Design and Test of a Thermosiphon Evaporator for Acid-Deficient Uranyl Nitrate

J. P. Drago
P. A. Haas

MASTER

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DESIGN AND TEST OF A THERMOSIPHON EVAPORATOR
FOR ACID-DEFICIENT URANYL NITRATE

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ABSTRACT

The reference process to recycle ^{233}U to HTGRs begins with the receipt of the $^{233}\text{UO}_2(\text{NO}_3)_2$ product solution from a reprocessing plant, which is fed to a resin loading system. The nitrate is removed by extraction with a liquid organic amine. By adding an evaporator to remove water, a continuous, high-efficiency process for loading uranium on ion exchange resin is possible. A vertical thermosiphon evaporator was designed, fabricated, and tested with natural uranium to meet the requirements of the HTGR fuel recycle Hot Engineering Test Facility (HETF).

The thermosiphon evaporator, which was designed to produce 60 liters of condensate per hour containing less than 1 ppm of uranium, performed satisfactorily and was simple to control. The uranyl nitrate solution was circulated continuously through the evaporator during operation of the resin loading system, with the liquid level being determined by a fixed overflow jackleg. The evaporation rate was controlled by regulating the steam supply pressure. The condensate contained less than 0.5 ppm of uranium, and the uranium decontamination factors (DFs) were 5×10^5 to 1×10^6 at boilup rates of 17 to 79 liters/hr. This evaporator design is adequate for remote operation, control of criticality, and other requirements for the proposed application.

1. INTRODUCTION

A full-scale system to simulate part of an HTGR fuel recycle Hot Engineering Test Facility (HETF) was designed fabricated, installed, and operated with natural uranium. One essential component

was a thermosiphon evaporator for concentrating acid-deficient uranyl nitrate solution. This report discusses the design and testing of the thermosiphon evaporator. The overall process is described briefly, and the requirements for the evaporator are reviewed more fully. The evaporator was designed to check critical concepts or requirements for remote operation 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 resins with uranium and carbonizing at controlled conditions. 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 ratio of ~ 2.2). The reference flowsheet for a ^{233}U recycle fuel facility at ORNL 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 . The addition of an evaporator to remove water (Fig. 1) makes it possible to obtain a highly efficient conversion of the purified $^{233}\text{UO}_2(\text{NO}_3)_2$ solution to loaded resin.

1.2 Objectives of Evaporator Development

The evaporator is part of an engineering-scale resin loading system that is full scale with respect to the Hot Engineering Test Facility. This system provides information relative to meeting the requirements for remote operation, control of criticality, material accountability, and interactions of individual operations. The engineering-scale system does not meet detailed requirements for containment, waste disposal, or shielding to allow operation with ^{235}U or ^{233}U and is therefore limited to use with natural uranium. Our intention was to

Fig. 1. Schematic reference flowsheet for resin loading.

obtain information on concepts or requirements that are unique to the evaporator, with little emphasis on requirements where generally accepted design solutions are known.

2. PROCESS REQUIREMENTS

The evaporator design must meet the requirements that result from application of the reference flowsheet (Fig. 1) to recycle ^{233}U . The feed and product stream compositions result from 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 also be considered.

2.1 Evaporator Performance

In most cases the feed to the evaporator will be about 0.3 M uranyl nitrate with NO_3^-/U mole ratios of 1.5 to 2.0. The engineering-scale resin loading system is designed for batch loading of resin, with 4 kg of uranium per batch. While the capacity of the pilot plant is based on one batch per day, the individual operations require a maximum of 4 hr per batch. The nitrate extraction system is continuous, with a capacity equivalent to at least 1 kg of uranium per hour. The evaporator must, on the average, have the capacity required for processing one 4-kg batch of uranium in 4 hr; the acid-deficient uranyl nitrate (ADUN) surge tank will accommodate the fluctuations resulting from the batch operations.

The water (in liters) added to the system for one 4-kg batch of uranium includes the following:

Uranyl nitrate feed plus rinse	50
Resin feed plus rinse	10
Uranium recovery scrub contactors	30
Resin wash	<u>30</u>
Total	120

Removal of this amount of water over a 4-hr period generates a minimum of 30 liters of condensate per hour. Rates up to 60 liters/hr would allow

more effective control of process concentrations; 30 to 60 liters/hr is the preferred range of capacity.

The evaporator was designed to meet the following requirements:

- (1) low maintenance, compact arrangement, and simple operation;
- (2) low uranium concentration (< 1 ppm) in the condensate;
- (3) low holdup (~ 5 liters);
- (4) continuous operation with a boilup of 30 to 60 liters/hr at a boildown ratio of 2; and
- (5) steam supply pressure not to exceed 30 psig (135°C).

About half of the condensate can be recycled in-cell to the uranium recovery contactors in the amine regeneration system and to the resin-washing operation (before drying). Condensate could be used for the caustic and final water scrub streams to the amine regeneration system; these streams result in NaNO_3 or NH_4NO_3 solution wastes.

The disposal of and the allowable uranium concentrations in the condensate waste have not yet been specified. A loss of 0.01% (0.4 g of uranium) would not be of economic importance. While the DF for uranium has not been specified, 10^4 is probably not acceptable; on the other hand, 10^6 is more than adequate. The feed contains approximately 10^5 ppm of uranium (on a weight basis). Thus, the uranium concentrations in the condensate would be about 10 ppm for a DF of 10^4 and 0.1 ppm for a DF of 10^6 . Experience has indicated that a well-designed evaporator can obtain DFs of 10^5 ; thus, the condensate was expected to contain about 1 ppm of uranium.

2.2 Other Considerations

The operating procedures should allow simple, frequent, automatic responses to control either the level or concentration of the ADUN surge tank. The level and/or concentration will show rapid changes; for example, 30 liters of resin wash would be added to the ADUN system in a short time. An "on-off" control of the heat supply to the evaporator appears most suitable. The use of reduced ADUN feed flow rates or other control procedures which could result in concentrated

nitrate solutions are undesirable. The concentrated solutions could contribute to corrosion, inventory or safety problems, and higher uranium contents in the condensate.

3. EQUIPMENT

3.1 General Description

Vertical thermosiphon evaporators are commonly used for radio-chemical applications because of their simplified, compact piping arrangement, low maintenance, and high performance. A schematic flow-sheet and a photograph of the vertical thermosiphon evaporator are shown in Figs. 2 and 3, respectively. Uranyl nitrate is supplied by a centrifugal pump to the evaporator feed nozzle, which is located near the liquid inlet of the reboiler. Concentrated uranyl nitrate is cooled to the resin system process temperature before being returned to the acid-deficient uranyl nitrate (ADUN) surge tank via a fixed jackleg (overflow weir). Vapor passes through the disengaging section and a wire-mesh mist eliminator. The entrainment-free vapor is condensed, and the condensate is collected. The overflow weir and condensate tank are located on a common vent line. The evaporator, of all-welded construction, is fabricated of type 304 L stainless steel. (A list of the engineering drawings is given in the Appendix). It is insulated to reduce heat loss to the surroundings.

A natural-circulation evaporator depends on density difference to produce flow through the reboiler. Partial vaporization of the process liquid in the heat exchanger tubes creates a two-phase mixture with a lower density than that of the liquid in the unheated leg. The hydraulic head resulting from this density difference causes the liquid to circulate through the reboiler. The high velocity of this two-phase flow reduces possible fouling and provides a high heat-transfer coefficient.

ORNL-DWG 75-17868R2

Fig. 2. Vertical thermosiphon evaporator for uranyl nitrate.

Fig. 3. Evaporator system without insulation.

3.2 Description of Equipment Items

3.2.1 Reboiler

The reboiler is an American Standard SSCF No. 03024 type 316 stainless steel, shell-and-tube heat exchanger consisting of fifty-six 61-cm-long by 0.64-cm-OD tubes. The heat transfer area is 0.678 m^2 (7.3 ft^2). The measured overall heat transfer coefficient, U_{AVE} , ranged from 500 to 800 $\text{Btu/hr}\cdot\text{ft}^2\cdot^\circ\text{F}$ (2450 to 3900 $\text{kcal/hr}\cdot\text{m}^2\cdot^\circ\text{C}$). Design boilup, 60 liters/hr, is attained at a steam-chest pressure of 11 psig. A plot of boilup rate vs steam pressure is shown in Fig. 4.

Piping of the tube-side nozzles of the reboiler was made in accordance with common design procedures. The flow cross-sectional area of the vapor-liquid outlet is approximately equal to the flow area of the tubes, whereas the flow area of the liquid inlet nozzle is approximately one-half of that of the vapor-liquid outlet. The vapor-liquid nozzle is fabricated from 1-1/2-in. sched 40 pipe, while the liquid nozzle is made of 1-in. sched 40 pipe. The process feed to the evaporator is piped near the reboiler liquid inlet. The liquid level in the downcomer leg is maintained at the same elevation as the top tube-sheet with a fixed jackleg overflow weir. The exit stream for the concentrated product has the same concentration as the liquid in the 3-in. sched 10 pipe downcomer leg.

3.2.2 Disengaging section and demister design

The vapor-liquid nozzle from the reboiler is positioned tangentially to the disengaging section. This feature is incorporated to reduce possible vapor locking, to provide a smooth entry to the disengaging section, and to separate the liquid droplets from the vapor. The disengaging section is a 6-in. sched 10 pipe with a 1-m space between the vapor-liquid nozzle and the wire-mesh mist separator. This space serves to reduce the liquid loading of the mesh. Vapor velocity through the 6-in. pipe is 1.4 m/sec at the design boilup rate of 60 liters/hr.

ORNL DWG 76-923RI

Fig. 4. Evaporator boilup performance.

Wire-mesh separators provide highly efficient entrainment removal and require little maintenance. When vapor and the entrained liquid droplets flow through a wire-mesh mist separator, the vapor passes easily through the pad; however, the liquid droplets, having greater inertia, contact the wire surfaces. As more droplets enter the pad, they coalesce, increase in size, and drain from the mesh. The vapor leaving the mesh pad is essentially free of entrainment.

The York Demister* Style 326 was selected because of its ultra-efficient fine mist removal characteristics. This device has an approximate bulk density of 0.13 g/cc, a void space of 99+%, and a wire diameter of 0.15 mm. A 30.5-cm-thick pad, the maximum thickness for mist eliminators,² was selected to obtain the maximum DF possible. The wire-mesh pad is constructed of type 304 stainless steel.

The optimum vapor velocity for a wire-mesh mist eliminator can be approximated by using the densities of the vapor and the liquid in the following equation:²

$$u_{OPT} = K \sqrt{\frac{\rho_l - \rho_g}{\rho_g}},$$

where

u_{OPT} = optimum velocity, fps;

K = constant, 0.35,

ρ_l = density of the liquid;

ρ_g = density of the vapor.

The process feed is usually 0.3 M uranyl nitrate and has a density of 1.06 g/cc at the boiling point. With a boildown ratio of 2, the uranyl nitrate concentration of the liquid in the unheated leg below the disengaging space is 0.6 M (density, 1.15 g/cc). The density of the vapor (steam) at atmospheric pressure is 0.0006 g/cc. Since the vapor is in contact with the concentrated liquid, substituting the densities

* Demister is a registered trademark of the O. H. York Co., Inc., Parsippany, N.J.

of the 0.6 M uranyl nitrate solution and the steam yields $u_{OPT} = 15.3$ fps (4.66 m/sec).

For the volume of vapor to be handled, the proper diameters for the vessel and the mist eliminator are calculated from the following:

Design boilup: 60 liters/hr

$$\text{Vapor flow rate: } \frac{(60 \times 10^3 \text{ g/hr}) 10^{-6} \text{ m}^3/\text{cc}}{(6 \times 10^{-4} \text{ g/cc}) 3600 \text{ sec/hr}} = 0.02778 \text{ m}^3/\text{sec}$$

$$\text{Flow cross-sectional area: } \frac{0.02778 \text{ m}^3/\text{sec}}{4.66 \text{ m/sec}} = 0.00596 \text{ m}^2$$

A 3-in. sched 10 pipe has a flow cross-sectional area of 0.005385 m².

The velocity utilizing a 3-in. sched 10 pipe is:

$$u = \frac{0.02778 \text{ m}^3/\text{sec}}{0.005385 \text{ m}^2} = 5.16 \text{ m/sec.}$$

Although this is slightly greater than the optimum velocity for the design boilup, the O. H. York Company claims that excellent performance will be obtained for velocities from 30 to 110% of the optimum.²

According to this recommendation, the minimum and maximum condensate rates using a Demister sized to fit in a 3-in. pipe are 16 and 60 liters/hr respectively. A full-cone spray nozzle is situated above the Demister to wash the wire mesh. Three 0.32-cm-diam stainless steel rods located above and below the wire-mesh pad are used to prevent the pad from moving.

3.2.3 Instrumentation

Temperature determinations were made by iron-constantan thermocouples positioned in the downcomer leg and around the heat exchangers; in addition, specific gravity, level, and pressure drop across the wire-mesh entrainment separator were measured. The temperatures in the evaporator circuit were recorded by a multipoint recorder. Typical temperatures for the reboiler exit and the downcomer leg were 101°C and 100°C respectively.

Specific gravity, level, and pressure drop over the wire-mesh pad were measured using Foxboro Differential Pressure cells. The air probes are constructed of 1/4-in. sched 40 pipe. Air flow rate to the probes was 3 to 6 std liters/hr. The specific gravity probes are located on a 30.5-cm separation in the downcomer leg (3-in. sched 10 pipe). The Foxboro Model 13A operates over the range of 0 to 254 cm H₂O. The unit is adjusted to 50.8 cm H₂O, corresponding to a maximum specific gravity of 1.667.

The upper density probe and the probe in the top of the disengaging section are used to measure the liquid level. The Foxboro Model 13A was also used for the level measurement and was adjusted to 63.5 cm H₂O, full scale. The liquid level in the evaporator is maintained at the same elevation as the top tubesheet of the reboiler.

The pressure drop across the wire-mesh pad was measured using the upper level probe in the disengaging section and the probe above the wire pad. The Foxboro Model 15A operating range of 0 to 25.4 cm H₂O was adjusted to read full scale at a pressure of 12.7 cm H₂O.

During operation of the evaporator, the specific gravity and level measurements showed fluctuations on the order of ± 3 divisions, corresponding to ± 0.05 unit and ± 2 cm respectively. The pressure drop across the wire-mesh pad varied with the condensate rate. For condensate rates of 17 to approximately 30 liters/hr, the pressure drop oscillated from 5 to 25% full scale (about 1/2 to 3 cm H₂O). At about 40 liters/hr, the pressure drop varied from 15 to 25% (about 2 to 3 cm H₂O). At the design boilup rate of 60 liters/hr, the pressure drop fluctuated from 30 to 50% full scale (about 4 to 6-1/2 cm H₂O). The pressure drop across the wire mesh occasionally exceeded 13 cm H₂O when the condensate rate was 79 liters/hr. According to the manufacturer,² the pressure drop is usually less than 0.25 cm to 2.5 cm H₂O, depending on the combined vapor and liquid loading.

3.3 Decontamination Factors for Uranium

The thermosiphon evaporator was tested by concentrating uranyl nitrate solution to determine the DF for uranium. Uranium concentration

in the condensate was examined as a function of the boilup rate, boildown ratio, and uranium feed concentration. Performance of the evaporator during resin loading runs (S-27 and S-28) and additional tests, EXP-1, -2, and -3, are presented in Table 1. The evaporator has now been used routinely for about 30 resin loading runs; no operating problems have been observed.

The degree of separation or purification is measured by the DF, is usually defined as the ratio of the concentration of a component in the feed stream to the concentration of that component in the product (purified) stream. Since the vapor in this evaporator is in contact with the concentrated uranyl nitrate, that is, the liquid in the downcomer leg, this concentration was used instead of the feed concentration in calculating the DF.

The uranium concentration of the feed was obtained from hydrometer measurements and from a plot of the specific gravity vs uranium concentration. The concentration in the downcomer leg (product stream) was obtained by multiplying the uranium concentration in the feed stream by the boildown ratio (feed rate/concentrate rate). The DF for each run was obtained by dividing the concentration of the uranyl nitrate product by the uranium concentration in the condensate. The effect of uranium concentration in the product stream on the uranium concentration in the condensate is shown in Fig. 5. As expected, increasing the uranium product concentration increases the uranium concentration in the condensate. Curves representing DFs of 2.5×10^5 , 5.0×10^5 , and 1.0×10^6 are included. If the two stray data points are omitted, the evaporator has an overall DF of 5×10^5 to 1×10^6 , producing condensate containing less than 0.5 ppm of uranium. The uranium analysis is accurate to within 10%. Condensate samples had a pH typically around 4.

Although the recommended boilup rate for the wire-mesh mist eliminator is 16 to 60 liters/hr, no significant difference in the DF of the evaporator was observed for condensate rates as large as 79 liters/hr. The pressure drop across the wire pad was greater than full scale (12.7 cm H_2O). In these instances, we believe that the wire mesh was becoming

Table 1. Performance of thermosiphon evaporator for uranyl nitrate solutions

Run number	Evaporator feed			Condensate		Concentrated product		Evaporator DF ^a
	Flow rate (liters/hr)	Specific gravity	Uranium concentration (g/liter)	Flow rate (liters/hr)	Uranium concentration (ppm)	Boil-down ratio (feed rate / conc. rate)	Uranium concentration (g/liter)	
S-27	94.3	1.062	48	47.8	0.16	2.03	97	6.1×10^5
	93.8	1.052	38	54.2	0.18	2.37	90	5.0×10^5
S-28	94.0	1.054	43	50.0	0.14, 0.13	2.14	92	6.6×10^5
	94.0	1.054	43	52.8	0.081, 0.078	2.28	98	1.2×10^6
	80.1	1.060	48	26.3	0.14, 0.15	1.49	72	5.1×10^5
EXP-1	47.8	1.084	64	17.4	0.22	1.57	101	4.6×10^5
	62.1	1.084	64	30.5	0.23	1.97	126	5.5×10^5
	95.6	1.084	64	42.7	0.25	1.81	116	4.6×10^5
	95.6	1.084	64	49.7	0.20	2.08	133	6.7×10^5
EXP-2	93.9	1.053	38	60.2	0.12	2.79	106	8.8×10^5
	95.4	1.056	45	55.9	0.18	2.42	109	6.1×10^5
	96.3	1.102	76	56.3	0.23	2.41	183	8.0×10^5
EXP-3	95.6	1.084	64	18.0	0.26	1.23	79	3.0×10^5
	95.6	1.084	64	39.5	0.40	1.70	109	2.7×10^5
	95.6	1.084	64	56.1	0.31	2.42	155	5.0×10^5
	126	1.084	64	68.9	0.28	2.21	141	5.0×10^5
	144	1.084	64	79.0	0.23	2.22	142	6.2×10^5

^aUranium concentration in the concentrated product divided by the uranium concentration in the condensate.

Fig. 5. Uranium concentration in the condensate and DF for the ADUN evaporator.

overloaded with liquid and vapor and that reentrainment would have occurred eventually.

4. CONCLUSIONS

Operation of the thermosiphon evaporator was simple and easy to control. The fixed jackleg maintains a constant liquid level in the evaporator and allows continuous circulation through the evaporator. The evaporation rate (and thus the uranium concentration in the resin loading system) was controlled by regulating the steam pressure while maintaining a constant feed flow rate to the evaporator. A high-quality condensate containing less than 0.5 ppm of uranium, for an overall DF of greater than 5×10^5 , is obtained by utilizing a tangential vapor entry to the large vapor disengaging section and a wire-mesh mist separator. The DF was not a consistent function of the condensate rate for rates of 17 to 79 liters/hr. This evaporator design meets the requirements of the HTGR fuel recycle Hot Engineering Test Facility.

5. ACKNOWLEDGMENTS

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7. APPENDIX - LIST OF DRAWINGS

Detailed engineering drawings were prepared for fabrication of the thermosiphon evaporator. The titles and ORNL Drawing Nos. are as follows:

Engineering-Scale Resin Loading: Thermosiphon Evaporator

Assembly and Plan	P 12307 CD 001 E
Subassemblies A & B	P 12307 CD 002 E
Subassembly C	P 12307 CD 003 E
Subassemblies D & E	P 12307 CD 004 E
Support Rack	P 12307 CD 005 E

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