nozzles, along with a new nozzle tested in FSSC Runs 27 and 28, are shown in Table A.7.

Although use of atomizing air to control liquid-feed rates has the advantage of eliminating an in-cell control valve, experience has shown that control of the liquid-feed flowrate solely by varying the atomizing airflow rate has disadvantages. The range of control using only atomizing air is very narrow, from about 260 L/h to 340 L/h for the Spraying System's Set-up No. 82 with a modified 0.95-cm orifice. The lower range value is limited by the maximum available airflow in the system, and the upper range is limited by the airflow required for good atomization. For any liquid flowrate outside this range, it was necessary to adjust manually a ball valve on the recirculation to vary the feed pressure and obtain the desired flowrate.

TABLE A.7. Nozzle Comparisons

Nozzle	Standard Set-Up No. 82	Modified Set-Up No. 82	Set-Up No. 152
Air-cap orifice size, cm	0.79	0.95	1.39
Approximate maximum liquid flowrate @ 25 psig for good calcination, L/h	320	360	>400(a)
Approximate airflow at a liquid flow @ 300 L/h, scfm	30	50	80

(a) Maximum flowrate has not yet been determined.

Since the melting rate of the ICM changes considerably as the canister fills up, it was necessary that the liquid feedrate be varied from 200 L/h up to 340 L/h. However, this is outside the range of control for use of atomizing air only. Another problem with feedrate control using atomizing air is that if a plug develops in the feed line, the atomizing-air control valve will close in an attempt to increase the liquid flow back up to the set point. Then, if the plug breaks free, there is a surge of liquid flow with no atomizing air before the control valve can react and increase the atomizing airflow. This situation can cause calcine build-up inside the calciner or cause a brief pressurization of the melter when a stream of liquid pours on top of the hot glass in the melter. Because of these problems, a control valve may be required either on the liquid feed line or on the feed recirculation line. After the 5-d run, an automatic controller was placed on the feed recirculation loop. This offers capabilities of either controlling feedrate with atomizing air or by varying the pressure on the feed recirculation line.

The amount of glass produced during the 5-d run agreed within 3.5% of the theoretical glass production calculated from the total amount of chemicals and frit used during the run. This material balance is shown in Table A.8. Table A.9 indicates typical power consumption in the calciner for various flowrates.

FSSC-26

This brief run was made to use the remaining feed left over from FSSC No. 25 to produce calcine. This calcine was shipped to SRL for their testing.

FSSC-27 and -28

Before these tests, modifications to the calciner feed system were made to eliminate the problems experienced in FSSC-25. The openings in the feed strainer screen were reduced from

TABLE A.8. FSSC-25 Material Balance

Materials	Kilograms
Total chemicals used	1740
Total chemicals in oxide form (calculated)	1325
Calcine made during canister changeouts	91.4
Total oxides to glass (total oxides - calcine made)	$1325 - 91.4 = 1233.6$
Frit used	3345
Theoretical glass yield based on raw materials (kilograms frit plus oxides to glass)	$3345 + 1233.6 = 4578.6$
Actual measured glass produced	4424
% Difference $\left(\frac{\text{theoretical} - \text{actual}}{\text{theoretical}} \right)$	$\frac{4578.6 - 4424}{4578.6} \times 100\% = 3.4\%$

TABLE A.9. Typical Power Consumption

<u>Liquid Flow- rate, L/h</u>	<u>Kilowatts Consumed</u>
0	75
200	245
300	300
340	320

0.48 cm to 0.32 cm to remove more of the large particles before they reached the 0.95-cm tubing that supplies feed to the calciner. Also, the strainer flush line was revised to allow the strainer to be flushed into a bucket to purge the feed system of any large particles that accumulate on the strainer. Previously, the strainer could be flushed only directly back into the feed tank, which must recycle the large particles. These modifications almost entirely eliminated plugging in the 0.95-cm tubing feed line and 0.40-cm orifice of the magnetic flowmeter. A new magnetic flowmeter with a 0.64-cm orifice was ordered to further minimize the chance of plugging.

In addition to determining the effect of the feed system modifications, the purpose of these runs was to test a higher capacity nozzle, Spraying System's 1-J fluid cap #6251000 and air-cap #11091547 with the standard 1.39-cm orifice. This 1-J nozzle set-up No. 152 is compared to the two nozzles used during FSSC-25 in Table A.7. Although ceramic air caps have been ordered for this nozzle size, stainless steel 316 was the only air-cap material readily available for these tests. Preliminary inspection of the air cap after about 30 h of atomization showed no detectable nozzle wear, which may indicate that this larger-size nozzle may not be as susceptible to abrasion from solids in the liquid feed as were the smaller nozzles. Runs 27 and 28 showed that the calciner could operate satisfactorily in excess of 375 L/h with this larger nozzle.

FSSC-29

This test was a 10-d spray calciner/ceramic melter run sponsored by SRL. A total of 59,600 L of simulated SRL waste slurry was calcined and 11,000 kg of glass was produced during the run.

Three different theoretical feed concentrations--46 g oxide/L, 55 g oxide/L, and 64 g oxide/L--were used to match the calciner output capabilities with the melter capabilities at different periods of the test. Lab analysis of slurry samples taken during the run indicated 15 to 20% lower slurry oxide content than what was expected from the theoretical feed concentration. The theoretical feed concentration is based on the specifications from the supplier, measured weights of the chemicals added during feed makeup, and theoretical conversion of each chemical to the oxide form. The major reason for the low oxide content was the $\text{Fe}(\text{OH})_3$ was suspected to be in a lower concentration than desired.

The performance of the calciner slurry feed system for the 10-d run was much improved over the 5-d spray calciner/in-can melter run. The major reason for this was that the

openings in the feed strainer screen were reduced from 0.48 cm to 0.32 cm to prevent large particles from becoming lodged in the 0.40-cm orifice in the magnetic flowmeter.

On the fifth day of the run (April 22, 1979), a furnace element failed, and it was noted that the west vibrator was not impacting properly. The reason for these occurrences was not apparent until inspection of the equipment after the run. The west vibrator impact pad that had been welded to the outside of the calciner barrel had fallen to the floor that supported the furnace and the calciner barrel, and was leaning against the bottom furnace element. After the furnace was removed to replace the furnace element and to reweld the impact pad to the furnace barrel, surface cracks were discovered in the calciner barrel around the impact pad weld. Repair of the barrel was accomplished by removing a 38.1-cm-dia section centered at the middle of the vibrator pad, and welding in its place a new 38.1-cm-dia piece of 1.91-cm-thick 310 SS metal. This new 1.91-cm piece was then used as a vibrator pad. This eliminated some of the stresses caused by welding a pad on the outside of the barrel.

Before failure of the furnace element necessitated a reduction in calciner feedrate, a slurry feedrate of 400 L/h was maintained for 6 h. Operating conditions at this flowrate are shown in Table A.10. After the heating element failure, the feed slurry concentrations were increased to allow the calciner to match the melter output capabilities and the run were successfully completed.

The atomizing air cap used for this run was a stainless steel 316 Spraying System #11091547 (Set-up No. 152). This air cap atomized the feed slurry satisfactorily throughout

TABLE A.10. Operating Conditions of Spray Calciner at 400 L/h

Parameter	Value
Atomizing Airflow	98 scfm
Sintered Metal Filter	
Pressure Drop	
65 μ	6.0 in. H ₂ O
10 μ	6.8 in. H ₂ O
Calciner Furnace Control Temperatures	
Zone 1 (bottom)	855°C
Zone 2	875°C
Zone 3	860°C
Zone 4 (top)	845°C
Calciner Barrel Outside Wall Surface Temperature	
Zone 1 (bottom)	750°C
Zone 2	735°C
Zone 3	760°C
Zone 4 (top)	780°C
Calciner Barrel Inside Wall Surface Temperature	
Zone 2	480°C
Zone 3	520°C
Zone 4 (top)	590°C
Sintered Metal Filter Outlet Temperature	180°C

the run. Inspection of the nozzle for wear after the run showed rounded internal edges on the upstream side of the air-cap orifice. Indications are that the air cap could have lasted at least another week before requiring shutdown for replacement. The wear characteristics of the larger stainless steel nozzles are an improvement compared to smaller stainless steel nozzles that failed after about a day.

FSSC-30

This test was a long-term spray calciner/ceramic melter run sponsored by SRL. The total elapsed time for the run was 25.6 d, with the equipment in actual operation 21.0 of those days. A total of 154,000 L of simulated SRL waste slurry was calcined and 35,000 kg glass was produced. The defined SRL feed composition is shown in Table A.11.

The performance of the spray calciner during the run was excellent. The calciner again demonstrated its reliability and ease of control. The calciner was not directly responsible for any downtime during the entire run. The only incident involving calciner-equipment shutdown resulted from a decision to reduce the calciner temperature and atomizing air pressure in an attempt to achieve better calcine/frit mixing. The object was to reduce the amount of calcine dust that settled to the outside of the melter's cold cap, which may have caused a calcine-rich region. Reduction of the calciner temperature and atomizing air pressure was successful in reducing the amount of "calcine dusting"--increasing the calcine particle size--but on the fourteenth day of the run the calcine was not being completely dried, and moist calcine accumulated on the cone section of the calciner. This caused the frit addition line to become

TABLE A.11. Savannah River Laboratory Feed Composition (Basis: 1000-kg Glass Product)

<u>Component</u>	<u>Kilograms</u>
Frit 211	720
Feed slurry components (oxide form)	<u>280</u>
	1000
Feed slurry components (chemicals used for feed makeup)	
Fe(OH) ₃	195.4
Al ₂ O ₃ 3H ₂ O	49.0
MnO ₂	39.0
CaCO ₃	17.8
NiO 2H ₂ O	25.2
NaNO ₃	8.2
Na ₂ SO ₄	4.0
Zeolite	<u>29.0</u>
TOTAL	367.6

To obtain the desired feed concentration, the above chemicals were mixed with sufficient water to yield 4286 L of feed slurry.

plugged. The frit backed up into the airlock section and sheared the rotating drive shaft on the bottom airlock valve when it attempted to close against the frit. A replacement shaft was not available, and the run was completed using only the top valve of the airlock assembly.

During the 18 h that the equipment was down for removal of the bottom airlock valve, the pressure drop across the sintered metal offgas filters increased greatly. The glass temperature during this time was $\sim 1250^{\circ}\text{C}$. Volatiles with a white smokey appearance could be seen rising from the glass surface. The calciner vacuum and atomizing airflow were also high during this time. The combination of these factors caused an adhesive layer to form on the surface of the filters and possibly within the pores of the filters. This layer was not removed during the filter blowbacks following startup of the calciner. The increased filter pressure drop limited the calciner feedrate that could be processed while maintaining a minimum vacuum of 1 to 2 in. of water. Therefore, for the remainder of the run, 300 L/h (70 kg glass/h) was the maximum rate processed.

During the latter part of the run, a warning light that indicated an imbalance of power consumption in one phase of the three-phase power supply was activated. This indicated that a furnace heating element had failed. The performance of the calciner did not degrade significantly after the warning light was alarmed.

Calciner Feed System Performance

The feed plugging problems experienced in previous tests with SRL feed were eliminated. A new magnetic flowmeter with a larger orifice size was installed just before this test. The larger orifice size allowed particles, that would have plugged the previous flowmeter, to pass through, while providing the necessary accuracy ($\pm 1\%$) needed for good feed flowrate control and measurement. Below is a comparison of the new and previous flowmeters:

<u>Magnetic Flowmeter Comparison</u>		
<u>Flowmeter</u>	<u>Orifice Size</u>	<u>Full-Scale Flow Range</u>
New flowmeter	0.64 cm	35 to 1000 L/h
Previous flowmeter	0.40 cm	15 to 400 L/h

A feed strainer with openings slightly smaller than the flowmeter orifice size is also used to strain out foreign material and large chunks of agglomerated feed chemicals. The strainer was flushed to the floor drain periodically to prevent excessive pressure drop through the strainer. The amount of feed chemicals lost from flushing the strainer was very small.

A problem that surfaced during this long-term run was erosion of the feed system piping. Two separate feed leaks occurred during the run in the feed recirculation line used for feed tank agitation. Holes were eroded through the wall of a 5.08-cm ball valve and through a threaded section of the recirculation line that is connected to the ball valve. About 2 h of downtime was caused by these leaks.

Atomizing Nozzle Performance

The atomizing nozzle air cap used for this run was Spraying System's Model #11091547 (Set-up No. 152), stainless steel 316, with a 1.39-cm orifice. Although the nozzle atomized well, it was replaced on the tenth day of the run for inspection.

Calcliner Operating Data

Operating data for various calcliner feedrates are given in Table A.12. The data show that the power consumption for the top two zones of the calcliner does not increase

TABLE A.12. Calcliner Operating Data

Parameter	Values at Various Feedrates					
	1	2	3	4	5	6
Calcliner slurry feedrate, L/h	257	279	300	321	343	364
Equivalent glass production rate, kg/h	60	65	70	75	80	85
Atomizing airflow, scfm	84	85	87	83	86	92
Power usage, kW						
Zone 1 (calcliner bottom)	44.7	56.6	84.4	91.3	92.2	102.5
Zone 2	61.8	80.5	90.6	98.3	98.3	111.4
Zone 3	63.2	57.4	55.8	60.3	59.5	61.3
Zone 4 (calcliner top)	57.8	53.1	53.2	57.6	56.9	60.4
TOTAL	227.5	247.6	284.0	307.5	306.9	335.6
Calcliner furnace temp., °C						
Zone 1 (bottom)	745	765	805	825	825	850
Zone 2	790	830	875	890	890	913
Zone 3	818	823	843	865	863	865
Zone 4 (top)	813	813	835	855	855	860
Calcliner barrel outside surface temperatures, °C						
Zone 1 (bottom)	650	690	710	710	715	730
Zone 2	650	680	725	750	745	745
Zone 3	700	720	740	745	750	750
Zone 4 (top)	745	750	775	795	795	790
Calcliner barrel internal temperatures, (a) °C						
Lower zone 3	400	460	500	510	530	500
Upper zone 3	430	460	520	530	555	500
Zone 4 (top)	530	550	590	590	600	560
Calcliner cone temp., °C (wall directly under calcliner barrel)	190	170	295	300	275	290
Filter housing temp., °C						
Sintered-metal filter inlet	170	190	210	240	195	200
Sintered-metal filter outlet	160	170	190	225	185	190

(a) Calcliner barrel internal thermocouples are about 0.32 cm from the inside surface of the barrel.

significantly with increased feedrates. On the other hand, the lower two calciner zones (Zones 1 and 2) consume much more power as the feedrate is increased. Possible explanations for this occurrence are: 1) the atomizing nozzle spray pattern is wider in the lower portion of the calciner, which increases the radiative heat transfer coefficient since more particles are exposed directly to the calciner wall; 2) the convective heat transfer coefficient is greater in the lower portion of the calciner from the high turbulence caused by the large volume of steam generated in the calciner; and 3) the general direction of the convection currents is upward next to the heated calciner wall (heated gases rise) and downward near the cooler center region of the calciner, which transfers heat from the lower zones to the upper zones and reduces the power required to maintain the calciner temperature set point in the upper zones. The actual explanation for the power consumption differences between the zones is likely a combination of the above effects.

Material Balance

The overall material balance for this test was excellent. Agreement between actual glass yields and theoretical glass yields (based on the measured weight of frit used and theoretical oxide content of feed slurry) was within 0.5%. Details are shown in Table A.13.

FSSC-31

This spray calciner/in-can melter run was completed using a 61-cm-dia canister with no heat transfer fins. The SRL glass and feed compositions were the same as those in FSSC-30.

The canister required 27 h to fill, and no downtime occurred during the run. The calciner feedrate was varied from 258 L/h to 129 L/h to match the melting capabilities as the ICM was filled. The average calciner feedrate was 192 L/h. The atomizing nozzle used was Spraying System's Set-up No. 82 modified with a 0.95-cm orifice in the alumina air cap.

TABLE A.13. Overall Material Balance

<u>Material</u>	<u>Total Amount</u>
Volume feed slurry calcined	154,000 L
Weight oxides from feed slurry (theoretical)	10,056 kg
Weight frit 211 used (measured by frit feeder)	24,790 kg
Theoretical glass yield (weight oxides + frit)	34,846 kg
Actual glass yield (from canister weights)	35,000 kg
Percent difference:	
$\frac{\text{actual} - \text{theoretical}}{\text{theoretical}} \times 100\% = 0.44\%$	

After the canister was filled with glass, the melter was separated from the calciner and operated another 4-1/2 h to produce calcine for use at SRL and at PNL. During this time, the calciner was operated at 300 L/h.

Equilibrium calciner operating data for flowrates sustained for more than 4 h is shown in Table A.14.

During the FSSC-30 spray calciner/ceramic melter run, the pressure drop of the sintered metal offgas filters increased dramatically. After the run, the filter blowbacks were allowed to cycle for several days with no offgas flow in an attempt to reduce the filter pressure drop. At the beginning of this spray calciner/in-can melter run, the filter pressure drop was nearly normal (~8 to 9 in. water), but slowly increased to greater than 20 in. of water by the end of

TABLE A.14. Calciner Equilibrium Operating Data

Parameter	Values at Various Feedrates		
	1	2	3
Calciner slurry feedrate, L/h	214	258	300
Atomizing airflow, scfm (Set-up No. 82 with modified 0.953-cm orifice)	40	37	36
Power usage, kW			
Zone 1 (bottom)	78	84	91
Zone 2	72	80	92
Zone 3	42	45	53
Zone 4 (top)	43	45	50
TOTAL	235	254	286
Calciner barrel outside surface temperature, °C			
Zone 1 (bottom)	760	750	770
Zone 2	740	730	755
Zone 3	750	735	755
Zone 4 (top)	785	(b)	820
Calciner barrel internal temperatures, °C(a)			
Lower Zone 3	610	(b)	575
Upper Zone 3	620	(b)	600
Zone 4 (top)	650	(b)	630
Calciner cone temperature, °C (directly under calciner bar- rel)	380	230	230
Filter housing temperature, °C			
Sintered-metal filter inlet	390	(b)	280
Sintered-metal filter outlet	385	(b)	260

(a) Calciner barrel internal thermocouples are about 0.32 cm from the barrel.

(b) Recorder not functioning.

the run. The filter blowbacks were again allowed to cycle for several days after the spray calciner/in-can melter run. Then, the filters were filled with water by using the filter blowback air lines to add the water to the upstream side of the filters. The filter blowbacks were then activated to drive the water through the filter pores in an effort to remove any water-soluble compounds. The flush water was collected at the bottom of the calciner cone. Analysis of the water-soluble compounds from this flush water is given in Table A.15.

The results of the chemical analysis indicate that Na^+ and Cl^- were the primary compounds flushed from the calciner with equal molar weights of each found:

$$\frac{11.7 \text{ g/L}}{23 \text{ g/mole}} = 0.509 \frac{\text{moles Na}^+}{\text{L}}; \frac{18 \text{ g/L}}{35.45 \text{ g/mole}} = 0.508 \frac{\text{moles Cl}^-}{\text{L}}$$

Methods for minimizing future NaCl volatilization include maintaining a cold cap on the glass melt surface (as much as possible), and reducing melter temperatures and offgas flow during periods of shutdown.

TABLE A.15. Analysis of Water-Soluble Compounds in Filter Flush Water

Chemical	g/L (a)
Al	1×10^{-4}
B	1×10^{-2}
Ca	1.4 ^(b)
Cr	4.3×10^{-3}
Cu	1.5×10^{-4}
Fe	1×10^{-4}
K	0.15 ^(b)
Li	2.8×10^{-2}
Mg	4.4×10^{-3}
Mn	2×10^{-5}
Mo	5.7×10^{-3}
Na	11.7 ^(b)
Pb	3×10^{-4}
Si	2.9×10^{-3}
Sr	3.1×10^{-3}
Ti	4×10^{-5}
Zr	4×10^{-5}
Cl^-	18 ^(b)

(a) Approximately 70 L of flush water was used.

(b) Major constituents--Ca, K, Na, Cl^- .

FSSC-32

This spray calciner run produced 565 kg of SPC-4 supercalcine. This supercalcine will be used for testing and comparing pellet-making processes. The feed composition for SPC-4 supercalcine is given in Table A.16.

The average calciner feedrate for the run was 279 L/h with 3400 L calcined in 12.2 h. A summary of run data is given in Table A.17. Tank depletion measurements taken during the run show that the flowrate indicated by the magnetic flowmeter was low by 15 to 20%. A calibration check of the flowmeter before the run also gave erratic results. It is possible that a trapped air bubble or particle was covering one of the flowmeter's electrodes. The flowrate inaccuracy must be considered when comparing data in Table A.17 with data from previous runs.

The sintered metal filter pressure drop was fairly constant throughout the run at 10 to 12 in. of water. This is greatly improved over the final few days of the June 25-day spray

TABLE 16. Supercalcine Composition

Component	Oxide Form	g/L Oxide	Supercalcine Molarity
Ag	Ag ₂ O	0.23	0.0020
Ba	BaO	4.05	0.0264
Cd	CdO	0.26	0.0020
Cr	Cr ₂ O ₃	0.89	0.0117
K	K ₂ O	2.95	0.0626
Fe	Fe ₂ O ₃	8.94	0.1120
HNO ₃	---	---	2.0
Mo	MoO ₃	16.48	0.1145
Na	Na ₂ O	0.31	0.0100
Ni	NiO	2.70	0.0362
PO ₄	P ₂ O ₅	7.10	0.1000
Sr	SrO	2.74	0.0264
Zr	ZrO ₂	12.78	0.1040
Co	CoO	0.73	0.0098
<u>Rare Earths (RE)</u>			
Ce	CeO ₂	11.32	0.0658
Other RE	Re ₂ O ₃	71.14	0.4260
<u>Supercalcine Additives</u>			
Al	Al ₂ O ₃	7.55	0.1480
Ca	CaO	3.47	0.0620
Si	SiO ₂	29.38	0.4890
Sr	SrO	6.27	0.0650
		189.29	

TABLE A.17. FSSC Run 32 Operating Data

Parameter	Values at Various Feedrates	
	1	2
Atomizing nozzle used	Set-up No. 82 with modified 0.95-cm orifice	
Calciner feedrate, L/h(a)	200	250
Atomizing air flow, scfm	34	34
Power usage, kW		
Zone 1 (bottom)	73.2	89.5
Zone 2	77.9	97.4
Zone 3	43.5	51.2
Zone 4 (top)	43.0	52.6
TOTAL	237.6	290.7
Calciner Furnace Temperatures, °C		
Zone 1 (bottom)	855	870
Zone 2	855	860
Zone 3	870	855
Zone 4 (top)	880	845
Calciner barrel outside surface temperature, °C		
Zone 1 (bottom)	758	788
Zone 2	765	790
Zone 3	773	780
Zone 4 (top)	790	793
Calciner barrel internal(b) temperatures, °C		
Lower Zone 3	625	615
Upper Zone 3	655	640
Zone 4 (top)	690	675
Calciner cone temperature, °C (directly under calciner barrel)	420	360
Filter housing temperature, °C		
Filter inlet	410	380
Filter outlet	358	326
Filter pressure drop (inches H ₂ O)	10.4	11.5

(a) Feed tank depletion measurements indicate that these flowrates are 15 to 20% lower than actual.

(b) Calciner barrel internal thermocouples are about 0.32 cm from the barrel inside wall.

calciner/ceramic melter run (FSSC-30) and the July spray calciner/in-can melter run (FSSC-31) when the filter pressure drop exceeded 20 in. of water. This improved performance is attributed to the filter water flush described in the FSSC-31 summary.

FSSC-33

A FSSC test was completed in which varied amounts of sodium metasilicate pentahydrate ($\text{Na}_2\text{OSiO}_2 \cdot 5\text{H}_2\text{O}$, a glass former) and cornstarch (a reducing agent) were added to the calciner slurry feed. The actual feed compositions used for the run are shown in Table A.18. Approximately 15 kg of the Batch 1 composition and of the Batch 4 composition were shipped to SRL for their testing.

The calciner functioned well with all of the feed compositions, except for Batch 5. This composition was much more viscous than the other compositions due to the high concentration of sodium metasilicate pentahydrate. The atomized feed particles did not dry sufficiently before striking the calciner wall, which caused them to stick to the surface. The buildup of this insulating scale was indicated by a drop in the calciner internal temperatures and power consumption. The calciner feed was immediately shut off and the scale was removed by vibration and by spraying water into the hot barrel, which caused the scale to fall off by thermal shock. Typical run conditions are shown in Table A.19. The run time per batch was not long enough to determine equilibrium power consumption or temperature data for each batch.

FSSC-34

This was a brief test (3.37 h) during which two batches of calcine containing boric acid were produced in an attempt to incorporate some glass-forming compounds into the feed slurry. The feed compositions are given in Table A.20. Previous sintering tests had determined that 3 g $\text{H}_3\text{BO}_3/\text{L}$ was the maximum concentration that could be processed without scaling with 800°C calciner wall temperatures. Since the calcine produced was not significantly different from the standard calcine composition not containing boric acid, further testing was not pursued.

TABLE A.18. Feed Compositions and Volumes

Feed Chemical	Composition, g/L				
	1	2	3	4	5
$\text{Fe}(\text{OH})_3$	45.6	45.6	45.6	45.6	45.6
$\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O}$	11.43	11.43	11.43	11.43	11.43
MnO_2	9.10	9.10	9.10	9.10	9.10
CaCO_3	4.15	4.15	4.15	4.15	4.15
$\text{NiO} \cdot 2\text{H}_2\text{O}$	5.88	5.88	5.88	5.88	5.88
NaNO_3	1.91	1.91	1.91	1.91	1.91
Na_2SO_4	0.93	0.93	0.93	0.93	0.93
Zeolite	6.77	6.77	6.77	6.77	6.77
Cornstarch	2.33	6.99	6.99	2.31	4.76
(g/g TDS calcine oxides)	(0.0357)	(0.107)	(0.107)	(0.0354)	(0.0729)
Sodium Metasilicate Pentahydrate ($\text{Na}_2\text{OSiO}_2 \cdot 5\text{H}_2\text{O}$)	0	0	17.25	17.25	35.51
(g/g TDS calcine oxides)	(0)	(0)	(0.264)	(0.264)	(0.544)
Actual Volume Used, L	600	200	280	520	130

TABLE A.19. Typical FSSC-33 Run Conditions

Parameter	Value
Calcliner average slurry feedrate, L/h	255
Atomizing airflow, scfm	30
Atomizing nozzle used	Set-up No. 82 with modified 0.953 cm orifice
Atomizing air nozzle pressure, psi	30 to 33
Slurry nozzle pressure, psi	15 to 20
Calcliner barrel temperatures (outside surface of wall), °C	760 to 800
Total run time, h	6.8

TABLE A.20. FSSC-34 Feed Compositions

Feed Chemical	Composition, g/L	
	1	2
Fe(OH) ₃	45.6	45.6
Al ₂ O ₃ ·3H ₂ O	11.43	11.43
MnO ₂	9.10	9.10
CaCO ₃	4.15	4.15
NiO·2H ₂ O	5.88	5.88
NaNO ₃	1.91	1.91
Na ₂ SO ₄	0.93	0.93
Zeolite	6.77	6.77
Cornstarch		2.33
(g/g TDS calcine oxides)		(0.0357)
H ₃ BO ₃	3.0	3.0
(g/g TDS calcine oxides)	(0.459)	(0.0459)

FSSC-35

This was a 5-d full-scale Spray Calcliner/Ceramic Melter (SC/CM) test (FSSC-35, CFCM-8). Several modifications were tested during the run:

- a new location of the frit entry point into the calcliner as shown in Figure A.4 to improve calcine/frit mixing;
- the use of a smaller frit particle size;
- the addition of cornstarch to the calcliner feed slurry for use as a reducing agent;
- the addition of sodium metasilicate pentahydrate as a glass-forming component introduced into the calcliner feed slurry rather than with the frit.

FIGURE A.4. Frit Addition Line

Frit Addition Modifications

The new location of the frit addition line decreased the angle (with respect to the horizon) of the line through which the frit falls. Air was pulsed through the line every 40 s to ensure that the frit flowed through the line. On the first day of the test, occasional plugs in the line occurred, which required extended bursts of air to remove them. After the air pulse time was increased from 0.3 s to 0.7 s on the first day of the run, the system worked well. Samples of calcine taken during filter blowbacks showed a significant amount of frit mixed with the calcine, which indicates that the modification improved the calcine/frit mixing somewhat. Also, a smaller frit particle size was used (50% by weight, 40 to 100 mesh, and 50% 100 mesh) to help improve frit/calcine mixing by allowing the frit to be more easily entrained with the calcine up to the filters.

The somewhat better mixing of calcine and frit provided by these modifications did not seem to decrease the amount of foaming experienced with the melter or significantly improve the glass product.

Cornstarch Addition

Initially, cornstarch was added to the calciner feed slurry at a rate of 1 kg/100 kg of glass produced. On the third day of the test, the cornstarch addition was switched from the feed slurry to the frit at 1 kg/100 kg of glass produced. Whereas in previous tests melter foaming was substantially reduced by the addition of cornstarch, foaming in this test could not be controlled by cornstarch addition to the feed slurry or frit. A possible explanation is that the cornstarch was being ignited in the calciner, regardless of the method of addition.

Sodium Metasilicate Addition

Sodium metasilicate pentahydrate was also added to the calciner feed slurry in an effort to obtain more intimate mixing of the glass-forming chemicals and the calcine. A short-term test (FSSC-33) made before the 5-d test indicated that this feed composition could be satisfactorily calcined. But during this long-term run, a calcine scale slowly accumulated on the inside surface of the calciner barrel. The power consumption in Zone 1 and the cone and filter temperatures began to slowly drop on the second day of the test. The atomizing nozzle was changed to eliminate the possibility that a damaged nozzle was the cause; however, the nozzle was in good condition. On the third day of the test, as a result of increased scale buildup, the calciner feed composition was changed to the standard TDS 211 without sodium silicate, and the frit was changed from 211-SS to 211. The feed and frit compositions are shown in Table A.21. It is likely that the sodium metasilicate pentahydrate reduced the sintering temperature (temperature at which the particles stick together), causing an insulating calcine scale that retarded the transfer of heat and resulted in a drop in power consumption.

Furnace Element Failures

After the feed composition was changed and the calcine scale was removed by barrel vibration and thermal shock (spraying water into the hot barrel), the power consumption did not show any further gradual drops. This indicated that no further scale was forming. However, several furnace elements failed during the test and resulted in low offgas filter temperatures and calciner cone temperatures. The calciner feedrate was eventually decreased, as a result of low temperatures, to 214 L/h (50 kg of glass/h) on swing shift of the fourth day. Inspection of the calciner after the run showed a deposit of calcine on the calciner cone, although the barrel was clean. A calciner run summary is given in Table A.22.

The failure of furnace elements during the test required that the furnace zones be removed from around the calciner barrel. Six of the nine furnace elements in Zone 1 (bottom zone) and in Zone 2 had failed. These failures were caused by the supporting edges of the furnace fire brick (repaired in January 1979 after FSSC-22) cracking again and causing several of the furnace elements to fall. The fallen elements then shorted out other elements. The fire brick and elements were replaced at this time in Zone 1 and 2. The furnace brick design is shown in Figure A.5.

TABLE A.21. Composition of TDS 211-SS and TDS 211 Feed Slurry and Frit

Makeup Chemical	kg Chemical/1000 kg Glass	
	TDS 211-SS	TDS 211
Fe(OH) ₃	195.4	195.4
Al ₂ O ₃ ·3H ₂ O	49.0	49.0
MnO ₂	39.0	39.0
CaCO ₃	17.8	17.8
NiO·2H ₂ O	25.2	25.2
NaNO ₃	8.2	8.2
Na ₂ SO ₄	4.0	4.0
Zeolite	29.0	29.0
Na ₂ OSiO ₂ ·5H ₂ O	74.9	---
Cornstarch	10.0	---
NaCl	4.8	4.8
NaF	0.5	0.5
TOTAL	457.8	372.9

Water--enough to make 4286 L of slurry.

Frit 211-SS Component	% Frit Weight	kg/1000 kg Glass
SiO ₂	58.8	398.1
Na ₂ O	18.7	126.6
Li ₂ O	4.7	31.8
B ₂ O ₃	11.8	79.9
CaO	6.0	40.6
TOTAL	100.0	677.0

Frit 211 Component	% Frit Weight	kg/1000 kg Glass
SiO ₂	58.3	419.8
Na ₂ O	20.6	148.3
Li ₂ O	4.4	31.7
B ₂ O ₃	11.1	79.9
CaO	5.6	40.3
TOTAL	100.0	720.0

Sintered-Metal Filter Failures

During the November SC/CM test and from its beginning, the venturi scrub samples were visibly high in calcine. After the test, the sintered metal filter assembly was removed. The filters were washed with water and an approximately 15.2-cm irregularly shaped piece was found missing from a 65-μ filter. The filters were then filled individually with water. Fifteen of the thirty-four 65-μ filters and two of the fifty-one 10-μ filters could not be filled with water, or leaked water considerably faster than the other filters, which indicated that

TABLE A.22. FSSC-35 Run Summary

Parameter	Value
Total run time, h	110
Downtime, h	30.5 (28%)
Melter foaming	20.5
Calciner scaling	10.0 (time required to change frit and feed com- positions)
Actual equipment run time, h	79.5 (72%)
Total glass produced, kg	4220
Total liters calcined (based on flowmeter measurements)	19,180
Average calciner feedrate while operating, L/h	241
Atomizing nozzle used	Air cap #11091547 (Spraying Systems Set-up No. 152)
Typical airflow	85 scfm
Typical air pressure	30 psig
Typical feed slurry pressure	10 psig

breaching had occurred in these filters. Several other filters were later found to have pieces missing. Analysis indicated that corrosion existed not only on the filter surface but had penetrated throughout the depth of the filter material. Chloride and sulfate are thought corrosion to be the primary causes, with high temperature oxidation also a possibility (Dierks et al. 1980). Stainless steel 316, the filter material, does not have good chloride resistance. Analysis of water used to flush the filters indicated that the filters were coated with a substantial amount of NaCl (see FSSC-31).

Results of analyses of three samples of material that coated the filters removed from the calciner after the November 5-d test are shown in Table A.23. The three samples of material were distinctly different in appearance and composition:

1. A white film of material that volatilized from the melter after the November 5-d test, and before the filters were removed, coated all surfaces. The material was 64% NaCl. The other major components of this sample, such as Fe and Al, were, likely, contaminants obtained when the white film was scraped off the coated surfaces. Chromium and Li were substantially higher in this sample than the other samples, which indicated that these components also volatilized from the melter. The source of Cr could have been an impurity in the calciner feed slurry or the refractory brick in the melter that had corroded badly.
2. A dark layer of calcine powder (under the white film) uniformly coated the filters. This was the normal calcine layer that coated the filters during operation.
3. Several hard chunks of material were caked between the filters. This material was higher in Na and Si than the normal calcine, which indicated that these chunks were

FIGURE A.5. Furnace Brick Design for Zones 1 and 2

formed during the first part of the November run while sodium silicate was being added to the calciner feed slurry. The sodium silicate apparently caused the calcine to sinter during periods of equipment shutdown, at which time the filter temperatures approached 500°C.

Since costs prevented procurement of new filters of more corrosion-resistant material, the failed filter elements were replaced with 6.35-cm-dia stainless steel 316 filters that were bought as spares during previous programs. While these filters were not expected to last any longer than the previous filters, they allowed the calciner to be made functional in about a month.

A comparison of the new filters with the previous filters is given in Table A.24. All new filters were 6.99 cm in dia, except for three Inconel filters, which were 2.08 cm in dia (these were test filters requested by SRL). Since the earlier 10-μ filters were all 5.08-cm in dia, more distance was needed between the new 6.99-cm-dia filters. Therefore, that portion of the tube sheet was plug-welded and redrilled to make a new filter spacing pattern for the

TABLE A.23. Analyses of Material on Filters After Defense Waste Run

Element	Percent ^(a)		
	White Film	Calcine Layer	Hard Chunks
Al	1.71	4.00	2.95
B	0.16	0.10	0.32
Ba	0.03	0.13	0.01
Ca	1.15	2.49	2.52
Cr	0.42	0.17	0.03
Fe	14.6	38.4	32.0
Li	0.44	0.04	0.14
Mn	0.35	6.01	0.51
Mo	0.10	0.21	0.048
Na	25.2	4.34	10.3
Ni	2.32	5.53	4.30
Si	0.74	2.13	5.52
Sr	0.05	0.19	0.91
Cl ⁻	39	3.8	0.17

(a) The results are expressed as percent of the total sample weight. The total percentages do not add up to 100% since elements, such as O₂, are not indicated in these analyses. For example, Fe actually exists as Fe₂O₃.

TABLE A.24. Comparison of New and Previous Filters

Filter	Pore Size, μ	Filter Size	Filter Material Thickness	Filter Material	Internal Support	No.	Surface Area
Previous Filters	10	5.08 cm dia x 91.4 cm	0.318 cm	316 SS	(a)	51	7.43 m ²
	65	6.99 cm dia x 91.4 cm	0.159 cm	316 SS	(b)	34	6.73 m ²
							14.16 m ²
New Filters	10	6.99 cm dia x 91.4 cm	0.159 cm	316 SS	(b)	28	5.62 m ²
	10	5.08 cm dia x 91.4 cm	0.318 cm	Inconel	(a)	3	0.437 m ²
	40	6.99 cm dia x 91.4 cm	0.159 cm	316 SS	(b)	33	6.62 m ²
							12.68 m ²

(a) Internally supported by a solid 316 SS center rod.

(b) Internally supported by a perforated 316 SS cylinder.

10- μ filter section. The new filter spacing pattern is shown in Figure A.6. A filter is located at each intersection of the lines in the figure.

Three Inconel filters were installed to determine if that material would be more resistant to the high temperatures and corrosive environment of the calciner than the 316 SS filters used in the past. Their location is shown in Figure A.6. At a future date, the filters can be destructively removed and inspected for corrosion.

NOTE: 40- μ FILTERS HAVE THE SAME SPACING PATTERN AS THE PREVIOUS 65- μ FILTERS. 10- μ FILTERS HAVE A WIDER SPACING PATTERN THAN THE PREVIOUS 10- μ FILTERS.

THREE TEST
INCONEL CANDLES
(FOR SRL)

FIGURE A.6. Spacing Pattern for New Filters

FSSC-36

This short-term run was made to check out the calciner after the furnace zones and sintered-metal offgas filters were repaired and reinstalled. The calciner functioned well during this run after a damaged spray nozzle was replaced. Detection of the damaged nozzle was difficult because moist spray was impinging on a section of the calciner barrel where no thermocouples were located. This experience indicated that, for proper detection of moist spray contacting the barrel, thermocouples need to be located 120° apart or less around the circumference of the barrel instead of the current 180° apart.

During FSSC-36, the feed slurry concentration was 144 g oxide equivalent per liter. This is 2.2 times as concentrated as the slurry composition previously tested. By more than doubling the feed slurry concentration, the output capacity of the calciner is also more than doubled. While no problems were encountered with the calciner feed system when feeding this concentrated feed for these short periods, accelerated abrasive wear is expected in the piping and feed pump during longer tests.

FSSC-37

Two canisters of glass were produced in this FSSC/ICM test. A total of 8000 L of feed slurry were processed in 33 h of operation for an average feedrate of 242 L/h. Details of the calciner operation are shown in Figure A.7 and Table A.25. The feed slurry makeup composition is shown in Table A.26. Although a more concentrated feed could have been used as demonstrated in FSSC-36, a feed concentration of 100 g oxide equivalent per liter was used to allow the calciner to operate at a higher flowrate. Lower rates were below the magnetic flowmeter's range for calibration accuracy.

FIGURE A.7. FSSC-37 Calciner Operating Parameters

TABLE A.25. Calciner Run Data (Run FSSC-37)

Parameter	Value
Nozzle used	Spraying System Set-up No. 82 (ceramic)
Typical slurry feed pressure, psig	25
Typical atomizing air pressure, psig	35
Typical atomizing airflow, scfm	31
Average slurry feedrate, L/h	242
Equipment operating time, h	33

TABLE A.26. Feed Makeup and Frit Compositions

Makeup Chemical	kg/1000 kg Glass	Oxide Form	kg/1000 kg Glass
Fe(OH) ₃	195.4	Fe ₂ O ₃	146.0
Al ₂ O ₃ ·3H ₂ O	49.0	Al ₂ O ₃	32.0
MnO ₂	39.0	MnO ₂	39.0
CaCO ₃	17.8	CaO	9.97
NiO·2H ₂ O	25.2	NiO	17.9
NaNO ₃	8.2	Na ₂ O	2.99
Na ₂ SO ₄	4.0	Na ₂ SO ₄	4.0
Zeolite	29.0	Zeolite	29.0
Na ₄ EDTA	22.7		
Cornstarch	1.6		
Anthracite Coal	8.1		
NaCl	4.6		
NaF	0.5		
CsNO ₃	0.2	Cs ₂ O	0.17
Sr(NO ₃) ₂	0.2	SrO	0.10
TOTAL	405.7		280.23

Water--enough to make 2082 L of slurry (100 g/oxide/L)

Frit-211 Component	% Frit Weight	kg/1000 kg Glass
SiO ₂	58.4	419.9
Na ₂ O	20.5	147.8
Li ₂ O	4.4	31.8
B ₂ O ₃	11.1	79.9
CaO	5.6	40.6
TOTAL	100.0	720.0

The calciner operated without a problem during this ICM test. However, auxiliary equipment problems caused significant downtime, and neither of the canisters had a continuous fill.

While filling the first canister, the lower airlock valve was not seating properly, which allowed air to bleed through when the top valve was open. The valve was adjusted to seal, and

this adjustment in turn required a limit-switch arm adjustment. This incident caused a shut-down of 4 h. Fortunately, it occurred in the early part of the run, which allowed the more significant rate demonstration at the top of the canister.

During filling of the second canister, the limit-switch arm adjustment again failed. The limit switch was not tripped, which prevented the upper valve from operating. Since the valve did not operate, the frit backed up into the solids feeder. The lower valve was readjusted and the feeder was cleaned out. In addition, a photocell, which is connected with the belt speed transducer, malfunctioned. The belt speed was interpreted as moving faster than it really was. This cut the frit addition to as much as one third the normal rate.

FSSC-38

The primary purpose of this test was to produce calcine for melter testing at SRL and PNL. The long-term operation of the calciner, independently of a melter, also allowed equilibrium conditions to be measured for various spray chamber-wall temperatures and calciner feed-rates. In addition, equilibrium was obtained while heating only the top three of the four calciner furnace zones--simulating a shorter calciner spray chamber. This simulation is only approximate since the bottom zone of the calciner spray chamber, while unheated, may still increase the heat transferred into the calciner by acting as a heat transfer fin. Nevertheless, significant data were obtained from this test.

Table A.27 shows the effect of the spray chamber-wall temperature and the number of heated zones on the maximum calciner feedrate. Operating experience has shown that an offgas temperature and calciner-cone temperature of greater than 200°C are required to produce a free-flowing calcine that will not build up in the calciner cone. Thus, the maximum calciner feedrate is defined as the feedrate that results in an offgas temperature of 200°C measured at the sintered-metal filters. If run conditions did not produce an offgas temperature of exactly 200°C, the maximum feedrate which would have resulted in this temperature is estimated (see the last column in Table A.27).

TABLE A.27. Effect of Wall Temperature and Height of Heated Spray Chamber on Maximum Calciner Feed Rate

<u>Date</u>	<u>Furnace Zones Used</u>	<u>Spray Chamber Wall Temperature, °C</u>	<u>Maximum Feedrate Used During Test at Stated Condi- tions, L/h</u>	<u>Equilibrium Offgas Temp. at Filters, °C</u>	<u>Estimated Maximum Feed Rate @ 200°C Offgas Temp. and Stated Conditions, L/h</u>
5/14/80	1 to 4	700	304	215	315
5/06/80	1 to 4	550	148	220	160
5/6/80	1 to 4	400	50	200	50
5/12/80	2 to 4 (Top 3 Zones)	700	208	195	205
5/07/80	2 to 4 (Top 3 Zones)	550	100	210	105

Operating data from test FSSC-38 are summarized in Table A.28. During this test, equilibrium operating data were obtained at spray chamber-wall temperatures of 400°C, 550°C and 700°C at various waste feedrates using all four furnace zones. Also, equilibrium data were obtained at 550°C and 700°C spray chamber-wall temperatures at various waste feedrates while the top three furnace zones were used. Zero power consumption for furnace Zone 1 (bottom zone) in Table A.28 indicates that only the top three zones (Zones 2 to 4) were in use at that time.

During FSSC-38, ~19,000 L of simulated waste feed was processed in 100 h, making for an average feedrate of 190 L/h. The average feedrate was lower than normal because of the 400°C and 550°C wall temperatures used during a portion of the test. The feed composition used is shown in Table A.29.

During heatup of the calciner before the test, a furnace element in Zone 2 failed. After the test, removal of the top three furnace zones revealed that the bottom element in Zone 2 failed because of arcing between the element and a metal support plate under the bottom row of fire bricks. The arcing was likely caused by a high-iron-content mortar that was used to hold the elements in place. This mortar had worked well in other locations of the furnace, but the relatively high electrical conductivity and close proximity to the metal support plate made the mortar unsuitable for the bottom furnace element.

The failed element in Zone 2 affected the power consumption slightly in that zone. However, the power consumption in other zones likely increased for a given temperature set point, making the total power consumption in Table A.28 comparable to other tests at similar conditions.

Fifteen barrels containing 1425 kg of calcine from this test were shipped to SRL for testing in their equipment.

Results of calcine analysis from test FSSC-38 are shown in Table A.30. While no direct correlations of calciner wall temperature and feedrate with weight percent S, Cl, F(ppm) and weight percent loss on ignition are apparent, higher weight percentages of carbon were generally found at 550°C and 400°C wall temperatures than at 700°C wall temperature. Some measured physical properties of calcine from FSSC-38 are shown in Table A.31.

Calcine particle size distributions were obtained by Quantimet projection analysis for calcine produced during run FSSC-38. Typical results are shown in Figure A.8. For this TDS-211 simulated waste slurry, 88% of the total number of particles are less than 11 μ , but account for only 2.8% of the total volume (mass). Since this neutralized waste is a slurry rather than a solution, the larger particles in the calcine represent the larger particles in the slurry. The very small particles are formed by water-soluble compounds dried from the highly atomized waste.

FSSC-39

This was a test using the FSSC and ICM in which two canisters of glass were produced (FSSC-39 and ICM-28 and -29). The feed slurry composition used is shown in Table A.32. A run summary is presented in Table A.33.

TABLE A.28. FSSC-38 Operating Data

Date	5/05	5/06	5/06	5/06	5/06	5/06	5/07	5/07	5/07	5/07	5/09	5/09	5/12	5/12	5/13	5/13	5/15	5/15
Time		0450	0900	1106	1756	2300		0322	0610	1152		1316	1225	1700	1135	1930		1300
Calciner slurry feedrate, L/h	236	123	148	75	50	50	148	75	100	123	192	168	208	208	255	275	285	304
Atomizing airflow, scfm(a)	31	36	35	45	54	54		44	39	41	37	38	36	36	31	31		31
Power Consumption, kW																		
Zone 1 (bottom zone)	79.0	43.5	50.6	30.9	18	0	0	0	0	0	0	0	0	70.3	79.8	83.5	88.0	86.2
Zone 2	61.0	31.5	37.5	26.0	15	33	67.4	34	43	63.3	78.8	73.1	81.6	57.9	60.6	63.8	67.5	70.2
Zone 3	49.7	26.0	30.0	22.0	13	23	48.2	25	33	43.7	55.3	51.6	58.5	45.5	53.4	56.4	52.3	56.6
Zone 4 (top zone)	39.7	24.4	24.4	14.6	9	17	36.4	20	27	34.0	42.4	38.9	47.4	37.2	42.4	43.3	44.5	45.3
TOTAL	229.4	125.4	142.5	93.5	55	73	152.0	79	103	141.0	176.5	163.6	187.5	210.9	236.2	247.0	252.3	258.3
Calciner furnace temp., °C																		
Zone 1 (bottom zone)	740	580	575	560	425	322		275	230	350	275	305	250	715	730			745
Zone 2	693(a)	545(a)	525(a)	520(b)	385(b)	527(b)		540(b)	550(b)	680(a)	716(a)	702(a)	710(a)	685(a)	675(a)			705(a)
Zone 3	783	627	620	620	473	616		625	637	740	793	780	790	775	792			795
Zone 4 (top zone)	753	620	585	580	437	595		600	615	765	753	743	760	745	757			755
Calciner chamber wall temp., °C																		
Zone 1	700	550	555	550	400	378		330	280	410	320	350	280	700	700	700		700
Zone 2	700	550	555	550	400	550		550	540	695	700	685	695	700	700	710		705
Zone 3	700	550	555	550	400	550		550	550	695	710	695	700	700	700	700		700
Zone 4	700	570	550	550	400	560		560	560	700	700	695	700	700	705	700		700
Calciner cone temp., °C (directly under spray chamber)	280	260	300	315	220	290		240	210	310	220	250	190	315	270			210
Offgas temp. at sintered metal filters, °C	260	240	220	265	200	250		230	210	280	225	255	195	290	260	240		215

(a) Spray System's Set-up No. 82 atomizing nozzle was used.

(b) These furnace temperatures are low--possibly because the thermocouple was not installed correctly and did not completely penetrate through the furnace wall.

TABLE A.29. Feed Slurry Composition

Makeup Chemical	g/L	Oxide Form	Equivalent g/L
Fe(OH) ₃	69.73	Fe ₂ O ₃	52.10
Al ₂ O ₃ ·3H ₂ O	17.49	Al ₂ O ₃	11.42
MnO ₂	13.92	MnO ₂	13.92
CaCO ₃	6.35	CaO	3.56
NiO·2H ₂ O	8.99	NiO	6.07
NaNO ₃	2.93	Na ₂ O	1.07
Na ₂ SO ₄	1.43	Na ₂ SO ₄	1.43
Zeolite	10.35	Zeolite	10.35
Na -EDTA	8.10		
Cornstarch	0.57		
Anthracite Coal	2.89		
NaCl (a)	1.6		
NaF	0.178		
CsNO ₃	0.071	Cs ₂ O	0.061
Sr(NO ₃) ₂	0.071	SrO	0.036
TOTAL	144.7		100.0

Oxide equivalent: 100 g/L

To produce 1800 kg calcine required 18,000 L of the above feed composition

(a) NaCl is supplied from impurities in the Fe(OH)₃. No additional NaCl is added.

TABLE A.30. FSSC-38 Calcine Analysis

Sample No.	Calclner Feedrate, L/h	Calclner Wall Temperature, °C	Number of Furnace Zones Used	%C	%S	%Cl-	F(ppm)	% Loss on Ignition (LOI)
1	236	700	4	1.52	1.09	0.27	320	13.98
2	255	700	4	1.67	0.74	0.23	350	15.81
3	280	700	4	1.10	1.31	0.36	290	13.62
4	123	550	4	4.42	0.51	0.12	300	16.35
5	75	550	4	1.81	1.20	0.36	275	7.46
6	50	400	4	2.62	0.73	0.21	320	20.51
7	50	550	3	2.41	1.36	0.38	330	10.25
8	75	550	3	3.03	0.78	0.26	290	11.33
9	123	700	3	1.06	1.38	0.39	310	6.30
10	100	550	3	1.46	1.14	0.32	320	10.10
11	208	700	3	1.56	1.00	0.20	310	16.29
12	255	700	4	1.25	1.11	0.36	235	11.95
13	304	700	4	1.98	1.16	0.38	330	15.23

TABLE A.31. Physical Properties of FSSC-38 Calcine

Properties	Value
Aerated Bulk Density	0.95 g/cm ³
Packed Bulk Density	1.27 g/cm ³
Compressibility	25%
Angle of Repose	27°

FIGURE A.8. Calcine Particle Size Distribution

The operation of an air sparge inside a canister to obtain a uniform glass composition was demonstrated with the first canister. Equipment operation for both the spray calciner and in-can melter went very smoothly. There were no feed interruptions or downtime during filling of the first canister.

Co-calcination (supplying frit in the calciner feed slurry) was demonstrated during filling of the second canister. Since the calciner wall temperatures were maintained at 400°C (compared to 700°C normally) to prevent the frit from sticking to the walls, processing rates were very low. While filling this canister, frequent losses in slurry feed pressure caused numerous temporary (~5 min) feed shutdowns. The losses in feed pressure were likely caused by a gradual buildup of the viscous feed slurry in the feed lines. Clearing the feed transfer lines with high pressure air (~40 psig) allowed the feed pressure to return to

TABLE A.32. Feed Slurry Composition

<u>Chemical</u>	<u>Chemical</u>	<u>Oxide</u>	<u>Oxide</u>
	<u>Form, g/L</u>		<u>Form, g/L</u>
<u>ICM-28</u>			
Fe(OH) ₃	69.83	Fe ₂ O ₃	52.17
Al(OH) ₃	17.52	Al ₂ O ₃	11.44
MnO ₂	13.93	MnO ₂	13.93
CaCO ₃	6.36	CaO	3.56
NiO 2H ₂ O	9.00	NiO	6.07
NaNO ₃	2.93	Na ₂ O	1.07
Na ₂ SO ₄	1.43	Na ₂ SO ₄	1.43
Zeolite	10.36	Zeolite	<u>10.35</u>
Cornstarch	0.57		
Coal	<u>2.89</u>		
TOTAL	134.8		100.0
<u>ICM-29</u>			
Fe(OH) ₃	69.83	Fe ₂ O ₃	52.17
Al(OH) ₃	17.52	Al ₂ O ₃	11.44
MnO ₂	13.93	MnO ₂	13.93
CaCO ₃	6.36	CaO	3.56
NiO 2H ₂ O	9.00	NiO	6.07
NaNO ₃	2.93	Na ₂ O	1.07
Na ₂ SO ₄	1.43	Na ₂ SO ₄	1.43
Zeolite	10.36	Zeolite	10.35
Cornstarch	0.57		
Coal	2.89		
Frit-211	<u>257.2</u>	Frit-211	<u>257.2</u>
TOTAL	392.0		357.2

<u>Frit Component</u>	<u>wt%</u>
SiO ₂	58.4
Na ₂ O	20.5
Li ₂ O	4.4
B ₂ O ₃	11.1
CaO	<u>5.6</u>
TOTAL	100.0

normal (≈20 to 25 psig). Also, the calibration for the magnetic flowmeter appeared to be ≈10% high when compared to tank dropout measurements during filling of the second canister. The calibration was made with slurry which did not contain frit. Apparently the solids-loading in the feed slurry affects the magnetic flowmeter flow indication. This erroneous flow

TABLE A.33. FSSC-39 Run Summary

Parameter	Value
<u>First Canister (ICM-28)</u>	
Operating time, h	14.3
Liters of waste processed	{1800 L @ 80.7 g oxide/L 2000 L @ 92.4 g oxide/L
Maximum feedrate, L/h	295
Average feedrate, L/h	271
<u>Second Canister (ICM-29)</u>	
Operating time, h	57
Downtime, h	8.9
Liters of waste processed	{1100 L @ 357 g oxide/L 1680 L @ 293 g oxide/L
Maximum feedrate, L/h	~67
Average feedrate, L/h	49

indication did not affect glass product composition since the frit and waste slurry were pre-mixed in the feed makeup tank. This is one advantage of placing the glass-forming components in the feed slurry. Other than the slurry feeding interruptions and incorrect calibration of the magnetic flowmeter, the equipment functioned well during this test.

FSSC-40

The purpose of this run was to produce SRL waste calcine for testing in alternative waste forms processes. The calciner was operated for 5.88 h with 0.43 h of downtime caused by a plugged strainer. No other problems were encountered.

Significant operating parameters for the full-scale calciner during the period from April 1977 to November 1980 are shown in Table A.34.

TABLE A.34. Summary of Waste Processing Runs With the Full-Scale Spray Calciner, April 29, 1977 to November 18, 1980

Run Number	Preliminary	Preliminary	Preliminary	FSSC-1	FSSC-2	FSSC-3	FSSC-4	FSSC-5	FSSC-6	FSSC-7	FSSC-8	FSSC-9
Date	4/29/77	5/25/77	6/23/77	7/26 to 7/28/77	8/3/77	8/8/77	8/12/77	9/16 to 9/27/77	12/1/77	12/6; 12/8/77	12/12/77	2/22/78
<u>Run Summary</u>												
Operating time, h	~2.5	5	5.17	15.7	5.5	0.25	3.58	252.4	1.9	16.2	4.35	11.3
Downtime	NA(a)	NA	NA	NA	NA	None	None	NA	None	None	NA	0.9
Reasons for downtime	NA	NA	NA	NA	NA	---	---	NA	---	---	NA	Vacuum blower breaker out
Total feed used, L	NA	700	NA	2068	905	48	380	19,647	400	3,137	929	2,770
Average Feedrate, L/h (while operating)	Up to 120	140	120 to 160	132	165	192	106	77.8	210	194	214	245
Type of melter coupled to	ICM	None	ICM	ICM	None	ICM	None	ICM		ICM	ICM	None
<u>Feed</u>												
Type	PW-7a	PW-7a	PW-7a	PW-7a	PW-7a	PW-7a	PW-7a	PW-7a	PW-7a	PW-7a	PW-7a	SRL "TNX"
Concentration, g oxide/L	NA	NA	NA	158	158	NA	158	~150	NA	173	173	See run summary
<u>Atomizing Feed Nozzle</u>												
Type	SU 72(b)	SU 72	SU 72	SU 72	External Mix 70	NA	SU 82	NA	SU 82	SU 82	SU 82	SU 82 ceramic
Typical airflow, scfm	SU 72	21	NA	NA	NA	NA	---	NA	NA	NA	NA	43
Typical air pressure, psig	SU 72	60	55	60 to 80	80	80	60	60	64	62	50	56
Typical feed pressure, psig	SU 72	NA	NA	34 to 50	NA	NA	37	37	43	35	37	40
<u>Calciner</u>												
Typical furnace temp., °C												
Zone 1	NA	~800	NA	800	800 to 900	800	800	800	820	800	NA	860
Zone 2	NA	~800	NA	800	800 to 900	800	800	800	812	850	NA	850
Zone 3	NA	~800	NA	800	800 to 900	800	800	800	845	900	NA	870
Zone 4	NA	~800	NA	800	800 to 900	800	800	800	920	900	NA	870
Typical power consumption, kW				@ 133 L/h		@ 120 L/h		@ 80 L/h		@ 232 L/h	@ 290 L/h	@ 250 L/h
Zone 1	NA	NA	NA	56.4	NA	NA	56	48	NA	66	75	67.2
Zone 2	NA	NA	NA	49.2	NA	NA	48	39	NA	80	78.5	68.8
Zone 3	NA	NA	NA	39.6	NA	NA	32	32	NA	60	63	66.0
Zone 4	NA	NA	NA	44.4	NA	NA	40	32	NA	60	63	59.6
Normal vacuum (in. H ₂ O) vibrator	NA	NA	NA	10 to 15	3 to 6	10	3 to 6	8	10	12		15
Cycle time, min	NA	3	NA	NA	NA	NA	NA	NA	NA	6 Total	6 total	6 total
Pulse time, s	NA	5	NA	NA	NA	NA	NA	NA	NA	NA	NA	4
<u>Offgas Filters</u>												
Blowback air pressure, psig	NA	70	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Blowback cycle, min	NA	11	NA	NA	NA	NA	NA	NA	NA	NA	NA	11
Blowback interval, min	NA	~0.02	NA	NA	NA	NA	NA	NA	NA	NA	NA	~0.03
Blowback pulse, s	NA	0.2	NA	NA	NA	NA	NA	NA	NA	NA	NA	0.25
Typical filter pressure drop (in. H ₂ O)	NA	<5	NA	NA	NA	NA	NA	0.8	NA	1.0	NA	2

(a) NA--data not available.

(b) Spraying Systems Company Nozzle Set-Up No. 72 (SU 72).

TABLE A.34. (contd)

Run Number	FSSC-10	FSSC-11	FSSC-12	FSSC-13	FSSC-14	FSSC-15	FSSC-16	FSSC-17	FSSC-18	FSSC-19
Date	3/20 to 3/23/78	3/23/78	4/12/78	5/22/78	5/22/78	6/1/78	6/23 and 6/26/78	7/20 and 7/21/78	8/9/78	9/11 and 9/14/78
<u>Run Summary</u>										
Operating time	62.3	14.6	2.28	3.0	4.67	4.58	~7.5	15.2	3.0	8.5
Downtime	NA	None	None	None	None	None	NA	NA	NA	NA
Reasons for downtime	NA	---	---	---	---	---	NA	NA	NA	Feed plugs
Total feed used	12,377	2,940	396	~500	~930	244	~760	3,133	320	NA
Average feedrate, L/h (while operating)	199	201	174	167	199	53.3	~100	206	107	NA
Type of melter coupled to	None	None		None	None	CM	CM	ICM	None	CM
<u>Feed</u>										
Type	SRL composite	SRL low-sodium composite	PW-7a	SRL high iron	SRL high-alumina	PW-7a	PW-7a	PW-7a	SRL composite	SRL
Concentration, g oxide/L	See run summary	See run summary	163	See run summary	See run summary	~150	~150	~150	NA	NA
<u>Atomizing Feed Nozzle</u>										
Type	SU 82 SS	SU 82 SS	SU 72 ceramic	SU 82 ceramic	SU 82 ceramic	SU 82	SU 82 SS	SU 82	SU 82	NA
Typical airflow, scfm	46	varied	31.6	28	28	50	NA	40.5	NA	NA
Typical air pressure, psig	60	varied	69	59	58.5	62	NA	57	58.1	NA
Typical feed pressure, psig	35	varied	45	40	40	25	NA	36	27.7	NA
<u>Calciner</u>										
Typical furnace temp., °C										
Zone 1	845	NA	NA	NA	850	NA	NA	NA	NA	895
Zone 2	855	NA	NA	NA	700(c)	NA	NA	NA	NA	600
Zone 3	855	NA	NA	NA	898	NA	NA	NA	NA	855
Zone 4	860	NA	NA	NA	918	NA	NA	NA	NA	855
Typical power consumption, kW	@ 200 L/h	NA	NA							
Zone 1	77	NA	NA	NA	74	NA	NA	NA	NA	NA
Zone 2	49.5(c)	NA	NA	NA	35.5(c)	NA	NA	NA	NA	NA
Zone 3	62.5	NA	NA	NA	70	NA	NA	NA	NA	NA
Zone 4	55.5	NA	NA	NA	49.5	NA	NA	NA	NA	NA
Normal vacuum (in. H ₂ O)	15	16	10.5	11	10	7	7	15	NA	NA
vibrator										
Cycle time, min	9.5 total	9.5 total	10 total	NA	10 total	NA	NA	NA	NA	NA
Pulse time, s	NA	2	2	NA	3	NA	NA	NA	NA	NA
<u>Offgas Filters</u>										
Blowback air pressure, psig	NA		NA	NA		NA	NA	NA	NA	NA
Blowback cycle, min	varied	15	NA	30	60	NA	NA	60	NA	NA
Blowback interval, min	varied	0.08	NA	0.08	0.08	NA	NA	NA	NA	NA
Blowback pulse, s	varied	0.3	NA	0.03	0.03	NA	NA	0.2	NA	NA
Typical filter pressure drop (in. H ₂ O)	varied	1.6	NA	5	6	3.2	NA	1.2	NA	NA

(c) Three failed heating elements.

TABLE A.34. (contd)

Run Number	FSSC-20	FSSC-21	FSSC-22	FSSC-23	FSSC-24	FSSC-25	FSSC-26	FSSC-27	FSSC-28	FSSC-29
Date	9/19 to 9/29/78	10/24/78	10/25 to 10/26/78	3/6/79	3/22/79	3/25 to 3/31/79	4/3/79	4/12/79	4/16/79	4/17 to 4/27/79
<u>Run Summary</u>										
Operating time, h	212.6	1.37	22.2	3.0	5	77.1 h	2.62	1.3	1.22	204
Downtime, h	13.4	None	None	2.0	None	43.8	None	0.5	None	35
Reasons for downtime	See run summary	---	---	nozzle change, ext. to int.	---	canister change- outs feed system plugs	---	feed system plugs	---	(e)
Total feed used	~28,900	~190	~3,300	~925	1,550	19,000	NA	NA	NA	59,600
Average feedrate, L/h (while operating)	~136	~139	~149	309	310	246	NA	NA	NA	292
Type of melter coupled to	CM	None		None	None	ICM	None	None	None	CM
<u>Feed</u>										
Type	SRL composite w/o sodium	SRL	SRL composite	PW-7a	SRL	SRL TDS	SRL TDS	SRL TDS	SRL TDS	SRL TDS
Concentration, g oxide/L		NA	NA	NA	70	70	70	70	70	46/55/64 ^(f)
<u>Atomizing Feed Nozzle</u>										
Type	SU 82 SS	SU 82	SU 82	SU 82 with ^(d) 0.95-cm orifice	SU 82 with ^(d) 0.95-cm orifice	SU 82 and SU 82 with 0.95- cm orifice	SU 152	SU 152	SU 152	SU 152
Typical airflow, scfm	NA	NA	NA	49	58	43	NA	97	83	93
Typical air pressure, psig	NA	NA	NA	53	64	54	NA	36.6	28.7	33.8
Typical feed pressure, psig	NA	NA	NA	38	37	30	NA	14	18	19
<u>Calcliner</u>										
Typical furnace temp., °C										
Zone 1	NA	NA	NA	800	840	820	785	NA	NA	855
Zone 2	NA	NA	NA	825	875	850	830	NA	NA	875
Zone 3	NA	NA	NA	815	865	845	825	NA	NA	860
Zone 4	NA	NA	NA	800	855	825	800	NA	NA	845
Typical power consumption, kW					@ 325 L/h	272 L/h		@ 300 L/h		
Zone 1	NA	NA	NA	83	87	82	NA	NA	NA	88
Zone 2	NA	NA	NA	92	99	86	NA	NA	NA	80
Zone 3	NA	NA	NA	68	73	60	NA	NA	NA	62
Zone 4	NA	NA	NA	60	67	54	NA	NA	NA	54
Normal vacuum (in. H ₂ O)	NA	NA	NA	10	10	10	10	10	10	8
vibrator										
Cycle time, min	NA	NA	NA	7.5 total	4.75 total	4.75 total	4.75 total	4.75 total	3.17 total	3.17 total
Pulse time, s	NA	NA	NA	1.5	1.5	1.5	1.5	1.5	1.5	1.5
<u>Offgas Filters</u>										
Blowback air pressure, psig	NA	NA	NA	40	40	40	40	40	40	40
Blowback cycle, min	NA	NA	NA	15	19	19	19	19	12.67	12.67
Blowback interval, min	NA	NA	NA	1.36 (avg.)	1.73 (avg.)	1.73 (avg.)	1.73 (avg.)	1.73 (avg.)	1.15 (avg.)	1.15 (avg.)
Blowback pulse, s	NA	NA	NA	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Typical filter pressure drop (in. H ₂ O)	NA	NA	NA	6	4.5	6	NA	8	7	7

(d) For increased liquid flow, the air-cap orifice was enlarged to 0.95 cm.

(e) Twenty hours to dissipate melter foam; 14 h to repair frit feeder airlock system; 1 h to remove atomizing nozzle for inspection (it was reinstalled).

(f) Three different feed concentrations were used.

TABLE A.34. (contd)

Run Number	FSSC-30	FSSC-31	FSSC-32	FSSC-33	FSSC-34	FSSC-35	FSSC-36	FSSC-37	FSSC-38	FSSC-39	FSSC-40
Date	6/4 to 6/30/79	7/30 and 8/1/79	8/28/79	9/28/79	10/29/79	11/5 to 11/9/79	2/29/80	3/4 and 3/6/80	5/5/80	8/21/80	11/18/80
<u>Run Summary</u>											
Operating time, h	504	2.7 with ICM, 4.5 without	12.2	6.8	3.37	79.5	4.48	33	100	71	5.88
Downtime, h	109	None	NA	None	None	30.5	4.0	7.19	0.5	8.9	0.43
Reasons for downtime	(g)	---	NA	---	---	(h)	damaged nozzles	(i)	loss of vacuum	feed system plugs	strainer plug
Total feed used, L	154,000	6,540	3,400	1,730	NA	19,180	NA	8,000	19,020	6,660	NA
Average feedrate, L/h (while operating)	306	208	279	254	NA	241	NA	242	190	94	NA
Type of melter coupled to	CM	ICM	None	None	None	CM	None	ICM	None	ICM	None
<u>Feed</u>											
Type	SRL TDS	SRL TDS	SPC-4	SRL TDS with additives	SRL TDS with additives	SRL TDS with and without additives	SRL TDS	SRL TDS	SRL TDS	SRL TDS & TDS with frit	SRL
Concentration, g oxide/L	65	65	189	varied	varied	varied	144	100	100	varied	
<u>Atomizing Feed Nozzle</u>											
Type	SU 152	SU 82 with modified 0.95-cm orifice	SU 82 with modified 0.95-cm orifice	SU 82 with modified 0.95-cm orifice	SU 152	SU 152	SU 82	SU 82	SU 82	SU 82	SU 82
Typical airflow, SCFM	87	40	34	30	80	85	NA	31	varied	varied	29
Typical air pressure, psig	30	34	30	32	27	30	NA	35	varied	varied	36
Typical feed pressure, psig	NA	NA	15	17	11	10	NA	25	varied	varied	26
<u>Calcliner</u>											
Typical furnace temp., °C											
Zone 1	805	825	855	875	NA	875	NA	730	varied	varied	NA
Zone 2	875	825	855	840	NA	875	NA	730	varied	varied	NA
Zone 3	843	825	870	853	NA	875	NA	730	varied	varied	NA
Zone 4	835	825	880	855	NA	875	NA	730	varied	varied	NA
Typical power consumption, kW	@ 300 L/h	@ 258 L/h			@ 260 L/h						
Zone 1	84	84	90	NA	NA	60	NA	70	varied	varied	NA
Zone 2	91	80	97	NA	NA	78	NA	53	varied	varied	NA
Zone 3	56	45	51	NA	NA	45	NA	44	varied	varied	NA
Zone 4	53	45	53	NA	NA	38	NA	35	varied	varied	NA
Normal vacuum (in. H ₂ O) vibrator	6	6	5	6	6	5	NA	10	5	10	7.6
Cycle time, min	5.0 total	5.0 total	5.0 total	5.0 total	5.0 total	8.8 total	NA	8.8 total	8.8 total	8.8 total	8.8 total
Pulse time, s	1.5	1.5	1.5	1.5	1.5	1.5	NA	1.5	1.5	1.5	1.5
<u>Offgas Filters</u>											
Blowback air pressure, psig	40	40	40	40	40	40	NA	40	40	40	40
Blowback cycle, min	10	10	10	10	10	8.8	NA	8.8	8.8	8.8	8.8
Blowback interval, min	0.91 (avg.)	0.91 (avg.)	0.91 (avg.)	0.91 (avg.)	0.91 (avg.)	0.8 (avg.)	NA	0.8 (avg.)	0.8 (avg.)	0.8 (avg.)	0.8 (avg.)
Blowback pulse, s	0.2	0.2	0.2	0.2	0.2	0.2	NA	0.2	0.2	0.2	0.2
Typical filter pressure drop (in. H ₂ O)	15	15 to 20	12	15	17	15	NA	4	3	NA	3.6

(g) Fifty-seven hours to dissipate melter foam; 21 h to repair frit feeder airlock system; 17 h for melter face heater replacement; 11 h for melter-to-canister and canister change-out problems; 3 h for atomizing nozzle change-out and feed system problems.
(h) Twenty and a half hours to dissipate melter foaming; 10.0 h to change feed composition as a result of feed buildup on the calciner spray chamber wall.
(i) One and a half hours as the result of plug in ICM connecting section; 0.92 h for airlock valve repair; 4.77 h for frit elevator repair.

NOTE

Two Bechtel reports are included in the following appendices (Appendices B and C). The appendices of these reports have been removed for the sake of brevity. Therefore, the reader is cautioned to ignore references to these appendices that may appear in the text of the reports.

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APPENDIX B

CALCINER FEED SYSTEM FOR THE SPRAY
CALCINATION/IN-CAN MELTING PROCESS

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PACIFIC NORTHWEST LABORATORIES
BATTELLE MEMORIAL INSTITUTE

THE SPRAY CALCINATION/IN-CAN MELTING PROCESS

REVIEW & RECOMMENDATIONS
FOR
CALCINER FEED SYSTEM
TECHNICAL REPORT NO. 2

Prepared by
BECHTEL INCORPORATED
SAN FRANCISCO, CALIFORNIA
JOB NO. 12400
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BECHTEL INCORPORATED
SAN FRANCISCO, CALIFORNIA
CONTRACT B-42999-AE

TECHNICAL REPORT NO. 2
REVIEW AND RECOMMENDATIONS
FOR
CALCINER FEED SYSTEM

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1.0 SCOPE

Battelle Pacific Northwest Laboratories has developed a Spray Calcination/In-Can Melting Process as part of its Waste Fixation Program for the nuclear fuel cycle. This report covers Bechtel's review and recommendations for the spray calciner feed system, considering liquid wastes from both defense and industrial facilities.

2.0 GENERAL

Bechtel review and recommendations are based on three possible feed streams to the spray calciner:

- o An acidic feed stream for an industrial facility.
- o A basic feed stream for an industrial facility.
- o A feed stream for a defense facility.

BNW is developing a spray nozzle as part of its Spray Calcination/In-Can Melting Process, and is considering the use of atomizing air with both internal-mix and external-mix nozzle designs.

As the actual nozzle configuration is not yet determined, Bechtel considered feed systems with both types of nozzles. The internal-mix nozzle requires delivery of the feed stream to the nozzle in the range of 20 to 30 psig and the external-mix requires a delivery pressure of less than one psig.

Equipment for the Spray Calcination/In-Can Melting Process is to be located in a remote cell, and a remote-operated crane is to serve as the only operating tool and major maintenance tool. Electro-Mechanical Manipulators are not to be used. Master-Slave Manipulators to be inserted through the cell walls can be used if required. Bechtel does deviate from the above basis in certain cases identified in the report and recommends the location of some equipment in a shielded equipment niche with its own viewing and handling facilities.

The feed strainer for removal of large particles that would plug the spray nozzle is not included in the scope of this report as it was agreed with BNW for the purpose of this report that the feed strainer would be considered to be located ahead of the calciner feed tank. In this location, after the HLLW and ILLW surge tanks but before the calciner feed tank, the blowdown from the strainer can be returned to the HLLW and ILLW surge tanks, and a means of size reduction and disposal developed. Tests would be required to demonstrate that the quantity of large particles requiring screening is not excessive and also that a practical size reduction step can be developed.

This report does not make a specific recommendation for equipment for a calciner feed system, but makes general recommendations for equipment as they are affected by the different feeds and spray nozzle requirements.

3.0 CONCLUSIONS AND RECOMMENDATIONS

Bechtel's conclusions and recommendations are summarized below where for each type of feed and spray nozzle requirement recommendations are made for feed tank agitation, feed pump and feed measurement and control. There are two alternative recommendations for Defense and Industrial-Basic Feed with internal-mix nozzle.

FEED AND NOZZLE TYPE		RECOMMENDATION		
Feed	Spray Nozzle Type	Feed Tank Agitation	Pump Type	Flow Measurement and Control
Industrial-Acidic	External	Air (Pachuca Tank)	Air Lift	Air Lift
Industrial-Acidic	Internal	Air (Pachuca Tank)	Remote Head Diaphragm	Remote Head Diaphragm Pump
Defense & Industrial-Basic	External	Air (Pachuca Tank)	Air Lift	Air Lift
Defense & Industrial Basic	Internal	Mixing Eductor	Centrifugal	Magnetic Flow Meter & Control valve
Defense & Industrial Basic	Internal	Air (Pachuca Tank)	Remote Head Diaphragm	Remote Head Diaphragm Pump

Bechtel's recommendations stress the use of air both for tank agitation and for an air lift to an external-mix spray nozzle. This results in the elimination of the need for mechanical equipment for tank agitation and pumping, reduces the maintenance requirements and increases the reliability of the calciner feed system at the expense of a modest increase in off-gas capacity. The air lift for feed to the spray calciner would

have to handle a low flow (approximately 1 GPM) of a stream averaging approximately 1% solids and the air lift system would have to be developed to assure that it does not plug with solids. Development of the feed air lift and the use of an "idler" air lift are discussed in Section 7.1 of this Report.

Bechtel urges that experimental work be done to confirm the adequacy of air agitation in the "Pachuca" feed tank and the development of air lift systems and operating procedures to minimize any plugging tendency in feed streams containing undissolved solids. Bechtel feels that the cost of development work as suggested in this Report for air agitation and air lifts would be modest. Bechtel also urges the development of BNW's external-mix spray nozzle to eliminate the need for any substantial pressure in the feed system as required for the internal-mix nozzle.

In the event future development work indicates minimal plugging in an air lift system cannot be avoided and an external-mix nozzle is desired, the same pumping and flow measurement and control systems as recommended for an internal-mix nozzle can also be used with an external-mix nozzle.

This report is based on eliminating all mechanical joints below the liquid level of the feed tank whose failure would lead to loss of feed tank contents. However, if mechanical joints were permitted below the feed tank liquid level and the cell liner and sump were relied on to contain the tank contents in the event of a mechanical joint failure, it would be possible to locate a remote head diaphragm pump or a centrifugal pump in an equipment niche below the feed tank. In this location a remote head diaphragm pump would not require a suction pot and its air lift and the centrifugal pump could have adequate suction conditions without having to be installed as a submersible pump in the feed tank. The equipment niche could also provide shielding for the centrifugal pump electric motor driver and extend its service life.

4.0

DEFINITION OF FEED

BNW has defined three spray calciner feeds for the purpose of this study:

o Industrial - Acidic:

An acidic feed stream for a future industrial facility based on the material balance shown on Allied General Nuclear Services' drawing 503E-A-001 attached to this report as Appendix A. This is a conceptual material balance developed by Allied General Nuclear Services for Sandia Laboratories under an ERDA contract.

o Industrial - Basic:

A basic feed stream for a current industrial facility as shown on Nuclear Fuel Services "Description of NFS Wastes and Storage Tanks", attached to this report as Appendix B.

o Defense (ERDA):

A typical feed stream for a defense facility (ERDA) described as follows:

Estimated Composition of ARHCO Terminal Liquor

<u>Chemical</u>	<u>Weight Percent</u>
H ₂ O	47.0
NaNO ₃	12.1
NaNO ₂	12.3
Na ₂ CO ₃	0.8
NaOH	14.3
NaAlO ₂	11.7
Na ₂ SO ₄	0.5
Na ₂ CrO ₄	1.2
 <u>Radiochemical</u>	 <u>Ci/Liter</u>
Cs-137	0.44
Sr-90	0.015
 <u>Specific Gravity</u>	 1.4

There will be some solids in the feed, but the amount and characteristics are not well defined now.

BNW has defined the feed rate for the three feed streams to the calciner to be about 250 l/hr, including the formic acid used in the Industrial-Acidic flowsheets and any other additives that may be used for the other flowsheets. BNW currently anticipates but has not yet demonstrated that the BNW full-scale calciner will handle this capacity at maximum calcination rates.

BNW has defined for the purpose of this report that the remote cells for solidification of waste are anticipated to have an average cell environment as follows:

Industrial - Acidic:	1×10^6 R/Hr
Industrial - Basic and Defense	1 to 5×10^4 R/Hr

Viscosities of the calciner feed streams have not been defined. However, Bechtel indicates in Section 9.2 the maximum viscosity the feeds could have before they would become significant in design and equipment selection.

5.0 PROCESS DESIGN

5.1 Bechtel Flow Diagrams

Process Flow Diagrams, Figures 1, 2 and 3, show proposed methods for feeding high-level wastes to the BNW spray calciner. Figure 1 uses an airlift and Figure 2 a remotely driven diaphragm pump, and include material balances based on Industrial-Acidic composition and feed rates (equivalent to a 5 MTUD reprocessing plant). Figure 1 could be used only with the external-mix spray nozzle because of the low head available from the feed airlift. Figure 3 is based on use of a tank-mounted centrifugal feed pump. The material balance for this flow diagram is based on ERDA waste. Composition and feed rate were specified by BNW.

5.2 Feeds

The feed in Figure 1 and 2 flow diagrams is a combination of Industrial-Acidic High-Level Liquid Waste at a rate of 150 gallons per MTU, and Intermediate-Level Liquid Waste at 75 gallons per MTU. The estimated fission product decay heat for the HLLW is 2.5 watts per liter and for the ILLW, 0.01 watts per liter.

The feed to Figure 3 Flow Diagram was set by BNW at 250 l/hr of the composition typical for ERDA waste. It is also somewhat representative of Industrial-Basic waste. The level of fission product decay heat is much less than that of the Industrial-Acidic waste case.

Figures 1 and 2 would be capable of handling any of the three contemplated waste streams. However, Bechtel considers Figure 3, using the centrifugal pump, satisfactory only for ERDA or Industrial-Basic feeds because the higher level of radiation from the Industrial-Acidic waste would rapidly damage the electric motor drive with its bearing lubricants and electrical components. The difference in expected cell radiation levels of 10^6 and 10^4 R/hr means that with radiation damage limiting, the time to failure using Industrial-Acidic waste would be 1/100th the time for ERDA and Industrial-Basic wastes.

5.3 Feed Tank

Two feed tanks of 24 hour capacity each are recommended. Tanks will alternate in service with one tank feeding the spray calciner and the other tank receiving feed from the feed preparation section or in clean-up and flushing. Factors weighed in this selection were operating convenience, cell space, maintenance, mixing and feeding requirements, and dissipation of decay heat without use of internal cooling coils.

A 24-hour tank is similar in size to the spray calciner itself, and therefore creates no special problems in maintenance or cell space. With jacket cooling alone it can dissipate up to 15 watts/liter of decay heat generation by the contents. This is over ten times the decay heat generation anticipated for the Industrial HLLW/ILLW feed mix.

To prevent collection of solids, particularly heat generating solids, in regions where they might be difficult to disperse by tank mixing or cleaning, the feed tank has a 60° cone bottom.

5.4 Mixing

The Industrial-Acidic feed mixture is expected to contain insoluble solids. Only approximate information on the quantity and the physical properties of these solids is available. The quantity is expected to be in the order of one percent. Two types of solids are expected -- granular and flocculent. Although the quantity of solids is relatively small, they are expected to contribute a significant proportion of the decay heat load. These considerations all dictate a reliable mixing technique to prevent plugging of equipment, unwanted settling, and to assure reasonable uniformity in the calcined product. The ERDA and Industrial-Basic wastes, since they are neutralized, will probably contain more insoluble solids than the Industrial-Acidic waste.

Bechtel recommends an air agitation method for this service that has been demonstrated in Pachuca Tanks used for leaching pulverized gold and uranium ores. This mixing method has been used for many years in industrial service and is quite adaptable to remote operation. The slurries circulated in Pachuca Tanks are heavier and probably more difficult to mix than the proposed HLLW/ILLW solutions. An article describing both the practical and theoretical aspects of the operation of these tanks is attached to this report as Appendix C -- A. G. W. Lamont, Air Agitation and Pachuca Tanks, Canadian Journal of Chemical Engineering, August, 1958.

The Pachuca Tank used the airlift principle to circulate the contents of the tank. Tanks have L/D proportions of about 2 and a 60° cone bottom. Air is sparged into the bottom of the cone. It may be sparged into the bottom of a center column that rises part, or all the way, to the top of the tank, or there may be no column at all. The Lamont article analyzes circulation characteristics of all three options.

For an Industrial, or Defense feed tank, Bechtel recommends a column extending to the top of the cone, with side perforations to permit circulation at low liquid levels. The specific features of this column should be optimized by experimentation.

A typical Pachuca Tank for leaching pulverized gold and uranium ores would be 22'-6" in diameter with about 45 feet of liquid depth including the 60° cone, and would use about 300 SCFM of air for mixing. There is no precise method for scaling this air rate down to the recommended tank size along a parameter of "equivalent mixing". Neither is there any way to determine the "degree of mixing" required by the HLLW/ILLW waste, or how their "mixability" relates to that of the heavy slurries mixed in the Pachuca Tanks. These factors must be resolved experimentally.

The Bechtel flow diagram is based on using 1.5 SCFM of air per square foot of tank cross section, as compared to 0.76 SCFM per square foot for the classic Pachuca Tank. This ratio is suggested by Figure 6 of the Lamont article, which indicates that decreasing the depth of liquid from 45 feet to 11 feet roughly halves the energy transfer for mixing imparted by a given quantity of air injected at the bottom of the tank. This is not a rigorous basis for relating the two services, and tests are recommended to determine the required air quantity for optimization of mixing.

The use of air for mixing in the feed tanks and airlifts for feeding the calciner obviously increase the amount of offgas to be treated. However, reference to the material balances on Figures 1, 2, and 3 shows that this is a relatively small increase. On Figure 1 and 2, where airlifts and air mixing are used, the total offgas from the process is 241 SCFM. On Figure 3 Flow Diagram where mixing and feeding is done by pumps, the total offgas is only reduced to 180 SCFM. On a basis of dry gas the difference is greater, but the flow in either case is small and should not result in a significant difference in equipment or operating costs.

For the centrifugal pump case, Figure 3, mixing in the feed tank is accomplished by the use of a circulating tank eductor. The pumped motive fluid to the circulating tank eductor is determined by equating the required power of the eductor motive fluid to the power released during tank agitation by the expansion of compressed air in the feed tank as shown on Figures 1 and 2. The eductor circulates a total of four times the quantity of the motive fluid. Liquid mixing eductors have been used commercially for many years.

6.0 FEED CONTROLS

The proposed controls of the liquid feeds to the spray calciner are different for all three flow diagrams. However, the frit feed and the formic acid feed when required, are ratioed from the liquid feed rate for all flow diagrams.

6.1 Figure 1, Airlift Case

Figure 1, the airlift feed case, all instrumentation for flow measurement is outside the cell. A two-stage calibrated air lift is used. The first stage head pot is held at constant level by overflow back to the feed tank, thereby providing constant submergence for the airlift delivering feed to the spray calciner. Feed rate is then a function only of air rate to the second stage airlift. The first head pot also contains a dip tube used for level recording and flow calibration. For calibration, the circulating air lift from the feed tank would be shut off for a short period and the rate of drop in head pot level measured. This procedure could be automated and computerized, and repeated as often as found desirable, several times an hour if necessary. The rate of drop in level in the main feed tank is, of course, another overall averaging check on the air lift calibration. Bechtel believes that this control system would hold the feed rate well within the allowable variation of $\pm 10\%$ sought by BNW.

6.2 Figure 2, Metering Pump Case

Figure 2 uses positive displacement diaphragm pumps to control the feed rate. Only the remote pump head is located in the cell. The drive motor and metering piston are outside the cell for radiation protection and ease of maintenance. A line carries the displacement fluid from the metering piston, through the cell wall to the diaphragm head. The diaphragm and ball checks in the head would be stainless steel, or some other material capable of resisting corrosion and radiation damage. There is a possibility that the pulsating flow from the pump could affect spray nozzle performance. If this is a problem, there are pulsation dampening systems which could be used. These are discussed later in this report.

As with the airlift feed system, the diaphragm pump system has the advantage of keeping all radiation-susceptible equipment outside the cell. It has the disadvantage of requiring maintenance on the remote pump heads in the cell. Also, it requires one stage of airlift to get the remote head high enough so that a leaking or open remote piping connector could not drain the contents of the feed tank on to the floor.

6.3 Figure 3, Centrifugal Pump Case

Figure 3 uses an inline magnetic flow meter and control valve in the feedline to control feed flow from the pump. This puts both a control valve and a flow meter in the cell. However, with a comparatively low level of cell activity, such as would exist for the Defense and Industrial-Basic wastes, a reasonable life may be attainable from this equipment.

SIGNATURE T B

Rev 8-12-77

DATE 4-29-77

TITLE Battle Northwest

JOB NO. 12,402

SUBJECT Spray Calciner Feed Studies - Airlift Case

SHEET NO. _____

Notes:

1. All head pots 4" ϕ x 3' x 60°
2. Basis for balance is:
 - Feeding calculator from #2 Feed Tank
 - #1 Feed Tank mixing & ready to feed
3. This case possible only with external mix nozzle.

FIGURE N^o 1

CALCULATION SHEET

129 (0512) 4-68

SIGNATURE T.B.Rev. 8-12-77
DATE 5-2-77TITLE Battelle NorthwestJOB NO. 12,400SUBJECT Spray Calciner Feed Studies - Metering Pump Case

SHEET NO. _____

Notes:

1. Head pots 4" ϕ x 3' x 60"
2. Basis for balance is
- Feeding calciner from #2 Feed Tank
- #1 Feed Tank Mixing and ready to feed

FIGURE No 2

CALCULATION SHEET

128 (0512) 4-68

SIGNATURE TD PROCESS FLOW DIAGRAM No. 3 Rev 8-12-77
TITLE Battelle Northwest DATE 6-8-77
SUBJECT Spray Calciner Feed Studies - Centrif. Pump Case JOB NO. 12,400 SHEET NO. _____

Material Balance Basis:
ERDA Waste, 250 l/hr

	①	②	③	④	⑤
Conc, g/l					
H ₂ O	658	Same	Same	658	None used for ERDA, NFS wastes
Na ₂ SO ₄	541			541	
NaOH	200			200	
Susp. Solids	TBD			TBD	
Total g/l	1400 (excl. solids)			1400 (excl. solids)	
l/hr (l/batch) (6000)	7,250	7,250	250		
Kg/hr (Kg/batch) (8400)	10,150	10,150	350		
GPM (Gal/batch) (1538)	32	32	1.1		
Batch Freq, hr	24				

	⑩	⑪	⑫
Calcine Flow, lb/hr			
O ₂	0.96	1.651	0.071
N ₂	4.11	4.376	0.266
NO	-	1.38	-
NO ₂	-	1.10	-
CO ₂	-	0.06	-
H ₂ O	-	21.57	-
Total lb/hr	5.07	30.137	0.337
lb/hr	145.8	658.3	9.72
Kg/hr	66.1	298.5	4.41
SCFM (32°F)	30.3	180.3	2.0 (avg)

FIGURE No 3

7.0 SPECIAL PROCESS FEATURES

7.1 Airlifts

The low point in the line of an airlift is vulnerable to collection of solids and possible plugging. The flow diagrams show an airlift design concept for prevention of this type of plugging. The downleg of the airlift is sloped to avoid horizontal flow runs, and joins the upleg at a sharp angle to localize the zone where solids would tend to collect. A small portion of the total airlift air, say 10 - 20%, is diverted through a restriction orifice to the apex of the juncture of the down and uplegs. This small flow of air would keep the apex agitated and prevent settling and possible packing of solids into the low point to form a plug. The primary air flow to the airlift maintains enough liquid flow to sweep the apex bleed air into the upleg, and there would be no tendency for the airlift to run backwards.

Bechtel recommends that this type of airlift be studied experimentally in transparent pipe before finalizing design. Both plugging and unplugging characteristics could be investigated. A number of design details could be optimized, and performance confirmed by such an investigation. A few possible configurations are shown on Figure 4.

During periods when a feed tank is full and the first airlift is circulating but the second airlift is not feeding forward, solids would tend to settle and plug the low point of the second airlift. To prevent this, an "idler" airlift should be installed from the low point of the second lift back to the constant level head pot to maintain circulation. The "idler" airlift could be shut off when forward feeding started. This feature was not included on the flow diagram because its function might be obscure and it might confuse the understanding of the fundamental operating mode of the process.

7.2 Feed Tank Bottom

The relative configuration of the feed tank bottom, the bottom outlet, and the circulation draft tube are areas where simulation experiments in transparent equipment could be helpful. The draft tube itself has many parameters to look at: length, diameter, perforations, height off the tank bottom, etc. Figure 5 shows several possible configurations of draft tubes and tank bottoms.

The configurations with the bottom outlet above the apex of the cone would require addition of a cleanout jet leg to completely empty the tank. The acceptability of an apex outlet may depend on provision of an idler airlift to continually circulate the outlet leg.

For the case using the tank-mounted centrifugal pump, it will be necessary to provide a vortex breaker in the tank bottom. This is a well known device, and its operation should not require experimental confirmation.

POSSIBLE CONFIGURATIONS FOR ANTI-PLUGGING

AIRLIFT FOOT DESIGN

(a)

(b)

(c)

(d)

(e)

FIGURE No 4

POSSIBLE FEED TANK BOTTOM DESIGNS

(a)

(b)

(c)

FIGURE № 5

8.0 FEED TANK

8.1 Feed Tank Mechanical Design

Bechtel recommends that the feed tanks be designed and constructed in accordance with the ASME Pressure Vessel Code, Section VIII, Division I, and the following:

Material: Type 304L stainless steel with two samples from each heat subjected to tests of corrosion resistant properties in accordance with ASTM A262, Practice C.

Design Pressure: Both internal and external design pressures would have to be established. As the feed tanks are vented to the offgas system and have external cooling water jackets, malfunctions and upset conditions in either the offgas system or cooling water system will determine the maximum internal and external operating pressures for the feed tanks. These can only be determined with certainty when the design of the off-gas and cooling water systems are known. Bechtel anticipates that the external pressure on the feed tanks from upset conditions in the cooling water jacket will control the design.

Design Temperature: The maximum temperature the tank would experience is about 240°F if the jacket cooling water was lost and the feed tank self boiled. Bechtel recommends that the design temperature be established as 300°F.

Corrosion Allowance: Bechtel recommends that a minimum corrosion allowance of 1/8 inch be used.

Nondestructive Testing: Bechtel recommends that all pressure retaining welds be 100% radiographed.

Seismic Design: The feed tanks themselves do not have to be designed for safety related seismic loadings as failure of the feed tank themselves would not create a substantial safety hazard as the tank contents would be contained by the cell floor. However, the feed tank structural support system should be designed for the full seismic loadings specified for safety-related components for the actual facility to assure that the feed tank does not fall and damage any items whose failure would be safety related.

8.2 Feed Tank Cooling

Two methods of maintaining the temperature of the self-heating feed stream in the feed tank were considered: internal cooling coils and external jacket. Internal cooling would have a higher overall heat transfer coefficient and require a smaller heat transfer area. However, it is preferred to have the minimum obstructions inside the feed tank so as not to impede the circulation in the tank for maintaining solids in suspension. External jacket for cooling is preferred if adequate cooling capacity can be provided.

To maintain the feed tank at a constant temperature, the total heat transferred is a function of both the heat release per unit volume of feed and the total tank volume. Considering external jacket cooling on the cylindrical portion of the feed tank, the heat flux (BTU/hr ft^2) varies directly with the heat release per unit volume of the feed and the tank diameter. Maintaining the tank contents at 60°C (140°F) and assuming cooling water conditions of 95°F inlet to the jacket and 105° outlet from the jacket, the value of the required overall heat transfer coefficient, "U", may be calculated for various feed heat releases and tank diameters. The results of this calculation are presented on Figure 6. Note that for the feeds considered in this study:

<u>Feed</u>	<u>Decay Heat</u>
Industrial-Acidic	1.551 w/l
Industrial-Basic	0.59 w/l
Defense	Not specified, but less than 1.551 w/l

the required value of the overall heat transfer coefficient, "U", is less than 5 BTU/hr ft^2 $^\circ\text{F}$. Adequate cooling is available with the external jacket for the feeds and feed tank diameter considered in this study. The heat release per unit volume would have to exceed 10 to 15 watts per liter before external jacket cooling becomes marginal with a 4'-6" diameter feed tank.

EXTERNAL WATER JACKETED FEED TANK

FEED TANK DIAMETER
VS

FEED HEAT RELEASE
FOR

VARIOUS OVERALL HEAT TRANSFER COEFFICIENT
WITH

FEED TEMPERATURE - 140°F

COOLING WATER INLET - 95°F

COOLING WATER OUTLET - 105°F

FIGURE No 6

9.0 PUMPS AND DRIVERS

The use of airlifts for controlling and delivering the feed to an external-mix spray nozzle has been previously discussed. Other types of pumping systems that can be considered are positive displacement and centrifugal type pumps.

9.1 Positive Displacement Pumps

The use of an internal-mix spray nozzle may require delivery pressure of the feed to the nozzle at 20 to 30 psig pressure. It is not practical to attempt delivery at these pressures by use of an airlift, but a positive displacement pump can be considered for these conditions. The positive displacement pump can also function as the flow control to the spray calciner by having an adjustable stroke which directly controls pump capacity. The type positive displacement pump Bechtel recommends for consideration is a diaphragm pump with a remote head. The pump motor and master diaphragm head are located outside the remote cell and are connected to the diaphragm head in the remote cell by a hydraulic transmission line filled with demineralized water.

Two manufacturers of remote head diaphragm pumps are Milton Roy Co. and Pulsafeeder. Literature on their pumps is attached as Appendices D and E. The selection of suitable diaphragm material for the remote head bears further study. Metal diaphragms are approximately 5 mils in thickness. Bechtel experience indicates that the use of cold worked Type 304L stainless steel for the diaphragm in nitric acid service is questionable due to the increase in intergranular corrosion of the cold worked material. Bechtel suggests that the suitability of more corrosion resistant materials for the remote head diaphragm, such as zirconium, titanium, or Hastelloy be developed with pump vendors.

The size of diaphragm pump should be selected so that its maximum capacity is more than twice the design feed rate to the spray calciner. This recommendation is to minimize the amount of flexing of the diaphragm and extend its service life.

The flow from a diaphragm pump is pulsating, and the effect of this pulsating flow on the performance of the spray calciner air atomizing spray nozzle is not known. Before using a diaphragm pump, its effect on the spray nozzle and calciner performance should be further investigated. The pulsations in the flow can be minimized by use of a pulsation dampener. Bechtel has considered both a separate vessel and another remote diaphragm head as pulsation dampeners. The conventional hydraulic pulsation dampener was rejected due to the limited radiation resistance of its rubber diaphragm.

The separate vessel, without a diaphragm, is also not recommended as it would require a means for controlling the air cushion pressure and instrumentation for monitoring the liquid level. These facilities would require piping connections to outside the cell, and a malfunction or operator error could lead to the transfer of pressurized radioactive feed out of the cell.

The remote head of a diaphragm pump, without inlet and discharge valves, can be used as a pulsation dampener. The diaphragm would function as a barrier between the feed stream and the air cushion. The air cushion can be controlled remotely from outside the cell. The air cushion in the remote diaphragm head and control air line will probably need modifications to provide adequate volume to function properly as a pulsation dampener. This can be achieved by adding a volume chamber in the range of 100 to 200 cubic inches to the system.

The remote head diaphragm pump would be installed with remote piping connectors. However, to avoid any mechanical joints whose failure could lead to loss of feed tank contents, these remote connectors must be elevated above the liquid level in the feed tank. To accomplish this, the feed is airlifted to a suction pot from which the remote head diaphragm pump takes suction. This is shown on Figure 2.

The remote head diaphragm pump and an additional remote head used as a pulsation dampener can be combined as a unit for installation and replacement. The combined unit could also be all-welded to minimize mechanical joints. It is anticipated that the diaphragm pump will require remote replacement, and the installation of this equipment in a shielded niche could be considered. This niche could have its own viewing and remote-handling facilities to facilitate replacement of the pump.

9.2 Centrifugal Pumps

The motor driver for a centrifugal pump limits its application in a remote cell. A motor with class H insulation is suitable for a total integrated dose of 1×10^9 R. In a cell with an environment of 1×10^6 R/hr, the expected life for a motor with class H insulation is six weeks, in a cell with an environment of 1 to 5×10^4 R/hr, the expected life is 2 to 11 years. Bechtel recommends that centrifugal pumps be considered only for Defense or Industrial-Basic waste streams where the anticipated cell environment is 1 to 5×10^4 R/hr and a pump life of one to two years can be accepted.

The centrifugal pump can be mounted on a flange on top of the feed tank with the pump extending into the feed tank and the motor driver mounted on top of the flange. Mounting both a centrifugal pump and a mechanical agitator on top of the feed tank is not very practical. Therefore, Bechtel recommends that a centrifugal pump alone be

installed with adequate capacity to meet both the needs for feed to spray calciner and feed tank agitation using a circulating tank eductor.

A centrifugal pump would have two head-capacity conditions to meet:

- o That required for feed to the spray calciner which would be a relatively high head and small capacity, approximately 1 GPM. The required head would be approximately:

Delivery Pressure @ Nozzle -	25 psig
Elevation Difference (allow) -	5 psi
Piping Friction loss (allow) -	5 psi
Control Loss (50% delivery & line loss) -	<u>15</u> psi
Total Head @ Pump Discharge	50 psig

- o That required for motive fluid to the circulation tank eductor for feed tank agitation. This requirement can be calculated by equating the power of the pumped motive fluid stream to the power released during tank agitation by the expansion of compressed air in a pachuca tank. Also, manufacturers of circulating tank eductors recommend that the minimum pressure at the nozzle be at least 20 psi greater than the static pressure. This calculation results in 25 GPM of motive fluid with 50 psig pressure at the nozzle and with these conditions the circulating tank eductor will circulate 4 times this quantity or 100 GPM. At the 100 GPM rate a full feed tank will be turned over in approximately 16 minutes. Appendix F is a brochure of a typical, commercially available, circulating tank eductor.

To meet both of the above conditions, a centrifugal pump would have to have a capacity of at least 26 GPM and a discharge pressure of 50 psig. However, to prevent the pump from running out on its performance curve and not developing the discharge pressure required for the feed to the spray calciner, the flow of motive fluid to the circulating tank eductor and pump capacity must be matched. The actual capacity of the eductor selected must be used to determine the pump capacity. Referring to Appendix F, if two 3/8" eductors are selected, they each have a capacity of motive fluid of 16 GPM at 50 psi differential pressure. The total required pump capacity, including 1 GPM for feed to the spray calciner, is then 33 GPM at 50 psi pump discharge pressure.

Bechtel recommends that the following types of centrifugal pumps be considered for this application:

- o Deepwell turbine-type canyon service pumps as described in Chapter XIV, Purex Plant Technical Manual.
- o Cantilever-type centrifugal pump as described in "Transfer of Radioactive Slurries" by C. B. Goodlett, SRL DP-1136.

The Crane Co. Deming Division cantilever type centrifugal pumps have not been recommended as the maximum setting for the pump is 16 3/4" below the tank mounting flange and the pump would require an extension suction pipe. This would create a priming problem whenever the pump suction was not flooded.

Viscosity of the feed has not been defined. However, viscosities up to 25 centipoises do not significantly affect centrifugal pump performance, and a viscosity problem is not anticipated in the proposed service.

10.0 CONTROL SYSTEMS

The control systems for three different feed schemes are shown on Figure 7, 8 and 9. These Piping and Instrument Diagrams are not complete and are only intended to show the controls for the feeds to the spray calciner.

10.1 Figure 7, Industrial and Defense Waste with External-Mix Nozzle

This scheme shows the use of air for tank agitation and a two-stage airlift for delivering the feed to the calciner spray nozzle. The use of an airlift to control flow was discussed in Sections 6 and 7. This figure shows the control of formic acid and frit in direct proportion to the calciner feed rate. The feed rate to the calciner can be the total from the airlifts from both feed tanks as at times one tank will be feeding the calciner and the other tank will be in the process of clean-up and flushing. The feed rate is a function of the quantity of airlift air; while this is not a linear function, it can be defined. The signal from the airlift air flow transmitter is sent to a function amplifier (see Appendix G) whose output signal would be linear with the feed rate to the spray calciner. The output signals from both function relays would be totaled in a summation relay whose output signal then would be linear with total flow. This signal can then be used to reset both the formic acid and frit controls.

Formic acid can be controlled by the use of a variable stroke metering pump as shown on Figures 7, 8 and 9 or by use of a rotometer flow transmitter and control valve as shown on Figures 1, 2 and 3. Bechtel would normally recommend a metering pump for the small flow quantity involved. However, the flow from the metering pump is pulsating, and the effect of these pulsations on the reaction between nitric and formic acids in the short duration they are in contact before the spray nozzle is not known. In this case, Bechtel recommends a metering pump only if it is established that the pulsating flow is acceptable. Either system can be reset from the total feed signal. The frit can be controlled by use of conventional bulk material metering equipment with a remote set point adjustment.

This control system will assure that the minimum quantities of formic acid and frit are being fed to the spray calciner. However, during periods where both tanks are feeding the calciner with one tank in clean-up or flushing, more than the minimum required formic acid and frit will be fed to the calciner. This is not expected to be a problem.

10.2 Figure 8, Industrial Waste with Internal-Mix Nozzle

This scheme shows the use of air for tank agitation and remote head diaphragm pumps for delivering the feed to the spray calciner. The remote head diaphragm pumps meter and control the feed to the spray calciner and would have remote set point adjustment. The set point for the pump can be adjusted by a manual loading station. The output signal from the manual loading station controlling each pump would be totaled in a summation relay whose output signal would then be used to reset both the formic acid and frit control. Control of formic acid and frit would be as previously discussed.

10.3 Figure 9, Defense Waste with Internal-Mix Nozzle

This scheme shows the use of a centrifugal pump for both tank agitation and feed to the spray calciner. It is recommended that this scheme be considered only for Defense waste with a cell environment of 1 to 5×10^4 R/hr due to the limited radiation resistance of the pump motor and lubricants and other electrical components. This scheme shows the flow to the spray calciner determined by a flow-measuring device and controlled by a throttling-control valve.

Magnetic flow meters and rotometers were considered for the flow measuring device. Orifice meters were not considered as the orifice would be too small at the low flow rates (approximately 1 GPM) of the calciner feed system and as they would also have stagnant lines where solids could accumulate.

Magnetic flow meters are available in corrosion and radiation resistant materials; and on special order it is possible to have the electrical insulation equivalent to that of radiation resistant Class H electric motors. It is recommended that a magnetic flow meter be selected on the basis of using the maximum size that will give a reasonable accuracy of $\pm 5\%$. The manufacturers recommend a minimum velocity of 3 feet per second for $\pm 1\%$ accuracy and a minimum velocity of 1 foot per second, for an accuracy of $\pm 5\%$. The increase in the diameter of the bore of the meter will reduce the tendency of solids to plug the meter. A manufacturer's brochure for a magnetic flow-meter is attached in Appendix H.

Rotometers are also available in corrosion and radiation resistant materials. Electronic and pneumatic rotometers have been used in the Purex process and their use is described in the "Purex Plant Technical Manual", Chapter XVII. Solids in the feed stream may be a problem with a rotometer due to the close clearance and high velocity between the float and tapered tube. The nature of the solids in the feed and their effect on a rotometer should be investigated before a rotometer is selected.

The throttling control valve for the feed should be sized on the basis that the pressure drop across the valve is $1/3$ to $1/2$ of the total frictional losses in the system. This includes the feed's pressure drop at the calciner's spray nozzle. This pressure drop and small flow quantity will result in the requirement for a control valve with "Microflo" valve trim. An offset body control valve with bellows seal as manufactured by Hammel Dahl and described in "Purex Plant Technical Manual", Chapter XVII, can be used for Defense plant neutralized waste feed. If the condition should arise where the feed to the calciner is acid, it is questionable if the cold worked 304L stainless steel bellows would resist intergranular corrosion attack. Hammel Dahl has developed a control valve design which has a 304L stainless steel body and a zirconium bellows. This valve design has been used at Allied-General Nuclear Services' Barnwell Fuel Reprocessing Plant and is shown in Appendix I. The valve is assembled without the need for any zirconium to stainless steel welds. The inner valve plug and stem bellows and all parts to which the bellows is welded are zirconium. Control air acts directly on the inside of the bellows; not all air passages are shown on the Appendix I drawing. Note that the control air pressure would have to be greater than the fluid pressure. This valve was used at AGNS in switching service, but the inner valve trim could be redesigned for throttling service.

Formic acid and frit would be controlled as previously discussed. Since the output signal from a magnetic flow meter or rotometer is linear with flow, the signal from each meter would be totaled in a summation relay whose output signal would reset the formic acid and frit control.

11.0 EQUIPMENT NICHE

Figure 9 shows an alternate concept of locating equipment items requiring maintenance, such as flow transmitters, block and check valves, control valves, etc., in an equipment niche. An equipment niche would have full viewing and handling capabilities including, if required, cranes, power manipulators, master-slave manipulators and T.V. It would also be possible to locate a remote head diaphragm pump and pulsation dampener in an equipment niche. An equipment niche with its more specific remote-viewing and handling equipment, located where it is not subjected to the same radiation exposure as the cell, would minimize the time required for remote maintenance and replacement.

APPENDIX C

SPRAY CALCINER VIBRATION ANALYSIS FOR THE
SPRAY CALCINATION/IN-CAN MELTING PROCESS

PACIFIC NORTHWEST LABORATORIES
BATTELLE MEMORIAL INSTITUTE

THE SPRAY CALCINATION/IN-CAN MELTING PROCESS

REVIEW & RECOMMENDATIONS
FOR
SPRAY CALCINER VIBRATION ANALYSIS
TECHNICAL REPORT NO. 1

Prepared by
BECHTEL INCORPORATED
SAN FRANCISCO, CALIFORNIA
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TECHNICAL REPORT NO. 1
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SPRAY CALCINER VIBRATION ANALYSIS

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APPENDICES

<u>Appendix</u>	<u>Description</u>
1	Bechtel Report - "Study Report on the Vibration Analysis and Creep-Fatigue Evaluation of BNW Spray Calciner"
2	Bechtel Report "BNW Vibrator Study - Vibration Test Report"
3.	Referenced Bechtel Interoffice Memoranda

1.0 INTRODUCTION

Battelle Pacific Northwest Laboratories has developed a Spray Calclner/In-Can Melting Process as part of its Waste Fixation Program. This development includes a spray calciner vessel with intermittently operated vibrators for removal of scale build-up on the vessel walls. This report covers Bechtel's review of some of the mechanical design aspects of the Spray Calclner and analysis of the long term effect of the vibrators on the spray calciner vessel due to creep, fatigue and work hardening.

2.0 GENERAL

This report consists of three parts:

- o Partial review of the BNW existing spray calciner design configuration for anticipated operating and maximum conditions to determine if any recommended, and BNW approved, design modifications were to be included in the finite element model used for dynamic analysis. This review is reported in the Mechanical Design Section of this Report.
- o The information on the force time history of the vibrators on the calciner wall was not available from BNW or the vibrator manufacturer. The vibrator is a Cleveland Vibrator Co. Model 1300-VMS modified by extending a shaft on the vibrator and it is mounted in a special spring support designed by BNW. Due to the modifications the vibrator manufacturer was not able to furnish any force-time history data. Bechtel was requested to test a modified vibrator in BNW's special spring support and determine the force-time-history for use in the dynamic analysis. Bechtel's test of the vibrator is described in the Report; "BNW Vibrator Study - Vibration Test Report" attached as Appendix 2.
- o A finite element model was developed for the spray calciner and static and dynamic analyses performed with the vibrator's force-time-history input to the calciner shell. The dynamic responses of the spray calciner were examined and an evaluation made for creep, fatigue and work hardening at critical locations in BNW's spray calciner on the basis that the calciner would operate for 8000 hours per year for a twenty year design life. The development of the spray calciner finite element model, static and dynamic analyses, results and evaluations, are described in Bechtel's Report; "Study Report on the Vibration Analysis and Creep-Fatigue Evaluation of BNW Spray Calciner," attached as Appendix 1.

The above Report also reference the following Bechtel Inter-office Memoranda and these are attached to this report in Appendix 3:

E. L. Gallagher IOM to P. C. Chen, June 14, 1977

E. L. Gallagher IOM to P. C. Chen, July 27, 1977

H. J. Mantel IOM to E. L. Gallagher, August 8, 1977

3.0 CONCLUSIONS & RECOMMENDATIONS

- 3.1 The limited review of the mechanical design which was conducted in this study has determined that the BNW Spray Calciner configuration is adequate, without stiffeners, for a maximum pressure of 1.45 psi (40 inches w.c.), internal or external, and is limited by the calciner shell at 850°C. If the maximum pressure were increased beyond 1.45 psi and the calciner shell wall thickness increased accordingly, the maximum pressure would then be limited at an internal or external pressure of 1.53 psi (42 inches w.c.) by the flat plate surfaces of the discharge cone and filter housing at 500°C. Stiffeners would have to be added to these flat plate surfaces to increase the internal or external maximum pressure beyond 1.53 psi.
 - 3.1.1 On the basis of the above it was agreed with BNW that stiffeners would not be included in the finite element model for the spray calciner.
 - 3.1.2 The maximum internal or external pressure loadings will be determined by the off-gas system. If the BNW design spray calciner is installed with an off-gas system which would permit the internal or external pressure to rise above 1.53 psi during upset conditions, adequate pressure and vacuum relieving systems should be added to limit the pressure to 1.53 psi.
 - 3.1.3 No attempt has been made to embark upon a complete and thorough mechanical design analysis of the BNW spray calciner, or of the design of a generic spray calciner for an industrial or defense facility, as these considerations were beyond the scope of this study.
- 3.2 Evaluations of creep, fatigue and work hardening from the results of static and dynamic analyses of the finite element model of the spray calciner with the defined vibrator force-time history input indicates that the BNW spray calciner assembly can withstand the vibratory loading for a twenty year operating life. The vibrators are not expected to induce excessive stresses in other parts of the assembly such as the filter housing and filter cartridges. The stress levels induced in the spray calciner by the vibrators are not unacceptable but could be further reduced by minor design modifications. It is anticipated that similar vibrator loadings would not create any creep, fatigue or work hardening problems with a similar spray calciner designed for an industrial or defense facility.

- 3.2.1 Although the analysis was made using the properties of the materials at the specified temperatures of 850°C for the calciner shell and 500°C for the discharge cone, filter housing and filter plenum, no thermal stress analysis was conducted as this was beyond the scope of this study. Bechtel's observations on the possible impact of thermal stresses on the results of this study are stated in Sections 3.2.2 and 3.2.3.
- 3.2.2 Thermal stresses are not anticipated to have a major impact on the evaluations made for fatigue and work hardening as thermal stresses are not cyclic (except for start-up and shut-down) and are self limiting. Large thermal stresses are not anticipated in the area of the calciner (near the reinforcing pad for the vibrator) which is critical for the fatigue and work hardening evaluations.
- 3.2.3 Thermal stresses could have an impact on creep evaluations. Thermal stresses are anticipated to be high in the base of the calciner cylinder where there is a transition and temperature gradient from the furnace heated calciner cylinder to the colder base plate. This is also the area of greatest creep damage from dead loads and it is possible that thermal stresses could affect the evaluation made for creep in this area.
- 3.3 Bechtel recommends as good practice that the mechanical design of a spray calciner for an industrial or defense facility be based upon the rules of the ASME Pressure Vessel Code (Section VIII, Division I and Code Case 1592). The maximum temperature of 850°C is beyond the scope of the Code, but the philosophy and rules of the Code can be followed in making the design. If Code Stamping should be required it would be necessary to apply for a special Code Case. All load conditions should be determined and evaluated including all normal and upset pressure and temperature combinations, all static, dynamic, thermal, seismic and vibrator loadings, as well as upset conditions for an overall facility housing a spray calciner cell.
- 3.4 The BNW design configuration is not optimum and it is recommended that for an industrial or defense facility the calciner assembly be modified to smooth geometric discontinuities and constraints and to reduce thermal gradients and resulting stresses. Other minor design features such as design of variable insulation thicknesses to establish acceptable temperature gradients and minimize thermal stresses, and provision of an unheated extension at the base of the calciner cylinder (at the cost of increasing the overall

height of the assembly) are considerations which should be evaluated in a complete design.

- 3.5 Corrosion rates for the spray calciner service are not well known and it is recommended that BNW continue to develop corrosion data as a feature of its spray calciner program.

4.0 MECHANICAL DESIGN

Prior to development of the finite element model and dynamic analysis for the effect of the vibrators, the mechanical design of the spray calciner was reviewed, primarily to determine if stiffening members were required for the anticipated external pressures that a spray calciner could experience in normal or upset conditions. These stiffening members, if required, on the large flat surfaces of the spray calciner, such as the discharge hopper, filter housing and plenum, could impact the finite element model and the dynamic analysis for the effect of the vibrators.

The mechanical design of the spray calciner is best checked by comparing it to the requirements of the ASME Pressure Vessel Code. Normally, Bechtel would recommend that a vessel in this service be designed and fabricated in accordance with the ASME Code for Unfired Pressure Vessels, Section VIII, Division 1. However, the chart for external pressure design in ASME Code Section VIII is limited to 1200°F (649°C), and procedures in ASME Code Case 1592-10 were utilized to check the design for external pressure at temperatures greater than 1200°F.

To check the spray calciner mechanical design the maximum pressures it can withstand were calculated based on the known wall thicknesses, allowable stresses and the maximum temperatures established from a review of the operating conditions reported by BNW. The calciner shell operating temperatures were reported to range from 600 to 800°C and the discharge cone, filter housing and plenum from 300 to 450°C. An increment of 50°C was added to the maximum of these operating temperatures to establish study temperatures, i.e. 850°C for the calciner shell and 500°C for the discharge cone, filter housing and plenum.

Allowable stresses are a function of the material and the design temperature. The allowable stress for type 310 stainless steel for the calciner shell is based on creep and has a relatively low value and was extrapolated beyond the maximum published 1500°F temperature. The allowable stress for type 304 stainless steel for other parts of the spray calciner at 500°C (932°F) is based on yield strength and has a relatively high value.

A joint efficiency of 70% was used for the calciner shell and no corrosion allowance was considered in the wall thickness. The maximum allowable operating pressures for various parts of the spray calciner determined from the above are tabulated below. The lowest of these values, 1.45 psi, establishes the maximum internal and external pressure for the spray calciner as limited by creep of the calciner shell at 850°C for the 20 year life of 8000 operating hours per year.

Calciner Shell @ 850°C: 1.45 psi internal & external pressure
Discharge Cone @ 500°F: 1.53 psi internal & external pressure
Filter Housing @ 500°F: 1.53 psi internal & external pressure
Filter Plenum @ 500°F: 4.41 psi internal & external pressure

The spray calciner is also subject to short term upset conditions and the maximum pressure and temperature for the calciner shell, can be affected by the duration of the short term upset condition as the allowable stress is limited by creep. The discharge cone and filter housing are limited to a maximum internal and external pressure of 1.53 psi and this value is not affected by the duration of the upset condition as it is based on the yield strength at 500°C. The spray calciner should be installed with internal and external pressure relieving systems to limit the pressure to 1.53 psi.

The BNW spray calciner was examined for the following anticipated upset conditions for an industrial or defense facility. The allowable stresses during the upset conditions, for the type 310 stainless steel calciner shell were determined by considering the data in ASME Code Case 1592 for type 304 and 316 stainless steel and extrapolating beyond 1500°F. using a creep limit of 1% for the total duration of the upset condition in the 20 year design life of the spray calciner.

- o Loss of furnace temperature control with calciner shell reaching a maximum temperature of 900°C, and an external pressure of 20 inches w.c. (0.72 psi) for a duration of 100 hours per year.

The spray calciner shell is adequate for these conditions as the actual stress is 54 psi compared to a 1% creep allowable stress of 500 psi and the incremental creep during this condition would be insignificant.

- o Loss of off-gas blower control or blocked feed to the calciner with the calciner shell at 800°C and a maximum external pressure of 200 inches w.c. (7.2 psi) for a duration of 10 hours per year.

The spray calciner shell itself is adequate for these conditions with an actual stress of 540 psi compared to a 1%

creep allowable stress of 4000 psi and incremental creep during this upset would be insignificant. However, the calciner discharge cone, filter housing and plenum would be overstressed at 7.2 psi external pressure and the spray calciner would have to be protected with a vacuum relief system to limit the external pressure to 1.53 psi.

- o Blocked outlet from calciner with calciner shell at a temperature of 850°C for a duration of 10 hours per year.

The internal pressure during this upset condition would have to be limited to 1.53 psi by a pressure relief system to avoid overstressing the discharge cone and filter housing. The calciner shell is adequate for the 1.53 psi internal pressure as the incremental creep during the upset is insignificant. The internal pressure is 20 psi for the 1% creep allowable stress (2000 psi) in the 200 hour upset duration.

The above design reviews were made to establish preliminary basic internal and external allowable pressures for the designated wall thicknesses and to determine if stiffeners were required for the flat plate sections of the assembly. Such items as flanges and nozzles, stresses due to concentrated reactions, thermal and seismic effects and calciner shell distortion due to cyclic thermal operations have not been addressed.

The limited investigation carried out indicated that stiffeners are not required on the existing BNW design spray calciner. The internal and external design pressures are limited to 1.45 psi (40 inches w.c.) by the calciner shell. If the possibility exists that during short term operational upset conditions the internal or external pressure could exceed 1.53 psi the calciner off-gas system should have pressure and vacuum relieving systems to limit the pressures to 1.53 psi. These findings were reviewed with BNW and it was agreed that stiffeners would not be included in the finite element model analysis.

The pressure and temperature conditions established early in the study (in consultation with BNW) for the finite element model dynamic analysis were 0.72 psi external pressure (20 inches w.c.) and 850°C for the calciner shell and 500°C for the discharge cone, filter housing and plenum. The analysis pressure was selected in consideration of the normal operating pressure of approximately 0.36 psi external pressure. The results of the finite element analysis were reviewed in the light of the overall study findings and the selection of a maximum pressure of 1.45 psi. This review showed that the finite element analysis and results would not be significantly affected by the higher pressure, that the conclusions were still valid and that a revised analysis was not justified.

Bechtel's work on this study has suggested a number of problem areas which warrant more detailed consideration in the complete design of a spray calciner assembly for an industrial or defense plant facility, as set out below.

- o Corrosion rates for the spray calciner are not well established. BNW has reported an estimated rate for the Type 310 stainless steel calciner shell of 0.003 ipy. BNW should continue to develop corrosion information for its spray calciner. If it is established that the corrosion rate for Type 310 stainless steel is substantial, wall thickness can be increased or more corrosion resistant and better high temperature property materials, such as Incoloy 800 and 802 and Hastelloy X, should be considered. In any event Bechtel recommends that even with corrosion resistant materials a minimum corrosion allowance of 1/16 inch be used in the mechanical design of a spray calciner.
- o In establishing the design conditions, the following should be considered in addition to the maximum operating pressure and temperature.
 - i. Loss of temperature control on calciner furnace
 - ii. Loss of off-gas blower control
 - iii. Loss of feed to the spray calciner
 - iv. Blockage in off-gas line from the spray calciner
 - v. Impact of a design tornado on both the internal and external pressures
- o Thermal gradients and resulting thermal stresses should be considered. High thermal stresses can be anticipated at the bottom of the calciner shell where it joins the colder support plate at the top of the discharge cone. Distortions in the calciner cylinder due to cyclic thermal operations should also be evaluated.
- o Seismic design should be considered. The spray calciner may not have to be designed for a design base earthquake loading which is specified for safety related components, since failure of the calciner itself may not create a substantial safety hazard as the calciner contents would be contained by the cell and cell floor liner plate. In this event the calciner could be designed for the usual loadings of the Uniform Building Code and its support brackets and the structural support system designed for a design base earthquake loading to assure that the calciner does not fall and damage any items whose failure would be safety related.

Bechtel does not anticipate any unusual design problems for seismic loadings from a design base earthquake. Bechtel's experience for similar vessels and support structures suggest that a design base earthquake will impose a horizontal loading of 0.4g to 0.6g. This loading is more severe than that of the Uniform Building Code and results in heavier support structures and vessel support brackets and reinforcing pads to adequately distribute the seismic support load into the vessel shell.

- o Some of the problem areas noted above can be minimized by revising the BNW spray calciner configuration to minimize discontinuities in the area of the bottom of the calciner shell. It is anticipated that this area will have high stresses from both concentrated support loads and thermal gradients. Changes in the support of the filter housing would also reduce concentrated support loads in the filter housing and discharge cone.

The following list indicates some of the modifications that can be considered. Some of these suggestions will affect overall height and space requirements for the calciner and the flow paths and velocities of the off-gas inside the calciner.

- i. Addition of stiffeners to the flat plate surfaces.
- ii. Change the flat plate supporting the calciner cylinder to a truncated cone or an ellipsoidal or flanged and dished head.
- iii. Develop a separate internal or external support system for the calciner cylinder and connect to the remainder of the calciner with an expansion joint.
- iv. Change single filter housing to multiple smaller housings symmetrically spaced around calciner shell.

Alternate conceptual configurations are illustrated on Figure 1.

Bechtel recommends that the complete design and analysis of all loading effects for a spray calciner for an industrial or defense facility be made on the basis of the ASME Code, Section VIII, Division 1 together with Code Case 1592. Note that these detailed design and analysis considerations have not been made in this study of the BNW spray calciner.

FIGURE 1: SPRAY CALCINER CONCEPTUAL CONFIGURATIONS

LETTER	DWG. #
A	S-953-R-0A
B	S-953-R-0B
C	S-953-R-0C
D	S-953-R-0D
E	S-953-R-0E

CUSTOMER NAME
BATTELLE PACIFIC

TITLE
VIBRATORY HAMMER KNOCKOUT

MODEL No.
VM-1300-AC-HK

DATE
6/17/74

DRAWN BY
AJB

APP. BY
JCL

SCALE
1

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APPENDIX 2

Bechtel Report

BNW Vibrator Study - Vibration Test Report

JOB NO. 12400

BNW VIBRATOR STUDY

VIBRATION TEST REPORT

August 8, 1977

by S.C. Lou

Introduction

In the stress analysis of the calciner vessel under the periodic impact of a vibrator, a force function is required as an input to the computation. Since no such information is available from the vibrator manufacturer, tests were run to obtain approximately the magnitude and periodicity of the force exerted by the vibrator on the vessel.

The vibrator tested was a model 1300-VMS manufactured by Cleveland Vibrator Company. It was mounted on 3/16" ϕ wire coil springs on two 1" ϕ shafts in a box, and a 1-1/2" ϕ ram rod is welded to the floating vibrator. The same entire assembly will be installed on the calciner to shake the calciner inner vessel.

Description of Test

A 12" x 4" x 3/8" target plate was attached to the vibrator assembly by 4-long, 1/2" ϕ threaded rods to simulate the calciner inner vessel. The location of this target plate with respect to the ram rod was varied from the initially barely touching position to clearances of + 1/16", - 1/16", - 1/8" and - 1/4", the negative clearances meaning precompression between the plate and the end of the ram rod.

The vibrator assembly was clamped down to a stand with sufficient mass to limit the amplitude of vibration. Accelerometers were attached to the target plate and to the vibrator proper. One accelerometer on the plate was connected to the integrator on a sound level meter to measure displacement. The other accelerometer on the plate and one on the vibrator were connected to two channels of a tape recorder. They measure the acceleration at these two locations.

Nitrogen was used to excite the vibrator. Tests were run for different positions of the target plate with the gas at pressures of 25, 50, 75 and 100 psig. For each pressure, a no load test was also run with the target plate backed away from the vibrator so that there was never contact between the ram rod and the plate during the run.

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When the vibrator was pressurized, the ram rod hit the plate whose displacement was displayed on the spectroscope and recorded by the X-Y plotter. This displacement consisted of bending of the plate, extension of the threaded rods and rotation of the vibrator and stand on the rubber mat. The displacement was also obtained by playing back the tape of recorded acceleration through the integrator on the sound level meter, the spectroscope, and an oscilloscope in the Acoustical Analysis Center.

The relation of force on the target plate and its displacement was determined by measuring the total deflection (bending + extension + rotation) when a known force is applied at the center of the plate. This spring constant was found to be 50 lb. per 0.04" or 1,250 lb./in.

The vibrator proper weighs 20 lbs. The assembly, including the box, the target plate and the threaded rods, weighs 112 lbs. The complete system, including clamps, clamping beams, H-block, etc., weighs 754 lbs.

Drawing BNW-VT-1 shows the experimental set-up and the instrumentation used.

Results

A typical displacement chart is shown in Figure BNW-VT-2, and an acceleration chart in Figure BNW-VT-3. These are all plotted against frequency. A displacement vs. time chart obtained from the acceleration tape is reproduced in Figure BNW-VT-4. From the last, a general pattern of the force is discernible.

This consists of a repetition of four cycles as shown in the rectangular frame during each of which the plate was hit by the vibrator rod twice and then vibrated for the remainder of the cycle. The magnitude of the forces is determined from the spring constant of the system.

Since the test conditions for the run represented by the attached displacement-time chart were considered very close to the actual operating conditions at the calciner vessel, the force function used for the stress analysis was based on this run. For simplicity, the force function was presented as follows:

Time, Sec	Force, lbs.	Time, Sec.	Force lbs
0	0	0.185	100
0.005	220	0.19	0
0.01	0	0.24	0
0.02	0	0.245	100
0.025	320	0.25	0
0.03	0	0.26	0
0.08	0	0.265	220
0.085	160	0.27	0
0.09	0	0.32	0
0.10	0		
0.105	220		
0.11	0		
0.16	0		
0.165	80		
0.17	0		
0.18	0		

SUMMARY OF VIBRATOR OPERATION

1. Spray Calciner operates 8000 hours per year
2. Vibrators operate for a 3 second duration every 3 minutes
3. Two vibrators on Calciner, 180° apart, alternate in operating every 3 minutes

Above cycle repeated from time 0.32 sec. on

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FORM NO. A-1

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No.	DATE	REVISIONS				BY	CHK	APPR
ORIGIN		BATTTELLE N-W LABS CALCINER VIBRATOR TEST				JOB No. 12400		
						SPEC/DES. GUIDE No		REV.
						BNW-VT-1		0

SHEET

6

DISPLACEMENT, IN.

Run no. 30

Accelerometer on Target Plate

Target Plate Location: $-\frac{1}{8}$ "

Gas Pressure: 100 psig

FREQUENCY, HZ

Fig BNW-VT-2

Fig. BNW-VT-3

Run no. 20

Accelerometer on Target Plate

Target Plate Location: $+\frac{1}{16}$ "

Gas Pressure: 75 psig

Scales: $X: \frac{1}{8}" = 0.01 \text{ sec.}$; $Y: 1" = 0.3"$

Fig. BNW-VT-4 Displacement - Time Chart

APPENDIX D

SPRAY CALCINER STRUCTURAL ANALYSIS

SPRAY CALCINER STRUCTURAL ANALYSIS

J. A. Griffiths, R. J. Shippell, C. A. Williams

GENERAL INTRODUCTION

Purpose and Scope

This section describes selected SC/ICM system structural analyses to help assess the equipment service life and structural integrity. Specific structural analyses highlight key stress areas in the SC or ICM equipment. Stresses were estimated for loading situations covering normal, upset, and emergency conditions. Loading conditions selected for analyses are generic in nature.

Normal operating conditions consider deadweight loading (1-g vertical loading), stresses due to internal pressure or vacuum, vibratory dynamic loading, and thermal stresses due to normal operating temperatures. Upset conditions are those conditions that include additional loading due to equipment malfunction with a moderate frequency of occurrence. Emergency conditions are more severe additional loadings, which have a low probability of occurrence. Roughly speaking, these three types of loadings correspond to the ASME Boiler and Pressure Vessel Code (ASME 1977) Level A, B and Levels C and D combined service loadings, respectively. The ASME Code does not strictly apply to these structures. Table D.1 provides the assumed loading conditions.

Three possible modes of structural failure are addressed: 1) excessive single-application stresses resulting in catastrophic structural failure or excessive deformation; 2) stresses exceeding cyclic-stress allowable limits, resulting in structural fatigue failure; and 3) stresses exceeding allowable creep stresses resulting in excessive deformation or creep-fatigue interaction failure.

Detailed designing and final design stresses are beyond the scope of this study. Basic design features are considered in the analysis. Some suggestions for detailing the final design configuration are provided later in this section based on normal design practices and experiences with operation of a prototype SC/ICM system.

General Analytic Approach

Finite-element model methods were used extensively for the structural analyses of the SC/ICM system. Where simple geometry permitted, closed-form analytic models were also used.

ANSYS (Swanson 1978), the computer code used predominately in the study, is a general purpose code based on a wavefront solution technique. ANSYS has both linear and nonlinear material behavior capabilities. ANSYS has both static and dynamic analysis provisions. In dynamic analysis, either response spectrum techniques based on structural modal participation analysis or direct time integration of the motion equations are available.

TABLE D.1. Assumed Loading Conditions for SC/ICM System

Component	Normal	Upset	Emergency
Calciner Assembly: Calciner Chamber, Cone, Filter Chamber	$\Delta P_{ext} = 2480 \text{ Pa}$ (0.36 psi) 1-g weight loading Periodic vibratory impact from 2 vibrators to remove calcine from walls Thermal stresses; maximum $T = 800^\circ\text{C}$	1) loss of furnace control-furnace power increases from 50-70 kW per zone to 150 kW per zone, maximum T increases to 900°C ; ΔP_{ext} increases to 4960 Pa (0.72 psi) 2) loss of filter chamber blower control or blocked feed inlet, vacuum relief system operates; ΔP_{ext} increases to 4960 Pa (0.72 psi) 3) blocked cone outlet, pressure relief system operates, maximum $\Delta P_{int} = 10,300 \text{ Pa}$ (1.5 psi) 4) Operating-Basis Earthquake (OBE)(a)	1) loss of filter chamber blower control or blocked feed inlet and loss of vacuum relief system; ΔP_{ext} increases to $4.96 \times 10^{-2} \text{ MPa}$ (7.2 psi) 2) blocked cone outlet and loss of pressure relief system; maximum $\Delta P_{int} = 0.138 \text{ MPa}$ (20 psi) 3) tornado depressurizes off gas duct; maximum $\Delta P_{ext} = 20,600 \text{ Pa}$ (3 psi), drop at 13,800 Pa/sec (2 psi/s) 4) tornado depressurization of hot cell; maximum $\Delta P_{ext} = 20,600 \text{ Pa}$ (3 psi), drop at 13,800 Pa/sec (2 psi/s) 5) Safe-Shutdown Earthquake (SSE)(a)
Calciner Assembly Support Structure	1-g weight loading	1) Operating-Basis Earthquake (OBE)(a)	1) Safe-Shutdown Earthquake (SSE)(a)
Piping/Jumpers	Internal pressure End displacements due to thermal expansion of calciner assembly 1-g weight loading of piping/jumpers plus contents	1) Operating-Basis Earthquake (OBE)(a)	1) Safe-Shutdown Earthquake (SSE)(a)
In-Can Melters	Thermal stresses* 1-g weight loading	1) Loss of furnace control 2) Operating-Basis Earthquake (OBE)(a)	1) Safe-Shutdown Earthquake (SSE)(a)

(a) Earthquake Loading - Two loading conditions were analyzed. First, pseudo-static loading was used with a maximum horizontal acceleration of 1.0 g and a vertical acceleration of 0.67 g. Second, a scaled El Centro-1940 earthquake response spectrum was used with a maximum horizontal ground velocity of 0.41 m/s (16 in./s), and a maximum vertical ground velocity of 0.27 m/s (10.7 in./sec). See text for details.

STRUCTURAL ANALYSES OF THE SPRAY CALCINER ASSEMBLY

Spray Calciner Assembly Model for Static and Seismic Loading Conditions

Introduction

A 3-D finite-element model of the spray calciner assembly is used to predict structural vibration modes shapes, associated natural frequencies, and corresponding seismic response. Static load cases considered include dead-weight loading and pressurization/vacuum under various calciner operating conditions. This section contains detailed model descriptions, engineering assumptions, and finite-element study results.

General Assumptions

General assumptions used in formulating the spray calciner assembly model are presented here. Other more detailed assumptions are discussed where appropriate along with the model detailing.

An approximate temperature distribution is applied to the calciner assembly finite-element model. Although the thermal stresses are addressed by a separate analysis, a simplified temperature distribution was used in this analysis to assign the appropriate temperature-dependent material properties. The generic material properties are taken from the ASME High-Temperature Code Case N47 (ASME 1977) and the Aerospace Material Handbook (DOD 1978).

Effects of pipe lines and other connectors to the calciner and filter chamber were modeled as lumped masses. Inertia contributions were present while the added support from the connectors was neglected. Both assumptions are considered conservative with respect to the overall structural response. Insulation on the filter chamber and cone was ignored; it provides no stiffness and little additional mass.

Finite-Element Model

The spray calciner system was modeled as an assemblage of 3-D elastic beam, shell, and generalized mass elements. The 3-D beam element is a uniaxial element with tension-compression, torsion, and bending characteristics. A 3-D shell element was the second type of element used in this analysis. This element is an elastic, flat, triangular element with both bending and membrane characteristics. The last element type used in this model was the generalized mass element, which is a point element having six degrees of freedom such as used for the above elements. A different mass and rotary inertia may be assigned to each coordinate direction or a mass matrix may be defined. The finite element model has 81 nodes and is made up of 62 beams, 84 plates, and 11 lumped masses.

The entire spray calciner system is shown in Figure D.1. The calciner frame, chamber, and filter chamber were represented by 3-D elastic beams. The frame is made up of 51 beam elements. The beam elements are "sized" to the appropriate geometry and material. General structural steel mechanical properties were taken from the ASME Code, Section VIII, Division II (ASME 1977). The calciner frame is connected to the foundation at the floor and wall. The frame was treated as a fixed connection at the floor (i.e., it was assumed that the floor bolts would restrict all translations and rotations). Results from subsequent analyses proved the validity of using this approach. The frame is connected to the wall via mechanical trunnions. Boundary conditions at the wall-frame interface allow vertical structural translation only. Model translations in the nodal x and y directions and rotations about the nodal x, y, and z axes were zeroed out. General vertical translation was allowed. This neglects the non-linear gap effect of possibly bottoming the structure against the trunnion.

Offsets in support-assembly beam elastic axes were ignored in this model. This effect would tend to couple bending and torsional deformations of the various assembly elements. The final geometry of the support assembly connections is subject to equipment detailing, so this effect was ignored for this generic structural modeling.

FIGURE D.1. Finite-Element Idealization of Spray Calciner System - Normal Orthographic and Isometric

Equivalent beam systems were used to model the spray calciner chamber and filter chamber. The spray calciner chamber and filter chamber were modeled using four and six beam elements, respectively. Actual inertia properties and densities were input to maintain proper mass distributions. The spray calciner chamber was of circular cross section with an outer diameter of 0.91 m (36 in.) a height of 3.3 m (130 in.), and a wall thickness of 9.5 mm (0.375 in.). The filter chamber was of rectangular cross section with nominal measurements of 1.78 m x 0.91 m x 6.35 mm (70 in. x 36 in. x 0.25 in.). Material properties for the Inconel-601 spray calciner chamber were taken from the Alloy Digest (1970). Due to the relatively rigid nature of the spray calciner chamber's response to the loading conditions, a 310 stainless steel spray calciner chamber would experience stresses similar to those the Inconel-601 chamber analyzed even though the two material properties differ somewhat. The filter chamber was a typical 304 stainless steel. These mechanical properties were taken from ASME (1977).

The interfaces of the spray calciner chamber and filter chamber with the horizontal base plate were modeled using rigid regions approximating the actual geometry of the spray calciner chamber and filter chamber. In this manner, the interface geometry with the base plate is realistic. This approach reduces modeling time at the cost of sacrificing chamber and filter chamber ovality mode deformation. Separate analyses (Bechtel, Inc. 1977b) have indicated that the frequencies of these ovality modes are beyond the seismic range (above 100 Hz); this assumption will therefore have little impact upon the overall structures' seismic response. The upper attached piping or braces to the spray calciner chamber and filter chamber were assumed to be sufficiently stiff to prevent any end spray calciner chamber or filter chamber translations except for vertical motion. Reaction forces in the braces proved to be sufficiently low to bear out this model. A worst-case study with free ends was also considered for seismic response.

The calciner base plate and cone section are represented by 3-D shell elements. The 2.54-cm (1-in.)-thick base plate model was comprised of 48 triangular shell elements. The plate used in the analyses was a 2.0 m x 1.2 m x 25.4 mm (80 in. x 48 in. x 1 in.) piece of 304 stainless steel. Mechanical properties for this material were taken from ASME (1977).

Two simplifying approximations were made in modeling the plate-frame interface. The first ignored the noncoincidence of the centroids of the shell and beam elements. This type of detailed study is most appropriately done following final structural detailing. The second approximation is matching the six degrees of freedom at the plate-frame junction. This approach simulates a welded interface while the actual structure should be bolted to permit thermal expansion of the baseplate relative to the colder frame. The number of bolts and bolting pattern can be chosen to limit edge rotation of the plate and approach the welded condition, thereby reducing the error involved in this assumption. This again must be determined at final structural detailing.

The cone section was modeled using thirty-six 6.35-mm (0.25-in.)-thick triangular shell elements. The model of the cone had an elliptical cross section with major and minor diameters of approximately 1.5 m (59 in.) and 0.86 m (34 in.). The sides of the cone taper to a point at the bottom. The cone section material was 304 stainless steel.

A conservative boundary condition was applied to the bottom of the cone section, i.e., that point was given full translation and rotational degrees of freedom. The diverter assembly bellows is considerably less stiff than the cone itself and therefore would afford no support of the cone section during an earthquake.

The furnace, filters, and external pipes were modeled as generalized mass elements. In all cases point masses were placed so as to produce near actual weight and mass distributions. An equivalent furnace mass was located around the perimeter of the chamber's rigid region. Due to the furnace "T" bar connection at the wall, furnace masses were constrained to have no lateral translation. Inherent in this model is the assumption that the relative motion between the furnace and plate would not overcome static, restraining frictional forces. Similarly, equivalent pipe weights were specified at the top of the spray calciner chamber assembly

and the top of the filter chamber. The filter masses were distributed through the filter chamber and across the length of the filters in a linear fashion.

Load Conditions for Analysis

The spray calciner assembly was analyzed for both static and dynamic loading conditions. The two static load conditions were gravity and pressure. Dynamic loads included seismic loading modeled by either the El Centro earthquake or any assumed pseudo-static loading. The following sections contain descriptions of various loading conditions and assumptions and idealizations for each. It is believed that the loading schemes selected result in a conservative estimate of the calciner's response. Refining the loading functions to reduce the uncertainties associated with the assumptions is not expected to substantially change the conclusions. Such refinement must be based on site-specific data.

Gravity Loads. Structural dead-weight gravity loads due to static body forces were computed from the mass density of the element materials and the element geometry. Lumped masses were used to account for attached equipment weights.

Pressure Loads. Pressure Loads postulated for calciner normal, upset and emergency operating conditions are described in Table D.1. Normal operating pressure was 2480 Pa (0.36 psi) vacuum. Upset pressures or vacuums can be generated from several fault paths. An upset vacuum of 4960 Pa (0.72 psi) can result from loss of the filter chamber blower control or a blocked feed inlet with the vacuum relief system operational. An internal upset pressure of 10,300 Pa (1.5 psi) can be encountered if the cone section becomes blocked to a degree that would cause the pressure relief system to operate. An emergency condition is encountered with the loss of the vacuum relief system in conjunction with either the loss of the filter chamber blower control or the blockage of a feed inlet. Either double fault condition could produce a vacuum of 4.96×10^{-2} MPa (7.2 psi). If the cone section outlet becomes blocked and the pressure relief system is lost, an internal pressure of 0.138 MPa (20 psi) will result, causing an emergency operating condition.

In summary, the pressures under consideration were: a normal operating condition vacuum of 2480 Pa (0.36 psi); upset conditions of vacuum 4960 Pa (0.72 psi) and 10,300 Pa (1.5 psi) internal pressure; and emergency conditions of 4.96×10^{-2} MPa (7.2 psi) vacuum and 0.138 MPa (20 psi) internal pressure.

Seismic Loading. Two methods were used to simulate seismic motion. First, for the case where the spray calciner chamber and filter chamber upper ends were considered constrained by attached piping and braces, lowest structural natural frequencies were sufficiently high to allow pseudo-static seismic loading. In this case a 1-g maximum horizontal acceleration was assumed for both horizontal directions and a 0.67-g maximum vertical acceleration was also supplied.

To check the sensitivity of the model to the spray calciner chamber and filter chamber constraints at their upper ends, the model was also run with these degrees of freedom unconstrained. Due to the reduced structural natural frequencies, an earthquake response spectrum approach was used. The El Centro earthquake was chosen and its velocity response spectrum

used (Harris and Crede 1976) in Figure D.2. A maximum horizontal ground velocity of 0.27 m/s (16 in./s) was assumed. No floor-response filtering was assumed, since this is highly site-dependent. This roughly corresponds to a 0.33-g maximum horizontal acceleration. Again, the vertical component was assumed to be two-thirds of the horizontal components.

Thermal Loads. An approximate temperature distribution was applied to the finite-element model. These temperatures were used in assigning the appropriate temperature-dependent material properties. Thermal stresses were not calculated using this model. Detailed internal stresses are calculated using a separate model. Due to the design of the structure, any thermal stresses are highly localized phenomena.

Results of Analyses

Study results are separated into a discussion of the static and dynamic analyses. The static load cases included dead-weight loading and dead-weight loading combined with system pressures of 2480 Pa, 4960 Pa and 4.96×10^{-2} MPa (0.36, 0.72 and 7.2 psi) vacuum and 10,300 Pa and 0.138 MPa (1.5 and 20 psi) internal pressure. Mode-shape predictions and seismic responses were studied under the dynamic section. Peak stresses and displacements for the various calciner components were noted for all load cases. Frame-foundation reaction forces were observed during the seismic study.

Static. Static load case results are summarized in Table D.2. The maximum stresses and displacements were identified for each load case. The maximum system vacuum of 4.96×10^{-2} MPa (7.2 psi) occurs during upset operation. This condition would cause a system peak stress of approximately 83 MPa (12,000 psi) to occur in the cone section. At this level of loading all members remain elastic. The maximum system pressure of 0.138 MPa (20 psi) occurs

FIGURE D.2. E1 Centro Earthquake Nondimensional Response Spectrum
Taken from Harris and Crede (1976)

TABLE D.2. Results from Static Analysis: Maximum Displacements and Stresses Corresponding to Various Combinations of 1-g_z Loading With Different Calciner Operating Pressures

Maximum Stresses and Displacements	Static Load Cases(a)					
	Deadweight ($\approx 1 \text{ g}_z$)	1 g _z + 2480 Pa ^{VAC} (1 g _z + 0.36 psi ^{VAC})	1 g _z + 4960 Pa ^{VAC} (1 g _z + 0.72 psi ^{VAC})	1 g _z + 10300 Pa ^{INT} (1 g _z + 1.5 psi ^{INT})	1 g _z + 4.96 x 10 ⁻² MPa ^{VAC} (1 g _z + 7.2 psi ^{VAC})	1 g _z + 0.138 MPa ^{INT} (1 g _z + 20 psi ^{INT})
Maximum x-displacement(b)	-0.20 (-0.77 x 10 ⁻²)	-0.24 (-0.95 x 10 ⁻²)	-0.51 (-0.20 x 10 ⁻¹)	1.09 (0.43 x 10 ⁻¹)	-5.08 (-0.20)	14.22 (0.50)
Location	SC Chamber	Cone	Cone	Cone	Cone	Cone
Maximum y-displacement	-0.08 (-0.31 x 10 ⁻²)	-0.10 (-0.40 x 10 ⁻²)	0.10 (0.40 x 10 ⁻²)	-0.30 (-0.12 x 10 ⁻¹)	1.27 (0.50 x 10 ⁻¹)	-3.56 (-0.14)
Location	Frame	Frame	Cone	Cone	Cone	Cone
Maximum z-displacement	-0.26 (-0.101 x 10 ⁻¹)	-0.20 (-0.80 x 10 ⁻²)	-0.20 (-0.80 x 10 ⁻²)	-0.69 (-0.27 x 10 ⁻¹)	2.31 (0.91 x 10 ⁻¹)	-7.11 (-0.28)
Location	Plate	SC Chamber	Cone	Cone	Cone	Cone
Maximum Frame Stress(c)	-9.00/-2.07 (-1300/-300)	-8.27/-2.55 (-1200/-370)	-8.27/-2.55 (-1200/-370)	-8.07/-2.48 (-1170/-360)	-8.72/-2.69 (-1265/-390)	-6.72/-2.07 (-975/-300)
Maximum SC Chamber Stress(c)	-0.31/-0.31 (-45/-45)	-0.41/-0.34 (-60/-50)	-0.45/-0.41 (-65/-60)	-0.10/-0.10 (-15/-15)	-1.48/-1.38 (-215/-200)	-2.90/-2.90 (-420/420)
Maximum Filter Chamber Stress(c)	-0.55/-0.52 (-80/-75)	-0.69/-0.59 (-100/-85)	-0.76/-0.62 (-110/-90)	-0.55/-0.41 (-80/-60)	-1.34/-1.24 (-195/-180)	1.93/1.90 (280/275)
Maximum Plate Stress	-8.55 (-1240)	-6.79 (-985)	-6.83 (-990)	-6.65 (-965)	7.48 (1085)	18.62 (2700)
Maximum Cone Stress	2.34 (340)	3.25 (470)	-8.00 (-1160)	-18.00 (-2610)	83.43 (12100)	234 (33870)

(a) Positive stresses are tension; negative stresses are compression.

(b) All units mm or MPa except within parentheses, where units become in. and psi.

(c) (Maximum Inplane Stress/Maximum Axial Stress)

during a possible emergency operation condition. Peak stresses again were observed to appear in the cone. Elastic theory predicts a stress level of about 234 MPa (34,000 psi). This would result in a localized yielding and stress relief effect and is not expected to affect the overall integrity of the components analyzed. Such a plastic deformation would probably be acceptable for the emergency shutdown loading case.

A static elastic buckling external pressure has been calculated for the calciner cone based on analogy with an equivalent cylinder (Roark and Young 1975, Table 35, Number 23). Based on the major diameter of the cone and its maximum slant height, a buckling pressure of 0.34 MPa (49 psi) is predicted for cone-operating temperatures of about 260°C (500°F). The maximum emergency-condition vacuum is 4.96×10^{-2} MPa (7.2 psi), well below the critical buckling pressure. Thus, the cone should not buckle under emergency loading conditions.

Dynamic

Mode Frequencies. Mode shapes and natural frequencies were predicted for two cases: a more realistic situation in which the tops of the spray calciner chamber and filter chamber were assumed to be constrained laterally (due to stiff piping attachments or seismic bracing); and a worst-case study that freed all six degrees of freedom at the ends.

A summary of the mode-frequency analysis for both cases is presented in Table D.3 below.

Typically, the cutoff frequency for seismic amplification effects is considered to be 33 Hz. The lowest natural frequency for the fixed-end condition is above this frequency. The free-end condition has four modes that can resonate to some extent during an earthquake.

The mode shapes for the first four modes of the free-end case are shown in Figures D.3 through D.6. The first mode shape indicated a y-z plane in phase rocking of the spray calciner chamber and filter chamber. The second mode showed similar motion, but a change in the plane of action from y-z to the x-z plane was noticed. Out-of-phase x-z rocking was observed in the third mode. The fourth mode indicated similar effects, but the plane was shifted from the x-z to the y-z.

The first mode for the fixed-end case (which occurs outside the seismic range) was a frame oscillatory mode. Except for vertical translation, the spray calciner chamber and filter chamber remains stationary for this mode, while the frame and legs move (Figure D.7).

Seismic Response. Fixed-end and free-end conditions of the spray calciner chamber and filter chamber required separate seismic studies. Since the fixed-end model's first mode

TABLE D.3. Mode-Frequency Analysis for Fixed and Free Cases

SC Chamber and Filter Chamber End Conditions	Natural Frequencies, Hz				
	Mode 1	Mode 2	Mode 3	Mode 4	Mode 5
Fixed	36	47	54	56	61
Free	15	19	29	33	39

FIGURE D.3. First Mode Shape at a Frequency of 15 Hz. (In-phase rocking of calciner chamber and filter chamber in y-z plane excited by El Centro Spectrum.)

was above the usual cutoff for spectrum analyses, a pseudo-static approach to the earthquake response was applied. Three static-load cases were studied: a 1-g loading in the global x direction, a 1-g loading in the global y direction, and a 2/3-g loading in the global z direction.

The outputs from the individual cases were summed using the Absolute Sum response-spectrum technique. The results from this procedure are summarized in the Tables D.4, D.5 and D.6.

This technique sums the results in a worst-case fashion. Examination of the results indicates a low (elastic) stress level in all components analyzed.

The free-end model had four modes that could possibly be excited by an earthquake. In this case, due to the lack of site-specific seismic data, a scaled El Centro earthquake velocity-response spectrum was applied to the structure. Two percent of critical damping was assumed.

The contributions of the various modes were summed using the Absolute Sum response spectrum technique. The results from this procedure are presented in Tables D.7 through D.9.

FIGURE D.4. Second Mode Shape at a Frequency of 19 Hz. (In-phase rocking of calciner chamber and filter chamber in x-z plane excited by El Centro Spectrum.)

Although the displacements' stresses and reaction forces were increased by the free-end assumption, the level of excitation still appears to lie well within the elastic design region.

Conclusions

The overall structural response generally indicated low (elastic) stress levels for all load cases considered. Some localized plastic stresses, however, would be developed in the cone section during emergency operation. Such localized yielding effects are anticipated to be acceptable for emergency conditions. However, some form of external bracing (Bechtel, Inc. 1977b) might be used to alleviate plastic deformation under emergency conditions.

Thermal Stresses in the Calciner Chamber/Base Plate Junction and Central Calciner Chamber

Introduction

The calciner chamber is heated up to a design temperature of 800°C. Actual operating temperatures may be less than this design temperature, depending on process conditions. The chamber is heated by a four-zone furnace, with the lowest several centimeters of the furnace having no active heating elements. In addition, most of the spray calciner support frame and chamber base plate are not directly heated by the furnace. Thus, a large thermal gradient

FIGURE D.5. Third Mode Shape at a Frequency of 29 Hz. (Out-of-phase rocking of calciner chamber and filter chamber in x-z plane excited by El Centro Spectrum.)

exists in the lower portion of the calciner chamber. Expansion of the lower edge of the chamber is constrained by the relatively cooler base plate. The thermal stresses at the spray calciner chamber/base plate junction are limiting stresses in the spray calciner design. A separate ANSYS finite-element model was used to estimate these stresses. Thermal stresses in the central spray calciner chamber due to radial thermal gradients are also significant and are discussed separately below. No other large thermal gradients exist in the assembly, so thermal stresses are not significant elsewhere.

Modeling Considerations

An axisymmetric model of the spray calciner chamber/base plate junction was used. Although this is not strictly representative of the actual geometry, the local nature of the thermal stresses at the junction makes this a good approximation. A total of 60 linear, elastic, 2-D, isoparametric, solid finite elements were used in the model. Figure D.8 shows the model geometry. Stainless steel alloy 304 material properties based on ASME Code (ASME 1977) design values were used for the 304L base-plate elements. Inconel-601 alloy was used for the

FIGURE D.6. Fourth Mode Shape at a Frequency of 33 Hz. (Out-of-phase rocking of calciner chamber and filter chamber in y-z plane excited by El Centro Spectrum.)

chamber. The Inconel-601 material properties were taken from Alloy Digest (1970) and the Aerospace Structural Metals Handbook (DOD 1978).

Due to limited material data, a classical bilinear stress-strain curve model was used for the Inconel-601 material with kinematic hardening. Temperature-dependent Young's Moduli were used. A more general, piecewise linear stress-strain curve was used for 304 stainless steel based on ASME Code Case N47 (ASME 1977) material hot tensile isochronous stress-strain curves.

The temperature distribution used for the analysis was based on actual spray calciner chamber temperatures measured by a series of thermocouples located near the base of a prototype spray calciner chamber during processing of nonradioactive, simulated waste. Temperatures for this run were somewhat lower than the design temperature. The actual high temperature in the lowest furnace zone for this run was 695°C. Of primary interest at the spray calciner chamber/base plate junction is the axial thermal gradient along the spray calciner chamber. The experimentally measured thermal gradient was linear over the lower 11.4 cm (4.5 in.) of the chamber. For analysis purposes, a linear axial thermal gradient was

FIGURE D.7. First Mode Shape With Laterally Constrained Calciner Barrel and Filter Chamber Ends at a Frequency of 36 Hz. (Frame oscillatory mode excited by pseudo static spectrum technique.)

assumed with the base plate at 510°C and the upper chamber at 695°C . Stresses due to radial thermal gradients in the central chamber are discussed separately.

It was further assumed that the base plate was mounted on the support structure in a manner that allows free outward expansion of the plate for the base plate temperature rise from 20° to 510°C . This can be accomplished by bolting the plate to the support structure with oversized bolt holes to allow for thermal expansion. If the plate is welded to the frame, additional compressive stresses arise in the plate due to the restraint in "radial" thermal expansion.

The model was analyzed for a four-step process as follows:

- a) The initial structure is at ambient temperature (20°C) and is initially stress-free.
- b) The structure is heated to the 510°C to 695°C temperature range, described above, resulting in some plastic deformation of the lowest region of the chamber.

TABLE D.4. Maximum Spray Calciner Component Displacement for Seismic Response (Fixed End)

Maximum Displacement	mm (in.)		
	Maximum x Displacement	Maximum y Displacement	Maximum z Displacement
Magnitude	0.24 (0.93 x 10 ⁻²)	0.24 (0.96 x 10 ⁻²)	0.16 (0.64 x 10 ⁻²)
Location	Frame	Frame	Plate

TABLE D.5. Maximum Spray Calciner Component Stress for Seismic Response (Fixed End)

Maximum Stress	Maximum Component Stress, MPa (psi)				
Magnitude(a)	5.58/2.21 (810/320)	0.66/0.21 (95/30)	6.31/0.34 (915/50)	6.52 (945)	2.38 (345)
Location	Frame	SC Chamber	Filter Chamber	Plate	Cone

(a) Maximum inplane stress (maximum axial stress)

TABLE D.6. Maximum Spray Calciner Component Reaction Forces for Seismic Response (Fixed End)

Maximum Reaction Forces	Maximum Reaction Forces, kN (lbf)		
	F _x	F _y	F _z
Magnitude	32.5 (7315)	18.8 (4230)	14.5 (3250)
Location	Frame	Frame	Frame

TABLE D.7. Maximum Spray Calciner Component Displacement for Seismic Response (Free End)

Maximum Displacement	mm (in.)		
	Maximum x Displacement	Maximum y Displacement	Maximum z Displacement
Magnitude	2.79 (0.11)	4.06 (0.16)	1.02 (0.4 x 10 ⁻¹)
Location	SC Chamber	SC Chamber	Plate

TABLE D.8. Maximum Spray Calciner Component Stress for Seismic Response (Free End)

Maximum Stress	Maximum Component Stress, MPa (psi)				
Magnitude(a)	16.38/9.38 (2375/1360)	8.90/0 (3500/30)	2455/0.21 (5760)	39.72 (1720)	11.86
Location	Frame	SC Chamber	Filter Chamber	Plate	Cone

TABLE D.9. Maximum Spray Calciner Component Reaction Forces for Seismic Response (Free End)

Maximum Reaction Forces	Maximum Reaction Forces, kN (lbf)		
	F_x	F_y	F_z
Magnitude	50.6 (11375)	23.4 (5270)	59.5 (13385)
Location	Frame	Frame	Frame

FIGURE D.8. Model Geometry for Spray Calciner Chamber/Base Plate Junction Thermal Stresses

- c) The structure is then cooled to the ambient (20°C) condition and residual stresses are calculated.
- d) The structure is then reheated to the 510° to 695°C condition. The strain range between the last two steps was used in the fatigue analysis.

This multi-step model was used to account for initial elastic shakedown due to material work hardening.

Predicted Stresses and Deformations

Stresses and deformations were predicted for the series of load cases previously described. Figures D.9 and D.10 show the distorted geometry following the second (cooldown) and third (reheat) load steps, respectively.

Note that both figures show exaggerated distortions with different magnification factors for the displacements. Figure D.19 shows the permanent set deformation due to plastic deformation in the lower spray calciner chamber on initial heating. The local restraint in spray calciner chamber expansion due to the cooler base plate is evident in Figure D.10. The maximum radial displacement at the outer base plate edge is 0.56 cm (0.221 in.) at temperature.

Figures D.11 through D.14 show the normal stress components and the stress intensity distribution at temperature for the region nearest the spray calciner chamber/base plate junction, where thermal stresses are the highest. The Inconel-601 chamber has been subjected to plastic deformation; the 304 stainless steel base plate remains elastic.

FIGURE D.9. Distorted Geometry of Spray Calciner Chamber/Base Plate Junction Following Cooldown

FIGURE D.10. Distorted Geometry of Spray Calciner Chamber/Base Plate Junction Following Reheating

FIGURE D.11. Junction Radial Stress Distribution at Temperature

Figures D.15 through D.18 show the residual normal stress components and the stress intensity distribution when the junction is cooled. Residual stresses in the spray calciner chamber are significantly higher than those in the plate, but all stresses remain elastic.

Creep-Fatigue Interaction Associated With Thermal Stresses

Two possible failure modes for the spray calciner chamber/base plate junction are creep and fatigue damage. The ASME elevated temperature Code Case N47 (ASME 1977) provides rules

FIGURE D.12. Junction Axial Stress Distribution at Temperature

FIGURE D.13. Junction Hoop Stress Distribution at Temperature

for assessing the combined effects of creep and fatigue. A combined usage factor approach is used. Although the Code Case does not strictly apply to the spray calciner assembly, this method of assessing cumulative damage is used in this analysis.

Limited information is available on Inconel-601 material fatigue properties at the service temperatures involved in the spray calciner assembly. In particular, fatigue data are highly dependent on test conditions such as specimen surface finish, mean stress level, and environmental conditions. However, to assess likely fatigue damage, three approaches were

FIGURE D.14. Junction Stress Intensity Distribution at Temperature

FIGURE D.15. Junction Residual Radial Stress Distribution Upon Cooling

used. In light of the relatively low equivalent strain range calculated for the highly stressed regions of the junction, low fatigue damage is expected under normal operation.

The first method compares the expected strain range directly with the Inconel-601 material fatigue curves from the Aerospace Structural Metals Handbook (DOD 1978). Test data are available for hot-rolled rod (both annealed and solution-treated) and cold-rolled and annealed sheet. Figure D.19 duplicates the test data. After correcting for test temperature differences, a worst-case service life of approximately 10^6 cycles is still predicted. This would indicate that expected fatigue damage to the junction is negligible under any reasonable service cycling.

A second approach is to use Manson's "Universal Slopes" low-temperature generalized fatigue equation (Bechtel, Inc. 1977b) to estimate fatigue life for a given strain range. Factors of 20 on the calculated number of allowable cycles and two on strain range are used as factors of safety to account for the use of this equation at a temperature of 510°C (950°F). For a calculated strain range of 0.00145, the fatigue life thus calculated is approximately 50,000 cycles. Fatigue damage due to startup/shutdown cyclic thermal stresses at the spray calciner chamber/base plate junction is therefore expected to be negligible.

FIGURE D.16. Junction Residual Axial Stress Distribution Upon Cooling

FIGURE D.17. Junction Residual Hoop Stress Distribution Upon Cooling

The third approach is to compare the predicted strain range against all materials for which design fatigue data are available in the ASME Code for the service temperatures involved. These materials are 304 stainless steel; 316 stainless steel, and Inconel 800H. The two design curves are simultaneously shown as Figures D.20. The predicted junction equivalent strain range is also shown. The minimum fatigue life thus predicted to 800 cycles based on Inconel 800H properties. This value will be used in service life predictions in order to be conservative.

Using the Aerospace Structural Metals Handbook (DOD 1978) creep data for Inconel-601 solution annealed material (Figure D.21) a creep rupture time of 60,000 h is predicted based on the maximum Von-Mises equivalent stress of 228 MPa (33 ksi) in the junction of chamber and plate.

Using the usage fraction approach, service lives are predicted for a weekly and monthly cycle of operation. The service lives are 4.7 years and 6.3 years are predicted, respectively, excluding shutdown time. If fatigue damage is neglected based on the Aerospace Structural Material Handbook Data, a service life of 6.8 years excluding downtime is predicted for either

FIGURE D.18. Junction Residual Stress Intensity Distribution Upon Cooling

case. From these analyses, the controlling factor in the spray calciner chamber life is expected to be creep damage, as opposed to fatigue damage.

A junction thermal stress analysis was also conducted for a 310 stainless steel chamber using ASME Code (ASME 1977) Type 304/316 stainless steel design material property values. Type-304 stainless steel has a lower Young's modulus at temperature than Inconel-601 but a higher coefficient of thermal expansion. Thus, thermal strains are higher in the 300-series spray calciner chamber than in an Inconel-601 spray calciner chamber, but associated stresses are lower. The high-stress region is also somewhat more extensive in the 300-series chamber than in the Inconel chamber due to the lower Young's modulus of the stainless steel.

The maximum equivalent strain range predicted by finite-element analyses for the 300-series chamber is 1.63×10^{-3} . This corresponds to an allowable number of cycles for fatigue damage of 3000. The corresponding minimum time to stress rupture for the maximum predicted Von Mises equivalent stress of 132 MPa (19,200 psi) is 200,000 h. Using the piecewise-linear creep fatigue envelope (Figure D.22) for 300-series stainless steels from ASME Code (ASME 1977), operating lives of 12 years and 19 years are predicted for weekly and monthly cyclic operation, respectively.

FIGURE D.19. Inconel-601 Fatigue Data Reproduced From Aerospace Structural Metals Handbook (DOD 1978)

Central SC Chamber Thermal Stresses Due to Radial Thermal Gradient

The central spray calciner chamber is essentially free to expand and has minimal axial thermal gradients. However, a radial thermal gradient exists due to the heat flux through the wall from the surrounding furnace. This radial gradient (assuming only radial heat flow in the chamber) is approximately 12°C (22°F) for the 70 kW per zone normal operating condition of the furnace and increases to 27°C (48°F) when, with loss of furnace control, the furnace section operates at its rated 150 kW per zone.

Using a linear elastic closed-form solution (Timoshenko and Goodier 1951), thermal gradient stresses are predicted as shown in Table D.10.

These stresses are well within elastic limits. Creep rupture life at these stresses for solution-treated Inconel-601 is approximately 40,000 h or 4.6 years of continuous operation based on peak bending stresses in the chamber. This is a highly conservative figure, since it is an elastic analysis. In actual practice, stress relaxation would occur that would lessen creep damage; creep life would be significantly better.

FIGURE D.20. ASME Code Fatigue Data at 510°C (950°F) Using Figures T-1430-1A and -1C of Code Case N47

FIGURE D.21. Creep Rupture Data for Inconel-601 Reproduced From Aerospace Structural Metals Handbook (DOD 1978)

Creep rupture at the inner surface of the spray calciner chamber would likely initiate cracking of this surface. However, due to the net compressive membrane stresses in the section under normal operating conditions, it is unlikely that cracks so initiated would propagate through the chamber wall.

A similar analysis was conducted for a 310 SS chamber. Due to a higher thermal conductivity of 310 SS over Inconel-601, the radial gradient in this case is estimated to be approximately 10°C (18°F) for the 70 kW per furnace zone normal operation and increases to 22°C

FIGURE D.22. Creep Fatigue Interaction Envelope for 300-Series Stainless Steels Taken from the ASME Code (ASME 1977)

(39⁰F) under loss of furnace control. The maximum predicted normal operating stresses are the axial and hoop stresses at the inner chamber surface, which are 16.8 MPa (2400 psi). Using the ASME Code (ASME 1977) minimum time to creep rupture curves for 304 SS, a design operating life of 27,000 h is predicted (3.1 years). This is somewhat less than that previously predicted for the Inconel-601 chamber.

Creep Buckling of Spray Calciner Chamber

Creep buckling of the spray calciner chamber was briefly investigated. The elastic critical external pressure for buckling of a thin tube with closed ends is predicted using a closed form model (Roark and Young 1975, Table 35, Number 20). For the spray calciner chamber the elastic buckling pressure is 1 MPa (144 psi) at 800⁰C. Using a model for creep buckling for cylindrical shells (Bargmann 1972), a critical time to complete creep collapse is predicted. A uniaxial steady creep power law with $n = 5$ is assumed for the Inconel-601 material (i.e., $\dot{\epsilon} = k\sigma^5$). An initial out-of-roundness of 1 cm for the chamber was assumed (equal to the wall thickness to account conservatively for initial imperfections). Using this model and a creep rate of 0.0001%/h at 800⁰C and 53 MPa (7600 psi) stress based on Aerospace Structural Metals Handbook (DOD 1978) data. A creep life in excess of 10¹⁰ years is predicted for the

TABLE D.10. Central Spray Calciner Chamber Thermal Stresses

Stress	Normal Operating Conditions	Upset Conditions Loss of Furnace Control
Inner surface hoop and axial stresses	24 MPa (3480 psi)	51.7 MPa (7500 psi)
Outer surface hoop and axial stresses	23 MPa (3340 psi)	49.6 MPa (7200 psi)

normal operating condition of 2480 MPa (0.36 psi) external pressure. Thus, the chamber is not likely to collapse due to creep buckling or elastic buckling under normal operating loads. A similar conclusion should be equally valid for a 310 SS chamber.

Vibratory Behavior of SC Chamber Under Dynamic-Impact Loading

Introduction

The purpose of this section is to identify the transient stresses caused by vibration of the calciner chamber for fatigue life evaluation. Two vibrators alternately impact the spray calciner at elevated temperatures. The dynamic analysis is performed at design operating conditions. This section describes the analysis approach, results of the analysis, and conclusions.

Method of Analysis

Loading Condition. The spray calciner was analyzed for static and dynamic loading at design operating temperatures (800°C). Dynamic loading is caused by two vibrators alternately striking the calciner chamber. The applied vibrator force function was taken from a previous Bechtel study of spray calciner assembly response to vibratory loading (Bechtel, Inc. 1977b). Briefly, the force function was experimentally determined by pressurizing the vibrator and striking a target impact plate. From a displacement versus time chart and experimentally determined spring constant, the magnitude of the forces was calculated. Although geometry changes to the structure may modify the actual loading function, the assumed loading function represents the best model available.

Finite-Element Model. Design specifications were followed as closely as available information allowed. An ANSYS finite-element model comprising one quarter of the chamber represents the spray calciner chamber with a height of 3.2 m (10.5 ft), a diameter of 91.44 cm (36 in.), a chamber thickness of 0.953 cm (0.375 in.) and an impact plate thickness of 2.54 cm (1.0 in.). The model shown in Figure D.23 is symmetric about a vertical plane through the vibrator impact points and a horizontal plane at mid-height. This models a condition of vibrator loading 180° apart. A localized stress distribution is to be reasonably expected from alternate vibrator loading at the design placement of 120° apart. Assumption of 180° loading points permitted use of an additional plane of symmetry in the model which considerably reduced modeling time and computer costs, but due to the local nature of the impact pad effects, this assumption should have little effect on predicted chamber stresses.

The calciner is modeled using three-dimensional, rectangular and triangular shell elements with both bending and membrane characteristics. Both elements have 6 degrees of freedom at each node: three components of translation and three components of rotation about nodal axes. A relatively fine mesh is used to model the impact area where higher stresses are expected. A coarser mesh is used to model the rest of the cylinder where stresses are not expected to be significant. The model uses 100 elements and 106 nodes. The impact plate is modeled with an assumed corner radius of curvature of approximately 2 in. Figure D.23, a plot of the model, shows the mesh refinement modeling the impact plate. Temperature-dependent material properties are input. The model experiences a uniform temperature rise to the design

FIGURE D.23. Finite-Element Model of Spray Calciner - One Quarter Axisymmetric Model

operating temperature of 800°C (1472°F) and vibratory stresses are evaluated using appropriate material properties. Thermal discontinuity stresses were not calculated using this model. The generic material properties are taken from the ASME High-Temperature Code Case (ASME 1977) and the Aerospace Material Handbook (DOD 1978). Results are similar for both 310 SS and Inconel-601 chambers, since their respective Young's Moduli do not differ significantly at temperature.

The top of the spray calciner chamber is restrained against all translation and rotation. Restraint is provided at the upper end of the chamber by the plate welded to the chamber and the spray nozzle acting as a shear pin tying the plate to the spray calciner assembly seismic bracing. The lower end of the chamber is welded to the base plate. At the midplane of symmetry in the model, the calciner is allowed to expand radially but is prevented from translating vertically and circumferentially.

Two checks were performed to assure the reliability of the finite element model in representing the behavior of the spray calciner. The fundamental natural frequency was calculated by using the ANSYS model and then compared with Bechtel's results (Bechtel, Inc. 1977b) and a closed-form solution using Donnell's equations (Kraus 1967). Both checks showed good agreement with the finite-element model results.

Results

Mode Frequency Analysis. The first seven natural frequencies are listed in Table D.11. The fundamental frequency computed by finite-element analysis is 106 cycles per second. It agrees very well with 111 cycles per second calculated using Donnell's equation. It is also in close agreement with Bechtel's results when scaled by appropriate factors for differing shell thickness and elastic modulus.

Figure D.24 is a plot of the lowest natural frequencies as a function of the number of circumferential waves calculated with Donnell's equation. It predicts the lowest natural frequency corresponding to $n = 3$. Figures D.25 and D.26 are plots of the mode shape of the fundamental frequency. Figure D.24 clearly shows that the lowest frequency occurs at $n = 3$. The mode shapes for modes two and three appear in Figures D.27 through D.30.

Dynamic-Response Analysis. A dynamic-response analysis determined the linear elastic transient stresses induced in an Inconel-601 chamber in the spray calciner assembly by vibrator impact forces represented in Figure D.31 (310 SS chamber stresses would be similar). Two

TABLE D.11. Natural Frequency of Spray Calciner Chamber

Mode	Inconel-601 Chamber Frequency, cps	310 SS Chamber Frequency, cps
1	106	98
2	137	126
3	149	138
4	156	144
5	167	154
6	184	169
7	195	180

FIGURE D.24. Natural Frequencies of Spray Calciner

FIGURE D.25. Mode Shape for Mode 1, $f = 106$ Hz

FIGURE D.26. Mode Shape for Mode 1, $f = 106$ Hz

vibrators are mounted per assembly, 120° apart. The two vibrators alternate operation every 3 min, with each vibrator being operated separately for a 3-s duration. Response to a single period of operation of one vibrator was analyzed. A previous Bechtel study (Bechtel, Inc. 1977b) found that the maximum spray calciner chamber response to such loading occurred in the first 3 s of operation. [Bechtel analyzed a similar spray calciner assembly, but with a

FIGURE D.27. Mode Shape for Mode 2, $f = 137$ Hz

FIGURE D.28. Mode Shape for Mode 2, $f = 137$ Hz

0.63-cm (1/4-in.) wall structure.] Therefore, the dynamic loading of Figure D.31 was repeated for nine cycles to simulate the applied loading to the calciner for the 3-s forcing period. Direct numerical integration of the equations of motion were used with a time step of 0.0005 s. Twenty master degrees of freedom were used in a reduced analysis.

Figure D.32 is a plot of the predicted radial displacement response at the point of vibratory hammer impact over 2.5 s. It indicates the periodicity of the response corresponding to

FIGURE D.29. Mode Shape for Mode 3, $f = 149$ Hz

FIGURE D.30. Mode Shape for Mode 3, $f = 149$ Hz

the vibrator's impact force period of 0.32 s. Figure D.33 is an enlarged view of the radial displacement response over one cycle of load. Figure D.34 plots the Von Mises equivalent stress response for one cycle of the outer shell surface under the point of impact. The maximum Von Mises equivalent stress developed by the structure is approximately 3860 MPa (560 psi) at the point of impact. The corresponding displacement is 0.00546 cm (0.00215 in.).

FIGURE D.31. One Cycle (0.32 s) of Vibratory Force Function (Bechtel, Inc. 1977b)

FIGURE D.32. Radial Displacement Response of Cylinder to Vibratory Loading at the Shell Surface at the Impact Point

FIGURE D.33. Radial Displacement Response for One Cycle of Vibratory Loading at the Shell Surface at the Impact Point

A contour stress plot of the impact plate and adjoining area, Figure D.35 shows two areas of stress concentration. As mentioned previously, the peak stresses occur at the point of vibratory loading. The closely spaced stress contours at the impact point indicate this stressed area. The plot shows a second area of stress concentration at a lower magnitude. The corner of the impact plate, develops a stress of approximately 3380 MPa (490 psi). This is to be expected at the discontinuity in shell thickness.

The Bechtel study (Bechtel, Inc. 1977b) predicted a maximum stress of 1330 psi at the weld around the impact plate for the thinner-walled spray calciner chamber. Two differences in the model geometry may account for the variation in the maximum stresses predicted by Bechtel and the present one. First, the Bechtel model used a square-cornered impact plate. The square corner induces a further stress concentration beyond that inherent in the abrupt change in wall thickness at the edge of the impact plate. The present model uses an arbitrarily chosen corner radius of approximately 5.08 cm (2 in.) for the impact plate. Second, a difference in shell thickness, 0.635 cm (0.25 in.) versus 0.953 cm (0.375 in.) contributes to the higher stresses in the Bechtel model.

FIGURE D.34. Equivalent Stress Response for One Cycle of Vibratory Loading at the Shell Surface at the Impact Point

Conclusions

Fatigue Life. The Manson "Universal Slopes" fatigue equation (Bechtel, Inc. 1977b) was again used to estimate fatigue life for the predicted stress level at 800°C (1472°F). Again, factors of 20 were used on the number of allowable cycles and two on strain range to account for uncertainties of using this equation in elevated temperature applications. Even so, a fatigue life of 5×10^{12} cycles was predicted for the Inconel-601 chamber and 5×10^{13} cycles for the 310 stainless steel chamber for the equivalent strain range associated with the highest peak stress in the transient that occurs once every 0.32 s of vibratory operation. Fatigue damage, therefore, was negligible.

Low stresses are predicted for the vibratory transient response. The ASME strain range associated with these low stresses is well below strain ranges for which design fatigue data are available (ASME 1977) for materials covered by the ASME Code. Thus, distortion and fatigue failure does not appear to be a significant consideration.

FIGURE D.35. Contour Equivalent Stress Plot Near Impact Plate Shows Peak Stresses Occur at the Impact Point and Corner of Weld Plate

Filter-Element Stress Analysis

A closed-form model of a cantilever beam with the other end hinged was used to predict bending stresses in the filter during vibratory loading. The lateral filter supports provide the hinged constraint at the lower filter end. The stiffness of the internal axial rod brace within the filter was not accounted for in the model.

Bending stresses equal to the Inconel-601 yield stress are reached for a solid filter (i.e., no porosity) under a 360-g static horizontal loading. This is well above vibratory or seismic loading conditions. Although, the model includes no microstructural effects on strength of the filter porosity, the high margin of safety inherent in the present calculation indicates the satisfactory design of the filters. In addition, porosity effects reduce filter mass (hence material loading) during vibration. This offsets to some extent the strength reduction due to porosity. It should be noted that no structural failures due to mechanical properties have occurred in the prototype spray calciner assembly filters during operation at PNL to date. Failure of the filters is more likely to be controlled by corrosion or other phenomena.

A simple cantilevered filter without lower-end support would be subject to four times the stress at the attachment point than predicted above. Therefore, the lower supports for the filters may not be necessary, since acceptable stresses occur during vibration loading without them; however, they provide a further margin of safety for the design.

OVERALL CONCLUSIONS

The main stresses under both normal and upset or emergency loading conditions have been predicted. Stresses are generally low enough to provide reasonable assurance of proper operation.

Careful attention should be paid to final detailing of the assembly to prevent undesirable stress concentration effects. For example, a prototype spray calciner chamber used for nonradioactive testing was fabricated of uniform wall thickness and impact pads were then fillet-welded to the sides of the chamber. These welds subsequently failed by crack propagation due to local stress concentrations associated with this type of construction. Essentially, a large "circular" crack under the plate was manufactured into the structure. The prototype chamber was modified to use a one-piece impact-plate insert with taper to the nominal wall thickness. Such an insert is less subject to stress concentration effects.

Two key stresses with respect to limiting spray calciner assembly service life are the radial thermal gradient thermal stresses in the central spray calciner chamber and the axial thermal stresses at the spray calciner chamber/base plate junction. Several steps can be taken to alleviate these stresses and extend chamber service life, if desired.

- a) The nominal chamber wall thickness can be reduced. For fixed heat flux, this reduces the radial wall thermal gradient, thereby reducing thermal stresses. Small reductions in stress at the elevated service temperatures can yield significant changes in time to creep rupture. However, care must be taken to ensure that vibratory stresses due to the impact hammers used to remove calcine are not increased to the extent that they become controlling.
- b) The calciner chamber can be increased in thickness near its lower junction with the base plate. This provides more material to accommodate the highly localized bending stresses there. A transition nozzle from 2.54 cm (1 in.) to the final central chamber wall thickness is suggested with no less than a 3:1 taper ratio. This option is even more necessary if option (a) above is also exercised.

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