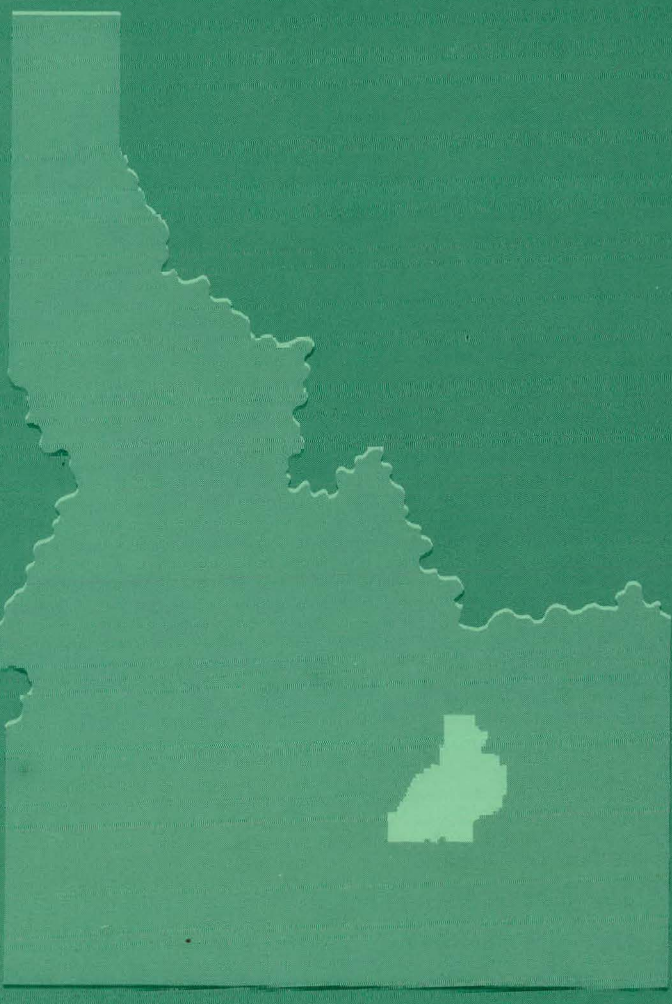
IDO-14539

PILOT PLANT STUDIES WITH A SIX-INCH DIAMETER FLUIDIZED BED CALCINER

D. R. Evans

April 20, 1961



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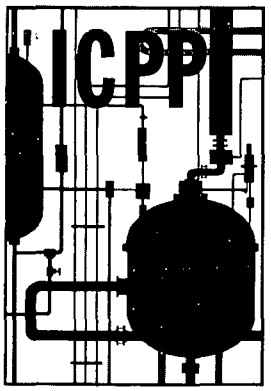

IDAHO CHEMICAL PROCESSING PLANT

IDO-14539
AEC Research and Development Report
Waste Disposal and Processing
TID-4500, Edition 16
Issued: April 20, 1961

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Operation and Development Work Performed By
E. S. Grimmett, Problem Leader
G. E. Lohse
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Atomic Energy Division
Contract AT(10-1)-205
Idaho Operations Office
U. S. ATOMIC ENERGY COMMISSION

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A B S T R A C T

The feasibility of the fluidized bed calcination process has been demonstrated at the Idaho Chemical Processing Plant. Solutions simulating wastes from the reprocessing of spent aluminum alloy, stainless steel, and zirconium fuels were successfully converted to free-flowing granular solids in a six-inch diameter fluidized bed calciner. The calciner was electrically heated, and a pneumatic atomizing spray nozzle was used to introduce liquid feed into the heated fluidized bed, where calcination took place. Removal of entrained particles from the off gas was achieved through the use of a cyclone and a venturi scrubber, although the cleaning achieved would not be adequate for calcining a radioactive waste. Dry fines removed by the cyclone were returned to the bed with the fluidizing air through an air jet on the dust discharge line of the cyclone.

The effects on the calcined product properties and on off gas solids loading of the degree of feed atomization, of bed temperature, and of returning dry fines to the calciner were noted while calcining simulated wastes from aluminum alloy fuels.

The distribution of ruthenium and the effect of ammonium ion were determined, and a slurry of a cesium complex with nickel ferrocyanide was dried on sand, then leached, as a method of cesium recovery.

Work done under Contract AT(10-1)-205 to the U. S. Atomic Energy Commission.

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PILOT PLANT STUDIES WITH A
SIX-INCH DIAMETER FLUIDIZED BED CALCINER

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

PILOT PLANT STUDIES WITH A SIX-INCH DIAMETER FLUIDIZED BED CALCINER

by

D. R. Evans

I. SUMMARY

Research and development work to demonstrate the feasibility of fluidized bed calcination as a possible step toward the ultimate disposal of radioactive wastes was initiated at the Idaho Chemical Processing Plant (ICPP) in May, 1955, using a six-inch diameter fluidized bed pilot plant calciner. The fluidized bed calcination process involves spraying an aqueous solution of metal salts into a heated fluidized bed where the salts are largely converted to oxides in the form of free-flowing granular solids. The solids are continuously removed from the bed, and the water vapor and volatile decomposition products leave with the fluidizing gas. Reduction in corrosion rates of the storage container, reduced mobility of the waste, and reduced storage costs are advantages of converting liquid wastes to solids; the combination of unlimited scale-up possibilities, continuous processing, versatility, and a lack of complex moving parts makes fluidized bed calcination unique among methods of conversion to solids. Although major effort was directed toward equipment development and process improvement, the following effects of known independent variables were noted while calcining simulated aluminum waste:

1. High nozzle air-to-liquid volume ratios in the pneumatic atomizing nozzle used to introduce feed into the calciner caused the average particle diameter of the fluidized bed to be low and the fine-particle loading in the off gas leaving the primary cyclone to be high. The opposite effects were noted with low values of this ratio.

2. Operating a dry fines return system caused the mass median particle diameter of the fluidized bed to be smaller than when the dry fines return system was not in operation. A higher fine-particle loading in the off gas leaving the primary cyclone was associated with dry fines return due to attrition in the return system.

3. A relatively large air annulus in the feed nozzle caused a relatively large average particle diameter in the fluidized bed at a fixed nozzle air-to-liquid volume ratio.

4. High values of bulk density and of product nitrate content were associated with low values of calcination temperature in the range tested, 185 - 500°C.

5. Increased fines elutriation at high fluidizing velocities was the only effect of superficial fluidizing velocity observed in the range 0.6 - 4.5 ft/sec; relatively high velocities (2.0 - 2.5 ft/sec) were necessary to decrease the probability of burnout of the electric heaters which supplied heat to the fluidized bed.

Tracer studies were conducted with simulated waste containing that concentration of ruthenium estimated to be in an actual waste; as much as 67 per cent of the ruthenium was forced to remain with the solid product by routing the fines return slurry from the venturi scrubber to the calciner bed with the feed while calcining at 400°C.

Calcination of zirconium and stainless steel simulated wastes was accomplished without any unusual operating difficulties in runs of short duration.

An operable and flexible system for the calcination of simulated aluminum reactor fuel reprocessing waste to a free-flowing solid of a reasonable bulk density (0.8 - 1.0 g/cc) and mass median particle diameter (0.3 - 0.6 mm) was developed at the ICPP. The process should also be useful for the calcination of zirconium and stainless steel wastes.

II. INTRODUCTION

Radioactive wastes from the aqueous processing of spent reactor fuels at the Idaho Chemical Processing Plant (ICPP) are currently stored in large underground tanks to prevent the release of large quantities of radioactive material to the surroundings. Although storage of aqueous waste by this means has been adequate, the need for safer, cheaper, and more permanent storage has long been evident.

Most approaches to improved storage involve conversion of the aqueous waste to a stable solid. Among these are batch calcination, continuous calcination in a fluidized bed, continuous calcination in a rotary calciner, adsorption of fission products on clays with subsequent sintering, and others. For most wastes, conversion to the solid phase offers the advantages of a large volume reduction and, potentially at least, a substantial reduction in the storage costs.

The fluidized bed calcination process originally conceived by the Argonne National Laboratory (ANL) Chemical Engineering Division[1] and first applied to simulated radioactive waste by the ICPP Technical Branch[2] offers the further advantage over other schemes of conversion of aqueous waste to solid of producing a granular, free-flowing solid which has a reasonably high bulk density and which may be easily handled by pneumatic transport techniques. Furthermore, the process is continuous and scale-up possibilities are nearly unlimited. Research and development efforts at the ICPP on the disposal of radioactive wastes have centered largely on the fluidized bed calcination technique for conversion of aqueous aluminum process waste to solid. Work was initiated by the ICPP Technical Branch Chemical Engineering Development Section in May, 1955, using a six-inch diameter fluidized bed pilot plant calciner.

The object of this report is to describe the fluidized bed calcination process developed during nearly five years of operation of the six-inch pilot plant calciner and to summarize the information and process data obtained.

A. Fluidized Bed Calcination Process

The fluidized bed calcination process at ICPP involves spraying an aqueous solution of metal salts through a pneumatic atomizing spray nozzle into a heated, gas-fluidized bed where the salts are largely converted to oxides in the form of free-flowing granular solids. The solids continuously leave the bed through an overflow pipe, and water vapor and volatile decomposition products leave with the fluidizing gas. Practical methods for furnishing heat to the fluidized bed include heating the calciner vessel walls (for small vessels) or placing heat exchangers in the fluidized bed (for larger vessels). The fluidizing medium can be any suitable gas (air, superheated steam, reducing or oxidizing gas), the selection depending upon the chemistry and economics of the particular process. (Normally air was used in the studies reported herein, although one successful run was made using superheated steam.) Remote handling (necessary when high-level radioactivity is involved) makes extremely desirable the simplicity of operation and continuous processing offered by the fluidized bed calcination technique.

Fluidized bed calcination as applied to aluminum alloy fuel process wastes converts the acidic aluminum nitrate solutions into free-flowing granular aluminum oxide with a resultant volume reduction in the range of 6- to 19-fold. Increased radiological safety and a significant reduction in storage costs are expected to be some of the results of this conversion of liquid to solid. Furthermore, corrosion problems associated with the liquid waste storage will almost surely be reduced by a significant amount, and the solids would be essentially immobile should the container rupture during storage.

B. Applications of the Fluidized Bed Technique

1. General Applications

Fluidized bed technology is a relatively new branch within the broad field of chemical engineering and has been found to have many applications in chemical processing because of the superior heat and mass transfer rates in a fluidized bed. Fluidized catalytic cracking, reforming, and alkylation have been used extensively in the petroleum industry and are largely accepted in today's technology. The iron and steel industry has investigated, and found promising, iron ore reduction in a fluidized bed. The calcination of limestone in a fluidized bed seems to be superior to the older rotary kiln technique[3]. The technical feasibility of processing of uranium ore concentrate to nuclear fuel using fluidized bed reducers, hydrofluorinators, and fluorinators has been demonstrated at ANL[4].

2. Radioactive Waste Calcination

The development of fluidized bed calcination at ANL and ICPP has opened new avenues of ultimate disposal of radioactive waste and fission product recovery and fixation, while simultaneously adding to the field of chemical processing a useful piece of equipment in which many unit operations and processes may be more effectively conducted.

It is recognized that aqueous storage of radioactive waste has many disadvantages, not the least of which are the corrosive nature of the waste effluents from aqueous nuclear fuel reprocessing, the danger to humanity if mobile aqueous wastes should be accidentally released to the ground water (by rupture of a storage tank, for instance), the inherent high expense of aqueous storage, and the large volume of water stored with the waste. Conversion of aqueous waste to solid product is therefore indicated, because solids are less corrosive than liquids, would be relatively immobile if the container ruptured, and should be less expensive to store because of the reduced storage volume made possible by removing the water.

Because Cs_2O and SrO, formed with alumina during the calcination of aluminum waste, are soluble in dilute nitric acid, it may be possible to recover Cs-137 and Sr-90 by leaching the calcined product. These fission products could presumably be concentrated and separated by ion exchange and reconverted to the solid state by the fluidized bed calcination technique. This method of recovering cesium and strontium indeed may be more economical than present methods of isolating these elements for use as radiation sources. It may also be a step toward unattended disposal of most of the alloying materials, corrosion products, and fission products from a reprocessing plant.

III. EQUIPMENT

A. Final Version of the Six-Inch Calciner System

The final version of the six-inch calciner system consisted of the following basic equipment:

1. The calciner vessel
2. The dry fines return system
3. The venturi scrubber and associated cyclone
4. The off gas condenser

The calciner vessel was a 6-inch, schedule 40 stainless steel pipe welded atop a 6-inch square by 7-inch high section in which eighteen 1000-watt cartridge heaters, 1/2-inch O.D. by 9 inches long and having a 6-inch heated length, were mounted on a triangular pitch through one side on 1 1/2-inch centers. The tangential off gas outlet was located near the top. Liquid feed was introduced through a pneumatic atomizing nozzle (Spraying Systems Co. 1/4 J Nozzle, Setup No. 2, consisting of a

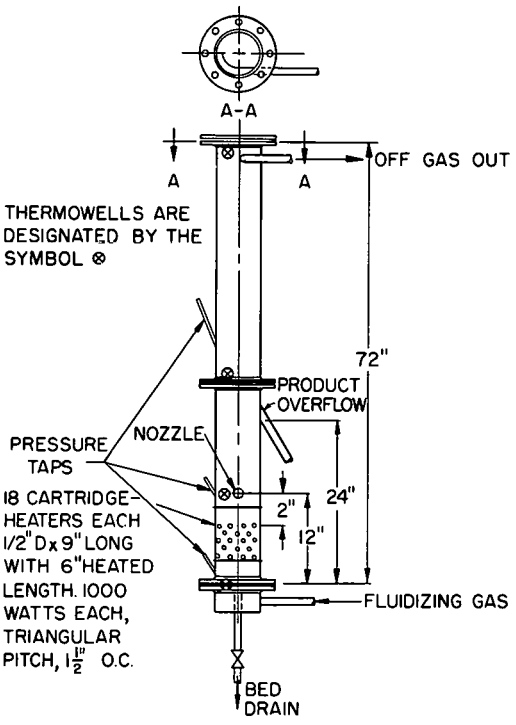

Fig. 1 - Final Version of Six-Inch Fluidized Bed Calciner Vessel

No. 70 air nozzle and a No. 2850 water nozzle). The granular product was removed through an overflow pipe located two feet above the bed support plate; this plate was a non-sifting bubble cap type adapted from a design developed by Anderson and Balduc(5). Figure 1 is a drawing of the final calciner vessel.

The dry fines return system consisted of a high-efficiency cyclone (primary cyclone) mounted external to the calciner vessel and having an air jet (dry fines return jet) to induce flow through the dust discharge. The dry fines return jet performed the following functions:

1. Returned particles from the primary cyclone to the calciner.
2. Recycled a substantial portion of the fluidizing gas to the calciner vessel so that the size of the off gas cleaning equipment downstream from the primary cyclone could be minimized.
3. Reintroduced to the bed seeds for particle growth which would otherwise have been lost.

The design of this jet was unique in that the particle-containing dry fines return gas was introduced axially to the jet, while the motivating gas was introduced concentric to this stream through an annular orifice. This design was intended to minimize wear to the jet both by the effect of a layer of solids-free gas along the walls of the mixing section and by the effect of introducing the solids axially. Extra resistance to wear was afforded by lining the wall of the mixing section with tungsten carbide. Figure 2 is a sketch of this jet.

The venturi scrubber and associated cyclone were installed as a means of removing as many of the submicron-sized particles in the off gas leaving the primary cyclone as possible. This equipment was extremely efficient in the reduction of the off gas solids loading(6).

The off gas condenser was a one-pass, shell-and-tube heat exchanger with condensing vapors on the tube side and cooling water on the shell side. This apparatus removed much of the nitrogen oxides and water vapor from the off gas.

Figure 3 presents a schematic diagram of the six-inch calciner system.

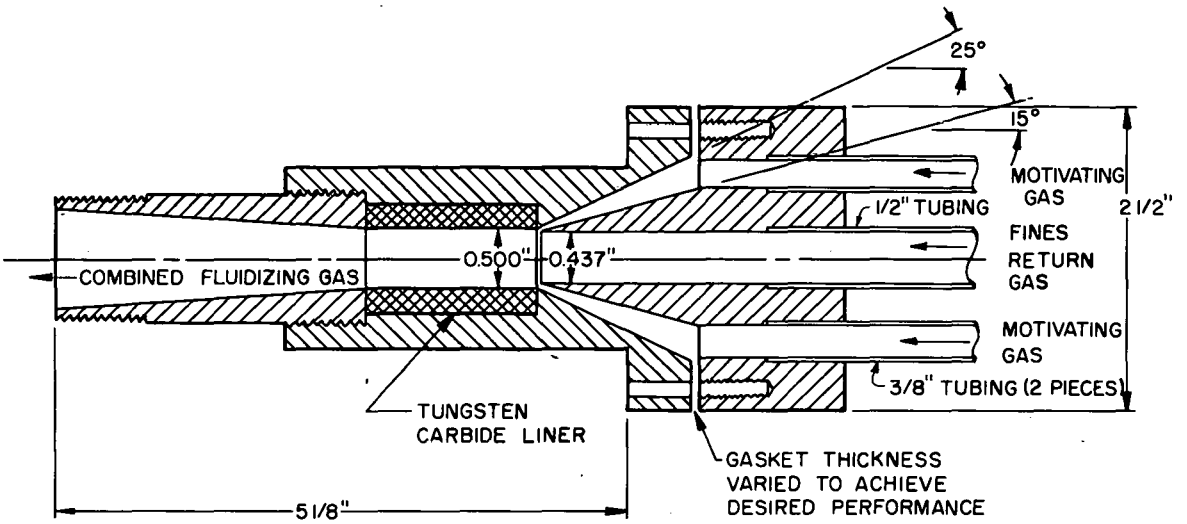

Fig. 2 - Dry Fines Return Jet

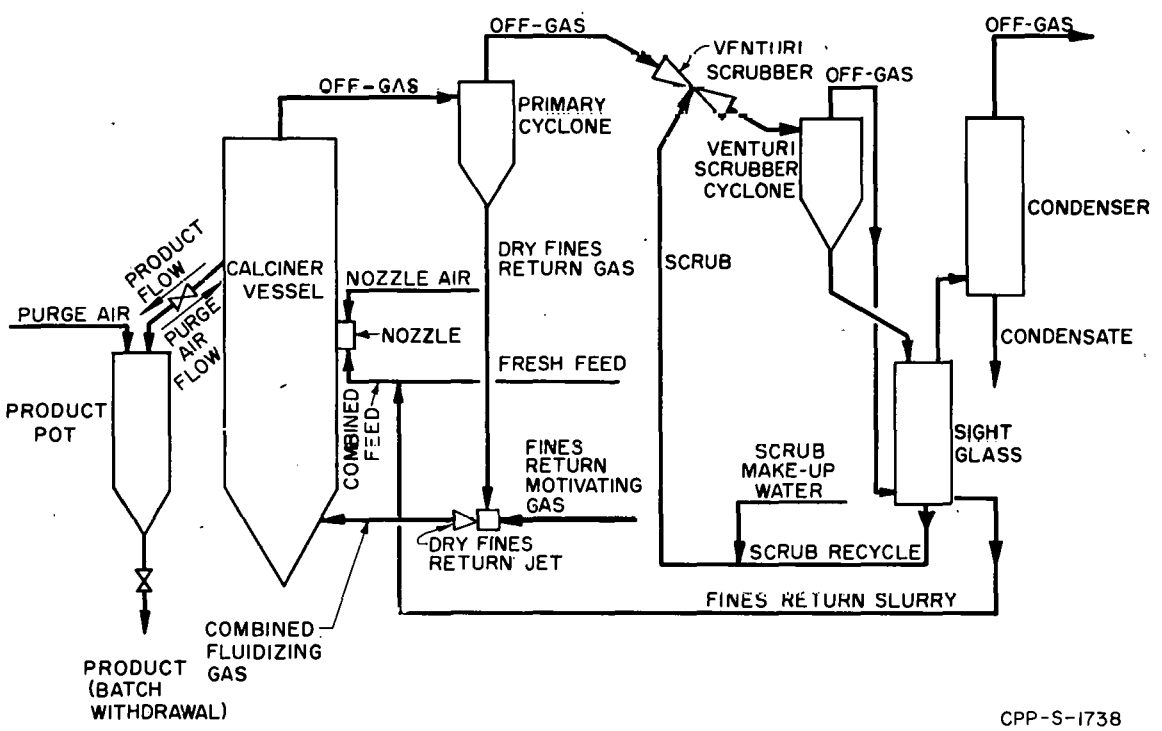

Fig. 3 - Schematic Diagram of Calciner System

This system, even in its final state of development, had a number of deficiencies. Among these were:

1. In order to operate at reasonable capacities, the cartridge heaters had to be operated at high flux densities and the resulting high surface temperatures. Under these conditions, if a significant reduction in the heat transfer rate (due to caking in the bed, poor fluidization, large particle size, etc.) occurred, even in a highly localized portion of the bed, the surface temperatures of the heaters in that portion of the bed would exceed the maximum safe temperature and the heaters would burn out. The replacement of heaters, in addition to being an expensive annoyance, required that many potentially useful runs be terminated prematurely.

2. Even under the best conditions, the feed rate was never greater than 8.2 l/hr, and the normal maximum feed rate was only about 6.0 l/hr.

3. The system lacked sufficient flexibility to be operated under a number of conditions. For example, operation under vacuum and operation with total off gas recycle were not possible.

For these reasons, larger pilot plant calciners (12-inch and 24-inch) have been designed with which various further studies have been and will be made. The inherent limitations of the six-inch calciner system dictated a shift in emphasis to larger, more refined calciner systems.

B. Equipment Testing

During the period of operation of the six-inch calciner (May, 1955, to February, 1960), a good deal of emphasis was placed on equipment development for improved operability (during initial stages) and on testing auxiliary equipment (during later stages). The important equipment that was tried for improved operability and rejected includes the following:

1. Bayonet-type, G-porosity filters of sintered stainless steel for off gas clean-up.
2. Manually-controlled electrical strip heaters fastened to the outside of the vessel.
3. A reciprocating positive displacement feed pump.

Two considerations motivated the rejection of the stainless steel filters.

1. The fluctuations in calciner pressure caused by switching from loaded filters to clean ones were considered undesirable.

2. Scale-up of a system utilizing filters would present considerable difficulty.

The capacity of the unit was increased by increasing the area available for heat transfer and by eliminating excessive heat losses when the internal cartridge heaters were substituted for the external strip heaters. Also, manual temperature control prohibited maintaining a reasonably constant bed temperature. Automatic adjustment of one of the four autotransformers was accomplished by installing an electronic-pneumatic controller sensing the signal from a thermocouple in the bed opposite the spray nozzle. The controller was connected by a flexible coupling to the shaft of the autotransformer through a pneumatic positioner.

Plugging of the feed nozzle often occurred as a result of the pulsating flow characteristic of a reciprocating positive displacement feed pump. The pulsating flow allowed nozzle tip temperatures high enough to cause partial evaporation of the feed, and create sufficient salt crystals to plug the feed orifice.

Auxiliary equipment tested included:

1. A primary cyclone of U. S. Hoffman-type design.
2. A packed gas-liquid contactor and an electrostatic precipitator for partial removal of the submicron-sized particles which escape the primary cyclone.
3. An oil-fired fluidizing air preheater.
4. A number of feed and slurry pumps.

A cyclone of design based on the U. S. Hoffman concept[7] was substituted for a cyclone of conventional design for use in the calciner dry fines return system. Although a thorough performance study of this modified design was not made, sufficient evidence was gathered to demonstrate the unsuitability of this particular cyclone in the calciner dry fines return system. Because the conventional cyclone had been satisfactory, it was re-installed.

The packed gas-liquid contactor was installed to remove particles from the off gas. However, since efficiency of this unit was limited, and since a venturi scrubber with associated cyclone proved to be highly efficient[6], no further serious consideration was given to the packed contactor. The electrostatic precipitator was tested separately from the calciner system and was found to be extremely efficient for the pressure drop incurred[8].

An attempt to minimize the fluidizing air requirement and to suppress ruthenium volatility was made by installing a high-pressure oil burner to preheat the fluidizing and jet motivating gas. The dry fines return jet recycled some of the off gas from the calciner vessel and combined it with the jet motivating gas so that less air was needed to maintain a given fluidizing velocity. Increasing the efficiency of the dry fines return jet by using high-temperature combustion gases as jet motivating gas increased the fraction of the off gas which was recycled. Fluidizing with combustion gases also furnishes a reducing atmosphere to suppress ruthenium volatility. However, the use of this heater was abandoned after several futile attempts to eliminate the clogging of the jet by soot from incomplete combustion of the fuel oil.

The initial run of the six-inch calciner made use of a reciprocating positive displacement liquid feed pump which was unsatisfactory because of the characteristic pulsating flow. A Moyno rotary positive displacement pump having a Type 316 stainless steel rotor turning against a neoprene stator gave good service pumping both fresh feed and combined feed. However, periodic replacement of the neoprene was necessary because of chemical attack. Later a Chempump (Model S-3/4 hp) in conjunction with automatic flow control performed well with fresh feed.

The addition of wet scrubbers to the six-inch calciner system added a problem to the many others associated with waste calcination in that an erosive and corrosive slurry (5 w/o Al_2O_3 - 40 w/o HNO_3) is the effluent from this type of scrubber. Practical considerations demand that a major part of the slurry be recirculated to the scrubber along with a relatively small amount of water to make up for that evaporated and that removed in the fines return slurry. Total recycle is, of course, impractical because the solids accumulating in the liquid must be removed as fast as they are added.

The Chempump was soon found to be unsatisfactory in slurry-pumping service because of excessive shaft and bearing wear. For this reason an attempt to use conventional slurry pumps was made, but the low flow rates involved in the pilot plant equipment could not be met by any available pump without extreme throttling or almost total recycling. However, the eventual necessity of pumping an alumina slurry prompted separate testing of several commercial slurry pumps. The extremely corrosive and erosive slurry from the venturi scrubber has not yet been pumped satisfactorily by any pump. However, three pumps were selected for testing and are listed below:

1. Johnston 2-Stage, Splined-Shaft, Deep Well Turbine Pump, Type GAXC, Drawing Number H-1644-C.
2. Wilfley Model AF Acid Pump, 1 1/2" x 1", Assembly 318-75.
3. Nagle 1" Sump Pump, Type CWOC, Frame 126-L.

For remote applications, the Johnston pump is attractive because little or no lubrication of the pump is necessary. However, conventional graphite bearings wore very rapidly while pumping a nitric acid-alumina slurry. Tests at Battelle Memorial Institute were made in which boron carbide was found to be resistant to this slurry. Boron carbide bearings have been designed and ordered for use with this pump, but have not yet been tested.

The Wilfley pump is considered to be suitable for non-radioactive service. However, the inherent leakage around the bearings while starting and stopping would be undesirable in radioactive service.

The Nagle pump has several advantages, among which are the following:

1. The lack of bearings or rubbing parts in contact with the solution being pumped would eliminate the need for special bearings.

2. Its characteristic low speed would reduce wear.

3. Large bulky parts would minimize the effects of wear.

The disadvantages of this pump are as follows:

1. Since this pump has no bearing at the impeller end of the shaft, severe vibration could result from shaft misalignment. (The Lawrence pump uses a similar design, but has a dual discharge, a feature which should minimize the possibility of excessive vibration and shaft whip.)

2. Periodic lubrication is necessary, although this could be done remotely.

IV. OPERATING PRECAUTIONS

Experience in the operation of the six-inch calciner pointed up the necessity of observing a number of precautions in start-up and shutdown of the unit. Major operating difficulties centered around plugging of various lines, and burning out of heaters. The more important of the techniques used to minimize these difficulties were:

1. The product pot purge air and nozzle air streams were never shut off completely. This was done to prevent plugging by fines of the orifices concerned.

2. The fluidizing air was always turned on before the electric heaters. This minimized heater burnout.

3. The maximum liquid feed rate was attained in not less than an hour, because more rapid increases in the liquid feed rate would cause heaters to burn out.

4. When shutting down, the feed line was flushed with water. Evaporation of liquid feed, if left in the lines, would cause crystallization and plugging.

5. A small flow of water to the wet scrubbers was always maintained to prevent plugging.

V. EXPERIMENTAL RESULTS

A. Effect of Process Variables

The known independent variables concerned with calciner operation are the following:

1. Nozzle air-to-liquid volume* ratio (NAR)
2. Calcination (bed) temperature
3. Superficial fluidizing velocity
4. Feed composition
5. Sample pot purge air rate
6. Return of dry fines to the bed

The most important measurable dependent variables are:

1. Product bulk density
2. Product particle size distribution
3. Loading of fine solids in the off gas

One of the unsolved problems encountered during the operation of the six-inch calciner was the failure to attain a steady state wherein the dependent variables had reached constant values and remained at those values so long as the run continued at fixed conditions of the independent variables. With the six-inch calciner, even when a particular run extended continuously over a period of from three to four weeks, a steady state was never attained.

The dependent variable which gave the most concern was the particle size distribution of the product. This property was followed closely throughout most runs, but without exception, the variation within a run was considerable. The mass median particle diameter (that particle diameter at which half of the particles by weight are larger and half smaller) was chosen as the most appropriate single value by which to represent the bed or product particle size.

Comparisons of the average mass median particle diameter have shown gross differences between runs. The mass median particle diameter fluctuated between 0.3 and 1.2 mm, and the best operation was characterized by median diameters in the range 0.3 to 0.6 mm. The bulk density of the product, however, reached relatively constant values on many runs. The bulk density on different runs varied between the limits of 0.5 to 1.1 g/cc; 0.8 g/cc was the most characteristic value of this property, with variations of $\pm$ 20 per cent within some runs.

The effects of the various operating variables on the dependent variables have not been determined in every case due to the difficulties described earlier. Where possible, measured effects are reported;

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* Volume of air calculated at metered temperature and calciner pressure.

otherwise the results of visual observations are reported in general terms. A summary of the original data from all runs made is presented in Table 1 in the Appendix.

1. Nozzle Air-to-Liquid Volume Ratio

The operating experience with the six-inch calciner has shown that increasing the nozzle air-to-liquid volume ratio (NAR) markedly decreases the product particle size and increases the loading of fine particles in the off gas. Figure 4 is a plot of the final mass median particle diameter as a function of NAR for runs made at 400°C and shows that the mass median particle diameter was in the range 0.3 - 0.6 mm when the NAR was in the range 300 - 750.

Particle size distribution plots for various average values of NAR are given in Figure 5. The curve for the low average NAR of 260 was obtained by averaging the particle size analyses of five runs at nozzle ratios between 230 and 300, the curve for the intermediate average NAR of 315 was obtained by averaging results from five runs with values of NAR between 312 and 320, and the curve for the high average NAR of 455 was obtained by averaging results from four runs with values of NAR between 450 and 465.

Figure 5 shows that the production of coarse particles is increased at the expense of fine particles at a low NAR. The reverse is true at high values of NAR. In fact,

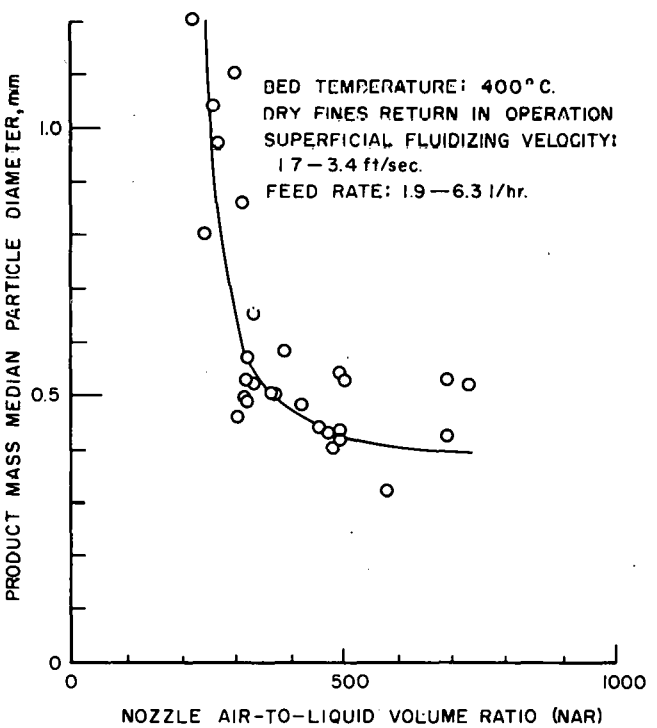

Fig. 4 - Product Mass Median Particle Diameter as a Function of Nozzle Air-to-Liquid Volume Ratio

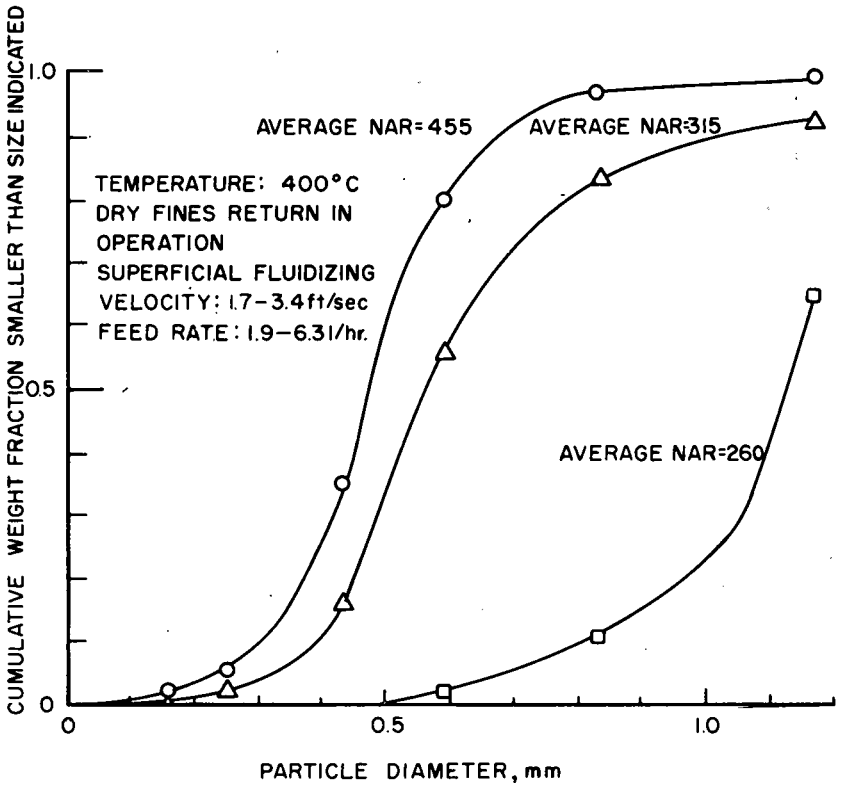

Fig. 5 - Particle Size Distribution at Various Average Nozzle Air-to-Liquid Volume Ratios

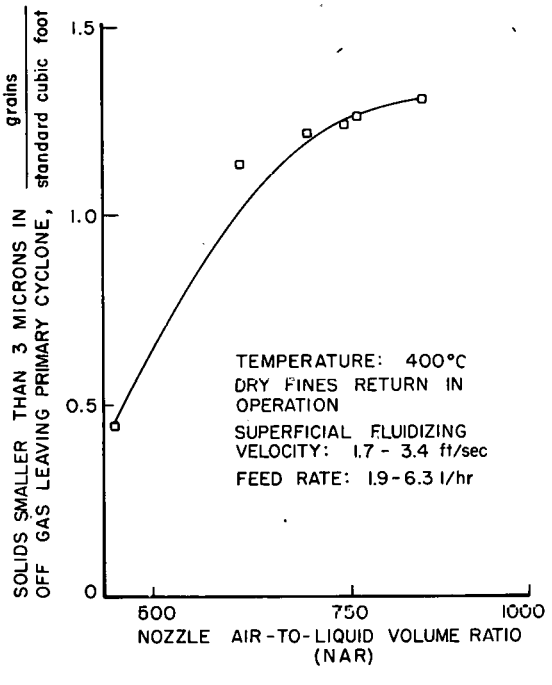

Fig. 6 - Concentration of Fine Solids in the Off Gas Leaving the Primary Cyclone as a Function of NAR

NAR has been found to be the most important criterion in the control of the mass median particle diameter. Proper adjustment of this value has been found to override the effects on the particle size of other independent variables.

The off gas loading of particles smaller than 3 microns as a function of NAR is presented in Figure 6. (The size 3 microns was arbitrarily chosen as an upper limit for defining "fine solids".) It may be seen that the fine-particle loading of the off gas increases with increases in NAR. Although the fine solids loading was not measured for every run, this loading (in weight per unit time) has never been over 15 per cent of the theoretical product rate for those runs in which it was determined, and is not considered to be intolerable.

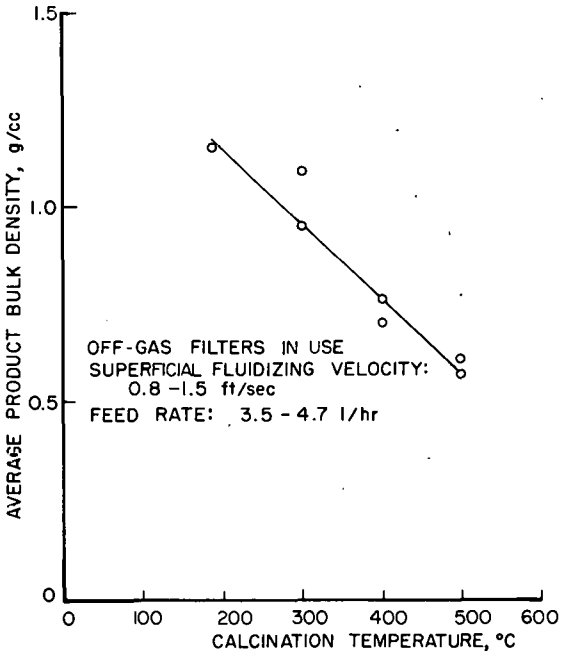

Fig. 7 - Average Product Bulk Density as a Function of Calcination Temperature

2. Calcination Temperature

Within the range of calcination (bed) temperature tested, 185 - 500°C, two trends were observed:

a. The average product bulk density decreased with increasing temperature, presumably because of an increase in intra-particle porosity. Figure 7 presents a plot of average bulk density as a function of temperature.

b. High values of product nitrate content were associated with low values of temperature; nitrate content was in the range of 5 - 6 per cent (as nitrogen) at 185°C and 0.8 per cent at 500°C.

3. Superficial Fluidizing Velocity

The superficial fluidizing velocity probably has an effect on particle size distribution because a change in the velocity should change the attrition rate between particles, and this in turn is related to the production of fine particles. Also, the largest particle size elutriated by the fluidizing gas is larger at high velocities. However, reliable data as to the quantitative effect of superficial fluidizing velocity are lacking, and as a result no valid quantitative statement can be made regarding this effect as a result of this work.

4. Feed Composition

The original feed used in the six-inch calciner simulated aluminum fuel waste and was made up to the following composition:

Aluminum nitrate, M	2.2
Nitric acid, M	1.25
Sodium nitrate, M	0.015
Mercuric nitrate, M	0.008

In January, 1959, the aluminum nitrate concentration was reduced to 1.95M, the sodium nitrate concentration was changed to 0.089M, and the mercuric nitrate concentration was changed to 0.006M to agree with revised estimates of the content of ICPP waste storage tanks. No significant effect of changing the aluminum nitrate concentration was observed, although no deliberate attempt was made to do so.

5. Product Pot Purge Air Rate

Some elutriation of fines from the product overflow back into the free space above the fluidized bed would be observed at sufficiently high purge air rates because the flow of this stream is countercurrent to the bed overflow. However, within the accuracy of normal screen analyses, this elutriation should have had a negligible effect on the size distribution of the solid product because the calculated diameter of a particle whose terminal settling velocity is equal to the highest overflow line purge air velocity (0.28 ft/sec) used is only 0.046 - 0.066 mm for bulk densities in the range 0.64 - 0.96 g/cc. In order to achieve a separation at 0.147 mm (100 Tyler mesh) for a bulk density of 0.8 g/cc, a purge velocity of approximately 1.4 ft/sec would be required.

B. Effect of Equipment Variation

Early in the program the operability of the six-inch calciner system was improved by using cartridge heaters, by changing the support plate, by replacing the bayonet filters with a cyclone, and by other equipment changes. However, a direct effect on the calciner dependent variables was seldom noted, because of the generally poor operability.

Once good operability had been achieved, two effects of equipment variation were noted and are given below.

1. Dry Fines Return

Because fine solids which would otherwise leave the system are returned to the bed through the dry fines return jet on the dust discharge of the primary cyclone, a finer bed was observed (at any given NAR) when the dry fines return system was operating than otherwise. Figure 8 shows this effect in a plot of mass median particle diameter as a function of NAR, with and without dry fines return. A higher concentration of solids smaller than three microns in the off gas leaving the primary cyclone was observed with dry fines return, because the increased quantity of solids of this size produced by attrition in the jet and cyclone are not removed appreciably by the primary cyclone. Figure 9 shows this effect.

2. Nozzle Air Annulus Size

The nozzle normally used to introduce the liquid feed to the six-inch calciner was a Spraying Systems Company Type 1/4 J Nozzle, Setup No. 2, consisting of a No. 2850 water nozzle and a No. 70 air nozzle. The liquid was introduced through a centrally-located orifice having a nominal diameter of 0.028 inch, and the atomizing gas stream was introduced through an annular orifice (concentric to the water orifice) having a nominal inside diameter of 0.050 inch and a nominal outside diameter of 0.070 inch. During maintenance on the calciner, the air orifice was inadvertently burred and subsequently repaired by enlarging it

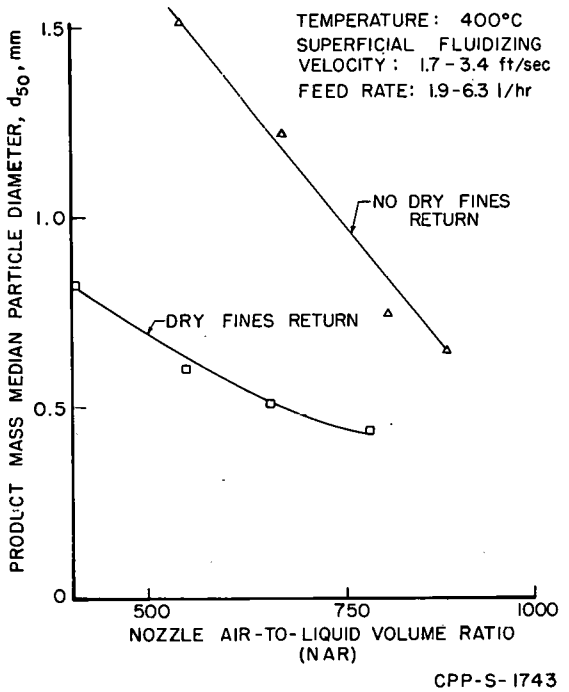

Fig. 8 - Product Mass Median Particle Diameter as a Function of NAR, Showing the Effect of Dry Fines Return

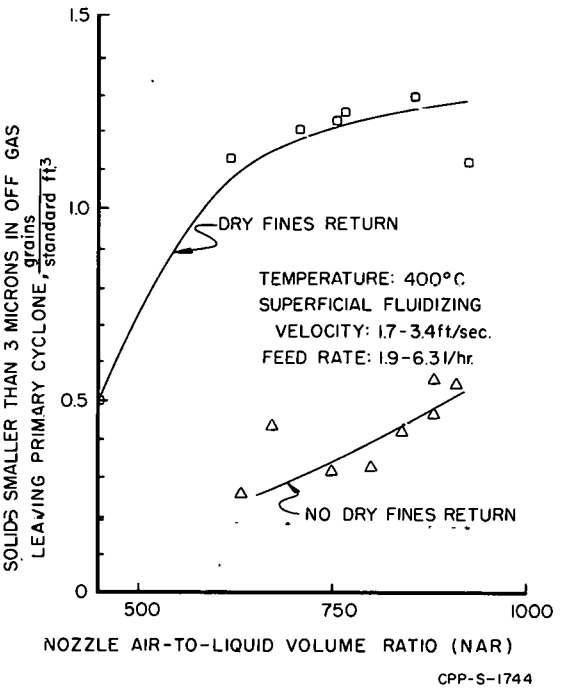

Fig. 9 - Concentration of Fine Solids in the Off Gas Leaving the Primary Cyclone as a Function of NAR, Showing the Effect of Dry Fines Return

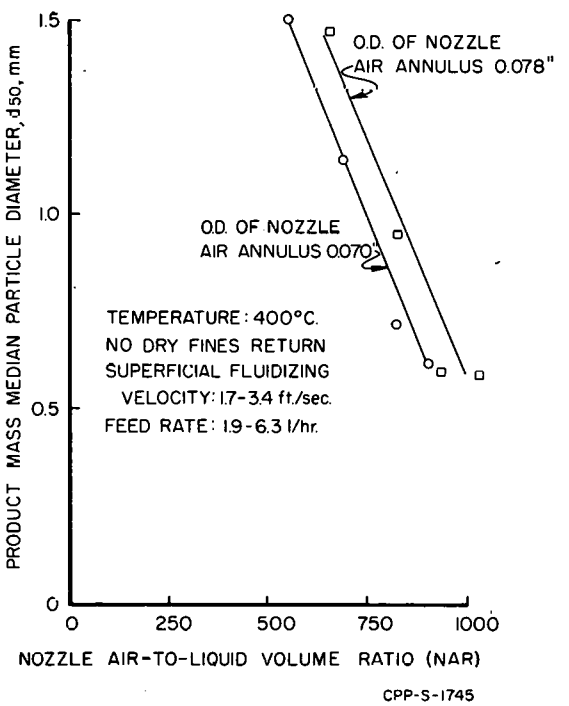

Fig. 10 - Product Mass Median Particle Diameter as a Function of NAR, Showing the Effect of Nozzle Air Annulus Size

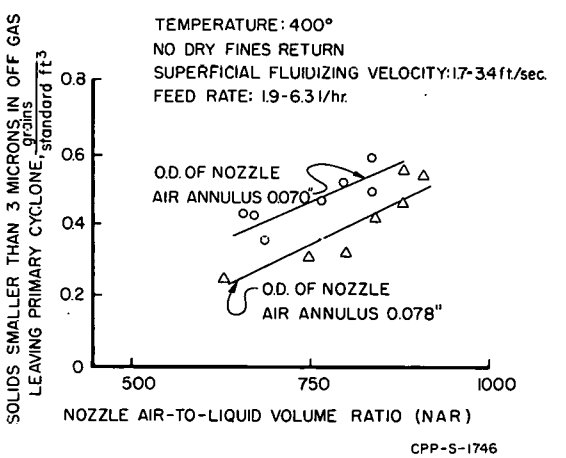

Fig. 11 - Fine Solids Loading in the Off Gas Leaving the Primary Cyclone as a Function of NAR, Showing the Effect of Nozzle Air Annulus Size

to 0.078-inch outside diameter, increasing the area of the air orifice from 0.00188 square inch to 0.00281 square inch, or a factor of about 1.5. At a given NAR, this would decrease the slip velocity between the air stream and liquid stream and thus increase the average droplet size in the spray, because the slip velocity is a major factor in atomization from a pneumatic spray nozzle[9]. Figure 10 shows that a finer bed indeed is obtained with the smaller, unmodified air orifice than with the larger one.

With a smaller average droplet size resulting from the smaller, unmodified nozzle air annulus, there should have been a higher proportion of droplets which were small enough to be spray-dried before they collided with and coated particles in the bed. These spray-dried particles are very small and any increase in their production would be reflected by an increase in the loading of solids smaller than 3 microns in the off gas leaving the primary cyclone. Figure 11 shows, as expected, a higher fine solids loading in the off gas leaving the primary cyclone with the unmodified orifice than with the larger one.

C. Related Programs

1. Distribution of Ruthenium

Escape to the atmosphere of the various radioactive fission products in ICPP waste would create an unacceptable hazard. Ruthenium, of all the fission products, presents a unique problem, since its tetroxide (boiling point 135°C) is volatile at usual calcination temperatures (400 - 500°C). Findings at ANL[10] demonstrate a much-reduced volatility of ruthenium at 500°C as compared to 400°C, presumably because of the higher equilibrium concentration of non-volatile species at 500°C. Retention of at least part of the ruthenium in the product at 400°C is desirable if operation at this temperature is to be considered seriously. (With a given calciner, a higher throughput is possible at the lower bed temperature of 400°C, because of the greater driving force available for heat transfer to the bed.) Therefore, five tests at 400°C have been made with ruthenium-spiked feed in the six-inch calciner, and the distribution of ruthenium among the various effluent streams was determined. The total concentration of non-radioactive and radioactive ruthenium added was 0.054 mg/ml, a concentration estimated to be actually present in waste.

The first three ruthenium tests indicated that most of the ruthenium in the feed escaped with the wet scrubber stream. A fourth test (in which this stream was returned to the bed with the feed) indicated that at least 24 per cent of the ruthenium in the feed remained in the calcined product. However, this value was suspect because of a poor overall material balance on the ruthenium, most likely caused by failure of the feed pump during the test. A fifth test, in which the conditions were identical to those in the fourth test, was made in an attempt to determine more definitely the disposition of ruthenium. An

acceptable material balance was obtained, and all equipment operated satisfactorily throughout the run. The venturi scrubber stream was recycled to the scrubber with makeup water, and a fines return slurry at a rate of 20 per cent of the fresh feed rate was maintained. Under these conditions, 67 per cent of the total feed ruthenium remained with the solid product, and an additional 28 per cent of the feed ruthenium was recovered in the condensate from the off gas condenser, for a recovery of 95 per cent. The ruthenium in the particulate matter in the off gas leaving the condenser was found to be only 9.0×10^{-7} grains per standard cubic foot of off gas, equivalent to less than 0.03 per cent of that entering in the feed.

2. The Effect of Ammonium Ion

The batch process formerly used at the ICPP for reprocessing aluminum fuels employed Hexone (methyl isobutyl ketone) in the first-cycle solvent extraction system. The residual nitric acid in the dissolver product stream was neutralized with ammonium hydroxide because of the chemical attack of nitric acid on Hexone. For this reason, ammonium ion is present in the first-cycle aqueous waste from the batch process.

In order to ascertain operating difficulties which might arise during the calcination of a waste containing ammonium ion, ammonium nitrate was added to the feed for two tests at concentrations of 0.078 and 0.18 molar, values which were originally estimated to bracket the actual concentration. Analyses made at a later date have shown the actual values to be much higher. (The actual concentration is about 1.4 molar.) However, no problems resulted from the presence of these small amounts of ammonium ion in the feed during these runs.

3. Calcination of Zirconium and Stainless Steel Wastes

Most of the planned power and military reactors will use fuel elements containing zirconium or stainless steel. In fact, wastes from processing such fuels are already accumulating at the ICPP. The dissolution of zirconium-bearing fuel elements involves the use of hydrofluoric acid (STR process), and the dissolution of stainless-steel-bearing fuel elements requires dissolution by hydrochloric acid-nitric acid mixtures with subsequent chloride removal (Darex process) or by sulfuric acid (SIR process). Electrolytic dissolution in nitric acid is also being extensively investigated at present for use with stainless steel fuel elements.

As a means of anticipating operating difficulties as a result of calcining wastes from these processes, simulated wastes from the first three processes mentioned have been calcined. The results of these tests indicate that these wastes can be successfully converted to solids in a fluidized bed calciner. The runs with simulated STR waste and simulated SIR waste were of only a few hours duration and no extensive data were obtained. However, a 360-hour run was made with simulated Darex waste and the data

indicated some characteristics of the granular solid product which are directly attributable to the type of feed. The product bulk density steadily increased during the run to a high value of 1.56 g/cc and was still increasing at the termination of the run. This compared to an average product bulk density of about 0.8 g/cc which has been observed during calcination of aluminum waste. The operation of the calciner throughout these runs was trouble-free.

4. Calcined Waste for Corrosion Studies

The final run in the six-inch calciner was made to produce 36 kilograms of calcined waste suitable for storage container corrosion studies. The feed for this run contained rare earth-group and platinum-group metal salts in quantities adequate to simulate the fission product content of actual ICPP waste.

This run presented no unusual operating problems.

5. Drying Nickel Ferrocyanide Slurry

Radioactive cesium may be precipitated selectively from a waste by the addition of nickel ferrocyanide. Drying a thickened slurry of this type on sand by the fluidized bed calcination technique destroys the ferrocyanide complex and converts the cesium to a leachable form. A number of tests made indicated the feasibility of this method. However, this approach was abandoned in favor of the more promising approach of direct leaching of the calcined solids[11].

VI. CONCLUSIONS

The following conclusions result from the operating experience with the six-inch pilot plant calciner:

1. An operable fluidized bed calciner for converting aqueous aluminum nitrate solutions to granular aluminum oxide was developed.

2. The calciner was operable at temperatures from 185 to 550°C, and at superficial fluidizing velocities from 0.6 to 4.5 feet per second.

3. The nozzle air-to-liquid volume ratio (NAR) was found to be a major operating variable. The calciner could be made operable with reasonable values of the other independent variables by adjusting the NAR.

4. The samples of solid product continuously overflowing to a sample pot were representative of the entire bed, within the accuracy of ordinary screen analyses.

5. When the dry fines return system was in operation, the mass median particle diameter of the bed was smaller and the loading of solids finer than three microns in the off gas leaving the primary cyclone was higher than when it was not in operation.

6. A larger product mass median particle diameter for the same value of NAR resulted from enlarging the air orifice on the pneumatic spray nozzle.

7. A run at 400°C made with ruthenium-spiked feed has shown that 67 per cent of the ruthenium introduced in the feed remained with the solid product, 28 per cent accumulated in the liquid stream from the condenser, and 5 per cent was not accounted for. This run showed that routing a fines return slurry to the calciner with the feed causes a good deal more ruthenium to remain with the solid product than otherwise.

8. The presence of 0.18 molar ammonium ion or traces of cesium, strontium, and ruthenium in the feed had no observable effect on the operation of the calciner.

9. Zirconium and stainless steel wastes can be calcined to granular solid material.

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TABLE 1

DATA SUMMARY

Run Number	Special Features of Run	Bed Temperature (°C)	Superficial Fluidizing Gas Velocity (ft/sec)	Average Feed Rate (l/hr)	Average Product Rate (kg/hr)	Length of Run (hr)	Nozzle Air-to-Liquid Volume Ratio*	Average Product Bulk Density (g/cc)	Concentration of Fine Solids in Off Gas Leaving Primary Cyclone (grains/ft^3)	Vessel Pressure (psig)	Final Product Size Distribution (per cent retained on screen) (Tyler Mesh) +14	+20	+28	+35	-60	+100	-100	Final Mass Median Particle Diameter (mm)
1 - 6	Short exploratory runs	~ 400	----	----	----	-----	----	----	----	-----	----	----	----	----	----	----	----	----
7	----	400 - 450	----	6.0	----	-----	----	----	----	2 - 6	----	10.7	----	25.5	56.3	3.3	4.2	0.41
8	Feed was simulated STR waste	375 - 400	0.90	4.8	0.46	3.7	210	----	----	2 - 6	----	2.8	----	64.2	31.3	1.7	----	0.45
9	Calcination of $Ni_2Fe(CN)_6$ slurry	450	----	7.0	----	1.0	----	----	----	2 - 6	----	0.1	----	0.8	21.7	74.2	3.2	0.21
10	Feed was simulated STR waste	375 - 400	1.57	2.7	----	5.0	----	----	----	2 - 6	----	1.0	----	61.5	24.5	13.0	----	0.41
11	----	400	1.60	3.0	----	8.5	400	----	----	2 - 6	----	----	----	----	----	----	----	----
12	First run with automatic temperature control	400	1.14	3.0	0.34	11.0	----	----	----	-----	----	----	----	----	----	----	----	----
13	$Ni_2Fe(CN)_6$ slurry	310	0.60	2.4	0.35	17.7	----	----	----	2 - 6	----	19.7	----	43.5	25.5	10.0	1.3	0.50
14	----	400	----	2.7	----	9.0	----	----	----	2 - 6	----	----	----	0.2	34.3	43.7	1.8	0.25
15	$Ni_2Fe(CN)_6$ slurry	310	0.92	4.2	----	9.0	----	----	----	2 - 6	----	----	----	----	----	----	----	----
Test	----	350	----	3.6	----	6.0	----	0.74	----	2 - 6	----	1.5	----	52.5	43.2	2.4	0.4	0.41
16	$Ni_2Fe(CN)_6$ slurry	400	0.92	2.0	----	4.5	----	----	----	2 - 6	----	----	----	----	----	----	----	----
17	Feed contained Cs-137 tracer	405	1.83	4.4	0.44	11.5	----	----	----	2 - 6	----	24.2	----	74.6	0.7	0.2	0.3	0.60
18	$Ni_2Fe(CN)_6$ slurry	450	1.72	4.6	0.23	9.7	----	----	----	2 - 6	----	----	----	----	----	----	----	----
19	$Ni_2Fe(CN)_6$ slurry	470	1.77	4.9	0.25	6.0	----	----	----	2 - 6	----	----	----	----	----	----	----	----
20	----	400	1.60	3.3	0.44	14.5	----	----	----	2 - 6	----	0.1	----	74.1	21.4	3.0	1.4	0.43
21	Feed was simulated SIR waste	400	1.37	4.4	----	11.5	----	----	----	2 - 6	----	----	----	----	----	----	----	----
22	Cs-137 tracer	360	1.08	4.0	0.51	13.0	----	----	----	2 - 6	----	1.0	----	81.1	15.3	1.7	0.9	0.46
23	----	360	1.50	4.0	0.36	35.7	----	0.77	----	2 - 6	----	29.6	----	66.9	1.8	0.3	1.4	0.62
24	----	360	1.08	3.5	0.56	8.0	----	0.88	----	1	----	14.3	----	46.2	26.0	11.7	1.8	0.47
25	----	500	1.32	3.5	0.43	29.7	325	0.58	----	2 - 6	----	91.0	----	7.5	1.5	----	----	0.93
Test	Attempt to form dibasic aluminum nitrate	185	0.78	4.7	0.74	16.0	----	1.15	----	2 - 6	----	4.5	----	61.4	25.3	7.4	1.4	0.43
26	Cs-137 tracer	400	1.30	3.5	0.48	46.5	537	0.71	----	2 - 6	----	3.2	----	53.5	36.8	6.0	0.5	0.42
27	----	300	1.12	3.6	0.36	49.5	519	0.95	----	2 - 6	7.1	73.4	----	18.9	----	0.6	----	0.90
28	----	400	1.30	3.8	0.42	74.0	494	0.77	----	2 - 6	----	4.8	----	60.6	26.1	8.5	----	0.45
29	Purge air was 0.28 ft/sec	300	1.12	3.6	0.40	66.0	519	1.10	----	2 - 6	----	3.0	----	19.9	23.9	34.3	18.9	0.28
30	----	300	1.12	3.7	0.48	67.0	502	0.96	----	2 - 6	1.0	83.5	----	15.5	----	----	----	0.90
31	Purge air was 0.28 ft/sec	500	1.50	3.6	0.48	28.0	354	0.62	----	2 - 6	0.9	4.9	34.3	20.4	24.3	10.9	4.3	0.50
32	----	400	1.30	2.3	0.30	51.7	799	0.75	----	2 - 6	0.6	2.9	9.0	22.4	43.2	12.4	9.5	0.37
33	First run with bubble-cap distributor plate	400	1.30	2.1	0.28	22.0	878	0.77	----	2 - 6	----	----	----	----	----	----	----	----
34	Cs-137 and Sr-90 tracer in feed	400	1.30	3.7	0.47	112.0	510	0.73	----	2 - 6	----	----	2.9	26.5	52.4	12.7	5.5	0.34
35	----	400	1.30	3.6	0.45	110.0	537	0.77	----	2 - 6	----	----	2.5	28.9	53.9	10.8	3.9	0.35
36	----	400	1.30	1.7	0.20	108.0	1074	0.81	----	2 - 6	----	----	13.5	24.2	33.7	14.6	14.0	0.36
37	First run with external cyclone and jet	400	1.72	3.6	0.35	242.0	468	0.73	----	3	----	4.0	14.2	31.5	38.4	10.6	1.3	0.43
38	----	400	1.72	1.9	0.18	202.0	666	1.04	----	3	----	2.7	16.5	30.3	36.1	10.8	3.6	0.42
39a	----	400	1.72	1.9	0.19	176.0	734	1.01	----	3	----	8.5	27.7	28.5	22.2	9.3	3.8	0.50
b	----	400	1.72	3.0	0.33	110.0	466	0.81	----	3	----	3.2	14.7	35.4	36.8	8.3	1.6	0.44
40	----	400	----	6.3	0.69	92.0	----	0.75	----	3	----	10.5	32.0	41.6	15.5	0.4	----	0.75
41a	----	400	1.72	4.0	0.39	87.0	365	0.70	----	3	45.6	15.6	21.4	12.4	4.0	0.7	0.3	1.54
b	----	400	2.60	5.7	0.53	14.0	254	0.70	----	3	31.6	54.5	12.1	1.4	0.4	----	----	1.02
c	----	400	3.40	6.0	0.72	352.0	247	0.78	----	3	19.1	27.5	27.5	19.2	6.3	0.2	0.2	0.82

* Air volume calculated at metered temperature and calciner pressure.

DATA SUMMARY (continued)

Run Number	Special Features of Run	Bed Temperature (°C)	Superficial Fluidizing Gas Velocity (ft/sec)	Average Feed Rate (l/hr)	Average Product Rate (kg/hr)	Length of Run (hr)	Nozzle Air-to-Liquid Volume Ratio*	Average Product Bulk Density (g/cc)	Concentration of Fine Solids in Off Gas Leaving Primary Cyclone (grains/ft^3)	Vessel Pressure (psig)	Final Product Size Distribution (per cent retained on screen) (Tyler Mesh) +14	+20	+28	+35	+60	+100	-100	Final Mass Median Particle Diameter (mm)
42a	Ruthenium tracer in feed	400	2.15	4.8	0.40	217.0	300	0.93	----	3	0.9	5.2	16.3	38.0	34.0	4.4	1.2	0.47
b		400	1.50	4.6	0.49	108.0	313	0.84	----	3	1.4	5.4	25.9	41.9	21.9	2.2	1.3	0.52
43a	----	400	2.82	3.6	0.30	62.0	472	0.91	----	3	T	2.0	5.3	35.9	48.8	7.0	1.0	0.39
b	----	400	2.82	8.2	0.80	21.0	207	0.78	----	3	5.5	33.4	45.2	13.4	2.5	-----	----	0.78
c	----	400	3.33	4.6	0.46	548.0	368	0.79	----	3	T	6.9	20.6	47.9	23.5	1.1	T	0.51
44a	----	400	3.45	5.3	0.55	48.0	318	0.77	----	3	T	9.1	35.7	41.9	12.8	0.5	----	0.58
b	----	400	3.45	3.7	0.34	48.0	328	0.83	----	3	T	1.4	26.3	53.0	18.8	0.5	----	0.52
c	----	350	2.75	5.2	0.51	48.0	264	0.89	----	3	13.6	75.9	9.0	1.3	0.2	----	----	0.98
d	----	350	2.75	3.6	0.27	48.0	378	0.94	----	3	1.2	13.5	38.9	27.9	15.4	2.0	1.1	0.60
e	----	400	2.75	5.4	0.55	48.0	315	0.80	----	3	T	1.4	22.8	51.9	22.7	1.2	T	0.49
f	----	400	2.75	5.3	0.54	48.0	255	0.80	----	3	----	2.3	21.6	51.7	23.2	1.2	T	0.48
g	----	350	3.45	5.1	0.45	48.0	333	0.88	----	3	4.5	23.7	28.9	26.6	14.6	1.2	0.5	0.66
h	----	350	2.75	5.3	0.44	48.0	482	0.91	----	3	0.4	2.6	17.7	37.7	36.4	4.4	0.8	0.45
i	----	400	2.75	3.7	0.33	55.0	453	0.80	----	3	----	0.5	9.8	50.0	36.0	3.0	0.7	0.43
j	----	400	2.75	4.3	0.43	48.0	397	0.75	----	3	----	6.7	42.8	38.8	11.3	0.4	----	0.58
k	----	400	3.45	3.6	0.33	50.0	474	0.79	----	3	----	3.9	27.4	44.9	23.2	0.6	T	0.48
l	----	350	2.75	4.3	0.37	51.0	398	0.87	----	3	75.9	12.4	4.7	3.5	3.3	0.2	----	1.14
m	----	350	3.45	3.6	0.33	44.0	477	0.99	----	3	1.8	27.7	33.9	18.7	13.8	1.5	2.6	0.66
n	----	350	3.45	4.8	0.43	14.0	356	0.88	----	3	3.9	2.5	58.4	22.6	6.7	2.8	3.1	0.70
o	----	400	3.45	5.5	0.60	46.0	310	0.88	----	3	24.5	27.7	30.7	13.3	3.6	0.2	----	0.87
p	----	350	3.45	5.4	0.58	46.0	470	0.89	----	3	5.2	10.9	28.7	38.3	15.9	1.0	----	0.62
45a	Ruthenium tracer in feed	400	2.6	3.3	0.28	47.0	361	0.99	----	3	2.1	8.2	21.7	37.1	22.4	4.6	3.9	0.52
b		400	2.6	5.3	0.56	43.0	318	0.82	----	3	<5.0	5.9	32.9	37.6	17.8	0.8	T	0.59
c		400	3.0 - 4.5	5.8 - 1.1	----	57.0	---	-----	----	-----	----	----	----	----	----	----	----	----
46a	First run with venturi scrubber and associated cyclone	400	2.42	4.4	0.38	35.0	230	0.72	0.15	8	55.0	44.0	1.0	----	----	----	----	1.13
b		400	2.81	4.1	0.41	64.0	455	0.83	0.31	7	T	T	7.4	43.0	39.7	8.1	1.8	0.44
c		400	2.42	1.8	0.15	70.0	450	----	0.15	8	2.5	4.2	31.0	37.3	19.2	2.9	2.9	0.54
d		400	2.23	1.9	0.16	63.0	300	0.85	0.31	8	35.8	55.8	6.4	T	T	T	2.0	1.05
e		400	2.23	1.8	0.18	05.0	667	1.00	0.11	10	T	2.9	35.4	26.7	21.9	4.7	8.4	0.49
f		400	2.23	3.2	0.36	96.0	400	0.85	0.09	10	T	0.8	18.0	49.5	28.4	2.7	0.6	0.47
g		400	2.23	3.0	0.29	07.0	587	0.78	0.06	7	T	T	2.8	22.9	50.4	17.1	6.8	0.33
h		400	2.23	4.1	0.41	92.0	332	0.65	----	8	2.8	20.4	39.8	26.5	10.0	0.5	T	0.64
Wlr-1a	Oil-fired fluidizing air preheater and U. S. Hoffman design cyclone installed	400	2.04	5.5	0.59	84.0	340	0.69	----	8	18.0	27.2	27.7	15.1	9.4	1.8	0.8	0.78
Wlr-1b		400	2.30	5.0	----	90.0	300	----	----	12	----	----	----	----	----	----	----	----
Wlr-2	----	400	2.30	3.6	0.30	53.0	671	0.72	0.96	8	2.5	8.2	31.1	32.1	21.6	3.7	0.8	0.57
Wlr-3	----	390	2.46	1.7	0.14	29.0	558	0.69	0.23	6	0.7	18.7	64.7	14.1	1.8	T	T	0.69
Wlr-4a	Nozzle air annulus O.D. changed to 0.078"	400	2.46	2.4	0.19	34.0	526	0.64	0.18	6	8.4	46.1	30.3	12.8	2.4	T	T	0.83
Wlr-4b		400	2.62	2.5	0.21	50.0	543	0.64	0.21	5	98.2	1.8	----	----	----	----	----	1.26
Wlr-4c		400	2.55	2.3	0.19	35.0	704	0.66	----	6	41.7	35.3	13.9	7.9	1.2	----	----	1.01
Wlr-5	Ruthenium tracer in feed	400	2.56	2.5	0.17	02.0	841	0.59	0.34	6	34.5	36.0	21.5	6.8	1.2	----	----	0.96
Wlr-6a	----	400	2.27	2.6	0.19	38.0	719	0.63	0.24	8	34.2	33.3	18.9	9.2	4.4	----	----	0.95
Wlr-6b	----	400	2.07	4.4	0.36	21.0	557	0.61	0.45	10	52.7	17.7	17.6	8.9	3.1	----	----	1.02
Wlr-7	----	400	2.24	2.4	0.16	48.0	769	0.63	----	8	17.8	25.7	32.8	18.1	5.6	----	----	0.81
Wlr-8	Conventional cyclone re-installed	400	2.24	2.7	0.20	25.0	692	0.64	0.12	8	10.2	36.6	40.6	11.2	1.4	----	----	0.83
Wlr-9	----	400	2.24	2.4	0.14	66.0	770	0.70	----	8	14.5	26.6	32.5	18.1	7.6	T	0.7	0.78
Wlr-10	----	400	2.32	2.5	0.18	26.0	426	0.68	0.17	7	57.3	38.6	3.2	0.5	0.1	T	0.3	1.13

* Air volume calculated at metered temperature and calciner pressure.

DATA SUMMARY (continued)

Run Number	Special Features of Run	Bed Temperature (°C)	Superficial Fluidizing Gas Velocity (ft/sec)	Average Feed Rate (l/hr)	Average Product Rate (kg/hr)	Length of Run (hr)	Nozzle Air-to-Liquid Volume Ratio*	Average Product Bulk Density (g/cc)	Concentration of Fine Solids in Off Gas Leaving Primary Cyclone (grains/ft^3)	Vessel Pressure (psig)	Final Product Size Distribution (per cent retained on screen) (Tyler Mesh) +14	+20	+28	+35	+60	+100	-100	Final Mass Median Particle Diameter (mm)
W1r-11a	----	400	2.31	2.6	0.22	60.0	636	0.69	----	7	34.8	30.5	19.3	10.3	4.2	0.4	0.5	0.94
W1r-11b	----	400	2.32	2.5	0.22	28.0	602	0.70	0.12	7	45.8	25.0	16.3	8.4	3.8	0.3	0.4	0.99
W1r-11c	----	400	2.27	2.5	0.21	23.0	530	0.70	0.12	8	66.5	22.7	6.7	2.4	1.7	----	----	1.12
W1r-11d	----	400	2.32	2.5	0.18	86.0	780	0.70	0.13	7	21.4	31.0	26.2	12.5	5.0	0.7	3.2	0.84
W1r-11e	----	400	2.34	2.5	0.23	34.0	896	0.70	0.11	7	28.4	27.1	20.8	15.0	6.2	1.2	1.3	0.86
W1r-12a	Electric fluidizing air preheater installed	400	2.55	2.4	0.18	112.0	1036	0.54	0.38	6	----	11.2	40.9	32.0	14.7	1.2	----	0.59
W1r-12b		400	2.32	2.3	0.17	43.0	969	0.58	0.35	7	0.6	21.7	44.3	22.7	8.4	2.3	----	0.67
W1r-12c		400	2.86	2.3	0.17	136.0	942	0.60	0.51	8	1.5	13.7	41.1	30.6	11.5	1.6	----	0.62
W1r-12d		400	2.48	2.5	0.23	38.0	639	0.62	0.45	8	26.6	46.7	14.0	4.7	6.6	1.4	----	0.94
W1r-13a	----	400	2.67	2.4	0.24	58.0	688	0.65	0.28	8	62.5	12.7	10.6	9.9	4.1	0.2	----	1.05
W1r-13b	----	400	2.48	2.3	0.24	129.0	828	0.58	0.37	8	29.0	31.6	22.0	11.6	5.0	0.8	----	0.90
W1r-14	Nozzle air annulus O.D. changed to 0.070"	400	2.65	2.4	0.23	110.0	821	0.61	0.51	8	10.1	26.0	33.7	19.3	9.3	1.6	----	0.65
W1r-15a	----	400	2.78	2.6	0.12	38.0	892	0.54	0.68	4	----	17.8	44.7	29.4	5.9	2.2	----	0.66
W1r-15b	----	400	2.84	2.5	0.24	147.0	668	0.60	0.48	4	51.7	20.8	14.5	8.0	4.1	0.9	----	1.01
W1r-15c	----	400	3.26	2.4	0.18	221.0	932	0.54	0.48	4	27.2	30.1	25.8	10.6	4.1	2.2	----	0.88
W1r-15d	----	400	3.20	----	----	96.0	----	----	0.29	4	16.8	24.7	31.4	16.5	5.6	5.0	----	0.79
W1r-15e	----	400	2.89	2.6	0.23	41.0	551	0.61	----	6	70.9	14.8	5.4	4.4	2.3	2.2	----	1.11
W1r-16	----	400	2.83	3.3	0.24	39.0	558	0.73	----	5	39.4	29.9	13.6	8.7	3.0	5.4	----	0.95
W1r-17a	----	400	2.80	2.8	0.27	48.0	664	0.79	----	5	3.5	7.2	18.2	26.2	10.3	22.3	12.3	0.45
W1r-17b	----	400	2.76	3.0	0.30	45.0	555	0.79	----	6	4.9	16.7	29.6	18.5	6.4	15.8	8.1	0.58
W1r-17c	----	400	3.01	2.7	0.29	18.0	795	0.82	----	4	3.7	7.9	17.8	22.4	18.9	16.3	13.0	0.46
W1r-17d	----	400	2.50	3.3	0.29	24.0	400	0.73	----	7	11.4	32.7	47.5	8.0	----	0.4	----	0.83
W1r-18	----	400	1.72	3.9	0.38	312.0	592	0.77	----	7	----	----	6.6	43.0	45.2	5.2	----	0.41
W1r-19	----	400	2.27	3.6	0.37	61.0	528 → 805	0.72	----	7	33.2	51.2	9.5	3.9	2.2	-----	----	1.01
W1r-Spl-1	Superheated steam used as fluidizing medium	400	2.03 → 2.43	4.5	0.32	380.0	346 → 800	0.77	----	7	1.0	----	15.8	53.8	24.5	3.9	1.0	0.47
W1r-20	Uranium spike in feed for particle growth rate studies	400	2.43	3.5	0.28	600.0	743	0.77	----	7	----	----	1.6	15.9	67.2	15.0	0.3	0.32
W1r-21	Ruthenium tracer in feed	400	2.43	3.7	0.20	408.0	605	0.79	----	7	----	----	6.1	49.6	41.6	2.1	0.6	0.42
W1r-Spl-2	Feed was simulated stainless steel waste	400	2.43	4.7	0.16	360.0	476	1.13	----	7	----	1.3	20.1	35.6	24.1	15.3	3.6	0.43
W1r-Spl-3	Calcined waste for corrosion studies	400	2.64	3.8	0.23	96.0	573	0.82	----	7	0.2	0.1	4.7	44.5	43.9	4.8	1.8	0.40

* Air volume calculated at metered temperature and calciner pressure.

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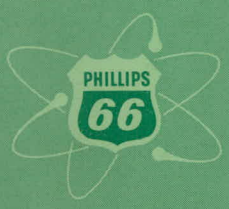

ATOMIC ENERGY DIVISION