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**PROCESS SPECIFICATIONS FOR CRITICAL MASS
CONTROL - PUREX PLANT (REVISION NO. 2).**

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PROCESS SPECIFICATIONS FOR CRITICAL
MASS CONTROL - PUREX PLANT
(REVISION NO. 2)

By

E. R. Irish, Manager
Purex Technology Operation
Research and Engineering Operation
CHEMICAL PROCESSING DEPARTMENT

July 30, 1958

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RICHLAND, WASHINGTON

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PUREX PLANT (REVISION NO. 2)

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PROCESS SPECIFICATIONS FOR CRITICAL MASS CONTROL -
PUREX PLANT (REVISION NO. 2)

1.01 INTRODUCTION

This document is Revision No. 2 of the "Process Specifications for Critical Mass Control - Purex Plant". It encompasses the changes in flowsheet, equipment and plant operating philosophy that have developed since the plant startup. It supersedes Document HW-44064 (Revision No. 1).

Plutonium-239 and uranium-235, either as the metals or as dissolved or solid compounds, are capable of spontaneous and violent nuclear reactions. Following initial dissolution of irradiated natural uranium fuel elements, the Purex Plant is normally involved in the processing of liquid solutions containing these isotopes. However, in Purex Plant solutions, uranium-235 is invariably associated with a large fraction of neutron-absorbing uranium-238, a condition which precludes the possibility of a nuclear reaction. (These specifications are based on the premise that no enriched uranium is processed in the Purex Plant.) Plutonium-239, on the other hand, is separated from uranium by means of the Purex process; and even prior (process-wise) to its separation, the ratio of plutonium to uranium must be controlled during the off-standard conditions of plutonium rework or recycle. Critical mass control in the Purex Plant is concerned solely with prevention of nuclear reactions of plutonium-239, which has been produced by irradiation of natural uranium fuel elements.

1.02 MINIMUM CRITICAL MASSES OF PLUTONIUM

1.02.01 Theoretical Minimum Critical Mass

Compared to the case of bare metallic plutonium-239, in general, a smaller mass of plutonium is required to support a spontaneous nuclear reaction when the plutonium is uniformly distributed as a compound in a liquid, e.g., as an aqueous solution. Under these conditions, neutrons released by fission of plutonium are moderated to lower energy levels by collisions with atoms of the liquid, and the fission capture cross-section of plutonium-239 is greater for these "slow neutrons" than for non-moderated "fast neutrons" (high-energy neutrons). Also, the minimum critical mass is dependent on the geometry of the mass, the presence of foreign ions or isotopes with the plutonium-239, and the influence of materials surrounding the mass. For the hypothetical limiting case of pure plutonium-239 uniformly distributed in a water moderator, the minimum critical mass is 509 grams of plutonium-239, provided it is contained in a 12.2-in.-diameter sphere which is completely surrounded by water. These conditions correspond to a plutonium concentration of approximately 123.5 g/gal. (32.6 g/l.) in the sphere. For smaller or larger spheres the minimum critical mass is greater, corresponding to larger and smaller plutonium concentrations, respectively. The effects on critical mass of other variables of interest to the Purex Plant are described below.

1.02.02 The Effect of Plutonium-240

Increasing the ratio of the isotope plutonium-240 to plutonium-239, such as would result from increase in total pile exposure of uranium fuel elements, causes a corresponding increase in the minimum critical mass of plutonium-239. For example,

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1.02.05 The Effect of Other Ions or Neutron-Absorbing Materials

In general, the introduction of chemical ions into an aqueous plutonium solution increases the critical mass because of the substitution of ions of lesser moderating power for the water moderator. The presence of uranium-238 with plutonium-239 inhibits plutonium criticality because of neutron absorption by uranium-238. If plutonium is in solution with uranium at a uniform concentration of less than 1800 grams of plutonium per ton of natural uranium, a neutron chain reaction cannot occur.

In the presence of certain neutron-absorbing solid materials the minimum critical mass of plutonium is increased. For example, some of the neutrons escaping from a plutonium-containing vessel may be absorbed by a surrounding mass of cadmium, or other neutron absorber; the effects of tamping and reflection by external objects are thereby reduced, and the allowable mass of plutonium in the vessel is increased. Again, by means of an arrangement of neutron-absorbing walls, or baffles, placed within a vessel, the geometry of the vessel may, in effect, be changed in the direction of increased critical safety.

1.02.06 Fast Neutron Reactions

At the present time, the experimental evidence available which bears on possible "fast-neutron" reactions of highly concentrated plutonium solutions (greater than 1900 g./gal) is considerably sketchier than the information bearing on "slow-neutron" reactions (characterized by relatively dilute solutions). Thus, geometries which are critically safe under conditions previously described above may be unsafe at plutonium concentrations above 1900 g./gal (500 g./l.).

1.03 PHILOSOPHY OF CRITICAL MASS CONTROL IN A CONTINUOUS SEPARATIONS PLANT

Specifications for Purex Plant critical mass control have resulted from application of principles discussed above, following a detailed review of the plant equipment by personnel of the Nuclear Physics Research Operation in cooperation with the Purex Technology Operation.

Changes in philosophy and techniques in the general field of separations plant process control have been necessitated during the past few years and by the trend toward continuous chemical processing. Specifications for critical mass control have been published and revised as required by the development of separations facilities. Likewise, a revised approach or philosophy has been required in the application of critical mass control.

Since the risk of nuclear accidents in a radioactive processing plant is not tenable, particularly in contact-operated and maintained areas, critical mass control of separations plants has its basis on the application of sizable safety factors to critical masses which are estimated from the current available data. Safety factors arise from competent and conservative analyses of these data, of the likelihood of human error in plant operation and control, of the accuracy of process control, and of the technological uncertainties related to the specification of allowable masses. The common sense approach to critical mass control is based on the recognition and maintenance of the factors of safety realizing that their existence is necessary for safe operation during occasional periods of off-standard operation which are part of the nature of manufacturing plant operations.

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- (a) Although there appears to be no chance that precipitation could occur, there is no guarantee. Even if precipitation did occur, probably not all the plutonium would precipitate from the solution. Precipitation would be extremely unlikely during plant operation because of the flow of process solution through the tank. If precipitation were to occur during plant shutdown, probably not all the plutonium would precipitate at once; and, in addition, during shutdown the tank is not always full or even up to operating level which reduces the plutonium holdup in the tank. However, if the possibility of precipitation could be discounted, the allowable mass could be increased approximately by a factor of seven.
- (b) Although the presence of foreign ions in TK-J5 is certain, a chemical composition with respect to these ions cannot be guaranteed, and the allowable mass has been adjusted accordingly. It is no simple matter to estimate the increase in allowable mass that could be charged to guaranteed chemical composition for a range of compositions. However, for a single hypothetical case an increase in allowable mass of fifty percent would be reasonable if it were also assumed that there is no analytical error.

In general, any mass and concentration specifications could be analyzed in the manner outlined above for the case of a remotely located vessel. It would be found that the determination of safety factors follows a similar philosophy for each case, although the safety factors are by no means numerically equal from case to case, and are always more conservative where men may be directly exposed to the equipment. The situation regarding allowable mass limits and safety factors may be visualized as analogous to allowable dose rates and safety factors for radiation protection. In either case, violation of a certain critical limit could result in catastrophe and safety factors are applied to prevent such an occurrence. The existence of the safety factor implies the possibility of violating the control specification. Strict adherence to the specification gives the maximum guarantee of safety. However, sufficient margin of safety is provided to insure routine detection and correction of a dangerous condition beyond any reasonable doubt.

1.04 APPLICATION TO THE PUREX PLANT

1.04.01 General Bases

The application of critical mass data to the Purex Plant is based on the processing of natural uranium irradiated at 200 megawatt-days per ton or greater under the current two cycle flowsheet conditions (Purex Plant Flowsheet IV), and excepts (a) any scheme for plutonium reflux in the columns of the Final Plutonium Decontamination Cycle, and (b) any scheme for recycle of a stream potentially containing trace or greater amounts of plutonium to a dissolver. In cases in which production rates enter into the analysis of a potential critical mass problem, a maximum rate corresponding to the processing of 26.7 tons uranium per day (8.33 tons/day design rate; 3.2 capacity factor) containing a maximum of 1500 grams of plutonium per ton of natural uranium has been assumed. Under flowsheet conditions, the possibility of formation of a plutonium precipitate (which conceivably might settle into a critically unsafe geometry) is considered extremely remote as evidenced by the fact that no appreciable amount of plutonium has been removed by equipment flushes. However, assuming a precipitate is possible, its routine detection might be considered suspect;

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the column when contacted by Fe^{+2} in the IBX stream and reoxidization to organic-preferring Pu^{+4} in the lower portion of the column when contacted by the oxidizing agent in the IBXF. Thus, the plutonium is effectively trapped within the column. The length of the column over which plutonium might be retained during such an off-standard operation is not known with certainty; consequently, the critical concentration could conceivably be exceeded if excessive plutonium accumulates. However, the neutron absorption of the normal uranium concentration in all but the bottom twenty inches of the column plus the neutron absorption of the nitrate ions associated with the uranium and plutonium increase the critical concentration of plutonium. The approximate amount of plutonium holdup in the IBX Column can be determined by material balance, and previous experience with plutonium retention in this column during periods of off-standard operation has indicated a critically-safe holdup of at least five kilograms. Consequently, critical mass control during periods of off-standard operation, during which plutonium reflux occurs in the IBX Column, shall be based on a material balance determination of the amount of the plutonium holdup.

1.04.04 IBS Column

In the 8-inch-diameter IBS Column the maximum plutonium concentration under any normal condition is at least a factor of four less than the limiting minimum critical concentration of 41.6 g./gal. (11 g./l.) of plutonium, estimated for the top disengaging section. Also, the minimum critical mass of 640 grams (200 MWD/T), based on accumulation of plutonium (such as by precipitation) in a portion of the 8-inch-diameter section, is approximately three times the normal maximum holdup in the column. Consideration of the flowsheet shows that a chain of events which could lead to criticality in this column is extremely unlikely and would be indicated by off-standard performance prior to the occurrence. For example, if plutonium precipitate could be formed, it would tend to fall into the "always-safe" slab-shaped bottom disengaging section; any tendency for its accumulation in a critical shape in the 8-inch section would also tend to plug the column sieve plates and cause flooding. No reflux mechanism has existed in this column to date, and no flowsheet changes can be visualized that might cause it.

1.04.05 2AF Tank

The 30-inch-diameter 2AF Tank, TK-J5, is a horizontal cylindrical tank designed for the continuous addition of chemical streams to the continuous IBP - 2AF process stream. The present tank has an upstream baffle which extends to within 3 inches of the bottom and 1-5/8 inches of the top of the tank to prevent crossflow in the compartment into which the process and chemical additions are made. Normally the tank contents are uniformly very low in plutonium concentration (less than 2.7 g./gal. (0.7 g./l.) maximum) compared to any limiting critical mass concentration. Consequently, critical mass control is based on the precipitation of plutonium on the tank bottom in the event of precipitate formation and an agitator failure.

The present spare 2AF Tank is the originally installed 2AF tank which was subsequently replaced to increase the allowable critical mass in the tank. This spare tank has an additional compartmental baffle which is welded to the bottom of the tank and extends to within 7-1/4 inches of the top. Critical mass control in this particular tank is based on the unlikely case of plutonium precipitation settling in a critically sensitive configuration on the tank bottom against the second baffle, if an agitator fails. On this basis, the minimum critical mass in the

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the 7-1/2-inch-I.D. jacket, criticality is possible (although exceedingly unlikely) because of the reflection and interaction effects of nearby vessels and other physical objects on this otherwise "always-safe" diameter. To insure criticality safety, the potential leakage of process solution into the jacket should be prevented by maintaining an adequate water pressure in the jacket at all times when the vessel contains significant quantities of plutonium.

The sloped floor of L-Cell drains to Sump S-1A, a 1-1/2 inch-wide groove with sloping bottom (approximately 8 inches, maximum depth) which runs the length of L-Cell (approximately 59 feet long) at the cell floor centerline. By virtue of its geometry, Sump S-1A is "always-safe" from slow-neutron criticality; thus, the minimum critical mass is based on the case of a full sump (19.25 gal.) at the maximum plutonium concentration known to be safe from a possible fast-neutron reaction, viz., 1900 g./gal. (500 g./l.). In the event of sump overflow of a uniform plutonium solution to the L-Cell floor, the minimum critical mass increases by an approximate factor of five (to a mass approximately equivalent to 4.5 days of production at maximum rates), and a slow-neutron reaction is possible. However, to allow for possible non-uniform distribution of plutonium, the working limit on the total plutonium allowable on the floor and in the sump, combined, is taken to be the mass allowable in the sump alone.

1.04.08 PR Room

PR Room process vessels, like those in L-Cell, were designed to be "always-safe" from slow-neutron criticality. However, unlike those in the L-Cell, PR Room vessels are more or less isolated from one another, and "interaction" effects are less pronounced. Consequently, contact maintenance restrictions are less severe.

Although a single loaded PR Can is sized to be "always-safe", this is not the case for two or more adjacent cans. The prevention of criticality due to "interaction" requires specifications governing the distance between cans during handling and storage, and the allowable number of cans in a particular array. Specifications given are based on the most restrictive case, viz., precipitation of plutonium in cans containing the maximum allowable mass (4.5 kilograms).

Principal PR Room process sumps include:

- (a) Sump S-1E, under the Plutonium Sampler Tank, L10;
- (b) Sump S-1F, under the Plutonium Sampler Tank, L9;
- (c) Sump S-1D, under the Vacuum Tank, L11. Both Sampler Tank sumps drain to Sump S-1D, which in turn may be emptied by vacuum.

All of the above sumps were originally of conventional, rectangular cross-section, open-top design, and thus could not be considered "always-safe" from the standpoint of criticality. In the case of Sumps S-1E and S-1F, however, at plutonium concentrations of 1900 g./gal. (500 g./l.) the minimum critical depths correspond to volumes greater than twice the volume of either Sampler Tank (L9 or L10), and control at lesser depths is considered practicable. But in the case of Sump S-1D criticality would have been possible because of potentially greater depths if it were not for a revised design which included a neutron-absorbing grid of baffles submerged in the sump. With this revision, the critical mass limitations of Sump S-1D are substantially greater than any visualized operating condition.

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- (a) These specifications apply to normal irradiated uranium processing and are void if U-235 enriched uranium is introduced into the plant.
- (b) Flowsheet conditions which permit the specified limitations to be met under steady-state operation shall be provided by the Purex Technology personnel of the Research and Engineering Operation.
- (c) Any process condition requiring a control method not defined by this specification shall be referred to the Manager, Purex Technology Operation.
- (d) Proposed alterations of revisions to equipment used for processing streams containing plutonium, during normal, rework, or off-standard operating conditions, shall be subject to review for criticality considerations by the Manager, Purex Technology Operation.
- (e) Rework of plutonium solutions shall be conducted within the limitations of these specifications.
- (f) Indication of precipitated plutonium in process tanks which are not critically safe by geometry shall be referred immediately to the Manager, Purex Technology Operation. In the event that plutonium precipitation in vessels is confirmed, the Purex Plant shall be shut down and the difficulty remedied.
- (g) The specifications in this document can be temporarily waived to accommodate an abnormal operating condition upon the written approval by the Manager, Research and Engineering Operation, Chemical Processing Department.

1.05.02 Dissolvers

Plutonium shall not be introduced to the Dissolver Tanks A3, B3, and C3 in any form other than as metallic plutonium contained in irradiated natural uranium slugs. Enriched uranium shall not be introduced in any form.

1.05.03 D-Cell Vessels through IBXF Tank, TK-J3

In vessels prior, process-wise, to the IBX Column, T-J6, and following the Dissolver Tanks A3, B3, and C3, the maximum allowable mass of plutonium shall correspond to a concentration of 1500 grams plutonium per ton of natural uranium. Vessels included are those in D, E, and H Cells, and the IBXF Tank, TK-J3, except the Backcycle Concentrator, E-H4.

1.05.04 IBX Column, T-J6

The normal flowsheet concentration of Fe^{++} in the IBX Column shall not exceed 0.035 M. The maximum plutonium holdup in the IBX Column shall not exceed five kilograms.

1.05.05 2AF Tank, TK-J5

When processing plutonium associated with uranium irradiated at less than 400 megawatt-days per ton, the total mass of plutonium in TK-J5 (containing no downstream baffle) shall not exceed 1650 grams. This corresponds to a full tank at a

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Entry into L-Cell

Entry of personnel into L-Cell during a time when the plutonium holdup in any L-Cell vessel (excluding the 2A Column, T-L1) may be greater than 500 grams shall be governed by the following provisions:

- (a) Entry within the inner double doors of L-Cell shall be limited to two men.
- (b) Within L-Cell, personnel shall not climb above floor level and shall not enter the spaces bounded by the principal structural supporting members (dunnage) of an equipment piece or an assembly of equipment pieces except to the extent described under (c) below.
- (c) Only one man at a time may reach within the spaces bounded by the equipment supporting members, and no portions of his body other than his hands and arms shall enter these spaces.

Whenever the plutonium holdup in each L-Cell vessel (excluding the 2A Column, T-L1) is known with certainty to be 500 grams or less, entry into L-Cell shall not be restricted by critical mass considerations. Special instructions for access to certain equipment pieces will be issued as necessary by the Manager, Purex Technology Operation, after consultation with personnel of the Nuclear Physics Research Operation, when access is required and the quantity of plutonium in the equipment is not known with certainty to be 500 grams, or less.

Product Receiver, TK-L6

The flow of cooling water to TK-L6 jacket shall be maintained during any time when the holdup of plutonium may exceed 500 grams.

L-Cell Sump

The total mass of plutonium in the L-Cell sump (Sump SL-A) shall not exceed 32 kilograms.

1.05.08

PR Room

Vessels

Approach by personnel to either Sampler Tank, TK-L9 or L10, or to the Vacuum Transfer Tank, TK-L11, shall be governed by the following specifications:

- (a) Operations in the spaces outside of the vessel enclosure shall not be limited with respect to the number of personnel.
- (b) Entry of personnel into a vessel enclosure shall be restricted to one man at any time when the plutonium holdup in the vessel may be greater than 500 grams. At any other time, entry shall not be limited.

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1.05.10 Recycle Hood and Storage Tanks

General

- (a) The maximum allowable plutonium holdup in an RC Can shall be 350 grams for plutonium produced at irradiation levels of less than 400 MWD/T, or 400 grams for irradiation levels greater than 400 MWD/T.
- (b) The maximum allowable number of loaded RC Cans in the Sample Gallery shall not be limited. However, RC Cans shall never be arrayed one above another, either during storage or during handling.
- (c) The number of bare loaded PR Cans in the Sample Gallery shall be limited to one, and the distance between a bare loaded PR Can and a loaded RC Can shall be a minimum of three feet, measured in a horizontal plane. Handling and storage of PR Cans in the Sample Gallery shall be otherwise governed by the applicable items under Section 1.05.08, "PR Cans", of this specification.

Recycle Storage Tank

The maximum allowable mass of plutonium in the Recycle Storage Tank shall be 650 grams. The addition of solutions of plutonium concentration greater than 13 grams per liter shall be further restricted, as follows:

- (a) Prior to the addition, the liquid level in the Recycle Storage Tank shall be less than one inch.
- (b) Following the addition, the contents of the Recycle Storage Tank shall be diluted, while agitating, to a plutonium concentration of less than 13 grams per liter. The current operating procedure will specify whether water or dilute nitric acid is to be used for dilution.

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Issued By:

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CHEMICAL PROCESSING DEPARTMENT

Date: 7/31/58

Approved By:

Paul F. Gast

P. F. Gast, Manager

Physics and Instrument Research & Development Operation
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December 9, 1958

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ADDENDUM TO HW-56913

With the advent of E-metal processing in the Redox Plant and subsequent calcination of the uranium in the UO_3 Plant, UO_3 recovered acid will contain some enriched uranium. Present critical mass control specifications, "Process Specifications for Critical Mass Control - Purex Plant (Revision No. 2)", HW-56913, prohibit any addition of enriched uranium to the Purex dissolvers. The attached addendum modifies HW-56913 in accordance with the most recent critical mass calculations, and permits the controlled use of UO_3 recovered acid, containing a limited amount of enriched uranium, in the Purex dissolvers. Please appropriately attach this addendum to your copy of the document.

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ADDENDUM TO HW-56913

I Page 9 Insert the following paragraph under:

1.04.02 Vessels Prior to the IBX Column

Uranium of low enrichment, 0.95 percent U^{235} or less, dissolved in 48 percent or greater nitric acid has no greater reactivity with irradiated natural uranium slugs than a water-moderated system, if the concentration of the dissolved low enriched uranium does not exceed 0.15 lb/gal. Consequently, UO_3 acid, recovered from the calcination of low enriched uranium, may be used under controlled conditions for the dissolution of irradiated natural uranium slugs.

II Page 14 Modify the last sentence under:

1.05.02 Dissolvers, to read,

Enriched uranium greater than 0.95 percent U^{235} shall not be introduced in any form.

Add the sentence:

Uranium containing 0.95 or less percent U^{235} may be added to the dissolvers in 48 or greater percent nitric acid provided the total uranium concentration of the acid does not exceed 0.15 lb/gal.

Reference:

Letter, R. L. Stevenson to E. R. Irish, "Allowable Uranium in Recovered Acid", December 1, 1958.

Issued By:

E. R. Irish
E. R. Irish, Manager
Purex Technology Operation
Research and Engineering, CPD

12/10/58
Date

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