HW-43081

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Released 1995

**Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830**

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RECUPLEX NUCLEAR SAFETY EQUIPMENT REVISIONS

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RECUPLEX NUCLEAR SAFETY EQUIPMENT REVISIONS

By

B. F. Judson

Z Plant Process Unit
Separations Technology Section
ENGINEERING DEPARTMENT

May 16, 1956

HANFORD ATOMIC PRODUCTS OPERATION
RICHLAND, WASHINGTON

Operated for the Atomic Energy Commission by the General Electric Company under Contract No. W-31-109-Eng-52.

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INTRODUCTION

A number of equipment revisions have been recommended by the Engineering Department for conversion of Recuplex to a Manufacturing facility.(1) These revisions include three items affecting the critical mass safety of the solvent extraction system:

(1) Replacement of the bottom disengagement section of the H-3 stripping column with an "always safe" unit.

(2) Replacement of the H-9 and H-10 intercolumn, organic phase surge tanks with "always safe" tanks.

(3) Replacement of the colorimetric plutonium monitors in the aqueous and organic raffinate streams with units insensitive to stream contaminants.

The following memorandum is intended to clarify the needs for these equipment revisions in achieving a safe and flexible operating system and to indicate the relative effects of revising each of the various equipment pieces separately. The general bases for the present criticality control measures in the solvent extraction system are reviewed briefly prior to discussion of the individual revisions.

SUMMARY AND CONCLUSIONS

Replacement of the H-3 bottom end section with an "always safe" unit would provide the maximum protection against the occurrence of critical conditions within the solvent extraction system and would simultaneously permit relaxation of the present operating limits. Alternatives to the actual replacement involve either the present severe operating limits or cumbersome and less effective interlock controls.

Replacement of the H-9 intercolumn, organic phase surge tank with an "always safe" unit is necessary to realize the maximum operating gains of an "always safe" stripping column and maintain design standards for criticality control. The original controls for this tank were based upon organic solutions exclusively, while recent considerations indicate that the measures to prevent the introduction of highly concentrated aqueous solutions are not sufficient in number to meet design standards. Retention of the present H-10 intercolumn surge tank, on the other hand, should require no operating limits, provided the monitoring systems for the raffinate receivers are operable.

The intended function of the colorimetric plutonium monitors in the aqueous and organic raffinate streams could conceivably be performed by instruments that would insure adequate salting of the feed, a safe composition of the solvent, and a low acidity of the stripping solution. The installation of such instruments could be considered as a possible alternative to the alpha scintillation monitors now under development.

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(1) K. M. Harmon, Memorandum to W. N. Mobley, "Recuplex Equipment Revisions", January 5, 1956.

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BASES FOR CRITICALITY CONTROL

The Recuplex solvent extraction system is comprised of a dilute feed tank, concentrated feed tanks, a strip feed tank, three pulse columns, four intercolumn surge tanks, product tanks, aqueous waste tanks, and two solvent feed receiver tanks. All these components are interconnected by continuously flowing process streams. Large quantities of plutonium are contained in the process streams, necessitating strict controls to prevent critical mass conditions. Batch size control cannot be employed throughout the system due to the lack of batch identity in a continuous operation. On the other hand, the use of special geometry vessels throughout the process is impractical since some of the streams are large in volume and low in plutonium concentration. Criticality control is, therefore, based upon a combination of controlled batch sizes for the dilute streams and specially shaped containers for the concentrated streams. Such a combination requires an interlock system superimposed on the normal operational control measures to prevent possible entry of concentrated plutonium streams into batch-controlled vessels.

In determining requirements for an interlock system or the need for "always safe" construction, the design basis has been that the component under consideration should still be sub-critical after the occurrence of two sequential operating errors or equipment failures. Thus, the dilute feed and the strip feed tanks are each protected from backflow of plutonium solutions from the columns by an interlock between the flow control instrument and the main control valve and by another interlock between the pump and a special valve. If the flow of solution from one of the sensitive tanks should cease because of instrument or pump failure, both valves in the outlet line automatically close. As the third preventative measure, a check valve is installed in the tank outlet line.

In addition to protection from mechanical failures, the criticality control system must provide protection from inadvertent process errors. Normal input to the dilute feed and strip feed tanks is from outside the continuously operated solvent extraction system and can, therefore, be regulated by plutonium batch sizing. Process errors within the system do not affect the control of these tanks, so that mechanical interlocks alone are adequate protection. The two components of the system that are "always safe" by virtue of their geometry -- the concentrated feed[1] and product tanks -- are also unaffected by intra-system process errors. The columns, the intercolumn surge tanks, and the waste receivers, however, each have individual equipment pieces which are not "always safe" and cannot be protected by mechanical interlocks alone; some process limitation is required or the degree of protection is reduced.

THE H-3 STRIPPING COLUMN

The three pulse columns were originally intended to be "always safe" from thermal chain reactions. When the vessel design proved inadequate due to unanticipated

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(1) The G-50 F-10-P receiver in the concentrated feed system should be either blanked off from the other tanks or revised to an "always safe" geometry. The system would then be uniformly "safe", and the danger of transferring excessive amounts of plutonium into G-50 through the bottom outlet valve eliminated.

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reflection and interaction effects, all the column end sections were revised to a "safe" geometry except the bottom disengagement section of the H-3 stripping column.(1) It was believed at the time that the mechanical failure and process error protection measures adopted for the organic raffinate tanks (cf below) were applicable to the H-3 end section. During the semiworks operation of the Recuplex facility, however, the failure of the H-1 pulse bellows and the rapidity with which the column drained demonstrated forcibly that the possibility of transferring the highly concentrated plutonium inventory from the top to the bottom of the H-3 Column during some unattended shutdown period is very real. The concentration of the product from the columns has, therefore, been reduced to below the limit for the bottom end section to insure safe operation.

The enclosed table includes the estimated minimum critical concentration and the process specification concentration limit for both low and high MWD/T plutonium in the present H-3 bottom disengagement section. Since the product concentration normally varies with product removal and inventory shifting, it is evident that the average product concentration necessary to prevent exceeding the process specification limit for the present equipment is much lower than the 100 to 110 gram per liter solution the system is capable of producing. Such an operational limit reduces process flexibility and increases the evaporator work load.

To remove this operational limit, the present H-3 bottom end section could conceivably be protected by a series of interlocks and operating practices. For example:

(1) Enclosing the pulse bellows in a sealed jacket.

(2) Installing a drain valve above the end section, which would be opened automatically by a decrease in the column liquid level.

(3) Displacing the H-3 Column inventory to the J-1 Tank prior to a shutdown.

Such a system is necessarily cumbersome.

Replacement of the H-3 bottom end section with an "always safe" unit would provide the maximum protection. To accomplish this would require a unit similar to those in the H-1 and H-2 Columns. The column height available for stripping would be reduced by approximately 18 inches (the present volume hold-up in the end section should not be reduced). In addition, the sensing instrument for the interface controller should perhaps be revised since a capacitance probe similar to the present unit could not be maintained without dissembling the column. A pressure differential system employing a Republic or Taylor transmitter located in the hood adjacent to the end section and using the heavier phase solvent as the sensing fluid has recently been tested and may be applicable.(2)

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(1) K. M. Harmon/B. F. Judson, Memorandum to W. L. Gallagher, "Recuplex Nuclear Safety Design Revisions", September 20, 1954.

(2) R. B. Richards, "Separations Technology Section Monthly Report - December, 1955:, Document No. HW-40692-H, p Fc-31, January 10, 1956.

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THE INTERCOLUMN SURGE TANKS

The four intercolumn surge tanks were also intended to be "always safe" by virtue of their geometry. The H-9 and H-10 organic phase surge tanks, however, were not replaced when the critically safe geometries were revised. Reliance for critical mass control in the two tanks was based upon the fact that 15 volume per cent tributyl phosphate in carbon tetrachloride has a theoretical plutonium saturation limit of 66 grams per liter. Although subsequent laboratory tests have confirmed this value,(1) two factors influencing the theoretical 66 grams per liter limit became evident during the semiworks program.

Operating experience has shown that the TBP content of the solvent cannot be controlled very closely. Concentrations as high as 30 per cent TBP, corresponding to a theoretical plutonium limit of 132 grams per liter, have been experienced during periods of lax solvent control (the initial uranium runs). Present techniques generally limit the TBP concentration to less than 17 per cent, which corresponds to a theoretical plutonium limit of 75 grams per liter, but are not reliable for closer control.

The second factor influencing the organic phase plutonium concentration is that the plutonium operating inventory in the system tends to shift between the H-1 and J-1 tanks. Moreover, the convenient practice of accumulating up to 30 liters of product solution in the J-1 tank before withdrawal from the system may result in a plutonium inventory as high as six kilograms with a 100 gram per liter product. If excess TBP is present in the solvent, the size and mobility of the inventory prevents the assumption that the plutonium concentration in the H-9 tank will always be less than the product concentrations (although under steady state conditions, the H-9 concentration will be one-half to one-third the J-1 concentration).

In addition to these two factors, recent consideration of the system indicates that it is mechanically possible to transfer aqueous product solution directly to the H-9 tank, either by a backsiphon through the outlet valve if the H-9/H-8 interlock fails upon a shutdown, or by pumping from J-1 to an empty H-1 column and from there to the H-9 tank.

To protect the present organic phase surge tanks, operational limits must be imposed upon the product concentration and the solvent composition. Based upon the estimated minimum critical concentrations(2) and the process specification concentrations presented in the enclosed table, suitable operational limits would be:

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(1) K. M. Harmon, "234-5 Development Sub-Unit Progress Report for September, 1954", Document No. HW-33750, p 12, October 11, 1954.

(2) The estimated minimum critical concentrations are based upon plutonium remaining in a true solution. The presence of solid plutonium seriously affects the limits in tanks that are close to "safe" dimensions. Thus, the minimum critical mass for a slurry of low MWD/T plutonium in the 38.5 liter H-9 tank is 1400 grams, corresponding to an over-all concentration of about 36 grams per liter, whereas the concentration limit for true solutions is 105 grams per liter. Although "cruds" have been present in the H-9 tank, there has been no evidence of solid plutonium compounds.

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(1) A maximum product concentration of 75 grams per liter for low MWD/T plutonium and 110 grams per liter for high MWD/T plutonium.

(2) The corresponding theoretical saturated solvent compositions of 17[1] per cent TBP for low MWD/T plutonium and 25 per cent TBP for high MWD/T plutonium.

These limits are above those now in force, since they assume a geometrically safe H-3 column. They are undesirably restrictive, however, especially for low MWD/T plutonium. Moreover, they do not meet the previous design basis that the component should be sub-critical after two failures or errors (viz pumping a 100 gram per liter solution of low MWD/T plutonium from J-1 to H-9 during a start-up would constitute two errors). Since mechanical interlocks are not applicable, use of the present tanks would result in a lower over-all degree of protection.

Replacement of H-9 above with an "always safe" vessel would remove the operational limits. Although the system would not be uniformly "safe", the H-10 tank would be protected from input of excess plutonium by the control measures for the solvent feed tank and by the interlock between the H-10 pump and outlet valve.

THE RAFFINATE RECEIVERS

Critical mass control for the aqueous waste receivers and the solvent feed receiver tanks is based upon preventing the accumulation and precipitation of plutonium in batch quantities greater than the limits specified in the accompanying table. The estimated minimum critical masses for the receivers are based upon plutonium present in solid form; the addition of a precipitating agent to the solutions with or without a prior analysis is considered to be one "error". Control measures are, therefore, required to prevent the introduction of excessive plutonium into the tanks from the solvent extraction system. For the aqueous raffinate tanks, these control measures assume that adequate extraction occurs if a suitable solvent is flowing and the aqueous phase is salted (plutonium(III) is not extractable as such, but is oxidized to the extractable plutonium(IV) and (VI) as long as a holding reductant is not present). Similarly for the organic raffinate tanks, the assumption is made that if stripping agent is flowing the solvent is strippable.

Present protection against mechanical failure for both the aqueous and organic raffinate tanks involves two interlocks between the extractant flow system and the sources of concentrated plutonium input to the appropriate columns. Thus, if the solvent flow ceases due to a pump or instrument failure, the concentrated feed and reflux-scrub pumps and the associated flow control valves are automatically shut down. Similarly, if the stripping agent flow stops due to a pump or instrument failure, the H-9 pump and outlet valve are automatically shut down.

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(1) This limit should probably be supplemented by the installation of a suitable specific gravity instrument to maintain a close control of the TBP concentration.

Safe operation requires protection against process errors, such as failure to add salting agents to the feed or the addition of excess acid to the stripping solution. Such protection is presently lacking, other than the normal control of feed and chemical preparation procedures. The colorimetric plutonium monitors installed in the feed lines to the L-2, L-3, and L-8 waste tanks and the K-1 and K-2 solvent tanks were intended for this function. Their application, however, has proven unsuitable due to the varying colors of the aqueous feeds (nickel corrosion products) and the organic solvent (iodine). Alpha scintillation counters suitable for these streams are under development and will be ready for plant installation between January and July, 1957.

As an interim, or possibly long term, measure, adequate protection against process errors may be gained by installation of suitable monitors to insure salting of the feed, composition of the solvent, and acidity of the stripping agent. An instrument measuring the specific gravity of the aqueous phase in the extraction system at the H-11 surge tank(1) could be employed if the assumption is made that solutions having a specific gravity of 1.25 or more are sufficiently salted (by comparison, 120 grams per liter product solution, 8 M nitric acid, and normal waste each has a specific gravity of about 1.25). Similarly, an accurate instrument for the H-10 surge tank(1) to limit the solvent specific gravity would insure that a gross excess or lack of tributyl phosphate is not employed. An in-line instrument to gauge the acidity of the stripping feed from the H-8 tank would also be required. Each instrument would be tied into an interlock system to prevent column operation unless the various solution limits are met. Such a scheme may prove cumbersome and restrictive for long term operation, but may be economically attractive.

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(1) The H-10 and H-11 surge tanks do not have specific gravity instruments due to the lack of sufficient solution height for an air purge system. Installation of a monitor in the G-10 feed tank, rather than H-11, would not account for the G-47, G-48, and J-1 sources of aqueous plutonium. Installation of a monitor in the K-1, K-2 tanks, rather than H-10, would limit allowable solvent pump-down and would require the two instruments to be connected to the pump circuit for alternate operation in the interlock system.

TABLE I

ESTIMATED MINIMUM CRITICAL CONDITIONS AND PROCESS SPECIFICATIONS

Tank No.	Estimated Minimum Critical Conditions		Process Specification Limits	
	<3% Pu^{240}	>3% Pu^{240}	<3% Pu^{240}	>3% Pu^{240}
H-3	70 g/l	95 g/l	65 g/l	85 g/l
H-9	105 g/l	150 g/l	75 g/l	110 g/l
H-10	100 g/l	150 g/l	75 g/l	110 g/l
L-2) L-3) L-8)	2.6 kg	2.8 kg	1.2 kg	1.3 kg
K-1) K-2)	1.2 kg	1.3 kg	0.55 kg	0.60 kg

Reference: O. F. Hill, "Process Specifications for Plutonium Isolation, Purification and Fabrication", HW-31967, Spec. No. 1.7.4.