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Drying of Uranium-Loaded Cation Exchange Resin with Microwave Heating

J. P. Drago
P. A. Haas

OAK RIDGE NATIONAL LABORATORY

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Refabrication Development - Task 300

DRYING OF URANIUM-LOADED CATION EXCHANGE RESIN

WITH MICROWAVE HEATING

J. P. Drago
P. A. Haas

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DRYING OF URANIUM-LOADED CATION EXCHANGE
RESIN WITH MICROWAVE HEATING

J. P. Drago
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ABSTRACT

The reference fuel kernel for recycle of ^{233}U to HTGRs (High-Temperature Gas-Cooled Reactors) is prepared by loading carboxylic acid cation exchange resin with uranium and carbonizing it at controlled conditions. The wet, uranium-loaded resin must be dried to a water content of 10 to 16 wt % prior to carbonization to minimize handling problems. Microwave heating was demonstrated to give controlled and reproducible dried resin in a vessel whose dimensions were safe for nuclear criticality (12.4 cm ID). A standard drying procedure was developed. The duration of microwave heating is controlled either by using an experimentally derived drying factor or by monitoring the amount of water removed from the resin. No significant difficulties were encountered in the operation of the dryer. Heat balance, microwave coupling efficiency, and minimum fluidization velocity were calculated and compared with experimental results or with the literature. Mixing of the resin during drying was required to ensure a uniformly dried product.

1. INTRODUCTION

Drying of cation exchange resins loaded with ^{233}U is required for the reference process of an HTGR fuel refabrication plant. The individual requirements for the drying process are not exceptional. However, the combination of requirements for controlled and uniform drying, remote operation in a radiochemical cell, control of nuclear criticality, and limited drying time are difficult to meet. Microwave heating of the washed resin appears to meet these requirements. This report covers the selection, design, and preliminary testing of a pilot-plant-scale dryer with microwave heating. The overall fuel preparation process is described briefly, and the requirements with respect to the dryer are reviewed in more detail. The dryer was designed to check the critical concepts or requirements for pilot plant use without being a prototype in all details.

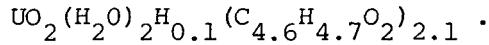
1.1 Preparation of HTGR Recycle Kernels

The reference fuel kernel for recycle of ^{233}U to HTGRs is prepared by loading carboxylic acid cation exchange resin with uranium and carbonizing it at controlled conditions. The reference resin used during these development studies was Amberlite IRC-72.* The optimum loading results are obtained with uranyl and hydrogen as the only cations in acid-deficient solutions of uranyl nitrate (NO_3^-/U mole ratios of <2). The purified $^{233}\text{UO}_2(\text{NO}_3)_2$ solution from a fuel reprocessing plant contains excess nitric acid (NO_3^-/U mole ratio of ~2.2). The reference flowsheet for a ^{233}U recycle fuel facility at Oak Ridge uses solvent extraction of nitrate by a 0.4 M secondary amine in a hydrocarbon diluent to prepare acid-deficient uranyl nitrate.¹ The amine nitrate is contacted with basic solutions (NaOH or NH_4OH) to regenerate free amine for reuse, and the nitrate is discharged in the form of waste solutions containing NaNO_3 or NH_4NO_3 . By adding an evaporator to remove water (Fig. 1), it is possible to obtain a highly efficient conversion of the purified $^{233}\text{UO}_2(\text{NO}_3)_2$ solution to loaded resin.

The carboxylic acid cation exchange resins are prepared by polymerizing acrylic or methacrylic acid with divinylbenzene. A typical composition for preparation of Amberlite IRC-72 may be:

- 10 wt % divinylbenzene
- 8 wt % ethylvinylbenzene
- 82 wt % acrylic acid.

From this composition and from analyses of uranyl-loaded resin, the average formula for resin dried to 110°C might be represented by:



Before the start of microwave heating, the drained resin retains about 12 molecules of additional water per atom of uranium in the porous resin structure.

*Trademark of the Rohm and Haas Company, Philadelphia, Pennsylvania.

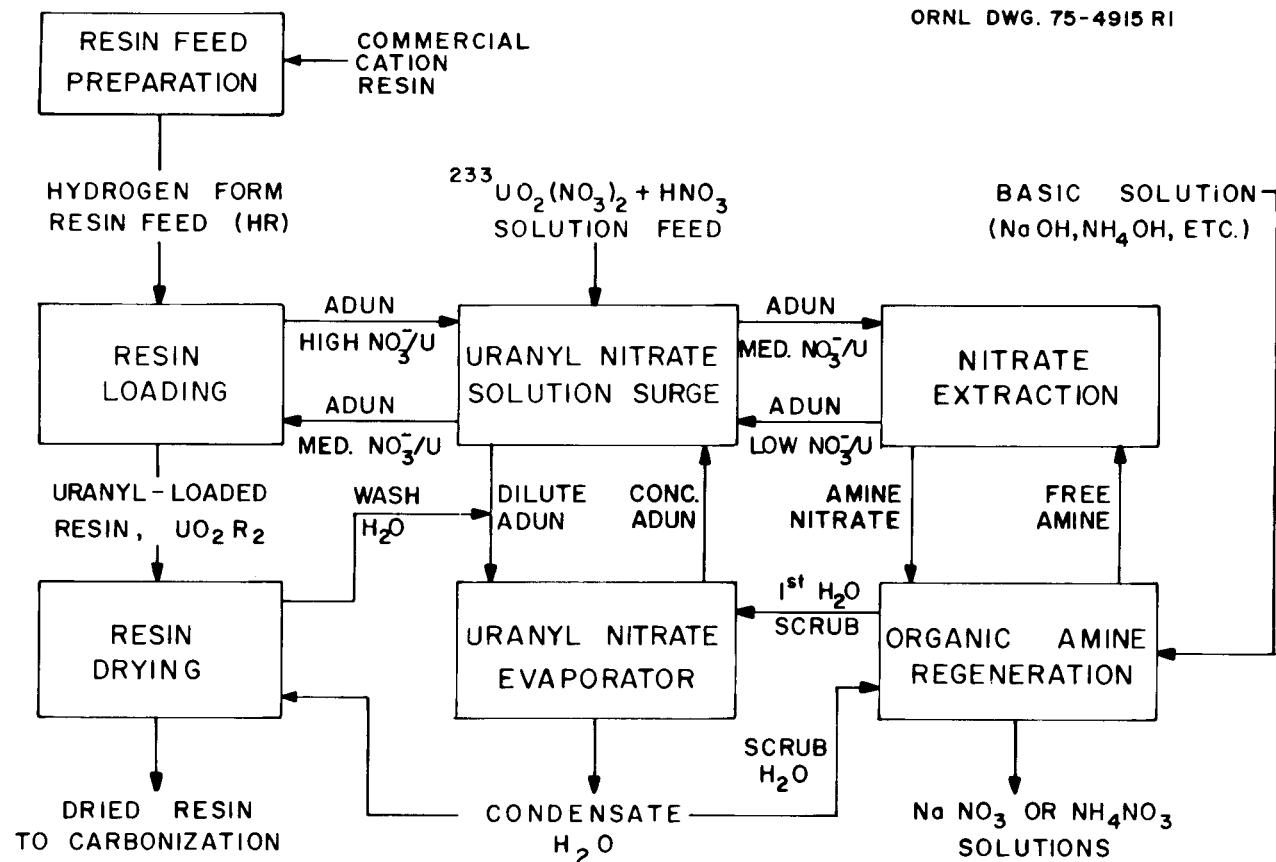


Fig. 1. Schematic reference flowsheet for FRPP resin loading.

1.2 Objectives of Dryer Development

The dryer is part of an engineering-scale resin loading system that is full scale with respect to the Hot Engineering Test equipment. The primary requirement of the dryer is to consistently produce dried resin of acceptable moisture content in order to allow pneumatic transfer of the material to the next processing step. The engineering-scale system provides information with respect to remote operation, the effects of criticality limitations, material accountability, and interactions of individual operations. The system does not meet the detailed requirements for containment, waste disposal, or shielding to allow operation with ^{235}U or ^{233}U and is therefore limited to natural uranium. The intention was to obtain information on concepts or requirements that are unique to the dryer without placing emphasis on requirements where generally accepted design solutions are known.

1.3 Characteristics of Microwave Heating

Microwaves are electromagnetic waves in the frequency range of 300 MHz to 300 GHz with corresponding wavelengths of 1 m to 1 mm. Microwaves have many of the same characteristics as light waves. They can be generated, reflected, transmitted, and absorbed, but there are basic differences in the materials that transmit and absorb them as well as in the manner in which each is generated.

Whereas the light bulb is a generator that produces light, the magnetron is a generator that produces microwaves. Microwaves, as well as light, are reflected by metallic objects. Materials such as glass, quartz, Teflon, silicon rubber, polycarbonate, and polypropylene are essentially transparent to microwaves. Water absorbs microwave energy while light is transmitted. Since water is one of the best absorbers of microwave energy, microwave heating is a potential aid for difficult drying processes.

When an electromagnetic wave is propagated in a dielectric material, the wave is attenuated. Power is thereby dissipated in the material in the form of heat. Water has certain molecular properties that enable the

material to absorb microwave energy. A water molecule is comprised of oxygen and hydrogen, and has a dipole moment. The charge of this molecule is asymmetrically arranged and the molecule is said to be polar. An electric field exerting a twisting force on a polar molecule attempts to align that molecule with the field. When the electric field is reversed, the molecule attempts to reverse its orientation; thus, frictional forces have to be overcome and the energy is dissipated as heat.

Microwave drying has several advantages over conventional heating which relies primarily on conduction-convection. These advantages include instant on-off capability, moisture leveling (i.e., preferential heating of the wettest material), and a shorter drying cycle without high temperatures. For this application with geometry restrictions, poor thermal conductivity of the resin, and uniformity requirement, microwave heating has satisfied our requirements better than any other methods considered.

1.4 Acknowledgments

These studies were part of the Thorium Utilization Program studies of the Chemical Technology Division. D. L. Million, R. D. Arthur, and T. V. Dinsmore participated in the experimental operation. Information from General Atomic Company personnel concerning their successful use of microwave heating to meet very similar requirements for resin-based fuel preparation was very helpful.

2. PROCESS REQUIREMENTS

The resin dryer must meet the requirements for ^{233}U -loaded resin in a fuel-recycle pilot plant using a reference flowsheet (Fig. 1). The dried resin must meet the chemical flowsheet requirements. The operating and control procedures must be acceptable for the remotely operated pilot plant. The accountability and criticality requirements for ^{233}U must be considered.

2.1 Capacity

The engineering-scale resin loading system is designed for batch loading of resin with 4 kg of uranium per batch. The pilot plant capacity is based on one batch per day, but the resin loading operations require 4 hr per batch or less. The nitrate extraction system is continuous with a capacity equivalent to at least 1 kg per hour of uranium. If the complete cycle for the resin dryer, which includes loading, washing, drying, and unloading, does not exceed 4 hr, a single dryer could serve a continuous nitrate extraction system with either multiple batch contactors or a continuous resin loading contactor.

The 4 kg of uranium per batch would require 11 to 13 liters of wet, loaded resin depending on the uranium loading per unit volume, which varies with the reference or alternate resin. About 2 kg of water would be evaporated to dry one batch of resin to a preferred water content (discussed in Sect. 2.2).

2.2 Dried Product Specifications

Specifications for the dried product have not yet been determined or verified. In general, the primary specifications are those for the carbonized and/or coated kernels; the dried, uranium-loaded resin must be suitable for carbonization, conversion, and coating to meet those primary specifications. The size, shape, sphericity, uranium content, and impurity content of the kernels are determined and controlled by resin feed preparation and resin loading operations. The most important specifications for drying of the resin are:

1. The shape of the kernels must not be degraded by cracking, clustering, or other changes during drying.
2. Nothing should be added that would add impurities to the carbonized kernels or that would result in uncontrolled variations in carbon and oxygen content after carbonization.
3. The water content of the dried resin should be within an optimum range for the requirements of accountability, handling, and carbonization operations.

The water content is generally determined as loss on drying (LOD) for overnight exposure to ambient air at 110°C. Specification of the water content for the dried, uranium-loaded resin is now 10 to 16% LOD. The primary requirement for accountability is that the LOD be reproducible and uniform. Treatment of the carbonization off-gases, including control of criticality, is simplified by minimizing the amount of water charged to the carbonization furnace. These two requirements resulted in an initial selection of <0.1 wt % LOD as a dried resin specification. At this level the dried resin shows static charge effects which make handling and transfer difficult. The resin literally climbs the walls of all surfaces exposed to it and cannot be poured or transferred pneumatically without leaving some of the particles behind. Handling tests were made after graphite powders were added to the resin, but this only alleviated the problem without eliminating it and is an undesirable process complication. Empirical tests showed that 10 to 20% LOD for uranium-loaded Amberlite IRC-72 results in acceptable handling properties. Static charge problems become increasingly noticeable as the LOD decreases below 10 wt %. The resin appears to become increasingly sticky from dampness as the LOD increases to about 20 wt %. The alternate resin, Duolite C-464,^{*} is slightly more porous and shows the same behavior at a slightly higher LOD, 12 to 24 wt % for optimum handling behavior.

2.3 Kinetic and Equilibrium Consideration

The uranium-loaded resins show typical behavior for drying of a porous solid. For a specified charge, microwave power input, and gas flow there is a short initial period during which the charge is heating up, a constant rate period during which the exit gas is saturated with water, and a declining rate as drying nears completion.

During the constant rate period, the equilibrium is determined by a thermal balance. The temperatures are constant with time, and the heat input equals the sensible heat of the air plus the heat of vaporization of the water. The exit temperature is that which gives saturated

* Trademark of the Diamond Shamrock Company, Redwood City, California.

air: that is, 100% relative humidity. The heat is generated throughout the resin particles. The gas is essentially transparent to microwave energy. The particles are slightly hotter than the gas. The temperature difference causes both heat transfer from the particles to the gas and mass transfer of water vapor.

As drying approaches completion, the drying rate decreases and the exit gas is not saturated with water. In this region, the resin has a lower vapor pressure of water in equilibrium with it than with the vapor pressure of free water at the same temperature. The water is held in the resin by capillary effects in small pores or by bonding. When the resin, which has been dried overnight at 110°C, is cooled to room temperature and is then added to room-temperature water, the resin will release enough heat to produce steam. Data reported by Carpenter² for exposure of dried resin at several relative humidities at room temperature provide an indication of both rates and equilibria (Fig. 2). Similar data for higher temperatures or for the removal of water are not available.

3. EQUIPMENT DESCRIPTION

A microwave energy system for drying uranium-loaded ion exchange resin was purchased from a commercial manufacturer* and tested. Features of the main components and service requirements are discussed.

3.1 Resin Vessel and Cavity

A schematic diagram illustrating the components of the microwave dryer system is shown in Fig. 3. The microwave cavity (applicator) is constructed of 0.16-cm-thick type 304 stainless steel that has dimensions of 1.2 m high x 1.2 m wide x 0.9 m deep. The resin container is a Pyrex tube, 13 cm OD x 0.3 cm wall x 1.2 m long. The column is a safe-geometry vessel with respect to nuclear criticality and provides ample free board to allow fluidization and mixing of the resin. At static conditions, the reference 11-liter resin batch occupies approximately 75% of the column. The width and depth of the cavity, as well as the location of the column,

* Gerling Moore Incorporated, Palo Alto, California.

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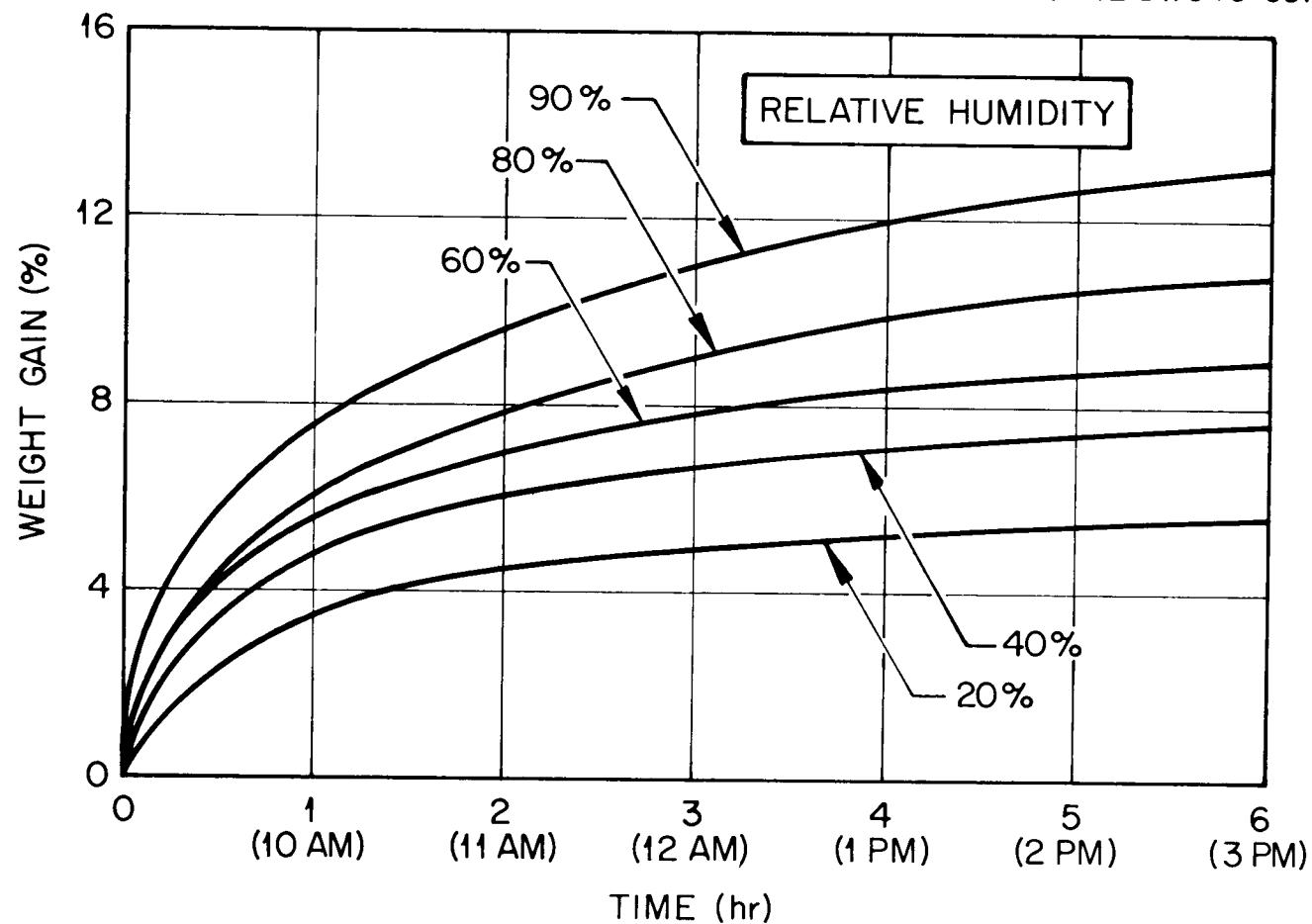


Fig. 2. Moisture pickup vs time at various relative humidities for loaded and dried Amberlite IRC-72 microspheres.

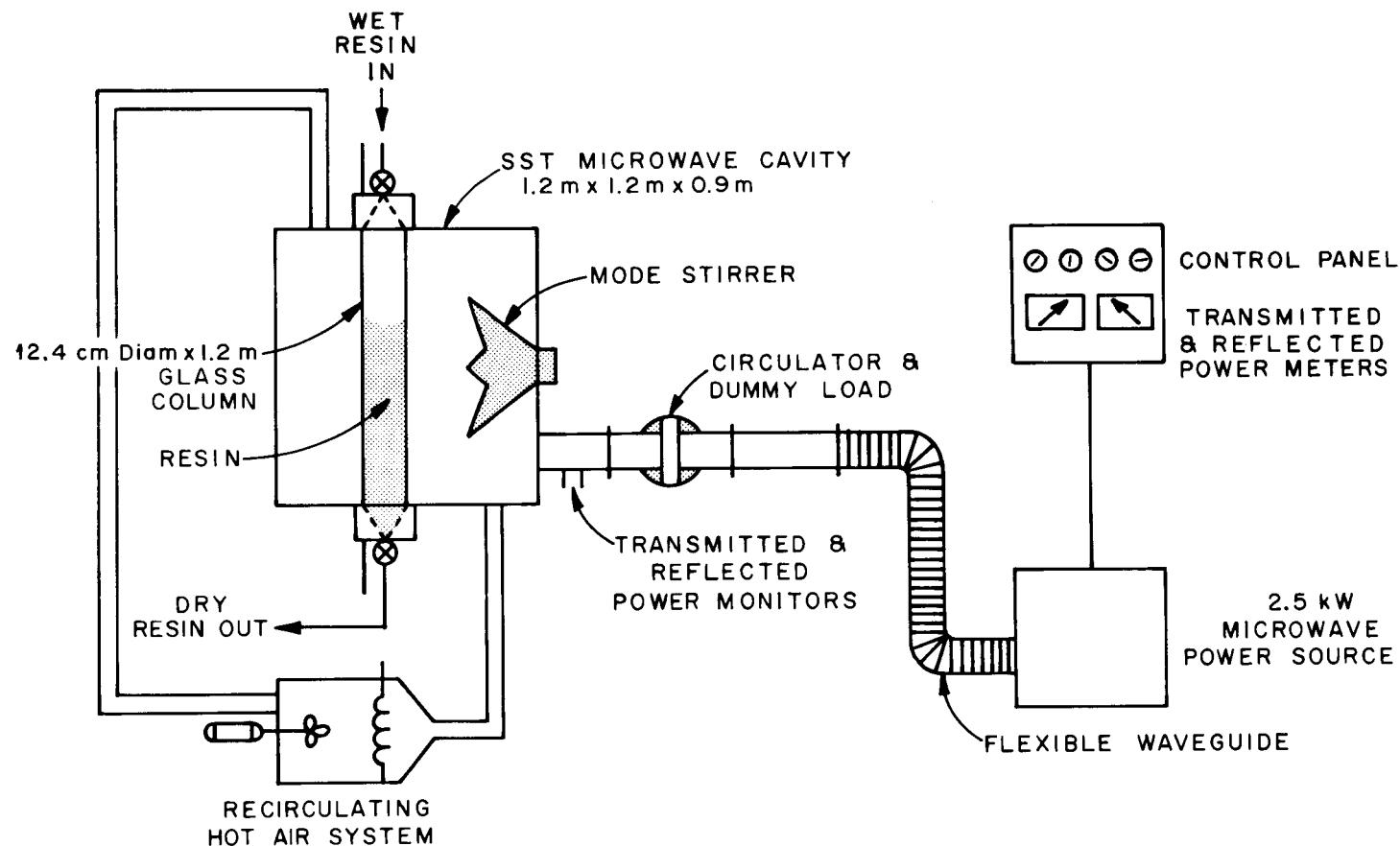


Fig. 3. Microwave heated dryer for uranium loaded, ion exchange resin.

were selected by the microwave system manufacturer. The cavity dimensions were chosen such that a large number of modes (electric field configurations) can exist at the frequencies near the operating frequency of the microwave source. The more modes a cavity has, the greater is the variety of electric field patterns that may be established in the cavity. This is desirable for uniformity of heating. However, when the energy is introduced into the cavity, the result is a stationary pattern of high and low intensities. To ensure that a large number of modes are excited, the microwave energy is directed on a blade of a rotating metallic device called a mode stirrer. This device continuously changes the microwave conditions in the cavity and varies the positions of the more intense zones such that the average conditions are uniform.

Provisions for solids addition and withdrawal (as well as liquid or vapor only, through screens) are incorporated in a special flange assembly (Fig. 4), which has a conical ($\sim 45^\circ$ slope), 120-mesh screen with a hole at the apex. A 1.9-cm ball valve is secured to the plate to allow retention and transfer of solids. Flange and screen are fabricated from type 304 stainless steel. During the drying cycle, instrument air is passed through the resin bed to aid in water vapor removal and to promote mixing of the material. The saturated air leaving the column is passed through a condenser and the condensate is collected. Thermocouples monitor temperatures of the gas streams entering and leaving the condenser. Four 0.64-cm-diam stainless steel tie rods secure the glass column and special end flanges in place. (Use of these metal rods inside the cavity is permitted, since they are securely fastened to the cavity walls; however, due to "shadowing effect," the number should be kept to a minimum.) Teflon gaskets and silicon rubber cement are used to seal the glass column to the flange since these materials are transparent to microwave energy.

When the load in a microwave cavity decreases, such as with the removal of water during a drying operation, both the current in the cavity wall and the voltage in the cavity increase. Depending on the input power level and load conditions, this energy may become sufficiently large to cause ionization of the air at certain locations in the cavity (e.g., sharp metallic points, sharp metallic edges and discontinuities, and gaps

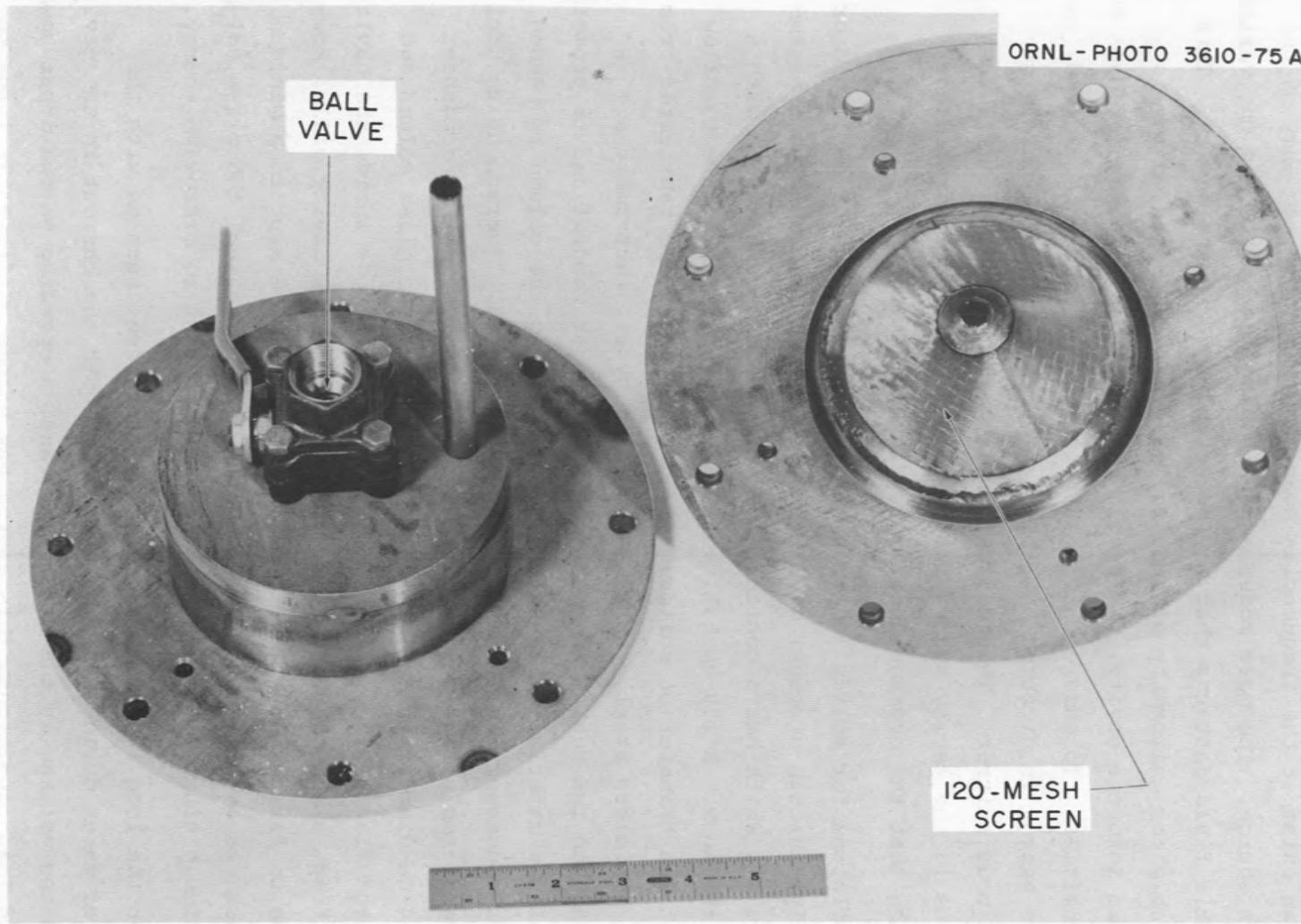


Fig. 4. End sections of resin dryer in microwave cavity.

between metal objects). At very high energy levels, arcing will cause spot welding at these locations. Arcing in the experimental apparatus was observed under no-load conditions at the gap where the tie rods are inserted through the flange assembly.

To reduce possible serious damage to the cavity, sharp metallic points and edges should be avoided. At metallic discontinuities, aluminum foil tape with a conductive adhesive such as 3-M Type 1170 is used. A special woven Monel gasket, Metex, was used between the special column-end flanges and the microwave cavity wall. This gasket is 1.9 cm wide with a 0.32-cm-diam bead of the woven material along each edge. Contact cement was used to secure the woven gasket on the bolt circle of the flange. Holes for the bolts were punched clean, and no stray wire ends remained. This gasket material was used along the bolt center lines of the removable view panel. This woven metal gasket is also available in stainless steel, called Technit.

A recirculating hot-air system reduces heat transfer from the resin column to the surroundings during the drying cycle. A 2-kW space heater and blower directs heated air into the cavity. The outside top and side walls of the cavity were insulated. The temperature inside the cavity is maintained at approximately the steady-state temperature of the column exit gas stream (typically, 70 to 80°C) during the microwave heating period. When this heater is not operated, water condenses on the glass column. The view port (20 cm x 100 cm) and flood-lamp ports are perforated screens of 0.40-cm-diam holes on 0.48-cm offset centers. These screens completely reflect the microwave energy.

3.2 Microwave Power Supply

The microwave power source (magnetron) is a continuously variable power generator with an operating range of 0 to 2.5 kW at 2.45 GHz. The 2.45-GHz band was selected over the 915-MHz band because the higher frequency produces a shorter wavelength, which results in more modes in a given cavity size and, consequently, more uniform heating. Standard rectangular, rigid waveguide, bends (elbows), and a 1 m length of flexible

waveguide were used to couple the power supply to the cavity. A quartz window diaphragm is located between the waveguide flange and the cavity wall to prevent material from entering the waveguide. Flexible waveguide is manufactured from silver-plated interleaved brass and can be fabricated in any length up to 1.5 m. This flexible waveguide can make a 90° twist or bend in 31 cm lengths. Standard rectangular waveguide is commonly fabricated from aluminum; however, stainless steel waveguide is available. The flexible waveguide cannot be fabricated from stainless steel.

To protect the power tube, a three-port circulator was installed to provide complete isolation between the reflected microwave power from the cavity and the power tube. The reflected power is directed by magnetically biased ferrites to the third port of the device where it is absorbed by the water-cooled dummy load. Since operation of the microwave transmitter and dummy load without coolant will result in damage to the system, a flow interlock requiring a minimum of 3.8 liters per minute of water is located in series between the dummy load and magnetron. Electrical requirements for the transmitter are 208-V, single phase, 60-Hz line having a minimum current handling capacity of 30 A.

Main features of the control panel include: microwave power timer, microwave on-time clock, magnetron current meter, microwave power adjust, power switches, and transmitted and reflected power meters. The transmitted and reflected power monitors are located in the waveguide segment connected to the cavity. A servo-controller was installed that was capable of operating in either of two modes, analog or limiting. In the analog mode, the unit receives an electrical signal that controls the output power level of the microwave transmitter between 0 and 2.5 kW. In the limiting mode, the transmitted power to the cavity is regulated such that the preset reflected power limit is not surpassed.

4. DRYING CYCLE AND MICROWAVE HEATING

In order to obtain the desired water content, a standard drying procedure and methods for determining the duration of microwave heating were developed. This procedure includes the transfer of the wet, uranium-loaded resin to the dryer, washing and draining the resin, microwave

heating, and cooldown prior to removal from the dryer. Instrument air is used during the heating period to aid in the removal of evaporated water from the resin and also to mix the material. A heat balance during the constant-rate drying period and the coupling efficiency of the microwave energy with the wet resin are discussed in this section. Uniformity and mixing of the resin are discussed in Sects. 5 and 6, respectively.

4.1 Charging the Dryer and Washing the Resin

Following the loading of the uranium on the resin, the fuel is transferred to the dryer as a slurry. The process solution, uranyl nitrate, is pumped into the top of the loading column and displaces the resin slurry through the bottom exit. The transfer may also be made by supplying 5 psig of air to the top of the loading column. The loading column and dryer are connected with 1.27-cm-OD stainless steel tubing. No clogging problems were observed during any transfers of the resin to the dryer. The glass column is vented to the process surge tank through the screen of the top flange.

After the transfer is completed, the resin is washed with the evaporator condensate that was collected during the loading operation. Upflow and downflow rinsing have been tested; however, the standard procedure uses downflow, since plug flow of the water through the bed is the more economical method of washing. Typically, 2 to 3 resin volumes of condensate are pumped down through the bed at 2 liters per minute, thus displacing the uranyl nitrate solution to the process surge tank.

The excess (interstitial) water remaining in the dryer column is then removed by downflow of instrument air at 35 slpm (standard liters per minute) for 10 min. This water is also sent to the process surge tank. This blowdown operation prior to microwave heating is used because it reduces the drying cycle time, but more important, it drains the resin to a reproducible moisture content (29 to 33 wt %).

4.2 Control of the Drying Cycle

One advantage in using microwave power for drying applications is that energy is preferentially absorbed by the wettest material. This is

due to the different dielectric properties of the water and the solid material. It was anticipated that this preferential heating would aid in achieving the desired water content uniformly throughout the batch. Operating signals and/or procedure to achieve this result were investigated. These include reflected power, temperature, drying factor, and the amount of water removed.

4.2.1 Reflected power

When all the incident energy of an electromagnetic wave is absorbed by a load, the load is said to be matched. There is no reflection from the load. However, when reflection takes place and interference of the incident and reflected waves occurs, only a proportion of the energy of the incident wave is absorbed by the load. The interference is constructive where regions of enhanced field intensity are produced and destructive in regions of reduced field intensity. Although the fields vary sinusoidally with time, when the spatial variation of field intensities remains stationary, this phenomenon is known as a standing wave pattern.

Standing waves are generally undesirable in applicators, since material exposed to the wave pattern is not uniformly heated; however, they provide a convenient means for measuring reflected power. The ratio of the electric field magnitude at positions of maximum and minimum in the standing wave pattern, the voltage standing wave ratio (VSWR), is directly related to the fraction of reflected power. The VSWR and reflected power are mathematically described as:

$$\text{VSWR} = \frac{E_i + E_r}{E_i - E_r}, \quad (1)$$

where

E_i is the electric field magnitude of the incident wave,

E_r is the electric field magnitude of the reflected wave, and

$$P_r = P_i \frac{(VSWR - 1)^2}{(VSWR + 1)^2}, \quad (2)$$

where

P_i is the power of the incident wave, and

P_r is the power of the reflected wave.

Thus, monitoring the VSWR is a means of determining the degree of mismatch of the load and, hence, the proportion of the power absorbed by the load.

In order to obtain the desired moisture content, it was anticipated that the duration of microwave heating could be determined by monitoring the increase in the reflected power, since the wet resin absorbed more microwave energy than the dry resin. However, the reflected power detector is not sufficiently sensitive to the change of water content of the resin to provide a reliable end point. At the initiation of 2.5 kW of microwave power to the cavity, the reflected power was stable, typically in the range of 30 to 40 W. At approximately 15 to 20 min into the microwave heating cycle, the reflected power did increase (usually to 50 to 70 W), but oscillated as much as 20 W. However, the reflected power remained in that range until the resin became much drier than desired.

4.2.2 Temperature

Instrument air is passed through the resin bed during the microwave heating cycle to aid in removing the water that has been evaporated. Since the amount of microwave energy absorbed decreases as the load in the cavity decreases, the exit gas temperature of the column should decrease correspondingly. However, the temperature of the exit gas did not vary with LOD of the resin until the resin was much drier than the required range of values. The temperature of the gas leaving the column increased from 25°C (the gas supply) to the steady-state operating temperature in less than 10 min. The air-flow exit temperatures were 65 to 70°C for 226 slpm, 70 to 75°C for 113 slpm, and 85 to 90°C for 43 slpm.

A relationship between the average water content (LOD) of the dried material and a drying factor, which enabled the operator to calculate the length of microwave heating for a measured wet resin volume and input microwave power, has been established experimentally (Fig. 5). For example, when the desired water content is 13% LOD, the drying factor is

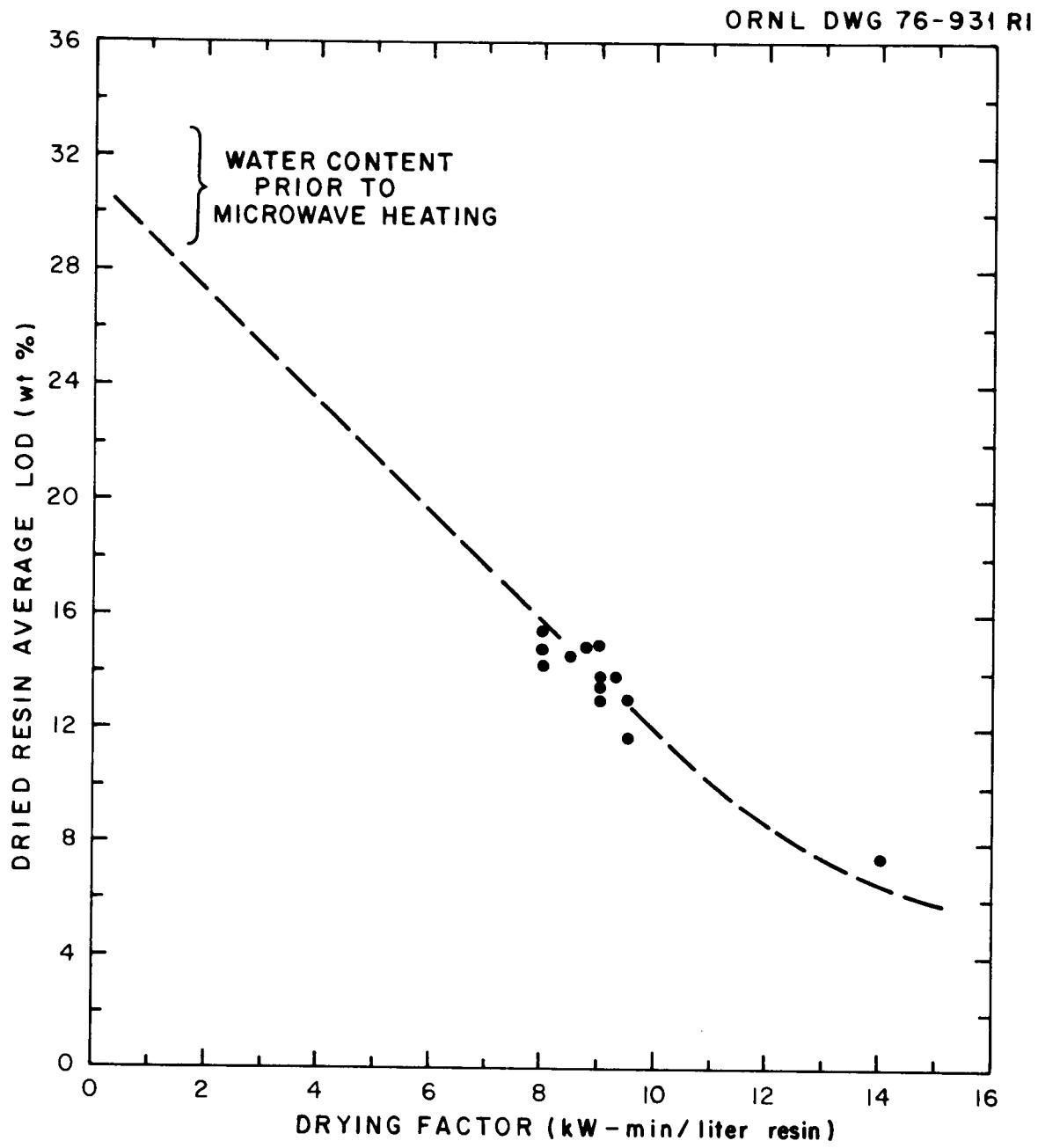


Fig. 5. Moisture content of the dried resin vs drying factor.

9.5 kW-min per liter of wet resin. The drying factor is normalized per unit volume because the volume of the wet resin varied from batch to batch. Multiplying the drying factor by the wet resin volume (11 liters for the reference batch) and dividing by the microwave power (typically 2.5 kW), the microwave heating cycle is 42 min.

Average LOD, resin volume, and microwave operating conditions of the data in Fig. 5 are presented in Table 1. All but two of the drying experiments were conducted at the rated output of the microwave power tube, 2.5 kW. Runs S-32 and S-33 were dried with 2.0 kW of microwave power. The drying factor, when used for a lower microwave power level, shows good agreement with the higher power level.

The graph shows the initial water content of the drained resin prior to microwave heating, 29 to 33 wt %. This range was calculated from a mass balance using the total water removed, the dried product weight, and averaged LOD for the batch. Samples for LOD analysis were taken from the bottom, middle, and top portions of the bed. Since the water removal rate is constant during the microwave heating cycle (see Sect. 4.2.4), a least squares curve is drawn from the initial water content through the average LOD of the dried material in the range of 10 to 16%. The nonlinear nature of this curve at low-moisture content can be explained in terms of coupling efficiency, which is the fraction of transmitted power absorbed by the load.

The percent of the microwave energy transmitted to the applicator that is absorbed by the load depends on factors such as the geometry of the load, the ratio of the volumes of load to the cavity, and wall losses. But the most important factor is the load in the cavity. For the system used for this study, the fully loaded cavity (containing more than 2 liters of water) should have a coupling efficiency (percent of the power that successfully heats the load) of approximately 80 to 85% of the input power, 2.5 kW.³ As the load decreases, the coupling efficiency decreases. Thus, this phenomenon must be considered when using the drying factor curve to obtain low-moisture content material.

Table 1. Moisture content of the dried resin
using the drying factor

Amberlite IRC-72 fully loaded with UO_2^{2+}

Drying factor (kW-min liters resin)	Average LOD ^a (wt %)	Wet resin, volume (liters)	Power (min)	Power (kW)	Run No.
8.0	14.9	11.1	44.5	2.0	S-33
	14.4	11.5	46.0	2.0	S-32
	15.6	11.5	37.0	2.5	S-35
8.56	14.7	11.25	38.5	2.5	S-39R1
8.8	15.0	9.65	34.0	2.5	S-28
9.0	13.8	10.95	39.5	2.5	S-38
	13.4	11.1	40.0	2.5	S-36
	13.6	11.4	41.0	2.5	S-34
	13.1	11.7	42.0	2.5	S-30
	13.9	11.8	43.0	2.5	S-37
	15.1	7.3	26.5	2.5	S-41
9.33	13.9	9.65	36.0	2.5	S-27
9.5	13.2	12.0	46.0	2.5	S-31
	11.8	10.25	39.0	2.5	S-29
	16.9	10.5	40.0	2.5	S-45 ^b
14.0	7.6	11.25	63.0	2.5	S-39

^aLoss on drying.

^bDuolite C-464 resin.

The resin loading system of a fuel recycle plant will be operated for a defined period producing the reference batch, 4 kg of uranium on 11 liters of resin. However, at the end of this series, called a campaign, a special loading and drying run will be required. The purpose of this smaller resin batch is to reduce the uranium inventory of the system prior to returning this remaining uranium to storage. A special loading and drying run (S-41) was conducted to simulate this requirement.

Using the standard drying procedure and drying factor of 9 kW-min/liter of resin, the 7.3-liter batch was dried to an acceptable moisture content of 15%.

One batch of the alternate resin, Duolite C-464, was dried successfully in the experimental apparatus (Run S-45). Using the drying factor, 9.5 kW-min/liter of resin, an acceptable average moisture content of 17% was obtained. The Duolite C-464 has more porosity and retains more water than Amberlite IRC-72 at similar conditions.

4.2.3 Amount of water removed

The duration of microwave heating necessary to obtain dry resin with the desired water content can be determined by monitoring the quantity of water removed during the microwave heating period. The only requirement for using this procedure is that the water content of the drained resin must be reproducible. From the drying experiments conducted, a relationship has been established between the average LOD of the dried resin and the water removed at the end of microwave heating (Fig. 6).

For example, in order to obtain dried resin with an average LOD of 13%, the microwave heating is concluded when 0.19 liters of water per liter of wet resin has been removed. In terms of the reference 11-liter resin batch, the microwave power is shut off when 2.1 liters of water has been removed. The abscissa is normalized as water removed per unit volume of wet resin because the batch size varied. Since the amount of water in the air leaving the condenser was not insignificant during the heating cycle (5 to 10% of the condensate), the total water removed from the resin, rather than the condensate alone, was used. The amount of water not condensed was calculated by using the dry air flow rate, the temperature of the saturated air leaving the condenser, and the psychrometric chart. Table 2 presents water balances for the batches dried using microwave heating.

The water content (29 to 33%) of the drained resin prior to microwave heating was calculated from a mass balance using the dried resin

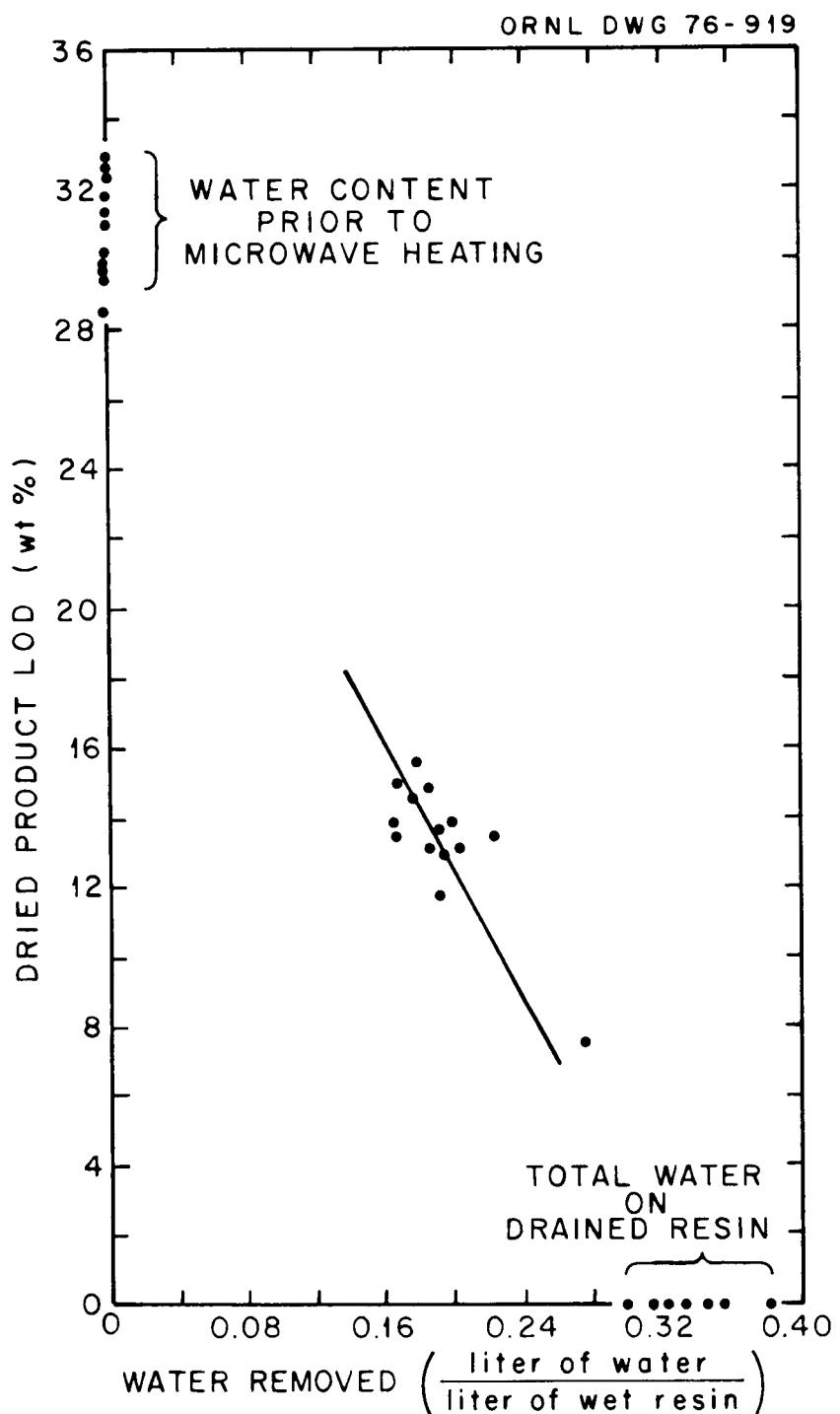


Fig. 6. Moisture content of the dried product vs water removed at the end of microwave heating.

Table 2. Water balance for microwave dried uranium-loaded resin

Water removed from resin			Drained resin						Run number
End of microwave heating (water (liters) ^a resin (liters))	Condensate (liters)	End of cooldown Condensate (liters)	Total water (liters)	Dried product LOD ^b (wt %)	Weight (g)	Volume (liters)	Initial LOD (wt %)	Total water (water (liters) resin (liters))	
0.163	1.70	1.98	2.14	13.4	8951	11.1	30.1	0.301	S-36
0.164	1.50	1.83	2.00	13.9	8806	9.65	29.9	0.335	S-27
0.174	1.85	2.08	2.27	14.4	10495	11.5	29.6	0.329	S-32
0.174	1.15	1.30	1.50	15.0	6437	7.3	31.1	0.338	S-41
0.176	1.85	2.08	2.33	15.6	10440	11.5	31.0	0.344	S-35
0.183	1.90	2.14	2.34	14.9	10097	11.1	28.5	0.319	S-33
0.186	1.90	2.10	2.22	16.9 ^c	8224	10.5	34.6 ^c	0.344	S-45 ^c
0.187	2.00	2.20	2.36	13.2	9188	11.1	30.9	0.322	S-40
0.189	1.80	2.10	2.26	11.8	8813	10.25	29.7	0.314	S-29
0.192	2.00	2.30	2.46	13.8	9265	10.95	31.9	0.324	S-38
0.193	2.10	2.30	2.50	13.1	10437	11.7	29.9	0.331	S-30
0.195	2.18	2.36	2.56	13.9	9038	11.8	32.9	0.324	S-37
0.204	2.25	2.50	2.74	13.2	10411	12.0	31.3	0.343	S-31
0.223	2.45	2.67	2.92	13.6	10363	11.4	32.6	0.380	S-34
0.277	2.92	3.07	3.32	7.6	8935	11.25	32.6	0.356	S-39

^aIncludes water in air leaving condenser.^bAverage Loss On Drying.^cDuolite C-464 resin, which has more porosity and more water than Amberlite IRC-72.

weight, total water removed, and the average LOD of the dried resin batch. Typically, 85% of the total water removal occurs during the microwave heating period. The remainder is removed during the cooldown portion. For run S-39, where the average LOD of the dried resin was 7.5%, 94% of the total water removal occurred during the microwave heating portion of the drying cycle. The total water on the drained resin prior to microwave heating was also calculated from a mass balance and was typically 0.32 to 0.36 liters of water per liter of wet resin. Since this corresponds to 0% LOD, these values are appropriately shown in Fig. 6.

The condensate was found to contain trace quantities of uranium and organics. Because of this contamination, the dryer condensate from a fuel recycle plant would probably be sent to the plant waste system.

The condensate rate was monitored for all experiments. Condensate rates of two runs, one using 2.0 kW and the other 2.5 kW of transmitted microwave power, are shown in Fig. 7. The condensate is normalized because the batch sizes were different: run S-32 was 11.5 liters, and run S-37 was 11.8 liters. During the microwave heating period the condensate rates are constant. The rates for the 2- and 2.5-kW runs are 0.21 hr^{-1} and 0.255 hr^{-1} , respectively. For the reference 11-liter batch this would be 2.31 liters/hr and 2.81 liters/hr for 2 and 2.5 kW, respectively.

For both runs, the standard blowdown procedure was used for removing the interstitial water in the resin bed (35 slpm for 10 min). When the microwave power was supplied to the cavity, the downflow of air was increased to 113 slpm. The temperature of the saturated air leaving the column increased from 25°C (the air supply) to the steady-state operating temperature of 70 to 75°C in less than 10 min. At the midpoint of the timed heating cycle, the air flow through the bed was reversed. The 30-min cooldown cycle was used for both runs prior to removal of the resin from the dryer.

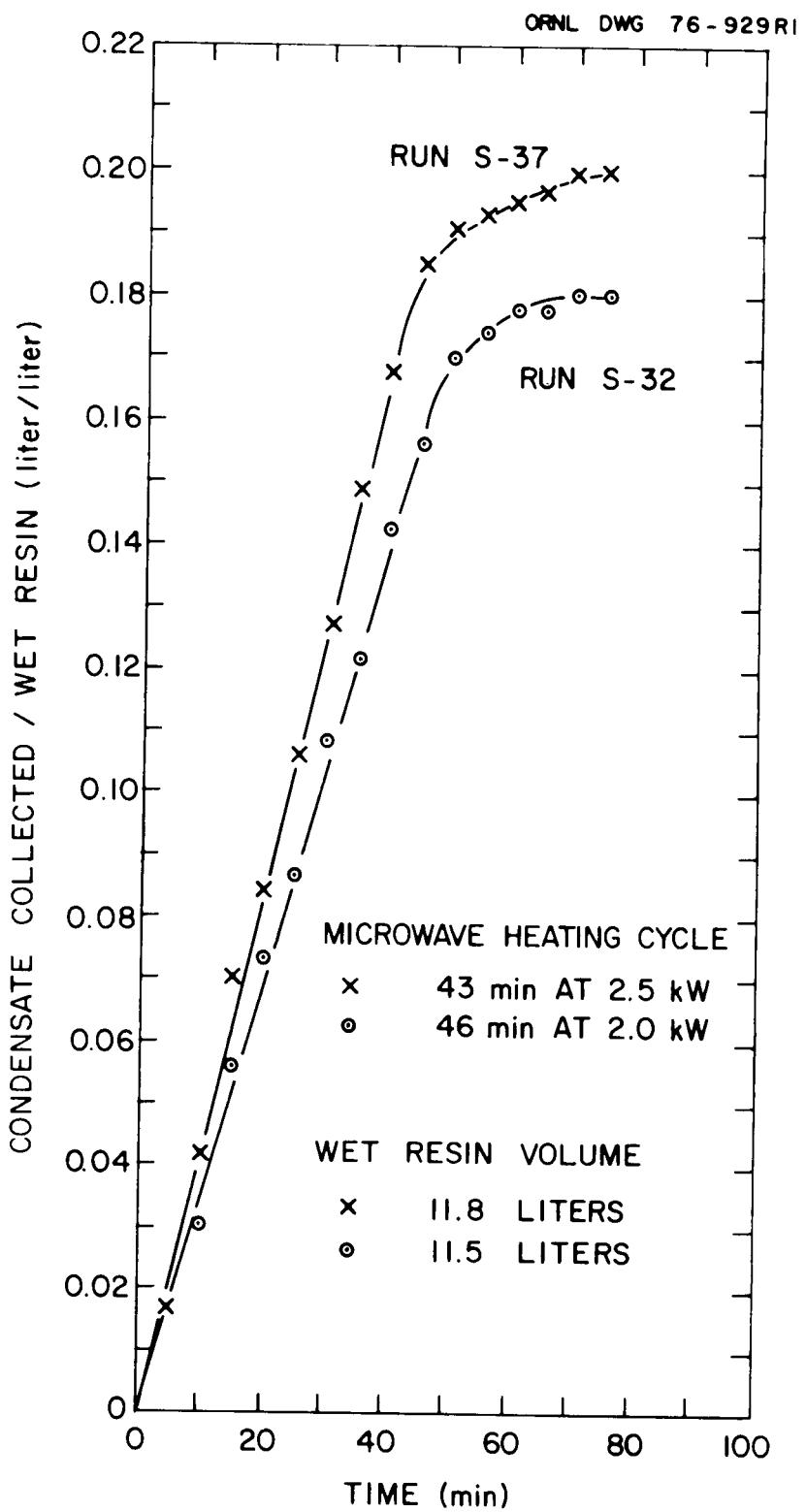


Fig. 7. Condensate rate for 2.0 and 2.5 kW of microwave power.

4.3 Cooldown and Removal

Following the microwave heating cycle, the resin is cooled down. Upflow of instrument air at 113 slpm for 30 min through the resin bed is sufficient to reduce the exit gas temperature to 30 to 35°C. When the dried resin has the preferred moisture content, 10 to 16% LOD, no clinging of the resin to the glass column, nor holdup on the screened flange was observed. Although the dried material flows easily from the column through the 1.9-cm ball valve by gravity, the rate of removal is greatly increased when there is a slight air purge (1 psig) through the screened portion of the bottom flange.

4.4 Heat Transfer and Coupling Efficiency

A heat balance using experimental results from a typical drying cycle is described. The coupling efficiency, which is the percent of transmitted microwave power absorbed by the wet resin, is calculated and compared with the literature value. A special test to determine the amount of microwave energy absorbed by dry (<0.1% LOD), uranium-loaded resin is also described.

The transmitted microwave power to the cavity for run S-37 was 2.5 kW. Instrument (dry) air was passed down through the bed for one-half of the 43-min microwave heating cycle and was then reversed. The saturated air leaving the resin bed was passed through a condenser and collected. The hot air blower warmed the microwave cavity to the anticipated steady-state operating temperature of the air leaving the column. Negligible heat transfer between the glass column and the surroundings is assumed. The coupling efficiency is calculated at steady-state conditions, which are attained with 10 min of supplying the microwave power to the cavity. Operating conditions and data are presented below.

Data from run S-37

Wet resin volume: 11.8 liters

Microwave power: 2.5 kW

Air flow to resin: Instrument (dry) ~ 0 g H₂O/g air at 25°C (113 slpm)

from resin: Saturated at 75 to 78°C

leaving condenser: Saturated at 25°C, 0.032 g-moles $H_2O/g\text{-mole air}$
Condensate: 3.0 liters/hr (167 g-moles/hr)

Air mass flow rate: 113 slpm (300 g-moles/hr)

Water in air leaving condenser: 300 (0.032) = 10 g-moles/hr

Mole fraction of water leaving the resin bed: $\frac{167 + 10}{300 + 167 + 10} = 0.371$

Partial pressure of the water: 0.371 (750 mm Hg) = 278 mm Hg

From the steam tables, this corresponds to 74°C. This is in excellent agreement with the measured temperature.

At steady state, the microwave energy absorbed by the wet resin is equal to the sensible heat of the air plus the heat of vaporization of the water.

Sensible heat of the air: 300(7)(74-25) = 102,900 cal/hr

Heat needed to evaporate the water: 167(18)(550) = 1,653,300 cal/hr

Total energy absorbed: 1,756,200 cal/hr (2.04 kW)

Coupling efficient: $\frac{2.04}{2.50} \times 100 = 82\%$.

According to the microwave manufacturer, the coupling efficiency of the microwave energy when our cavity is fully loaded is 80 to 85%.³

A special test was conducted to measure the amount of microwave energy absorbed by very dry resin (<0.1% LOD). In order to avoid the handling difficulties associated with resin of this low moisture content, wet resin was transferred to the cavity where it was dried to this low moisture content by microwave heating. This test used 7.5 liters of uranium-loaded resin.

After 60 min of 2.5 kW of microwave heating, the column exit-gas temperature started to increase from the steady-state operating temperature of 75°C at about 1°C per min. The upward gas flow rate through the bed was 113 slpm. The microwave power was reduced from 2.5 kW to 0.75 kW at 80 min in the cycle because severe arcing was observed. The reflected power reading for 2.5 kW and 0.75 kW transmitted to the cavity were 200 and 80 W, respectively. Since no arcing was observed using 0.75 kW of microwave power, the heat balance was conducted at this power level.

Using 750 W of microwave power and an upward gas flow rate through the bed of 212 slpm, the dried resin attained steady state when the column exit temperature stabilized at 65°C. The cavity was heated to this temperature. Assuming no heat transfer between the resin and the surroundings, the amount of microwave energy absorbed by the resin can be calculated as follows:

Air temperatures, inlet: 25°C
outlet: 65°C

Air flow rate: 212 slpm (570 g-moles/hr)

$$\begin{aligned} Q &= (570)(7)(40) \\ &= 159,600 \text{ cal/hr (185 W),} \end{aligned}$$

and the coupling efficiency is

$$\frac{185}{750} \times 100 = 25\%.$$

From an energy balance, using the transmitted microwave power, the power absorbed by the resin, and the reflected power from the cavity, the difference indicates that approximately 500 W is absorbed by the metallic walls.

Following the test, a sample of the dried resin was found to have a moisture content of <0.1 wt %.

5. UNIFORMITY OF DRYING

Following the microwave heating and cooldown cycles, three samples of the dried resin were taken for LOD analysis. Samples which were believed to be representative of the bottom, middle, and top portions of the bed were taken during the removal of the resin from the column (Table 3). The preferred moisture content for the dried, uranium-loaded resin is 10 to 16% LOD. Almost all of the resin samples are within that range.

Prior to microwave heating, all batches were drained by downflow of air at 35 slpm for 10 min. When the microwave power was supplied to the cavity, the downward flow of instrument air through the bed was increased to 113 slpm. At the midpoint of the timed heating cycle, the

Table 3. Moisture content uniformity using microwave heating

Air flow: 113 slpm downflow for one-half of time at power

Microwave power: 2.5 kW

Resin: UO_2^{2+} loaded Amberlite IRC-72

Run number	Drying factor $(\frac{\text{kW-min}}{\text{resin (liters)}})$	LOD (wt %) ^a Sample location			Average	Remarks Upflow until removal (slpm)
		Bottom	Middle	Top		
S-33 ^{b,c}	8.0	14.7	15.2	14.8	14.9	142
S-32 ^{b,c}		7.7	15.6	13.2	14.4	113
S-35		15.5	16.2	15.1	15.6	226
S-39R1	8.5	14.8	14.8	14.4	14.7	113
S-28 ^c	8.8	14.2	15.4	15.4	15.0	113
S-38	9.0	9.6	14.2	13.4	13.8	113
S-36		13.7	13.3	13.2	13.4	113
S-34		14.0	14.5	12.3	13.6	226
S-30 ^c		12.5	13.4	13.4	13.1	113
S-41		15.0	15.1	15.0	15.1	226
S-27 ^c	9.3	13.6	14.6	13.6	13.9	113
S-31 ^c	9.5	13.8	14.2	11.6	13.2	113
S-29 ^c		10.2	12.7	12.4	11.8	113
S-45 ^{d,e}		17.6	16.2	16.9	16.9	d
S-39	14.0	8.4	7.6	6.9	7.6	113

^aLoss On Drying.^bMicrowave power 2.0 kW.^cIncreased upward air flow to 226 slpm for 1 min.^dDownward flow of 43 slpm for one-half time at power; upward flow of 43 slpm for one-half time at power; upward flow of 226 slpm until removal.^eDuolite C-464 resin.

air flow through the resin bed was reversed. For most of the experiments, the air flow rate was maintained at 113 slpm; however, other flow rates were also tested. Upon reversing the air flow, slugging and channeling rather than smooth fluidization is observed. It will be shown later in this section that mixing of the resin is necessary in order to obtain a uniform moisture content. When air was supplied only through the screen, a small amount of wet resin was observed during the removal of the batch from the dryer. Introducing air through the ball valve as well as through the screen eliminates this problem.

The considerably lower moisture content sample taken from the bottom of the column (S-32, S-38) is believed to be the first quantity of resin removed from the dryer. Since dry instrument air is used to mix the material, preferential drying of the resin bed near the air port is believed to be the reason for this low moisture content. Other samples representative of the bottom of the column were taken following removal of about 50 to 100 ml of resin. These low-moisture content samples were not included in obtaining the average moisture content of the batch, since these samples probably do not represent one-third of the entire batch. This is substantiated by the results from a multiple sampling of another dried resin batch, S-40.

Following the 30-min cooldown using upflow of air at 113 slpm, samples were taken for a moisture content profile along the axis of the resin column. The first quantity of resin removed from the column (No. 1) was found to be considerably drier than other samples taken; particularly the second sample (see Table 4). During the removal of the remainder of the resin from the column, five additional samples were taken, each representative of one-fifth of the column.

The skewed moisture-content profile with the wettest resin in the lower fifth can be explained as follows. First, downward flow of air was used for three-quarters of the 40-min microwave heating cycle. This tended to dry out the top portion of the bed faster than the middle and lower portion. Second, since the top portion of the resin bed is near the center of the cavity, and is therefore exposed to more microwave energy than the middle and still more than the lower portion, one expects

Table 4. Moisture content uniformity using microwave energy with and without mixing

Drying factor $\left(\frac{\text{kW-min}}{\text{liter resin}} \right)$	LOD (wt %) ^a						Remarks
	Sample position in column						
Bottom	1	2	3	4	5	Top	Average
<u>A. With mixing (air upflow of 113 slpm)</u>							
9.0	5.8	15.0	14.2	13.5	11.7	11.4	13.2
							Run S-40
							Wet resin vol: 11.1 liters
							Microwave cycle: 40 min at 2.5 kW
							Air purge procedure -
							Downflow: 30 min at 113 slpm
							Upflow: 40 min at 113 slpm
<u>B. Without mixing (air upflow of < 45 slpm)</u>							
10.2	16.2	14.0	13.8	13.7	12.0	12.2	13.7
							Run S-39R2
							Wet resin vol: 11.25 liters
							Microwave cycle: 46 min at 2.5 kW
							Air purge procedure -
							Downflow: 9 min at 11 slpm
							Upflow: screen - 9 min at 11 slpm, valve - 80 min at 40 slpm
8.5	20.8	17.6	16.7	15.8	14.1	12.0	16.2
							Run S-39R3
							Wet resin vol: 11.25 liters
							Microwave cycle: 38.5 min at 2.5 kW
							Air purge procedure -
							Downflow: 10 min at 11 slpm
							Upflow: screen - 10 min at 11 slpm, valve - 10 min at 45 slpm
							Downflow: 40 min at 11 slpm

^aLoss On Drying.

the bottom portion to be the wettest. For these reasons, the average moisture content of the batches S-32 and S-38 was calculated without using the bottom sample.

Two experiments were conducted to determine the uniformity of the moisture content of the dried resin when the resin bed remains static throughout the entire drying cycle. The principal difference between these two tests was the procedure of purging the resin bed of the evaporated water. In one test, essentially all the air purge was supplied upward through the bed, whereas in the other test, the quantity of air upflow and downflow was equal. Previously dried material was rewetted, and transferred to and drained in the dryer using the standard blowdown procedure. Operating conditions and LOD analyses of samples taken from the batches are given in Table 4.

It was anticipated that the bottom portion of the resin column would not receive as much microwave energy as the top and middle sections for two reasons. Although the microwave power distribution is level throughout the bulk of the cavity, the power decreases rapidly within approximately 7 cm of the walls. In addition to this phenomenon, since a small portion of the resin lies in the screened cone of the bottom flange (see Fig. 3), it is expected that the microwave power level in this area will be still lower than near the walls. Second, since the top portion of the resin bed is located near the center of the cavity and is therefore exposed to microwave energy entering through the flat face as well as the sides, the top portion is also expected to become drier than the middle, and significantly drier than the bottom portion.

Therefore, to assist the microwave heating to obtain a uniformly dried product, a procedure was tested that was aimed at preferentially contacting the wettest resin (located in the lower portion of the bed) with the dry instrument air (run S-39R2). Approximately 95% of the total gas flow was upward through the resin bed. The gas flow rate through the bottom screen was nominally one-tenth of the flow used in the standard drying procedure. When air was supplied through the bottom ball valve, a higher flow rate (45 slpm) was necessary in order to retain

the resin in the dryer. For experiment S-39R2, microwave power to the cavity was terminated when 2 liters of condensate was collected. During the drying cycle, the cavity was heated by the recirculating hot air system.

As the resin was removed from the dryer, the batch was divided into six equal portions, and each portion was mixed and sampled for LOD analysis. Three distinct moisture content regions were observed (Table 4). The wettest portion of the resin bed was the bottom sixth section, while the driest portion was the top third. The remaining three samples from the column contained essentially the same moisture content, which was approximately the average of the wettest and driest sections.

The purpose of the second experiment (S-39R3) was to examine the moisture uniformity of the dried resin when equal amounts of upflow and downflow of air are used. Table 4 shows the air purge procedure and flow rates. The length of microwave heating was calculated by multiplying the wet-resin volume (11.25 liters) by the drying factor (8.5 kW-min/liter resin) and dividing by the microwave power (2.5 kW). The cavity was heated by the hot air system to prevent heat transfer from the resin to the surroundings.

As in the previously mentioned static-bed uniformity test, the dried resin batch was divided equally into six portions, and each portion was mixed and sampled for LOD analysis. Although the duration of microwave heating in the experiment was less than for the previous test and, subsequently, less water was removed from the resin, the relative moisture content profile along the axis is similar. As expected, the bottom section was the wettest, the top portion the driest, and resin samples from the middle section contained an intermediate water content.

In conclusion, for this resin-vessel geometry and location of the resin in the cavity, mixing of the material is required in order to ensure a uniformly dry product. Although microwave energy is preferentially absorbed by the wettest material, the above mentioned factors appear to override this effect.

One design improvement for a future resin dryer would be to relocate the bottom flange such that it protrudes approximately 7 cm into the cavity. This modification would increase the microwave energy intensity in the lower portion of the resin vessel and thus allow the microwaves to heat the column more uniformly.

6. FLUIDIZED BED MIXING

The standard drying procedure utilizes downward air flow through the bed for one-half of the timed microwave heating cycle. For the remainder of the drying cycle the air flow through the bed is upward. Most of the experiments were conducted using 113 slpm of instrument air; however, other gas flow rates (142 and 226 slpm) were also tested. At the initiation of upward flow of air at 113 slpm, slugging and channeling, rather than smooth fluidization, was observed. At this flow rate, bursts of resin were seen to extend approximately 7 to 13 cm above the fixed bed height. Higher gas flows resulted in more violent slugging, sometimes expelling resin a few centimeters below the screened outlet. This slugging and channeling is due to the geometry configuration and physical properties of the fluid and solid material. During the heating cycle, the sticky nature of the wet resin also contributes to the slugging behavior.

The minimum fluidization velocity was calculated⁴ to be 18.6 cm/sec for air at 25°C and for the properties of the dried product resin (Table 5). The minimum fluidization flow rate for the 12.4-cm-ID resin vessel is 135 slpm. This agrees with the behavior observed near the end of the microwave heating and during cooldown where the total gas flow from 113 slpm of air is about 200 liters per minute of saturated air at 70 to 75°C.

7. CONCLUSIONS

Microwave heating was successfully demonstrated to give controlled and reproducible drying of 11-liter batches of uranium-loaded (4 kg of

Table 5. Properties of uranium-loaded resin

Uranium per bead, g	60×10^{-6}
Wt % uranium of dried resin, 0% LOD	0.47
Weight of dried bead, 0% LOD	1.3×10^{-4}
Weight of dried bead, 13% LOD, g	1.5×10^{-4}
Particle diameter, cm	0.05
Particle density, g/cc	2.29
Bulk density, 13% LOD, g/cc	1.42

uranium) resin in a vessel of safe dimensions for nuclear criticality (12.4 cm ID). The dried resin must have a water content of 10 to 16 wt % to minimize handling problems. The microwave heating evaporates water throughout the resin bed, with preferential heating of the wettest resin, and allows short drying cycles.

In order to obtain the desired water content, a standard drying procedure and methods for determining the duration of microwave heating were developed. Using an experimentally derived drying factor or monitoring the amount of water removed from the wet resin has been found to provide a reproducibly acceptable dried product. Temperature or reflected power were not very reliable in obtaining resin of the preferred moisture content.

A heat balance on the resin dryer system and the coupling efficiencies of the microwave energy with the wet resin were calculated and found to agree well with experimental results and the manufacturers literature. The minimum fluidization velocity was also calculated and compared favorably with experimental observations. It was necessary to mix the material in order to ensure a uniform moisture content. No significant operating difficulties were encountered.

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