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## Testing of Unique Fuel Reprocessing Equipment Designs

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## TESTING OF UNIQUE FUEL REPROCESSING EQUIPMENT DESIGNS

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### ABSTRACT

Unique equipment designs have resulted from the application of revised criticality safety criteria to nuclear fuel reprocessing at the Idaho Chemical Processing Plant (ICPP) located at the Idaho National Engineering Laboratory (INEL). The unique designs resulting include slab and annular tanks, slab decanters, and parallel heat exchange tube bundles on a thermosyphon evaporator. These designs were tested in full-sized mockups to verify proof-of-concept, operating capacities, and operating scenarios. These unique equipment designs have been used in the design of a new fuel reprocessing facility under construction at the INEL.

### INTRODUCTION

Administrative controls, nuclear poisons, and favorable vessel geometries have been used to prevent the occurrence of nuclear criticalities in nuclear fuel reprocessing facilities at the Idaho Chemical Processing Plant (ICPP) located at the Idaho National Engineering Laboratory (INEL). However, in the future it is planned to further enhance criticality safety at ICPP by the sole use of favorable vessel geometries wherever feasible.

Favorable geometries at the ICPP have generally been defined as single vessel geometries which will remain sub-critical by neutron leakage under worst credible conditions when containing a maximum concentration of 500 g/L (as UNH solution) of U-235. The potential for water flooding of the process cells with the associated full reflection of the neutrons is also considered in the criticality analysis. Specific geometric constraints resulting from these criteria are a maximum cylindrical vessel diameter of 12.70 cm (5 inches), a maximum slab thickness of 6.35 cm (2.5 inches), and annular vessels with a 6.35 cm (2.5 inch) thick annulus. Application of these geometries with the associated size constraints has resulted in design of slab and annular tanks for solution storage, a slab geometry decanter, and a thermosyphon evaporator with two parallel heat exchange tube bundles and an annular disengaging head. Since the applications of these geometries to nuclear fuel reprocessing is relatively new, mockups of the equipment were built and tested to verify that the proposed operations could be performed in the equipment and to measure operating parameters for the equipment.

#### SLAB TANK

A slab tank 9.6 m (31.5 feet) tall, 3.51 m (11.5 feet) wide, and 6.35 cm (2.50 inch) between inside surfaces of the slab and having a volume of 1960 L was constructed of stainless steel. The bottom of the tank was sloped 15° to facilitate solids removal. Four sparge lines were installed inside the tank; two near the deep end, one near the center, and one in the shallow end. Two additional lines were located in the deep end for level and density measurement. Ninety-six spacer pins and a jet suction nozzle were installed within the tank to simulate vessel internals. Seven sample ports were located on one side of the tank for obtaining solution samples. An isometric view of the vessel is given in Figure 1.

Solution density mixing tests were performed in the slab tank to verify the ability to adequately mix solutions for process operability and nuclear material accountability purposes, and to determine required mixing times. The tests were performed by adding aluminum nitrate solution of known specific gravity to demineralized water in the tank (in a 1 to 4 volume ratio, respectively) and mixing the solution by air sparging using various combinations of tank volume and sparger air flowrate. Samples were drawn from the sample taps at predetermined time intervals to obtain time-dependent density profiles for the respective sample locations, and the specific gravity was measured using a high accuracy analysis method. Time-dependent density profiles were generated from the data and used to determine mixing times.

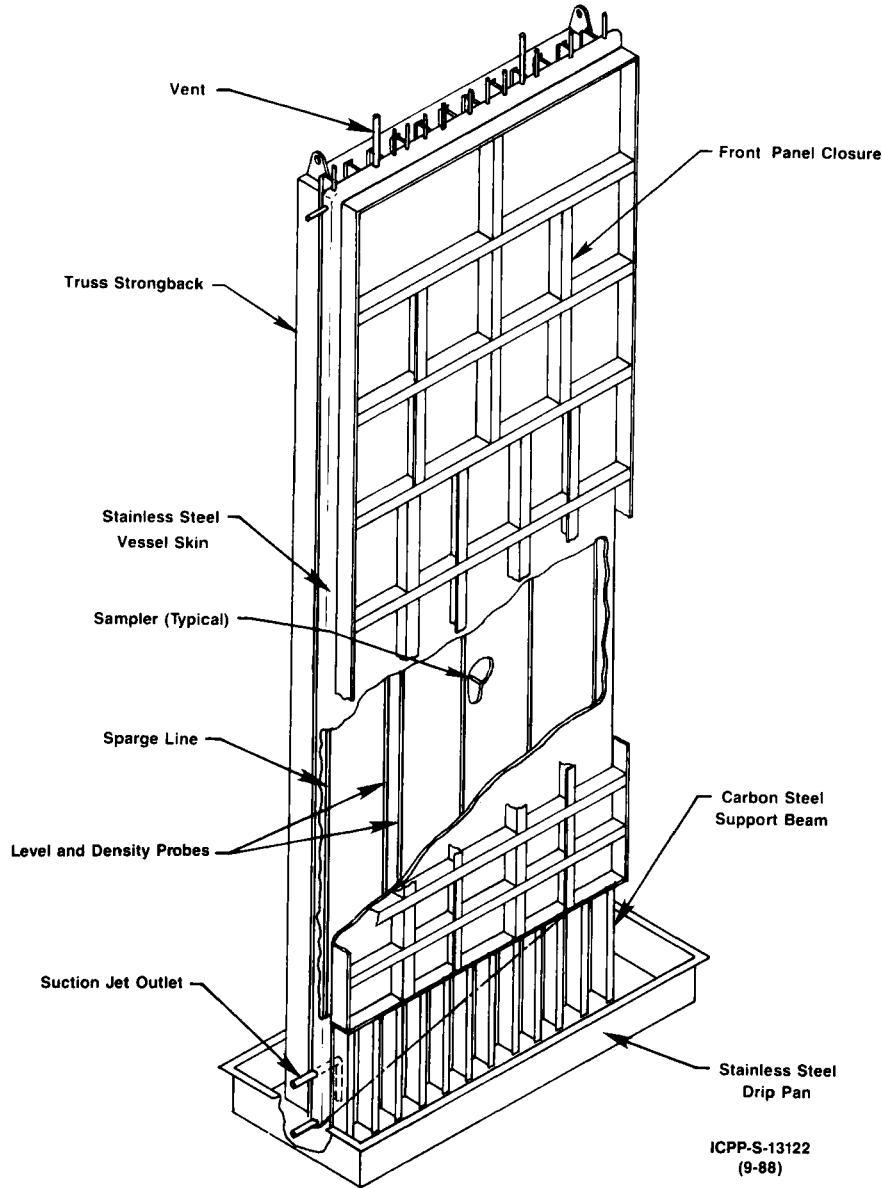


Figure 1. Slab Tank

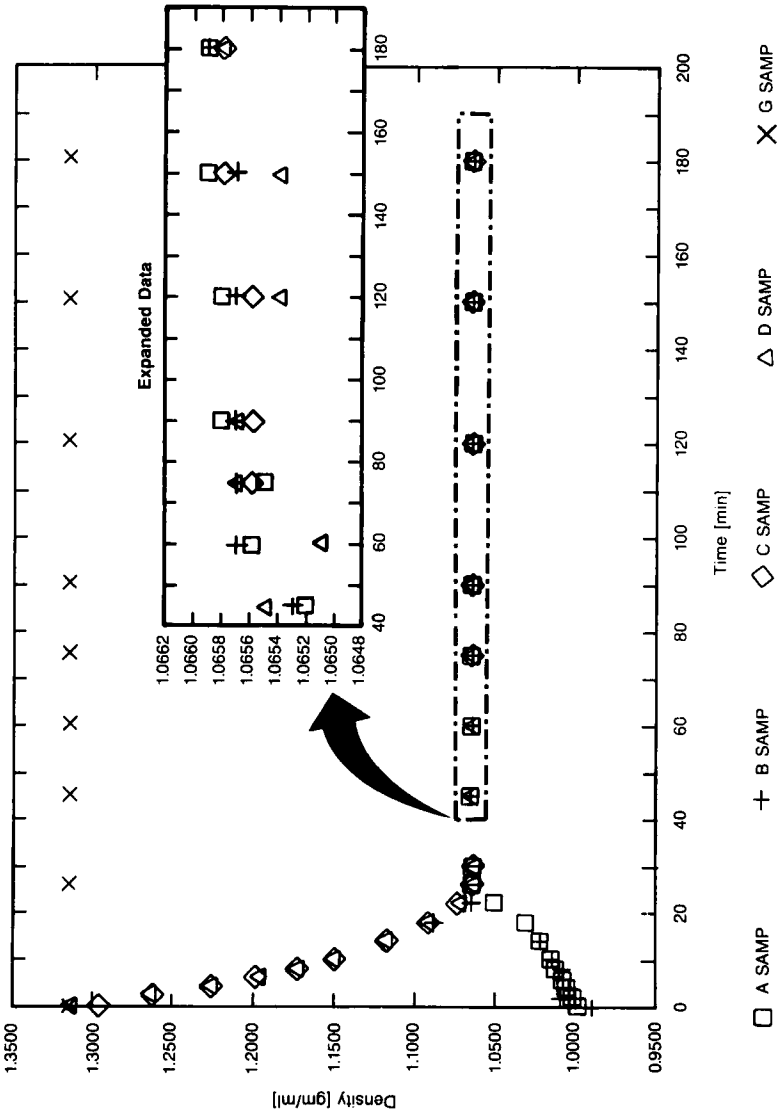
Solution density data from the slab tank showed an exponential convergence to an average value except for several tests where 1 sample point (G sample in Figure 2, sparged at 1.16 standard cubic meters per hour) indicated little or no mixing. Close examination of the data and the vessel design revealed an unmixed region in the deep corner of the tank. A jet suction line in the tank restricted circulation in this region. Forced recirculation of solution from this region to the top of the tank via a drain line allowed complete mixing of the tank contents in the test. The volume of the region is small and will not adversely effect process sample results or can be included as unmixed volume in accountability calculations. Mixing times required to achieve a uniform specific gravity ranged from 12 to 115 minutes depending on the liquid level and sparge air flowrate. An airflow rate of 40 SCFH per sparge was found to provide adequate mixing in a reasonable time while minimizing total off-gas flow. A full tank could be adequately mixed for ordinary operational purposes in 15 minutes and for nuclear material accountability in 30 minutes.

#### ANNULAR TANK

The annular tank was 7.32 m (24 feet) tall, and 1.50 m (59 inch) diameter at the inside face of the outer cylinder wall with an annular gap of 6.35 cm (2.50 inches) and a working volume of 1800 L. The center of the vessel was constructed of stainless steel and the outer cylinder of Plexiglass. The bottom of the tank was sloped 15° to facilitate solids removal. Four sparge lines spaced 90° apart and three level and density measurement probe lines were installed in the tank. An isometric view of the tank is given in Figure 3.

Acid-base mixing tests were performed in the annular tank to visualize solution mixing patterns. The tests were performed by alternately adding small volumes (spikes) of nitric acid and sodium hydroxide to a solution in the tank containing bromothymol blue indicator and then mixing the solution by air sparging using various combinations of sparges. Mixing patterns were observed by watching the color change of the indicator as the spike mixed through the tank.

Mixing flow patterns during air sparging were easily observed in the annular tank. Circulation cells in the tank were observed with some regions of minimal mixing in the center of the cells. The cells were bounded by solution rising near an active sparge line and by solution circulating (falling) about halfway between active sparges. Mixing times (defined as the time required to accomplish a complete color change) varied from 1.5 to 7 minutes



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Figure 2. Typical Slab Tank Mixing Test

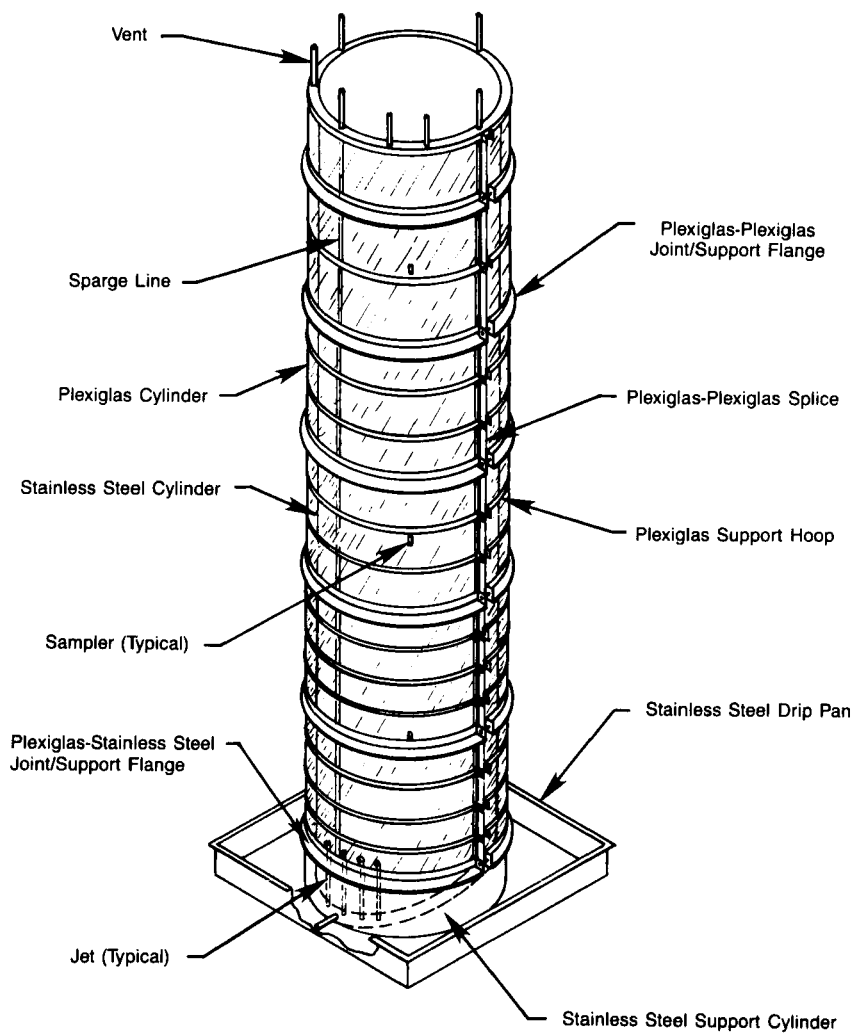


Figure 3. Annular Tank

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depending on the volume in the tank and the number and location of the active sparge lines. The longer mixing times were associated with use of two sparge lines and a quarter-full tank. No dead zones were observed in the tank during the tests.

Solution density mixing tests were also performed in the annular tank to verify the ability to adequately mix solutions of widely varying densities for process operability and nuclear material accountability purposes, and to measure mixing times. The same procedure used for slab tank mixing tests was used for the annular tank.

The density profiles were similar to those from the slab tank, showing an exponential convergence to an average value as shown from typical test results in Figure 4 (0.59 standard cubic meters per hour), however, no unmixed regions were observed. Mixing times ranged from 5 to 140 minutes depending on the tank level, sparge air flowrate, and location of active sparges. An air flow rate of 40 SCFH per sparge provided adequate mixing in a reasonable time while minimizing off-gas flow. A full tank could be adequately mixed for operational purposes in 10 minutes and for nuclear material accountability in 20 minutes.

#### SLAB DECANTER

The slab decanter was 1.22 m (4 feet) wide, 1.17 m (46 inches) tall, 6.35 cm (2.50 inches) between inside surfaces, and the bottom was sloped 15° to facilitate solids removal. The total volume of the vessel was 75 L and it was designed to provide at least 10 minutes of residence time for an aqueous flow of 300 L/hr. The organic weir was located at the top of the vessel in the shallow end, and an external pipe jackleg was used as the aqueous weir. A feed manifold entered the decanter on the deep side 0.79 m (31 inches) up from the bottom of the decanter. The feed manifold was constructed using 1 inch 16 BWG stainless steel tubing. Holes in the manifold were spaced to allow even distribution of the two-phase solution entering the decanter. A vent line connecting the decanter vessel and the aqueous jackleg was used to equalize pressure. A side view of the decanter is given in Figure 5.

Operational verification testing of the slab decanter was performed by feeding the decanter with aqueous/organic mixtures at varying flowrates and aqueous to organic ratios. The organic used was 30 volume percent tributyl phosphate (TBP) in n-dodecane. Organic flowrates ranged from 0 to 100 L/hr. Three different compositions of aqueous solution were used in the tests; water (Sp. Gr.



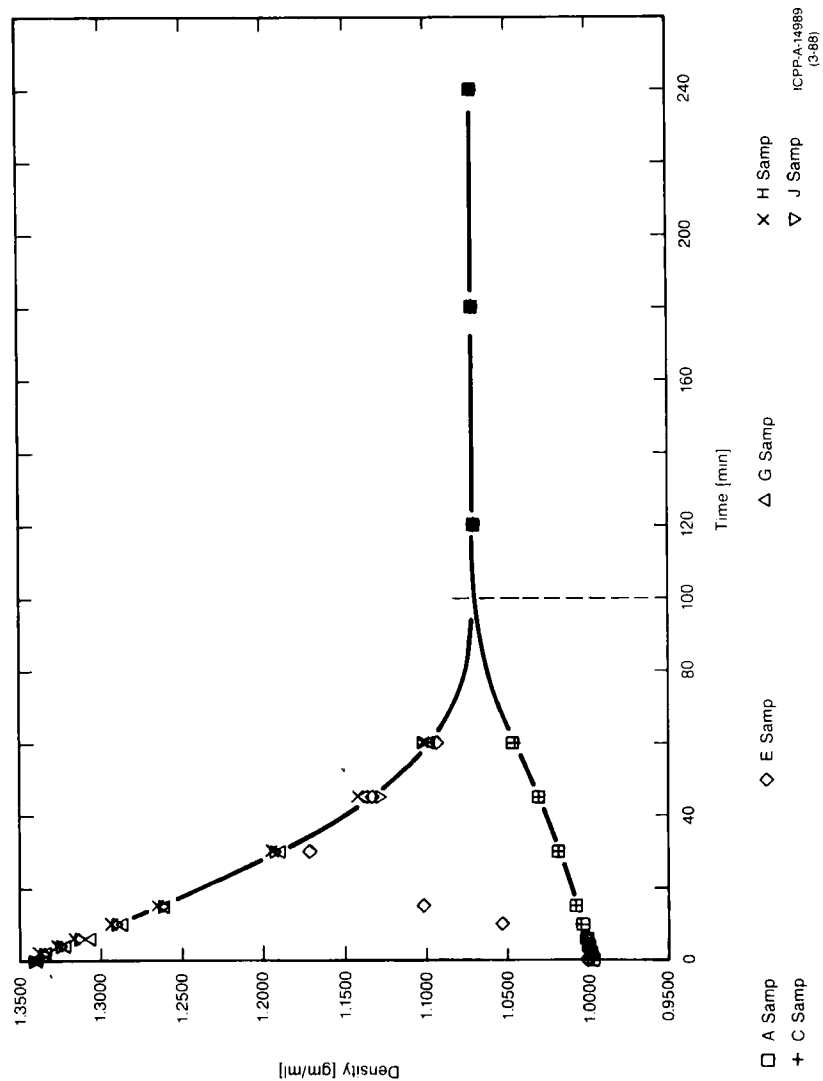
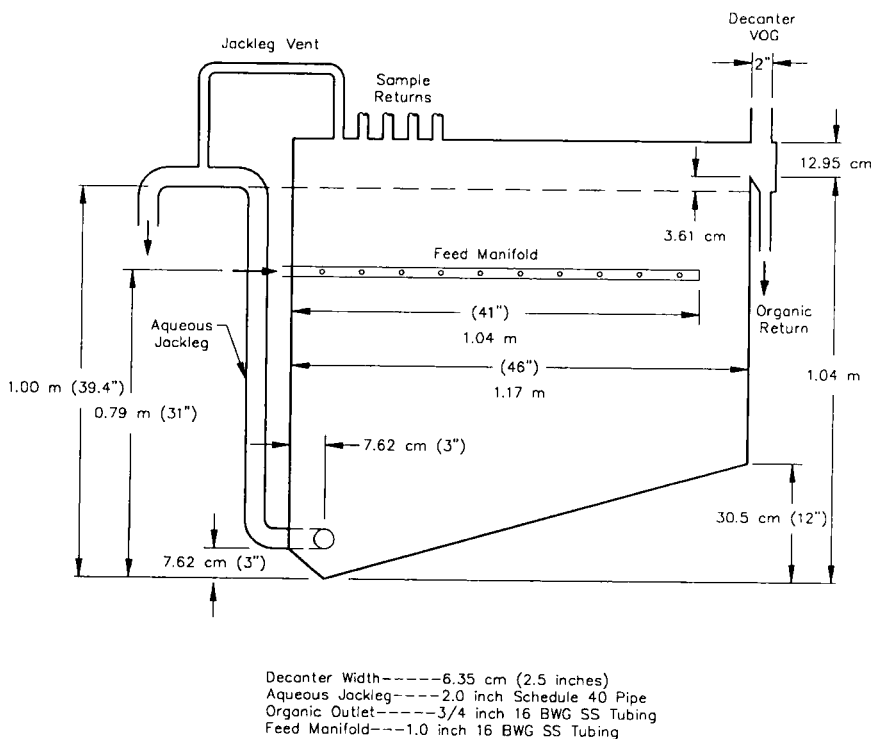


Figure 4. Typical Annular Tank Mixing Test

= 1.0), 3.2 M nitric acid (Sp. Gr. = 1.10), and a mixture of nitric acid and aluminum nitrate each at 1.3 M (Sp. Gr. = 1.24). Aqueous flowrates ranged from 25 to 1450 L/hr. The tests were performed at high aqueous to organic volumetric ratios (between 6:1 and 35:1) in the feed stream. The interface level was measured at flow conditions and the effluent streams were sampled to measure cross-phase entrainment.

The test decanter was able to separate mixed phase feed streams at flowrates more than twice the design flowrate without exceeding a 0.1 % entrainment limit set for the effluent streams. Flooding of the decanter occurred when the total flow to the decanter exceeded 1200 L/hr. Flooding of the decanter was defined as the point where aqueous solution passed over the organic weir. The



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Figure 5: Slab Decanter

specific flow at which flooding occurred depended on the aqueous density. The interface position was primarily affected by flow-rates and aqueous density for the conditions tested. The resistance to flow through the aqueous jackleg and over the organic weir added to the static head of each phase to affect the interface position.

#### THERMO-SYPHON EVAPORATOR

The test evaporator was a thermo-syphon type consisting of two heat exchangers, a central column, and an annular vapor-liquid disengaging head. The shell of each heat exchanger tube bundle was 15.4 cm (6.065 inches) inside diameter (ID) and contained 23, 3/4 inch OD 10 BWG tubes, 3.66 m (12 feet) in length. The total surface for heat transfer based on outside tube diameter was 10.0 m<sup>2</sup> (107.6 ft<sup>2</sup>). Supply steam entered the top of the tube bundle shell and flowed countercurrently to the process fluid rising in the tubes. Steam condensate collected at the bottom of the tube bundle shell side, then was forced by the live steam to flow through a 3/4 inch schedule 40 pipe to the top of the tube bundle where it exited. The central column was a 4 inch schedule 40 pipe, 4.95 m (16.25 feet) long. Feed entered the lower half of the central column through a downcomer from the bottom tray in the vapor-liquid disengaging head. Concentrated product exited the central column through an overflow line at about the same elevation as the top of the heat exchange tube bundles. The annular vapor-liquid disengaging head was 1.13 m (44.5 inches) outside diameter (OD) by 1.03 m (40.5 inches) ID. A wire mesh mist eliminator was located at the top of the head. Four bubble cap trays with 60 bubble caps per tray spaced equally around the tray were also located in the head. Sample ports, pressure taps, and thermocouples were located throughout the evaporator for collecting solution samples and providing operating data. A flow meter was located in one of the heat exchange tube bundle inlet lines to allow measurement of recirculation rates. A schematic of the evaporator is given in Figure 6. Evaporator operating pressure was maintained by a steam jet (coarse vacuum) and air bleed system (fine vacuum control).

Proof-of-concept and evaporator capacity tests were performed by operating the evaporator at different boilup rates varying from 62 to 668 L/hr. Both water and nitric acid were used as process fluids for the tests. Feed rates for the acid tests ranged from 256 to 422 L/hr with a concentrate (bottoms) acid molarity of about 5.6. The evaporator was operated at steam pressures ranging from 73.1 to 99.3 kPa (10.6 to 14.4 psia). The evaporator was started using liquid level as the setpoint for steam flow control until the desired bottoms concentration was reached. Then the bottoms density was used as the setpoint for control. The product stream was

then allowed to gradually increase until it began to overflow from the evaporator. Acid stripping in the disengaging head was tested by feeding the evaporator on one of the top three bubble cap trays. Operation for each test proceeded until steady-state was reached (10 to 12 hours) and then was maintained for 1.5 to 2 hours. Samples of the process streams and of the liquid on the bubble cap trays were then taken.

Heat transfer coefficients measured in the tests ranged from 300 to  $1700 \text{ W/m}^2\text{K}$  and compared favorably to heat transfer coefficients reported in the literature<sup>(1,2)</sup>, see Figure 7. No surging or other hydraulic instabilities were observed during the tests indicating that parallel heat exchange tube bundles can be used. Data from the disengaging head indicated that the bubble cap trays were effective at stripping nitric acid from the feed yielding a

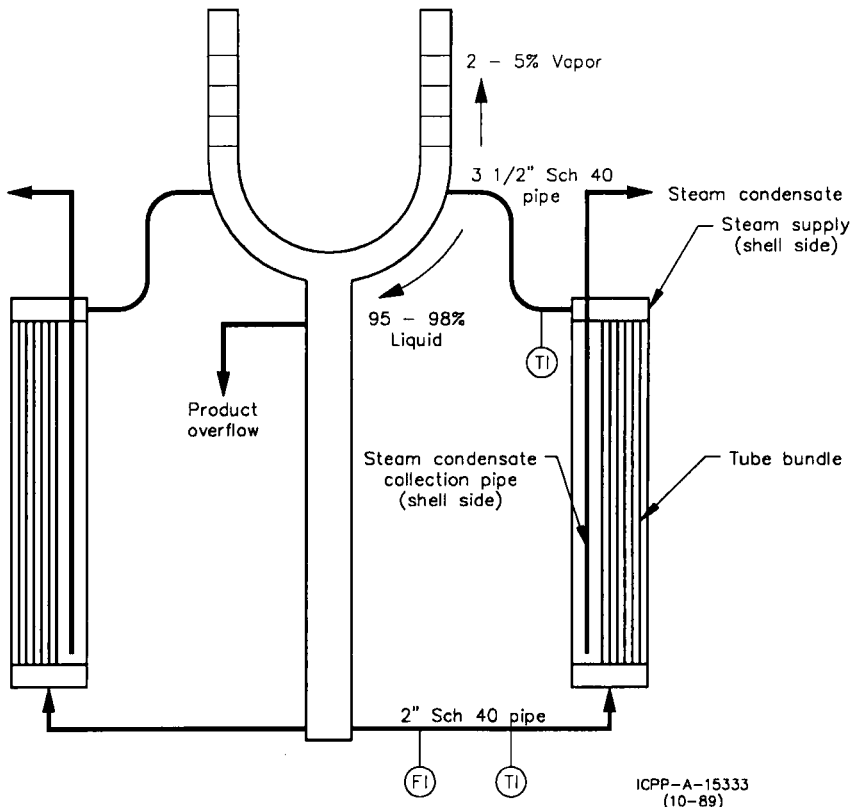
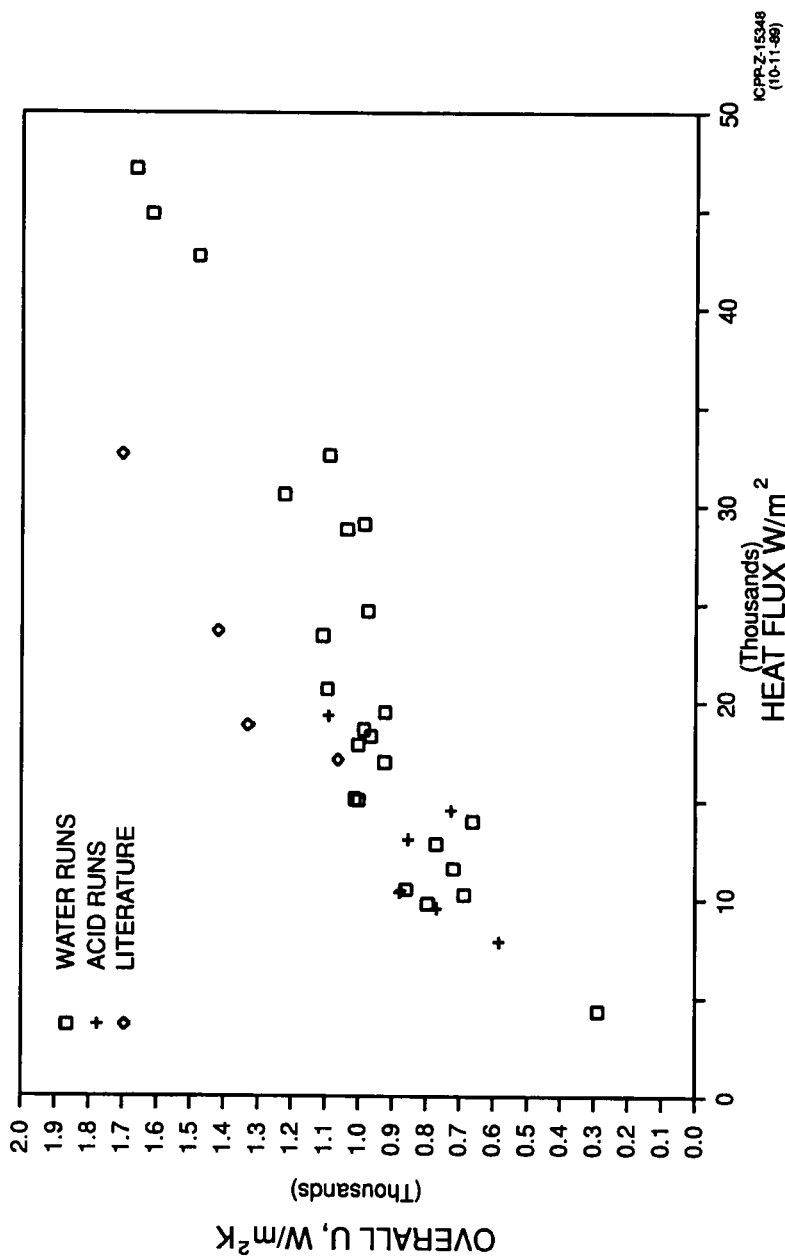


Figure 6. Thermosyphon Evaporator Schematic



## FIGURE 7. OVERALL U vs HEAT FLUX

low acid overhead stream (0.0026 M nitric acid) suitable for recycle within the process.

### CONCLUSIONS

These tests showed that unit operations typical of nuclear fuel reprocessing could be accomplished in favorable geometries. Problem areas in the equipment design were identified and action taken to resolve them. Operating parameters for the various pieces of equipment were also measured providing operating experience with the equipment.

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