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Development, Design, and Preliminary Operation of a Resin-Feed Processing Facility for Resin-Based HTGR Fuels

MASTER

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HTGR FUEL RECYCLE PROGRAM (189a OH045)
Refabrication Development - Task 500

DEVELOPMENT, DESIGN, AND PRELIMINARY OPERATION OF A RESIN-FEED
PROCESSING FACILITY FOR RESIN-BASED HTGR FUELS

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ABSTRACT

Fuel kernels for recycle of ^{233}U to High-Temperature Gas-Cooled Reactors are prepared by loading carboxylic acid cation exchange resins with uranium and carbonizing at controlled conditions. Resin-feed processing was developed and a facility was designed, installed, and operated to control the kernel size, shape, and composition by processing the resin before adding uranium. The starting materials are commercial cation exchange resins in the sodium form. The size separations are made by vibratory screening of resin slurries in water. After drying in a fluidized bed, the nonspherical particles are separated from spherical particles on vibratory plates of special design. The sized, shape-separated spheres are then rewetted and converted to the hydrogen form. The processing capacity of the equipment tested is equivalent to about 1 kg of uranium per hour and could meet commercial recycle plant requirements without scale-up of the principal process components.

1. INTRODUCTION

The reference fuel kernel for recycle of ^{233}U to High-Temperature Gas-Cooled Reactors (HTGRs) is prepared by loading carboxylic and cation exchange resins with uranium and carbonizing at controlled conditions.¹ One important advantage of this process is that the kernel size, shape, impurities, and perhaps other properties can be controlled or verified for the resin particles before uranium is introduced. These operations can be carried out in a nonradioactive resin-feed processing facility which would supply resin to a remotely operated refabrication plant.¹

The development of resin feed processing of this type has been part of the HTGR fuel recycle program at the Oak Ridge National Laboratory.

The properties required for the feed resin in uranium loading are much different in type and degree from those usually used in ion exchange resins. The necessary properties first had to be identified and measured. Special equipment and procedures were then developed and tested to give the required control of these properties. Finally, a resin-feed processing facility was designed, installed, and operated to supply resin for fuel recycle development and for a future remotely operated pilot plant.

Both development studies and the design and preliminary operation of a resin feed processing facility are reported here. The process requirements and the overall flowsheet are reported first. The individual process operations are reported in the order of use with results from both development tests and the preliminary operation of the facility.

1.1 Acknowledgments

The resin feed processing facility was developed, designed, and installed as part of the Thorium Utilization Program. J. A. Carpenter, Jr., and R. L. Beatty of the Metals and Ceramics Division contributed information and experimental tests for several of the process operations. G. M. Lawson and P. E. Stein of the Engineering Division supervised the preparation of drawings for the construction of the facility. J. D. Flynn and L. R. Sullivan of the Plant and Equipment Division supervised the installation. R. D. Arthur, T. V. Dinsmore, and J. R. Parrott, Jr., participated in development and operation of the equipment.

2. PROCESS REQUIREMENTS AND DESCRIPTION

The HTGR fuel recycle program has included both the procurement and evaluation of commercial ion exchange resins and the processing of the commercial resins to meet the specifications for size, shape, composition, and quality assurance. The resin-based fuel concept was initially tested with strong acid (sulfonic) resins, but the sulfur remaining in the carbonized kernels was a major limitation.² The initial carboxylic acid resins tested were either nonspherical or did not give high loadings of uranium.³ The resins, the specifications, and the procedures described in the following sections are compromises between what would be optimum for fuel refabrication and the cost and technical feasibility of restrictive specifications. All resins tested were developed by the resin manufacturers for other possible commercial uses. The only manufacturing variations to meet HTGR fuel requirements were those needed to improve the size and shape yield of resins of an otherwise standard type.

2.1 Resin Specification

The specifications for the product of the resin feed processing are derived from specifications for carbonized recycle fissile kernels and are therefore dependent on the carbonization behavior. These fissile particles are spheres with the chemical composition of ~50% UO_2 --50% UC_2 in a carbon matrix and are prepared by controlled carbonization and conversion⁴ of the uranium-loaded resin. Particle specifications impose requirements on the feed resin as follows:

1. The shape persists through carbonization, and the feed resin must meet the shape specification. Also, the shape must not

be degraded by cracking, clustering, or other effects during process operations including the uranium loading and carbonization.

2. The composition of the resin must result in carbon and oxygen as the only important constituents remaining with the uranium after high-temperature carbonization. This requires use of carboxylic acid type cation exchange resins. Impurities are limited by their effects as nuclear poisons (boron equivalent specifications), by unacceptable chemical reactions during coating or irradiation (iron, chlorine, chromium, and nickel must be limited), and by problems during reprocessing (the principal source of ^{14}C is nitrogen impurities).
3. The size after carbonization (in combination with the uranium content per unit volume) is a complex function of many variables, but is proportional to the feed resin size when all other variables are fixed.
4. The uranium content in both weight percent and density must exceed the minimum required to give the fuel loadings needed. Since both the weight percent of uranium and the density show very large changes during carbonization, any untested resins must be loaded and carbonized to check the acceptability of the carbonization behavior.

These requirements result in the proposed specification for the resin to be purchased for resin feed processing. The reference Amberlite

IRC-72^{*} resin has met these specifications, but the Duolite C-464^{**} alternate resin has a lower uranium capacity of borderline acceptability.

2.1.1 Chemical composition and impurities

The resin shall be the sodium form of a carboxylic acid type cation exchange resin prepared by polymerization of acrylic acid and divinylbenzene. Conversion to the hydrogen form using HNO_3 shall leave carbon, hydrogen, and oxygen as the only major constituents. Impurities in dried hydrogen-form resin shall not exceed:

Fe, Cr, Ni	≤ 20 ppm each impurity
Total equivalent boron content	≤ 1 ppm

2.1.2 Uranium loading capacity

The uranium content of hydrogen-form resin contacted for ≥ 2 hr with 0.3 M uranium as uranyl nitrate with $\text{NO}_3^-/\text{U} = 1.7$ shall exceed both of the following:

1. The uranium content of resin dried in air to 120°C shall be ≥ 45 wt %.
2. The uranium content of washed, uranium-loaded resin in water on a bulk volume basis shall be ≥ 1.25 moles of uranium per liter.

2.1.3 Size

The specification for the converted product spheres is equivalent to 65×10^{-6} g of uranium per sphere in the mean diameter, and a mean

^{*} Trademark of the Rohm and Haas Co.

^{**} Trademark of the Diamond Shamrock Chemical Co.

diameter of 340 to 380 μm . This requires sodium-form feed resin of about 740- μm mean diameter for the two resins tested (Amberlite IRC-72 or Duolite C-464). The size range listed in the converted product specifications is equivalent to about 40 to 100 μg of uranium per sphere or 640 to 840 μm diam for the sodium-form resin. Therefore, the resin shall be >25 wt % of 640- to 840- μm diam spheres in the sodium form, and the unit price shall be computed on the basis of resin within this range.

$$\text{Unit price} = \frac{\$/\text{ft}^3 \text{ for sodium-form resin}}{\text{fraction of 640 to 840 } \mu\text{m diam}}$$

2.1.4 Shape

The ratio of maximum diameter/minimum diameter shall be used as a specification of shape. For the size range specified (640 to 840 μm diam in the sodium form), $\geq 90\%$ of the particles shall have the ratio ≤ 1.1 .

2.1.5 Structure, uniformity, and density after carbonization

The structure, uniformity, and density of the spheres after conversion shall be as good as the lower range of values observed for the reference Amberlite IRC-72 resin after conversion. The converted product has a carbon matrix with uniformly distributed UO_2 , UC_2 , and $\text{UC}_x\text{O}_{2-x}$, and a uniform porosity and high surface area. The density of the converted product should be $\geq 1.9\text{-g/cm}^3$ bulk density or the mercury pycnometer density at 75 psi should be $\geq 3.0\text{ g/cm}^3$. Either the resin composition and manufacturing procedure shall be identical to previous lots of resin which were shown to meet these specifications or a test lot shall be supplied for determining the structure, uniformity, and density after carbonization.

2.2. Commercial Cation Exchange Resins

Ion exchange resins from the commercial manufacturers are processed at ORNL to meet the specifications for size, shape, composition, and quality assurance. Amberlite IRC-72 is being used as the reference weak-acid resin. The uranium loading behavior of the Duolite C-464 is also suitable, but it shows significant differences from the Amberlite IRC-72. The costs of both resins are in the range of \$0.05 to \$0.10 per gram of uranium, depending on the allowable range of particle-sphere diameter.

Both manufacturers have modified their preparation procedures to increase the particle size and shape yields for our requirements. Therefore, the size and shape results reported are for specific batches only and are not typical of the commercial resins. For the Amberlite IRC-72, these changes have resulted in narrower particle-size distribution and fewer very small spheres. Apparently, these changes can be accomplished by small process variations and do not result in any increase in price. For the Duolite C-464, the size and shape yields are greatly improved by the manufacturing changes and appear to meet the current product specifications without any further resin feed treatments. Since the yield for standard commercial resins is 15 to 20%, the six times higher unit price of the specially prepared Duolite C-464 is equivalent to the same cost per gram of uranium. These two resins do not have large commercial uses and the reproducibility of the best (most recent) lots will have to be confirmed for future orders.

The two resins are described in the manufacturer's literature as follows:

Amberlite IRC-72. Acrylic-divinylbenzene matrix structure, a weakly acidic carboxylic acid type cation exchange resin, sodium form, pH range of 5 to 14, total exchange capacity of 2 meq/ml wet, 43.6 kilograins of $\text{CaCO}_3/\text{ft}^3$, apparent density of 0.74 g/cm^3 , effective size of 0.40 to 0.55 mm, 71 to 73% moisture, 85% swelling for $\text{H}^+ \rightarrow \text{Na}^+$ conversion, faster adsorption, and improved hydraulic characteristics as compared to Amberlite IRC-50.

The best lot for HTGR fuel preparation was identified as Rohm and Haas Lot No. 2-6681.

Duolite C-464. Polyacrylic matrix, a macroporous, weak-acid, carboxylic cation exchange resin, hydrogen form,* pH range of 5 to 14, total exchange capacity of 3.5 meq/ml (wet) or 10 meq/g (dry), specific gravity of 1.15, +20 -50-mesh size, 52 to 57% moisture, outstanding resistance to osmotic attrition, and high capacity.

The best lot for HTGR fuel preparation was identified as Diamond Shamrock Lot No. 2-60.

The requirements for HTGR resin-based fuels have been reviewed repeatedly with several manufacturers of ion exchange resins. We now believe that there is little chance of any significant overall improvement in the characteristics of the two resins described. The capacity is limited by the chemical formula of the polymer. The most recent lots of both resins (as received) have contained more than 99% perfect spheres. A

* Resin purchased for HTGR fuels has been delivered in the sodium form.

narrower size range would be preferred, but the development and pilot plant programs do not require large enough quantities of resin to make special manufacturing procedures economically feasible. Equivalent resins are now available from other resin manufacturers in the United States and foreign countries. However, the two descriptions given are not sufficient to assure equal performance. Other resins with the same apparent descriptions would not allow high loadings of uranium, were not spherical, or would show large amounts of cracking at the uranium loading and drying conditions used.³

2.3 Process Capacity

The resin feed processing facility was sized to meet the present capacity requirements using part-time operation of the equipment. Since the installed equipment is still of moderate size, little saving in the installation cost would result from the use of smaller equipment. In fact, the larger equipment results in a lower overall cost to the development program because of lower costs for labor. In addition, the requirements for a commercial recycle plant would not require scale-up since the principal process equipment components are already full-scale.

The fuel recycle development program, the hot pilot plant, and the first commercial plant are based on batch operations with 4 kg of uranium as the reference batch size. The reference recycle particle contains about 65×10^{-6} g of ^{233}U per sphere; it requires wet resin with an average diameter of about 730 μm in the sodium form, or an average diameter of 570 μm in the hydrogen or the uranium loaded forms. Therefore one batch contains about 70×10^6 spheres and requires 11 liters of wet hydrogen-form

resin, 25 to 30 liters of wet, sodium-form resin which goes to the dryer, and 150 to 200 liters of wet resin which goes to size separation. Capacities equivalent to one batch per 4 hr would allow efficient use of operating labor for the present requirements and would permit six batches per day (or 24 kg of uranium per day) to meet the production requirement of a commercial plant. The resulting capacities per unit time for 240 min are 45 cm³/min for wet, hydrogen-form resin, 120 cm³/min for wet, sodium-form resin which goes to the dryer, 0.7 liter/min for wet resin which goes to size separation, and 5000 spheres per second of product resin.

2.4 Other Requirements

The product of resin feed processing must be packaged in the amounts and containers required by the pilot plant or other ²³³U loading facility. It is expected that resin transfer containers will be charged with measured amounts (equivalents) of resin feed. Control of the resin loading process results in reproducible fractional loadings of uranium (i.e., equivalent of uranium/equivalent of resin) independent of the variations in the amount of resin present. However, the amount of resin must be measured and controlled to assist with uranium accountability and for the convenience of other process operations.

The procedure and equipment for product storage, quality assurance measurements, and metering into transfer vessels were not included in the initial installation of the resin-feed processing facility. These additions will have to be designed and installed to be compatible with the corresponding facilities of remotely operated ²³³U-loading pilot plants or full-scale facilities.

2.5 Overall Process Flowsheet

The overall resin-feed processing flowsheet involves three major treatments (Fig. 1). The wet-screening treatment determines the size of the wet, sodium-form resin. The shape-separation treatment rejects the nonspherical resin particles. The conversion treatment rewets the sodium-form resin and converts it to a metal-free hydrogen form for exchange with acid-deficient uranyl nitrate. The fluidized-bed drying and dry screening are auxiliary operations required for the shape separations. Each of these operations will be described in a separate section of this report, which includes information on process development and selection of equipment. The composition and exchange capacity are limited by those of the commercial resin supplied to the resin-feed processing facility. The intention is to use only resin types which have been tested for HTGR fuel fabrication and to require duplication of the tested properties as part of the resin purchase requisition. The composition and exchange capacity of the hydrogen-form resin would be checked before it was used for recycle fuel fabrication.

3. WET-SIZE CLASSIFICATION

The first treatment of the commercial ion exchange resin is wet screening by feeding a slurry of sodium-form resin in water to a vibratory screening unit. The removal of large amounts of oversize and under-size resin greatly reduced the capacity required for the subsequent process operations. The problems that resulted when unsized commercial resin was dried and screened are described in Sect. 4. The use of a large volume ratio of water/resin provides a suspension and removal of fine resin

Fig. 1. Resin feed preparation process flowsheet.

particles which other size classification procedures do not achieve. Sizing of the wet resin gives a reproducible control of the exchange capacity per resin bead. Resin beads which appear to be dry show a large change in diameter with changes in water content, and this introduces a large additional variable with respect to controlling the wet size by use of dry screening.

3.1 Description of Equipment

For the wet-size classification, the slurry of resin in water is fed to a three-screen separator. Three screen fractions are discharged to collection tanks with small excesses of water, and a slurry of the smallest size is discharged to a settler (Fig. 2). The size separator is a SWECO^{*} 0.76-m (30-in.)-diam vibratory separator of a standard design. All surfaces that contact the resin are stainless steel or polymeric gaskets and seals. The screen discs are interchangeable (Fig. 3). The screen cloths are made of tensile bolting cloth (TBC) with TBC-mesh sizes as listed in Table 1. The fines settler is a 55-gal drum with welded inlet, exit, and overflow nozzles. All valves for resin slurries are ball valves. The only other special fabrication item needed for the size classification system was a modification of the polyethylene resin tanks to replace flat bottoms with conical bottoms. The assembly details are shown by construction drawings (Appendix 9.1).

^{*}Southwestern Engineering Co., Model LS30S6666, Los Angeles, Calif. 90051.

Fig. 2. Resin size and shape classification flowsheet.

Fig. 3. Photograph of 0.76-m-diam wet-screen separator.

Table 1. Tensile bolting cloth mesh sizes for 0.76-m screens

Southwestern Engineering Co. Model No. LS30S6666
vibratory screen separator

Screen material: TBC

Mesh (openings/in.)	Wire diameter (in.)	Openings (μ)
18	0.009	1184
22	0.0075	965
24	0.0075	869
26	0.0075	787
28	0.0075	716
30	0.0075	681
32	0.0075	605
34	0.0075	556
36	0.0075	516
38	0.0075	477
40	0.0065	470
46	0.0055	411
60	0.0045	310
200	0.0016	86

3.2 Procedures for Wet-Size Classifier

Resin is fed to the 0.76-m-diam vibratory screening unit by a water ejector (Fig. 2).^{*} The vibratory screening unit is located approximately 3 m above the three resin holding tanks (110-liter capacity each). The resin flow to the ejector is controlled by a ball valve and a vacuum pressure gage. An additional water supply line to the screening unit is used in order to obtain higher water/resin flow ratios. The resin slurry and additional water flows enter a small beaker located at the mouth of the screening unit to break the velocity and control the distribution of the resin onto the screen. The water leaves the screening unit through the bottom spout to an inclined 55-gal tank (fines settler). Surge capacity for the recycle water stream is provided with an additional tank. In-line filters are located in the pump discharge line.

3.3 Wet-Size Classifier Results

The 0.76-m-diam vibratory screening unit operated well; no important problems were encountered. In order to obtain reasonable resin flow rates and still have a high (40:1) water-to-resin volume ratio, a bypass line was used around the jet ejector. The water flow was maintained at 100% of the rotameter capacity (32 liters/min). The resin flow to the jet ejector was controlled by a ball valve to obtain a vacuum of 30.5 cm Hg. At these conditions a 40:1 flow ratio was obtained. The resin flow rate was 0.82 liters/min. Screening capacity for each 8-hr shift was 400 liters (13.9 ft³, or two 55-gal drums). The manufacturer's rated capacity is

^{*}Hydraulic ejector, Penberthy Model LH1.

200 liters/min and 5 liters/min of water and resin respectively. Motive pressure to the jet was 34 psig.

Two banks of double length (Cuno CT-102) in-line filters were found to be unusable due to rapid plugging. Filter openings of 40 and 150 μm were found to clog rapidly. Currently, screenings are performed without filters and the fines are discharged as settler bottoms or in the overflow of excess water.

Two of the polyethylene storage tanks failed by cracking near shop welds that were made when the conical bottoms were added. The tanks were repaired by welding and were reused.

A 200-liter (7 ft^3) batch of Amberlite IRC-72, lot No. 2-6681, was screened with 18, 22, and 38-mesh screens, and the 22/38 fraction was rescreened with 26, 30, and 34-mesh screens. The size distribution for this batch (Fig. 4) showed a mean particle size of 720 μm , and the yield of the preferred material ($-26 +30$ mesh or $730 \mu\text{m} \pm 50 \mu\text{m}$) was 18-1/2 vol %. A photograph of the as-received resin and the wet classified product ($-26 +30$ mesh) is shown in Fig. 5. Also shown in this figure is this sized material as it leaves the fluidizer dryer. Other size separation results are tabulated in Table 2.

4. RESIN DRYING AND DRY SCREENING

The wet, sized, resin must be dried to give free-flowing individual beads for the particle-shape separation described in Sect. 5. Damp resin is sticky, while completely dried resin shows static charge effects which result in unacceptable particle-shape separator operation. Therefore, the dryer must give acceptable control of the amount and uniformity of

Fig. 4. Size distribution of Amberlite IRC-72, lot 2-6681.

Y-140345

800 μ m

Fig. 5. Feed and product photographs for size separation of Amberlite IRC-72.

Table 2. Size data for cation resins in the sodium form

Manufacturer's lot No.	Nominal size	Mean diameter (μm)	Yields (vol %)		
			26/30 TBC $735 \pm 50 \mu\text{m}^a$	24/32 TBC $735 \pm 130 \mu\text{m}^b$	30/34 TBC $620 \pm 60 \mu\text{m}^c$
I. Amberlite IRC-72 (results from wet screening)					
2-5612	20/50 mesh	820		24	10
2-5990(I)	20/50 mesh	640	15	30	18
2-5990(II)	20/50 mesh	680	14	34	11
2-6633	20/50 mesh	770	11	28	9
2-3947	20/50 mesh	710	16	31	15
2-6681	20/50 mesh	720	18	40	14
II. Duolite C-464 (most results from microscopic examination)					
05-03-4H	20/50 mesh	830	10	20	9
1-4J	720 μm	720	>90	>95	small
5-4H	800 μm	800		>95	
--	700 μm	700		>95	small
2-60	730 μm	730	>90	>95	
3-61	730 μm	740	>90	>95	

^aUranium capacities of (52 to 78) $\times 10^{-6}$ g/sphere.

^bUranium capacities of (36 to 100) $\times 10^{-6}$ g/sphere.

^cUranium capacities of (28 to 52) $\times 10^{-6}$ g/sphere.

drying. Sticking together or clustering of two or more spheres is also troublesome if the resin is not agitated. The maximum temperature must be limited to prevent thermal degradation. As a result of these requirements, drying in a rotary-drum vacuum dryer, with microwave heating, and in a fixed bed were all unacceptable. The LOD (loss on drying) of the resin must be lowered to approximately 20% or less, but the resin must not be dried to the point of excessive static charge development or resin damage. The wet, drained resin has a moisture content of approximately 3.8 lb of water per pound of dry resin or an LOD of 79%. To reach an LOD of 20%, 3.6 lb of water per pound of dry resin must be removed.

Fluidized-bed drying was selected as a method offering acceptable control of drying temperature and product dryness, as well as maintaining an agitated bed to keep the particles from forming clusters. The suitability of fluidized-bed drying was confirmed using a rental laboratory unit.⁵ The carboxylic acid resins in the sodium form were dried to 100°C and rewet with no detectable change in exchange capacity; hydrogen-form resins show a loss of capacity.

4.1 Fluidized-Bed Dryer Equipment and Procedures

The fluidized-bed drying is performed in a commercial, packaged drying unit^{*} as shown schematically in Fig. 6. The product container, with a capacity of 2.9 ft³, is filled with wet resin and sealed pneumatically into the dryer. The electrically driven blower pulls room air through the bed at 800 to 1200 cfm, and the air is heated at the inlet

* Model FA-75 Fitzaire Fluid-Bed Dryer, the Fitzpatrick Company, Elmhurst, Ill.

ORNL-DWG 76-570

Fig. 6. Fluidized-bed dryer used for ion exchange resin.

by the finned-tube steam heater. The inlet air temperature can range from ambient to 230°F. The temperature is controlled with a proportional controller and an air-actuated valve on the steam inlet. Air flow and fluidization are controlled by a damper directly preceding the blower.

A typical drying run begins with the loading of the wet resin into the bed. After the product container is sealed into the dryer, the air flow begins and the inlet air temperature is set. While drying can be done at other temperatures, 65°C is preferred for the inlet air because it results in a reasonable drying time with no damage to the resin. The damper is manipulated to produce a well-agitated bed, as observed through viewports. Due to the sticky nature of the wet resin, a smoothly fluidized bed cannot be achieved at the beginning of the run. There is always a good deal of slugging, channeling, and expulsion of material from the bed.

Conditions and procedures for use of the pilot-plant scale fluidized-bed dryer were tested as a problem for the MIT School of Chemical Engineering Practice at Oak Ridge.⁶

4.2 Fluidized-Bed Dryer Results

Three drying periods can be observed. At first, the bed is merely warmed to equilibrium with the fluidizing air. In the second period, the drying reaches a constant rate, and the outlet air temperature remains constant at about 40°C. Finally, a falling rate period is entered. The resin shrinks in volume by a factor of 4.3 and begins to fluidize more evenly. This shrinkage is apparent in photographs of the resin (Fig. 5). The outlet temperature begins to climb, and, at an outlet temperature of

about 60°C, the dryer is shut down and the product removed. At this point, the resin has an LOD of 10 to 20%. The elapsed time of drying ranges from 1 to 3 hr depending on the batch size, atmospheric conditions, and the initial wetness of the resin.

The initial drying tests were made with inlet air temperatures of 90 to 95°C and a final exit temperature of about 80°C. These test batches showed a few cracked spheres after uranium loading, but there was no detectable cracking at lower drying temperatures. Resin batches dried to 63°C exit air temperatures showed 14- to 16-wt % LOD for Amberlite IRC-72, and 21- and 14-wt % LOD for Duolite C-464 (Table 3). The alternate Duolite C-464 resin showed surface cracks when it was dried. Shape separation does not reject spheres with surface cracks, and these Duolite batches showed sphere fragments of up to 30% after rewetting, conversion to the hydrogen form, and uranium loading. Apparently, the Duolite C-464 cannot survive our processing for shape separation and will have to meet the shape specification as purchased.

Two dryer runs were made with unsized Amberlite IRC-72 resin. The effect of the wide range of resin size was very troublesome. The filter bag for the exit gas became coated and plugged, thus restricting the gas flow. When drying sized resin, the particles that reached the filter dropped off without causing any difficulty. The unsized resin penetrated and adhered to the filter either because of the fines present or because of the lack of washing which occurred during wet screening. Large amounts of resin fines also dropped through the support screen and collected in the bottom of the dryer entry plenum. This difficulty could be controlled by changing the support screen to a finer mesh. The overall yield of

Table 3. Fluidized-bed drying and particle-shape separation results
for resin feed processing facility

Resin name and batch No.	Drying results				Dry screening results (wt %)		Shape separation reject (wt %)	Shape reject after UO ₂ ²⁺ loading (wt %)
	Maximum exit air temperature (°C)	Wt loss (%)	Volume loss (%)	LOD ^a (wt %)	+26	-50		
					TBC mesh	TBC mesh		
Amberlite IRC-72 No. 2-6681	63		58	14	5.6	1.5	3.40	0.47, 0.49
	63				7.1	0.8	1.38	0.41
Duolite C-464 No. 3-61	63	64	58	21	0.5	0.1	0.95	20
Duolite C-464 No. 1-4J	63	59	70	14	<0.1	1.9	17.6	30

^a Loss on drying to 120°C in air.

size-and-shape-separated resin was reduced as some resin fines adhered tightly to large resin spheres and caused them to be rejected during particle-shape separation. None of these three problems was significant for the reference drying conditions of -26 +30 mesh resin.

4.3 Dry Screening

The clusters and fines which are present after the drying operation are removed by dry screening using a 0.46-m-diam vibratory screen unit.* The resin feed rate to the screen is controlled by a ball valve and is adjusted to supply approximately 1/2 kg of resin per minute. The screened material is continuously removed from the unit. The unit is equipped with 26- (785- μ m openings) and 50- (370- μ m openings) mesh screens to remove the clusters and fines, respectively. The dry screening of resin is satisfactory for these scalping conditions in which most of the particles are much smaller than the large screen and much larger than the small screen.

The amounts of oversize and undersize material have been typically 2 to 15 and 0 to 3 wt %, respectively. The undersize includes very small spheres that were attached to larger spheres during the wet screening, and the amount of undersize depends on the wet screening conditions. The oversize is clusters and can be mostly reclaimed as usable spheres if the clusters are broken up by tumbling or some other mild mechanical treatment. The quantity of such clusters is decreased to the lower end of the range listed for good wet screening and dryer operating conditions (Table 3).

* KASON Corp., Model K18-3-SS.

Screening of dry resin was tested as a primary means of size control in place of wet screening. Since the resin is dried for shape separation, it appears that dry screening might result in fewer process operations. Dry screening is less satisfactory than wet screening for two major and several minor reasons.

1. Operation of the fluidized-bed dryer was unsatisfactory (see Sect. 4.1) when large quantities of resin fines were present.
2. The diameter of "dry" resin varies greatly with the residual water content so that uniform and controlled drying is necessary if the dry sizing is to result in good control of the exchange capacity per particle.
3. Drying of all the resin requires a much larger dryer, and this is more expensive to install and operate than is the wet screening equipment.
4. Dry resin blinds the usual vibratory screen separators, and special types of screening equipment or accessory equipment are required (see Sect. 9.2).
5. Dry screening abrades the surface of the resin (the significance of this change in appearance is not known).
6. The alternate Duolite C-464 resin cannot survive drying (see Sect. 4.2).

The conditions and results for the most promising dry screening tests are in Appendix 9.2.

5. SEPARATION OF NONSPHERICAL PARTICLES FROM SPHERES

The separation of spherical from nonspherical particles is accomplished on the smooth surface of an almost horizontal vibratory feeder. The spherical particles roll rapidly downhill with little effect from the vibration. The nonspherical particles cannot roll and are moved slowly by the vibration to a different exit slot from the spheres. The vibration also provides a controlled flow of particles through a feed slot to the separator. This shape-separator concept has been applied to HTGR fuel particles since 1966,⁷ but units of higher capacity and improved reproducibility have been developed for a resin-feed preparation facility.

The flow rate to a single feed point is limited since the spherical and nonspherical particles must have room to move past each other to their separate exit points; thus, only a small fraction of the plate surface is covered by particles. The amplitude of the vibration is an important operating variable; therefore, multiple feed points to a single plate are practical only if the plate has a uniform amplitude over the entire surface area being used. Vibratory feeders for normal uses do not have such uniformity. The amplitude of vibration and the two angles (to the horizontal) are the three primary variables for controlling the shape separation.

5.1 Equipment Description

Steel plates, 18 x 76 x 1.27 cm, were machined to allow five feed points (Fig. 7). These plates were sent to a manufacturer of vibratory feeders^{*} who was contracted to mount and adjust vibratory units which

^{*}Eriez Magnetics, Erie, Penn. 16512.

Fig. 7. Principal shape separator details.

could provide a useful range of uniform amplitudes of vibration. Although a single vibrator for each unit was proposed initially, the manufacturer recommended and delivered each unit with two vibrators on a single-control rheostat.*

The vibrators with the plate were mounted on a stand designed to satisfy three requirements (Fig. 8). The slope may be adjusted by three screws acting as a tripod. This slope is most conveniently measured as the angles of the two plate axes to the horizontal. The two exit streams were collected in separate containers. The five feed points were fed by gravity from a single vessel with feed nozzles (described in Sect. 5.2). Three identical units were installed as part of the resin-feed facility. The dry screening and shape separators were installed in a room with controlled humidity to prevent excessive changes in moisture content of the resin during these operations (Fig. 9).

5.2 Particle-Shape Separation Procedures

The operating procedures for the particle-shape separators are easy to teach and optimize by visual observation, but cannot be reduced to quantitative formulas. The controlled variables cannot be measured with accuracy. The effects of the particle properties (size, density, surface texture) are also variables. The performance of the five-unit shape separators was reproducible and did not normally vary with time nor with overnight shutdowns of the units. The initial adjustments do not result in any loss of materials because any poorly separated materials are simply returned to the feed vessel.

*Eriez Model 45A-2 with GS-115 Control Unit. (Each vibratory: 115 V, 60 Hz, 30 W, 1.75 A).

Fig. 8. Photograph of five-unit shape separator.

Fig. 9. Dry screening equipment and shape separators in a controlled humidity room.

The capacity and the sensitivity of the separation can be changed by varying the power input to the vibrators and the inclination of the two plate axes to the horizontal. The vibration power must be sufficient to move the nonspherical particles to the discharge slot, but excessive power bounces the slightly nonspherical particles so that they may roll and be discharged with the spherical particles. The discharge of nonspherical particles is assisted by setting the plate at a slight axial angle downhill toward the reject discharge slot. The cross angle is adjusted so that the spherical particles roll across the plate at an approximate 45° angle. Typical values for good particle-shape separation of the ion exchange resin were: 1° axial angle, 1.5° cross angle, and 60% on the control rheostat (full power is 30 W/vibrator). The amplitude of the vibration appeared to be about 0.2 mm. Increasing the angles or the vibration amplitude increases the capacity with some loss of separation efficiency. The resin particles separate into two distinct streams, with some nearly spherical particles following intermediate paths between the two streams. By observation, the angles and vibration can be adjusted to maximize this separation.

The effects of extreme values of the three control variables are primarily as follows:

1. High-power input (high amplitude of vibration) bounces the particles so that the separation between the streams of spheres and nonspheres is less distinct.
2. Low-power input (low amplitude of vibration) results in inadequate or no movement of nonspherical particles so that the plate surface becomes overloaded.

3. High axial angle results in poorer separation of the streams of spheres and nonspheres.
4. Low axial angle results in inadequate or no movement of nonspherical particles so that the plate surface becomes overloaded and also reduces the feed rate.
5. High cross angle allows more cross movement of nonspherical particles and thus results in less effective separation or acceptance of slightly nonspherical particles.
6. Low cross angle results in a change in angle of the stream of spheres so that the stream is nearer the shape reject slot and some spherical particles are rejected.

5.3 Results

These units provided a satisfactory and reproducible shape separation. The five separation areas on a single plate are usable for a range of operating conditions which are easily identified by observation of the particle behavior. Although there are differences between the three units and between the end and center areas of a single unit, these differences are within the range of acceptable conditions and do not compromise the operation.

Irregular shapes (halves of spheres, fragments, clusters, extruded shapes) are separated from spherical particles very efficiently. For rounded particles, the degree of nonsphericity can be measured as the ratio of maximum diameter to minimum diameter. Even when this ratio is as small as 1.1/1, particles that are chipped, egg-shaped, or have small teats can be rejected with high efficiency. Spherical particles with

surface cracks as the only defect are not separated effectively. If the accepted material from good operation is separated a second time, the second-pass reject fraction will generally be $<1/10$ of the first pass, and nearly all the particles will contain very small defects (Table 4).

The movement of nonspherical particles is much slower than that of spherical particles. As a result, the capacity of the shape separators decreases as the fraction of nonspherical particles increases. Using the three units in the resin feed facility, shape separation performance was excellent for a feed rate of 2.5 kg per hour of resin containing less than 1% nonspherical particles but was no more than adequate for 1.4 kg per hour of resin containing about 30% nonspherical particles. These feed rates were about 8000 and 5000 particles/sec, respectively (the total for 15 feed points).

Some alternatives were tested for fabrication or design variables. The finish of the separation surface must be good. For the ion exchange resin, a 32- μ in. ground finish was not adequate, whereas a finish of about 16 μ in. was acceptable. The plate with the 32- μ in. finish was made acceptable by coating it with a standard automobile paint. The most satisfactory feed arrangement was a small block clamped to the plate with a rubber line to the feed vessel and an approximately square port open to the separation area (Fig. 10). Development units were fabricated with an adjustable spring to vary the opening size, but the final three units were fitted with fixed 1.6-mm-high by 3-mm-wide openings. The openings must be at least three times the average particle diameter

Table 4. Results for rerun of shape-separation product fractions

Resin: Amberlite IRC-72, Batch 2-6681

Shape separation feed stream	Product stream	Yield (wt % of feed)	Appearance (visual)
26/50 Dry screening	Accept	98.6	All perfect spheres. No cracks.
	Reject	1.38	Mostly clusters. No cracks.
First shape separation accept fraction	Accept	>99.8	All perfect spheres.
	Reject	<0.2	Nearly all would meet shape-accept specifications; spheres with small teats.
First shape separation reject fraction	Accept	47	Mostly good spheres; some spheres with teats.
	Reject	53	Nearly all clusters or teats on spheres.
+26 Dry screening	Accept	7.1	Nearly all clusters.

ORNL-DWG 76-14632A

Fig. 10. Feed nozzle for shape separator.

to avoid flow stoppages from bridging, and any grossly oversized particles must be removed by presizing. Flexible rubber lines were used, and the weights of the feed blocks were kept to a minimum in order to avoid affecting the vibration characteristics of the plate. The slot angle shown in Fig. 7 resulted in better discharge of nonspherical particles than did a slot at a right angle to the plate axis.

Some shape separation results for different resin batches are tabulated (Tables 4 and 5). The shape defects typical of the two resins are shown by photographs of Amberlite IRC-72 (Fig. 11) and Duolite C-464 (Fig. 12). This uranium-loaded Duolite C-464 does not show the cracking which occurs when Duolite C-464 is dried in the sodium form.

6. RESIN CONVERSION

The conditions for this resin conversion have not been optimized. The resin-feed preparation facility was designed for a batch conversion because the remainder of the feed preparation is batch. The conversion is done in three steps:

1. The resin volume is measured. About 1.8 moles of HNO_3 per liter of resin is circulated for about 30 min and then decanted to waste. For routine operation, this acid would be the stored acid from the second step of a previous conversion.
2. A second acid addition is made with circulation for more than 3 hr with pH less than 0.5. For routine operation, this batch of acid is stored for use in the first step of the next conversion; a large excess of acid can thus be used without being wasted.

Table 5. Summary of shape separation results vs resin lots and treatments

Resin	Mfg's lot No.	Size range of wet, Na ⁺ form	Form when shape separated	Rejects (wt %)	Feed resin pretreatments or remarks
Amberlite IRC-72	2-6681	26/30 TBC	Na ⁺	0.56, 0.65	No pretreatment
			UO ₂ ²⁺	0.4	Dried 160°F, shape separated
			UO ₂ ²⁺	3.33	Dried 200°F, shape separated
	2-3947	26/30 TBC	Na ⁺	15.8	No pretreatment
			UO ₂ ²⁺	1.5	Dried 200°F, shape separated
			UO ₂ ²⁺	28.6	No pretreatment
		22/26 TBC	UO ₂ ²⁺	9.9, 6.5	No pretreatment
			Na ⁺	24.3	No pretreatment
			UO ₂ ²⁺	10 to 15	M&C Division results
	2-5612		UO ₂ ²⁺	0.27, 0.46	Dried and shape separated
Duolite C-464	2-60	740 μm mean	Na ⁺	8	No pretreatment
	1-4J	720 μm mean	Na ⁺	<0.1	No pretreatment
			UO ₂ ²⁺	0.25	No pretreatment
			UO ₂ ²⁺	14.1	No pretreatment
			UO ₂ ²⁺	9.3	M&C Division results

Y-140347

Fig. 11. Shape classification of Amberlite IRC-72.

Y-140344

800 μ m

Fig. 12. Shape classification of uranium-loaded Duolite C-464.

3. The converted resin is washed with demineralized water to a pH above 3.

6.1 Batch Conversion Equipment and Procedures

The resin conditioning part of the resin processing facility (Fig. 13) was manifolded to allow flexibility of operation. The use of three (Na^+ to H^+ to Na^+ to H^+) or more conversions was considered a possibility, but is now believed unnecessary. The resin conditioning tank (Fig. 13, T-1) has a cone-bottom screen with a center discharge opening to a ball valve. The dried, shape-accept resin is added to water in tank T-1. The operations listed in Sect. 6 are carried out as needed by using fluidizing conditions in T-1 to provide mixing and good solution-resin contact. The first acid addition is normally the solution in tank T-5 remaining from a previous conversion, and the depleted acid- NaNO_3 solution is sent to tank T-3 for neutralization and discharge. The new acid used to complete the conversion is stored in tank T-5 for use in the next conversion run. Washing is by fluidization with demineralized water. The conditioned resin is discharged through the bottom valve for storage or transfer.

Three spectrochemical analyses of batch converted resins show 2, 5, and 40-ppm sodium (Table 6), although chemical analyses have more commonly shown 20 to 80 ppm sodium. Any exchangeable Na^+ remaining in the resin should exchange with UO_2^{2+} during resin loading and large amounts would accumulate in the uranyl nitrate solution before the equilibrium amount in the resin was significant. Such accumulations could result in increased recycle of uranium as waste solutions, but it provides protection against excessive sodium contamination of the uranium-loaded resin.

Fig. 13. Resin conditioning system flowsheet.

Table 6. Spectrochemical analyses of resin by the ORNL
Spectrochemical Laboratory at the Y-12 Plant

Resin name	Amberlite		Duolite		Amberlite
Resin type No.	IRC-72		C-464		IRC-72
Batch No.	2-5612, 2-5990		5-4H		2-5612
Resin form	Hydrogen		Hydrogen		UO ₂ ²⁺
Concentration units	<u>μg/ml</u>		<u>μg/ml</u>		<u>μg/g</u>
Al	25	30	30	10	10
B	4	3	10	1	3
Ca	20	30	20	20	20
Co	<3	<0.5		3	3
Cr	70	12	100	40	40
Cu	3	12	2	3	5
Fe	100	15	200	150	400
K	1	9	1	3	30
Li	0.3	0.03	0.2	0.2	0.3
Mg	10	60	5	1	1
Mn	2	6	5	3	3
Mo	5	0.09			
Na	5	40	2	60	80
Ni	4	9	7	3	5
P	5		2	20	50
Si	100	150	200	40	40
V	≤1	<0.1		3	0.1
W	20	<0.5			
Zn	3	12	4	15	15
Zr	12	<0.5			
Rare earths	All <1				

Duolite C-464, which was dried in the sodium form and shape separated, showed large amounts of fine fragments of spheres during conversion.

6.2 Continuous (Higgins) Column Results

A continuous, 2-in.-ID contactor of the Higgins type was tested for continuous resin conversions in place of the reference batch conversions. Results obtained for the conversion of Amberlite IRC-72 from the sodium to the hydrogen form are excellent. An in-line pH measurement for the effluent NaNO_3 solution allows easy adjustment and control of the resin/ HNO_3 ratio. For about 10% excess HNO_3 , the washed product resin showed 0.5 to 5.0-ppm sodium. The best initial charge procedure is to fill the column with sodium-form resin, then start the normal acid feed rate, and finally start the resin feed when the solution effluent drops to the selected control pH. The resin remaining in the column when all of a batch of resin has been fed can be easily processed to completion. Acid flow is stopped when the effluent pH indicates low utilization of acid. Wash water is continued until the effluent pH indicates good washing, and the resin is then discharged completely using the valve settings for the resin-pulse part of the operating cycle. The large volume change (hydrogen-form resin has half the volume of sodium-form resin) causes no difficulty. More detailed results are reported elsewhere.

8

For these tests, the flow rates were 0.1 to 0.2 liter/min of 1.24 or 1.30 N HNO_3 , 0.2 liter/min of wash water, and 0.025 to 0.12 liter per minute of hydrogen-form resin product. The effects of process control procedures, startup and shutdown procedures, and resin volume changes were examined.

In theory, either the resin rate or the acid rate could be varied to control the conversion operation. The concentrations of the feed acid and feed resin are preset constants. The pH of the effluent solution is easy to measure but the percent conversion of the resin is not. The load time directly controls the resin rate, and controlling the effluent pH by varying the load time is very logical and effective.

The traditional method of process control is to measure and control the solution flow rates and control the resin rate by varying the load time and the pulse time by setting timers. The load time controls the number of pulses per hour, and the pulse time controls the size of each pulse (see Fig. 14 for the effect on effluent pH that results from varying these two plus the acid feed rate). The product of these two yields the resin rate. The timers required frequent adjustments to maintain the effluent pH within desired limits. With experience the correct timer settings could be determined. Since the effluent pH was the variable of concern, switching to manual control of load time based on observation of effluent pH was a logical alternative. This alternative method required the operator to observe the effluent pH and to initiate a pulse when the pH reached a certain value (typically 1.0). This manual feedback control proved effective and provided consistent and reproducible operation. The traditional control method requires damping action from the operator. The amount of damping would be highly dependent on the skill and experience of the operator.

The second method, although tested for manual control only, required only an automatic response. An automatic feedback control system would duplicate the manual feedback control and would maintain good operating

Fig. 14. Exit solution pH for sodium to hydrogen-form conversion of Amberlite IRC-72 using HNO₃.

conditions unattended. For example, if the acid throughput suddenly decreased or increased, the original system would not respond automatically; however, the proposed system would automatically decrease or increase the resin throughput to match the new acid throughput. The efficiency of acid utilization was 79 to 99% for these tests but was not of primary concern. The proposed control procedure from the effluent pH can easily be used to achieve a high efficiency.

The Amberlite IRC-72 resin undergoes a volume reduction of about 50% upon conversion from sodium form to acid form. This phenomenon did not hinder continuous acid conversion.

Startup presented no problem and is mentioned only because one of the two methods used was more desirable for acid conversion than the other. One method began with the contactor full (to the feed point) of sodium-form resin. Acid was fed through the resin bed until the effluent pH reached a certain value; then normal operation was started. The other method began with a column full of acid and resin was gradually introduced into the column. Once enough resin was present, normal operation was started. It was noted that a larger quantity of acid was lost (some acid was pushed out the resin exit) with this second method, thus making the first method the preferred startup procedure.

Completion of the conversion at the end of a batch of resin was also simple to accomplish. The acid flow was continued until the exit solution was acidic ($\text{pH} < 0.7$), at which point the acid was shut off and wash water continued until a $\text{pH} > 3.5$ was reached. The resin was then flushed from the column by a continuous pulse flow of several minutes duration.

6.3 Quality Assurance

Measurements must be made to verify that the product of the resin-feed processing facility meets the specifications for recycle fuel preparation. The purchase requisition for resin will require that the manufacturer certify that the composition and manufacturing procedures duplicate those of a previous lot of acceptable resin. The only measurements planned for the resin as purchased are the cation exchange capacity and a microscopic comparison of size and shape.

The converted hydrogen-form resin product will be sampled and stored while quality assurance measurements are completed. These quality assurance procedures have not yet been specified. The importance of some properties are presently being determined by irradiation tests. Examples of spectrochemical analyses are shown in Table 6.

It is probable that one quality assurance test for processed resin will be a loading of a small batch with natural or depleted uranium followed by carbonization, conversion, and measurements of the converted resin. The commercial resin is produced in lots which will provide specification resin for 150 kg or more of uranium per lot, and one test with depleted or natural uranium should qualify a complete lot.

7. SUMMARY AND CONCLUSIONS

Process development was completed and a facility was designed, procured, and installed for resin-feed processing to meet HTGR recycle fuel specifications. The capacity of the individual process components are equivalent to about 1 kg of uranium per hour. This allows efficient use

of operating labor to meet present requirements, and a commercial recycle plant will require little scale-up of the principal components.

The specifications for the resins are derived from those for carbonized, recycle fissile kernels. The resins are commercial carboxylic acid cation exchange resins in the sodium form, but the suitability of a specific brand and type number must be demonstrated experimentally. The compositions, microstructure, and loading behaviors of a specific commercial resin appear to be reproducible. The resin feed processing includes operation to control the resin size, to remove nonspherical particles, and to convert to the hydrogen form.

The sodium-form resin is fed as a slurry (water/resin volume ratio of 40) to a 0.76-m-diam vibratory screen separator. The uranium capacity per sphere of about 65×10^{-6} g of uranium requires wet sodium-form resin of 730 μm for either Amberlite IRC-72 or Duolite C-464. Drying of unsized resin followed by dry screening is much less satisfactory with difficulties for drying, blinding of screens, and variable shrinkage during drying.

The separation of spherical from nonspherical particles is accomplished on the smooth surface of almost horizontal vibratory feeders. The capacity and sensitivity of the shape separation are changed by varying the power input and the inclination (two angles) of the vibratory plates. Three units of five feed points each gave the desired shape separation capacity (5000 spheres/sec). As a preliminary to this shape separation, the sized sodium-form resin is dried in a fluidized-bed dryer. The Duolite C-464 resin shows excessive cracking from drying in the sodium form and will have to meet the shape specifications as purchased.

The sized and shape-separated sodium-form resin is rewet with water and converted to the hydrogen form using a nitric acid solution. The facility was designed for batch conversion, but a continuous conversion and washing column (Higgins type) was also tested. The product quality assurance, storage, metering and packaging procedures, and equipment to meet the requirements of a ^{233}U -recycle facility have not yet been selected and installed.

8. REFERENCES

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9. APPENDIXES

9.1 Appendix A: Drawings of the HTGR Subtask 210 Resin Preparation

P-11708-EE-001	Engineering flowsheet
002 R1	Equipment layout
003	Resin preparation tank T-1
004	Resin preparation tank T-4
005 R1	Tank T-1 support and operator platform
006	Settling and surge tank
007	HNO ₃ & NaOH tanks T-10A & B
008	Surge tank T-12
010	T-9 resin conditioning tank
013	Waste neutralization tank T-11
016	Equipment elevation
S-11708-EB-001	Tank supports
A-11708-EB-002	Resin shape, separator room, plans, section, and elevation
A-11708-EB-001	Resin preparation room environmental control diagram
M-11708-EM-001	Vacuum transfer tank T-6
002	Vacuum transfer tank T-6, details #1
003	Vacuum transfer tank T-6, details #2
EJN-20527 E3E20527-D014	Resin preparation power plan (electrical)

9.2 Appendix B: Size Separation of Dry Resin

The problems and disadvantages of dry screening as the primary size separation procedure were discussed in Sect. 4.3. One resin loading run was made with resin processed by dry size separation. The uranium loaded resin did not show any detectable cracking or other changes in behavior other than an abraded surface appearance. The behavior during drying is reported in Sect. 4.2. The size separation was carried out in the Metals and Ceramics Division laboratories and the results reported^{*} included the following:

Initial tests have been completed to determine whether our Sweco^{**} Turbo-Screen Particle Classifier could be used for upgrading unloaded as-received resin. There was nothing in the results to indicate that it could not.

The tests were conducted in the following manner. The resin in one of the two barrels you supplied was fed into the machine rigged with the Sweco No. 32 screen. The oversized material was given repeated passes until only a small change in the weight was observed. The total undersize material was weighed and a small portion was riffled from it and shape analyzed. The remaining undersize material was then used as feed to the machine rigged with the No. 38 screen and so on with the No. 46 and 50 screens. The numerical results are presented in the attached table along with some pertinent test parameters. These parameters were selected from our experience with Ni-loaded resin. There was no opportunity to determine optimum conditions. Except for the initial passes with the No. 32 screen the feeding was done at a constant rate of about 270-280 g/min by means of a vibratory feeder. Feeding in the case of the passes with the No. 32 screen was done by hand pouring the resin at a more or less constant, but unknown, rate.

You may note that we worked down in screen size, contrary to your suggestion. This was done for two reasons. First, it was noticed that the as-received resin contained a large

^{*} J. F. Carpenter, Jr., internal memo to P. A. Haas, Nov. 11, 1976.

^{**} Southwestern Engineering Company.

number of clumps and clusters and it was felt best to start with No. 32 screen in order to remove these. It was because of these that the vibratory feeder could not be used on the first passes. Second, our experience with screening Ni-loaded Amberlite IRC-72 had shown that the efficiency of separating undersize from a given feed decreases as the percentage of the undersize in the feed decreases. By working down, therefore, we could maintain conditions in which most of the feed was oversized and, thus, high efficiencies of undersize removal could be achieved. This is apparent from the weights of the oversize after the several passes using screens No. 38, 46, and 50. I believe the corresponding results for the No. 32 screen are not the results of an efficiency phenomenon but, rather, a manifestation of the machine's ability to break the clumps and clusters.

Because of the inherent nature of the action of this device, particular attention was paid to the possibility of particle breakage in these present tests and earlier ones with Ni-loaded resin. As seen in the table, shape rejects of 2-4% by weight were observed. For comparison, the shape reject in your loading batch S51 was measured to be 0.14%. You stated that S51 used resin from the same supply lot as the resin used in the present tests but that the resin used in S51 had been wet screened. Clearly the reject appears higher in this present case. However, microscopic examination of the reject in the present tests showed that it was not, in fact, broken particles but mostly very small whole microspheres such as are sometimes seen as "satellites" adhering to larger microspheres. Such small particles were observed in the clumps and clusters of the as-received material. From this I presume that the machine's ability to break up these clumps and clusters is the major reason for the appearance of the small microspheres. The reason why they appear as shape reject is probably that the neck where the particle was attached to a larger microsphere constitutes a fairly extensive flat area on the surface of the small particle. I suspect that your wet screening helps disperse the clumps and that these very small microspheres appear in your final undersize cut.

The good material from the shape analyses was also inspected. The major features observed were a general erosion of the surfaces of the microspheres with exposure to the machine and the presence of irregularly shaped particles of "dust" on the surfaces. Some cracks in the surface and volume of some of the particles were observed but were rare. It is not clear whether this dust is created by the machine or is already present in the as-received resin.

Assessment of Sweco Turbo-Screen Particle Classifier for Upgrading As-Received Unloaded Resin^a

Test Parameters:

Fan Speed : 1600 rpm (2.0 setting)
Wand Pressure : 20 psi
Secondary Air Valve : Fully open
Oversize Chute Cover: Removed

Sweco Screen Number	Size (μm)	Starting Weight (g)	Feed Rate (g)	Weight Oversized (g)	Weight Undersized (g)	Shape Analysis of Undersized		
						Weight Good (g)	Weight Reject (g)	Reject (%)
First pass too coarse to use feeder; merely poured in.								
32	630			6184.6 (1st pass)	12,385.6	372.5	6.5	1.715
				4399.8 (2nd pass)				
				3867.4 (3rd pass)				
				3565.7 (4th pass)				
				3149.3 (5th pass)				
38	503	12,006	273	4761.2 (1st pass)	7527.7	898.4	19.3	2.10
				4509.4 (2nd pass)				
				4440.0 (3rd pass)				
46	412	6,609.7	275	2646.2 (1st pass)	4135.5	501.3	19.2	3.69
				2503.1 (2nd pass)				
				2468.3 (3rd pass)				
50	368	3,617.5	278	1052.9 (1st pass)	2674.1	310	10.5	3.28
				975.6 (2nd pass)				
				948.2 (3rd pass)				

^aJ. F. Carpenter, Jr., internal memo to P. A. Haas, Nov. 11, 1976.

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