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SPECIFICATIONS FOR CRITICAL MASS CONTROL
REDOX PLANT - E-METAL

AUTHOR

M. K. Harmon

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SPECIFICATIONS FOR CRITICAL MASS CONTROL

REDOX PLANT - E-METAL

By

M. K. Harmon

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Redox Technology Operation
Research and Engineering Operation
CHEMICAL PROCESSING DEPARTMENT

By Authority of CG PR-2,
WASnyder, 1-6-94.

By Jerri Maly, 1-10-94.

Verified By J E Savely 1-31-94

July 1, 1959

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SPECIFICATIONS FOR CRITICAL MASS CONTROL

REDOX PLANT - E-METAL

INTRODUCTION

The specifications given in HW-42300(19) are for the processing of irradiated natural uranium; the specifications given in this document cover the processing of E-Metal and are supplementary to those in HW-42300. These specifications include process limits based on plant experience with the precycle-type flowsheet and make allowance for the most advanced flowsheet proposals(17).

E-Metal is defined as natural uranium which has been enriched with U-235 to a concentration of 0.95 percent. The higher U-235 content makes the E-Metal more reactive than either natural or irradiated natural uranium. This difference in reactivity places a definite limit on the amount of massive metal that can be charged to the present Redox dissolvers. Once the slugs have been dissolved, the processing of E-Metal is essentially the same as the processing of natural uranium. Thus, except for the dissolver charge size, the following specifications reflect few actual changes in numerical limits. One change that needs to be recognized is that the amount of U-235 plus plutonium in the metal solution is much closer to the maximum "always safe" ratio than with irradiated natural uranium (see Section B 1.01). Once the E-Metal uranium has been separated from its accompanying plutonium, no critical mass of uranium can be assembled because the enrichment is below the minimum critical.

The plutonium isotope, Pu-239, and the uranium isotope, U-235, are capable of both slow-neutron and fast-neutron chain reactions. Slow-neutron chain reactions result when the neutrons released by the fission of a Pu-239 or a U-235 nucleus are moderated to thermal energy levels by collisions with other kinds of atoms before capture by other Pu-239 or U-235 nuclei, which in turn fission and continue the reaction. Fast-neutron chain reactions are basically the same, except that the free neutrons undergo little or no moderation and thus lose little or no energy before capture. A certain minimum quantity of plutonium and/or uranium must be present for a given geometry, moderation, and reflection before a chain reaction can take place. This quantity is called the minimum critical mass. The fission capture cross-sections for plutonium and uranium are smaller for fast neutrons than for slow neutrons. Thus, the critical mass for a fast-neutron reaction is larger than for a slow-neutron reaction -- not less than 5 kilograms of plutonium for a fast-neutron reaction as compared to a minimum of approximately 0.5 kilogram for a water-moderated slow-neutron reaction. Fast-neutron reactions can take place only at high plutonium concentrations (well above 500 grams per liter) with atomic ratios of hydrogen to plutonium well below 50 and are thus impossible under the conditions of the Redox process.

The prevention of a slow-neutron chain reaction in the Redox Plant and in the 233-S Plutonium Concentration Building is based upon the application of several basic principles. These principles are given in detail below. Most of the equipment in the Redox Plant proper is of sufficient size to permit criticality, whereas the geometry of all but two of the 233-S Building vessels precludes criticality for systems of solutions of less than 500 grams per liter plutonium concentration and limited neutron reflection. Therefore, the solution compositions in the Redox Plant and the plutonium content of two 233-S vessels must be limited to prevent chain reactions.

The limits are applied to the non-safe vessels wherever possible in such manner that two or more highly improbable off-standard conditions must exist simultaneously to promote criticality. Thus the plutonium concentration is held below the always-safe value, precipitating agents are not piped to tanks containing uranium-free plutonium, and many of the plutonium vessels are continuously agitated. The amount of plutonium which may be stored in any vessel is held below the conservatively estimated minimum critical mass when such mass is based on the assumed formation of a layer of homogeneous precipitate which has not settled to the bottom of the tank. This assumption provides complete reflection for neutrons both above and below the precipitate. These critical masses are smaller than would be obtained if precipitates settled to the bottom of the E-Cell tanks because all have inverted dished bottoms. The dished bottoms in the Feed Tank (L-1) and the Recycle Addition Tank (L-16) necessitate more conservative bases for the minimum critical masses to allow for the "improved", i.e., less safe geometry. Process conditions have been investigated and redefined with regard to precipitation during normal processing. In tanks containing solutions of uranium and plutonium, the ratio of plutonium to uranium is held below the always-safe value. The controls thus take advantage of the neutron poisoning effects of U-238 but do not allow for the beneficial effects (increased critical masses) which result from other neutron poisons such as nitrate ions, which are normally present in process streams.

There is a variation of critical mass with the exposure history of the metal due to the variation in the ratio of Pu-240 to Pu-239, and the specifications are therefore based on two ranges of exposure. (Significant flowsheet economies result from the use of the higher critical mass of the higher exposure metal whenever applicable.)

The nomenclature of the Redox Plant is used throughout these specifications instead of the HW-No. 5 Flowsheet⁽¹⁴⁾ nomenclature used in the previous specifications⁽¹⁹⁾.

BASES

B 1.01 Always-Safe Ratio of Plutonium-to-Uranium Concentration

There is a ratio of plutonium to E-Metal uranium (Pu/U) concentration below which the neutron-absorbing capacity of the uranium prevents criticality. In other words, an "always-safe" solution is one whose components when precipitated, will not result in a critical concentration no matter what quantities are involved.

In conjunction with the always-safe ratio, experimental work⁽²⁰⁾ determined the ratio of plutonium to uranium in solution before and after partial precipitation with sodium hydroxide. Using dissolver solution with greater than 90 percent of the plutonium in the tetravalent state, the precipitate was found to be rich in plutonium due to the formation of plutonium(IV) polymer at the lower acid concentrations. Under the proper conditions, the plutonium concentration relative to uranium in the precipitate may be increased by as much as a factor of two. With greater than 90 percent of the plutonium in the hexavalent state, solution was found to be rich in plutonium after precipitation occurred. The plutonium enrichment of the solution is probably due to the slightly

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higher solubility of plutonium(VI) versus uranium at the lower acid concentrations. Therefore, the neutralization of high-acid dissolver solution with caustic should only be performed after the oxidation of the plutonium to the hexavalent state.

Nitrate solutions resulting from the dissolution of E-Metal in nitric acid are safe solutions as long as they are not precipitated. Once the plutonium in the dissolver solution has been oxidized from the tetravalent to the hexavalent state, the solution is always safe. This always-safe solution will remain always-safe as long as the plutonium is in the hexavalent state and as long as the always-safe ratio of Pu/U is not exceeded by the addition of more plutonium, e.g., Z-Plant recycle, rework plutonium from waste or from the Plutonium Concentration Building (233-S).

The margin between the always-safe and not always-safe ratios is very narrow at the higher exposure levels. This narrow margin precludes the addition of very much, if any, plutonium rework via either the head-end process or the LAFS Concentrator (F-2). The always-safe ratio has been calculated for various pile exposure levels and is plotted in Figure I versus the grams of plutonium per ton of uranium (g/T) to be expected from the most efficient pile at HAPO. As can be seen from Figure I, the always-safe ratio is greater than the expected g/T up to 800 g/T at which point the two are equal. Above 800 g/T (approx. 1000 MWD/T) the actual ratio of plutonium to uranium in the "as-received" irradiated E-Metal is the always safe ratio; thus no plutonium may be added to this system prior to uranium-plutonium separation.

Thus, to assure nuclear safety of E-Metal processing between the dissolvers and the 1B Partition Column, the following two conditions should be assured: first, the plutonium in the dissolver solution should be oxidized to the hexavalent state as soon as practical, but definitely before the addition of a caustic solution, and second, the always-safe ratio of Pu/U should not be exceeded.

B 1.02 Always-Safe Plutonium Concentration

In an infinite volume of solution, a minimum concentration of 8 grams per liter of plutonium in water is required to produce a neutron chain reaction⁽¹³⁾. Nominal flowsheet conditions have been established so as to limit all Redox Plant solutions (except for certain streams in the 233-S Plutonium Concentration Building) to a maximum plutonium concentration of 23 grams per gallon (6 grams per liter) or 75 percent of the minimum critical concentration⁽¹¹⁾. In real, finite vessels, the minimum critical concentration is greater than 30 grams per gallon (8 grams per liter) and depends upon metal exposure, vessel geometry, tamping, and the presence of other ions. The safety margin involved in the use of an upper limit of 23 grams per gallon is thus greater than 25 percent.

The maintenance of a subcritical plutonium concentration should not be considered an adequate safeguard against criticality. Therefore, wherever the possibility exists that plutonium might be precipitated due to a combination of operational errors, limits are established on either the total

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amount of plutonium or the plutonium-to-uranium ratio in all vessels not geometrically safe. Also, limits are placed on plutonium concentrations and solution storage times to minimize the possibility of precipitate formation by reaction with hexone (see Section B 1.05, below).

As indicated in Section B 1.06, below, the flowsheet conditions also provide safe solution concentrations in hexone phases for those vessels in which two-phase systems could accidentally occur.

B 1.03 Always-Safe Geometry

The results of critical mass studies have indicated that slow-neutron criticality is impossible using plutonium solutions of less than 500 grams per liter concentration in infinitely long, water-surrounded cylinders with inside diameters not more than about 5 inches, and air-surrounded cylinders with inside diameters not more than about 8 inches. Such cylinders are said to be of always-safe geometry and this term is applied to any configuration in which slow-neutron criticality is not possible. In determining that a vessel is always safe from criticality by virtue of its geometry, it is not sufficient to consider the vessel's configuration by itself. Rather, the possible reflecting, moderating, or absorbing effects of supporting members, nearby equipment, piping and personnel must be taken into account. It is therefore not possible to define simply and completely the dimensional limits for safe geometry in real systems of near-optimum size.

B 1.04 Batch Size Control

For nuclear safety, only a limited quantity of fissionable materials may be stored in a vessel which is not of always-safe geometry. The procedure of limiting the total amount of this material in a vessel to less than the minimum critical mass using analysis and volume or weight measurements is known as batch control. Batch control is exercised for both solid fuel elements as charged to the dissolvers and for the storage of solutions.

The allowable weight of solid fuel elements that may be charged to the present Redox dissolvers (original design) depends upon the amounts of U-235 and Pu-239 present in the elements. At the optimum irradiation level, the equivalent U-235 content (the plutonium content converted to U-235 equivalent plus the remaining unconverted U-235) is used as the basis for the minimum critical mass calculations. In practice, a maximum of 45 percent of the minimum critical mass value is used as the dissolver charge size. This value allows a safety margin for double charging and for the presence of a small undissolved heel.

The allowable plutonium batch in any vessel is a fraction of the minimum critical mass for the vessel calculated on the basis of a neutron-reflected precipitate layer(3,8,9,10,11). Batch control is effected for the plutonium vessels by either a) adjusting stream flow ratios to obtain plutonium concentrations corresponding to the storage of no more than an arbitrarily established limit (90 percent) of the calculated minimum critical mass when

the vessels are filled to the overflow, or b) controlling solution transfers so that vessels never contain more than that fraction of the calculated minimum critical mass.

A more rigorous control, limiting the amount of plutonium to less than one-half of the minimum critical mass, is applied to the Recycle Addition Tank (L-16). This is discussed in Section B 1.08.02.

As indicated in Section B 1.02, no vessel under batch control routinely receives plutonium solutions which exceed the always-safe concentration.

B 1.05 Avoiding Precipitation of Fissionable Materials

The capacity of the Redox Plant is such that multiples of the minimum critical mass may be processed through many of the vessels of non-safe geometry in a 24-hour period. These process solutions of uranium and plutonium or plutonium alone are designed to be critically safe, but undetected partial precipitation on a continuing basis could lead to the accumulation of a critical mass. The gradual accumulation of fissionable materials in solid form, proceeding unnoticed is one of the greatest potential critical hazards. The accumulation of a potentially critical amount of plutonium and enriched uranium or plutonium alone through precipitate formation is avoided as follows:

- (a) Precipitating agents are added only to metal solutions with an always-safe ratio of Pu/U.
- (b) No agents which precipitate uranium or plutonium are piped to the metal solution storage tanks (H-7, H-8, or H-9), to the plutonium cycle tanks, or to the Plutonium Concentration Building (233-S).
- (c) Flowsheet variables such as plutonium and nitric acid concentrations are established to avoid conditions favoring plutonium precipitation.
- (d) The maximum amounts of plutonium allowed in the plutonium cycle tanks at any time are less than the critical amounts.
- (e) Time limits are imposed on the storage of hexone-bearing plutonium solutions.

The pick-up of 20 kilograms of plutonium from the 233-S Building during November, 1958, demonstrates quite clearly that a gradual accumulation of fissionable material in a process vessel is possible. The methods of detecting such a build-up are by means of a) material balance, b) neutron monitoring, and c) thorough nitric acid flushing. Wherever possible, all three methods are used. Since the first two methods are not always accurate or available, 60 percent nitric acid flushing of all uranium and plutonium processing equipment is performed at scheduled six-month intervals to assure that no deposition of uranium or plutonium has occurred. Non-routine acid flushes are also made if large material balance discrepancies are observed, which cannot be logically explained.

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In the case of the plutonium concentration equipment in the 233-S Building, if there is an apparent "loss" of plutonium between the 3BP Sampler Tank (E-3) and the Concentrated Plutonium Sampler Tank (L-6) greater than five (5) kilograms of plutonium exclusive of normal heel holdups, the 233-S Building equipment is repeatedly flushed with 60 percent nitric acid until the plutonium accumulation has been removed or proved not to exist. Although five kilograms of plutonium is the maximum allowable in the 233-S Building (based on the 7-inch diameter L-2 Steam Stripper containing a solid plug of plutonium polymer) it is very strongly recommended that any "loss" of plutonium over and above normal heels should instigate immediate remedial action.

The lack of concrete evidence of plutonium precipitation in process vessels under conditions heretofore successful in preventing precipitation does not remove the need for continuing alertness to this possibility. Abnormal analytical results on plutonium streams or the recovery of larger-than-normal amounts of plutonium in vessel flushes may be indicative of plutonium precipitation.

B 1.06 Avoiding Two-Phase Systems

More than seven years of operating experience has revealed no tendency toward accumulation of hexone layers in the 2AF (E-5, E-6, and E-7) and 3AF (E-1) Tanks. Nevertheless, it is important that the operating conditions assuring single-phase systems in these vessels be continued so as to avoid possible non-representative samples of the tank contents and the undetected accumulation of potentially critical amounts of plutonium. The plutonium(VI) distribution between the organic and aqueous phases in such systems favors the hexone layer by a factor which is a function of the aqueous phase $\text{Al}(\text{NO}_3)_3$ and HNO_3 concentrations. If the agitation in the vessels containing two phases is inadequate, the aqueous phase samples represent a value of the plutonium content of the tank well below the actual value because the plutonium concentration in the hexone phase is higher than in the aqueous phase. Moreover, the likelihood of obtaining a sample of the organic phase is slight. The possibility of accumulating plutonium in potentially critical amounts in undetected hexone layers is minimized by combinations of the following controls:

- (a) The columns feeding the vessels where hexone layers are particularly undesirable are normally operated well below their flooding velocities and process specifications do not permit operation beyond 90 percent of the flooding velocities.
- (b) The 1BP and 2BP Butt solutions are hexone-free aqueous solutions and therefore dissolve hexone existing in the 2AF and 3AF Tanks.
- (c) The concentration of plutonium(VI) in a hexone phase which is in equilibrium with an aqueous phase is primarily a function of the aqueous phase $\text{Al}(\text{NO}_3)_3$ and HNO_3 concentrations. The 2AF and 3AF compositions

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are varied to control the plutonium distribution so that plutonium concentrations in hexone phases (if any exist) are below the minimum critical values for homogeneous systems in the corresponding tanks.

- (d) The 1B and 2B Columns are provided with instruments which reveal flooding or instability and it is required that these instruments be read hourly. Flooding conditions of the more common types are revealed by readings of the column static pressure or air pressure to the let-down valve. A large loss of hexone from the 1B Column noticeably affects the (recorded) density of the 1BU stream.
- (e) Plutonium in the LBP Tank (E-6) is maintained in the aqueous-favoring trivalent state by the presence of ferrous ion. The LBP Tank (E-6) contents are not agitated and the tank thus acts as a phase separator, so that little or no hexone as a separate phase is transferred to the Cross-Over Oxidizer (E-7) and the 2AF Tank (E-5). From time to time the E-6 Tank is jettied empty and any accumulated hexone is returned to the organic system.
- (f) The sudden accumulation of large amounts of plutonium in hexone layers in the 2AF or 3AF systems would be reflected by decreased plutonium concentrations in the routine 2AF and 3BP samples and would be shown by the 3BP in-line monitor. Hexone in the 3AF system is highly unlikely due to the hexone stripping normally provided by the Plutonium Ozonator (E-2).

Hazards inherent in organic-aqueous two-phase systems are controlled by avoiding flooding or inadequate phase disengagement in the 1B and 2B Columns. If these column control precautions were not observed, hexone layers could accumulate in aqueous feed systems. However, the plutonium concentration in the hexone phase would be no more than 90 percent of the minimum critical concentration for a homogeneous system (by virtue of control item (c), above). The safety of a deep hexone layer with 90 percent of the minimum critical plutonium concentration would depend strictly upon maintaining or reducing that concentration. Effects, such as precipitation or stripping into smaller volumes of an aqueous phase, could lead to criticality simply by raising the plutonium concentration somewhat in excess of 11 percent. Hexone layers should thus be treated promptly but with extreme caution to avoid concentration effects. The plutonium content of such a hexone layer could be reduced to safe levels by successive equilibrations with portions of 0.9 M $\text{Al}(\text{NO}_3)_3$ solution of 0.10 M HNO_3 and at least 0.01 M $\text{Na}_2\text{Cr}_2\text{O}_7$ concentration. The suggested scrub solution would provide a plutonium distribution coefficient of unity between the two phases.

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B 1.07 Limitations on Column Stream Compositions and Flow Ratios

The non-safe dimensions of many of the solvent extraction columns require that some flowsheet limitations be imposed to preclude criticality during normal operation or during conceivable non-standard operation. Flowsheet specifications affecting nuclear safety are presented here as a control over future flowsheet revisions. Before any changes in stream compositions and flow ratios are made, the possible hazards of plutonium reflux or accumulation must be considered and allowances made in the flowsheet design or in the sampling schedule. "Hybrid" flowsheets and dual-scrub type of column operation in particular present the hazard that plutonium may be fed to a system from which there is no "escape" until high reflux concentrations are attained.

Plant experience and laboratory studies have demonstrated that plutonium may be reduced in valence and precipitated from hexone solutions of low acidity even in the presence of an aqueous phase containing dichromate ion(18,19). Since the reduction reaction is a function of acid concentration, a lower limit is set on acid concentrations for the upper scrub solutions in contact with hexone solutions. The success of such control method has been demonstrated.

B 1.07.01 Plutonium Stability in Hexone

Preventing plutonium deposition from the hexone phase overflowing the 1A and 2D Columns and plutonium reflux in the scrub sections of these columns requires that the hexone contain a finite quantity of nitric acid. Nitric acid concentrations in hexone above a given minimum result in stable hexone and plutonium solutions. Nitric acid concentrations in hexone below this minimum, result in solutions which are stable for periods of time which decrease as the nitric acid concentration decreases. Thus, when a hexone solution of plutonium has been in contact with a 0.2 M acid-deficient aluminum nitrate solution, the period of stability is about 30 minutes. In tests performed in the laboratory and later confirmed in the acid-deficient 1A and 2D Columns, it was shown that if the 1AA and 2DA streams were acidified to 0.1 M nitric acid, a hexone solution of plutonium with a stability of 10 hours would result. Nitric acid concentration in the hexone phase is a function of its flow rate and the flow rate of the counter-current stream with which it comes in contact. Therefore, when any changes are made in either the 1AA or 2DA composition or flow ratio or in the flow ratios of the 1AX or 2DX, samples of the hexone phase leaving the given column are obtained and tested for plutonium deposition characteristics(20).

The significant differences between the distribution coefficients (E_d) for uranium(VI) and plutonium(VI) with low salt concentrations present possibilities for plutonium reflux in the 1A and 2D Column scrub sections. Thus, an increase in the concentration

of plutonium relative to that of uranium beyond the ratio in the feed streams is possible. In the absence of precipitating conditions in the columns, the hazards of plutonium refluxing are not great and are reduced to negligible proportions by one or a combination of the following:

- (a) establishing a minimum for salt concentration in the scrub stream;
- (b) maintaining the ratio of unsalted aqueous flow to organic flow below a safe maximum which is a function of the stream compositions; or
- (c) installing head-tank instruments (and/or establishing sampling schedules) so that two independent operating errors have to be made before salt-free scrub can be fed to the column.

Flowsheets which employ low-salt (or unsalted) scrub streams and method (b) are tested for freedom from excessive plutonium reflux or deposition in laboratory studies. The laboratory studies involve four scrub stages or more depending upon the plutonium profile throughout the scrub section.

The equipment requirements of method (c) apply to flowsheets which use salted scrub streams and which would be unsafe because of plutonium reflux if water scrubs were accidentally used.

Also, since hexone is slowly decomposed (particularly in acidic systems containing dichromate ion), solvent streams containing plutonium should be stripped of their plutonium as soon as possible to prevent the decomposition of hexone and subsequent precipitation of compounds of plutonium with the products of this decomposition, e.g., oxalic acid. Under normal operating conditions, the hold-up time between extraction and stripping is about 20 minutes at a processing rate of 10 tons per day.

B 1.07.02 LBX Ferrous Ion Concentration

A maximum ferrous ion concentration in LBX must be set to prevent plutonium reflux and possible criticality in the extraction section (or in the disengaging section) of the LB Column in the event of cessation of LBX flow. The normal hourly column instrument readings should reveal the condition before critical quantities of plutonium can accumulate, but the limit on ferrous ion concentration prevents criticality in the event of an undetected LBX flow failure or other cause for a stagnant aqueous phase in the LB extraction section.

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B 1.07.03 2AS (3AS) Composition

The plutonium reflux in the 2A Column scrub section depends upon the salt content, acidity, and aqueous-to-organic ratio (L/V) in the scrub section. The non-safe diameter of the 2A Column upper disengaging section requires that appreciable plutonium reflux be avoided by employing only certain combinations of 2AS compositions and L/V values. As in the 2A Column, the composition of the upper scrub stream in the 3A Column must be controlled to avoid plutonium reflux.

B 1.07.04 IOX Flow

Maintenance of the IOX flow is necessary to avoid possible criticality in the IO Column in the event of significant carry-over of plutonium in the organic waste (particularly 2BW and 3BW) streams.

B 1.08 Plutonium Concentration Facility (233-S Building)

The Plutonium Concentration Facility (233-S Building) is composed of vessels critically safe by geometry -- within certain limitations -- with the exception of the Feed Tank (L-1) and the Recycle Addition Tank (L-16). The calculations of critical mass limits of safe vessel geometry are based on a plutonium concentration of 500 grams per liter. The bases are discussed below.

B 1.08.01 233-S Equipment and Personnel Approach Thereto ⁽¹²⁾

The bases for the safe geometry of many of the vessels in the 233-S Building include limits on the proximity of other plutonium-bearing vessels and limits on the proximity of personnel. Such limits must be imposed because of possible neutron reflection effects (which were excluded or defined at limited levels, corresponding to air tamping, in calculating the always-safe dimensions of the equipment⁽⁷⁾) which could promote criticality. Thus, certain broad access limits which are always safe have been established, based on nearness of personnel to plutonium-bearing vessels and the number of personnel concerned, vessel geometry, and total plutonium and plutonium concentration in the vessels.

B 1.08.02 Feed Tank (L-1) and Recycle Addition Tank (L-16)

The Feed Tank (L-1) and the Recycle Addition Tank (L-16) are not of always-safe geometry. It is therefore necessary to limit the amount and concentration of plutonium in these vessels by means of batch control. Since the L-1 Tank normally receives solution only from the 3BP Sampler Tank (E-3), batch control is easily maintained.

The Recycle Addition Tank (L-16), on the other hand, receives plutonium solution from sources outside the Redox Plant via recycle and PR Cans and also from the 233-S Building process sump (IS-1). Thus, the possibility of plutonium precipitation in L-16 cannot be dismissed as easily as in the E-Cell vessels, for which more than seven years' operating experience can be drawn upon. The plutonium assay of material in the recycle cans added to the Recycle Tank may be seriously in error, or, due to operational error outside the Redox Plant, a plutonium-precipitating agent or plutonium polymer may be present in a recycle can. For these reasons the maximum batch size for the Recycle Addition Tank is established (Specification 1.04) at less than half the critical mass for this vessel. Other safety factors in the Recycle Addition Tank (L-16) are the continuous agitation of the tank contents and the frequent draining and flushing of the tank.

Addition of solution from the Greenhouse Sump (IS-1) is arbitrarily limited, due to the possibility of high concentrations of plutonium in this solution. The installation of the 1.2-gallon Sump Recirculation Tank (L-17) and its very accurate specific gravity and weight factor instrumentation makes it possible to determine the maximum amount of plutonium that could be present in the IS-1 solution. It is of the utmost importance that the L-17 instrumentation be maintained in good operating condition.

B 1.08.03 Greenhouse Sump and Load-Out Hood Pan

The Greenhouse Sump (IS-1) is critically safe. However, when the sump overflows and liquid backs out into the stainless steel floor pan, interaction with other pieces of process equipment becomes possible. Therefore, it is necessary to limit the amount of liquid permitted on the Greenhouse floor.

The load-out hood pan does not have an always-safe geometry. Therefore the depth of liquid in the pan must be limited.

B 1.08.04 Recycle Cans

The critical safety of recycle cans is assured by limiting the quantity of plutonium per can, by requiring that loaded cans be normally kept in their carriers, and by limiting the assembly of cans. In general, the same considerations govern the handling of recycle cans as apply to PR Cans.

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B 1.09 PR Cans⁽¹⁶⁾

In the absence of interaction with other plutonium-containing vessels, an individual PR can is critically safe by virtue of its geometry. Interaction effects are minimized by the lead-cadmium alloy shielding around the can and by the 23.5-inch diameter can carrier.

Restrictions on the plutonium content of PR Cans and on the maximum number of cans which may be assembled are designed to prevent criticality in the event of either of the following possible accidents: (1) a PR Can springs a leak, allowing plutonium solution to drain into the carrier, or (2) plutonium is precipitated in a PR Can or carrier.

Filled PR Cans should generally be handled and stored only in their carriers. This procedure will permit containment of leaks and spills, will provide shielding, and will prevent neutron interactions due to accidental placing of a PR Can in excessively close proximity to another plutonium-containing vessel.

SPECIFICATIONS

Over-all Redox Plant critical mass control shall be based upon the following general specifications:

- (a) These specifications apply to E-Metal uranium (0.945 percent U-235 initial enrichment) processing and are void if higher U-235 enriched uranium is introduced into the plant.
- (b) The Process Specifications for Operational Control - Redox Plant⁽²¹⁾, shall conform to the requirements of these Critical Mass Control Specifications.
- (c) All chemical processing operations (including the rework of plutonium solutions) in the Redox Plant shall conform to the Critical Mass Control Specifications regardless of whether or not such operations are within or outside the limits given in the Process Specifications.
- (d) Proposed new equipment or revisions to existing equipment used for processing streams containing plutonium, enriched uranium, or a mixture of the two during normal, rework, or off-standard operating conditions shall be subject to review for criticality considerations by the Manager, Redox Technology Operation.
- (e) Indications of precipitation of plutonium, enriched uranium, or a mixture of the two in process tanks shall be referred immediately to the Manager, Redox Technology Operation. In the event that the precipitation is confirmed, the Redox Plant shall be shut down and the difficulty remedied as directed by the Manager, Redox Technology Operation.

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- (f) Any process condition requiring a control method not defined by this specification shall be referred to the Manager, Redox Technology Operation.
- (g) The specifications in this document can be temporarily waived to accommodate an abnormal operating condition upon written approval by the Manager, Research and Engineering Operation, Chemical Processing Department.

S 1.01 Always-Safe Ratio of Plutonium-to-Uranium Concentration

Before the always-safe ratio of plutonium to uranium can be guaranteed in dissolver solution, the plutonium must be oxidized to the hexavalent state. Therefore, before the addition of any precipitating agent, e.g., sodium hydroxide, the dissolver solution must be made 0.01 molar in sodium dichromate, and heated to boiling. This hexavalent plutonium oxidation state should be maintained until the plutonium and uranium are separated in the 1B Partition Column.

For vessels in which safety is assured by use of the always-safe ratio, the plutonium content shall not exceed that determined from Figure I. This limit applies to the Oxidizer (H-4), Centrifuge Feed Tank (G-5), 1AF Adjustment Tank (H-1), 1AF Feed Tank (F-7), 1SF Feed Tank (F-8)*, 2DFS Concentrator (F-2), and 2DFS Feed Tank (F-1).

S 1.02 Always-Safe Plutonium Concentration

None of the plutonium solutions in the Redox Building shall exceed a plutonium concentration of 23 grams per gallon (6 grams per liter). In the 233-S Building, the plutonium concentration in the Feed Tank (L-1) and the Recycle Addition Tank (L-16) shall not exceed 23 grams per gallon.

S 1.03 Always-Safe Geometry

A complete definition is not possible for all safe geometry conditions and therefore equipment changes involving safety by virtue of geometry require the approval of the Manager, Redox Technology Operation.

S 1.04 Batch Size Control

The maximum amount of E-Metal (0.95 percent U-235 before irradiation) that may be charged into the Redox Plant dissolver vessels of standard design, is 2.0 tons⁽²³⁾.

The amount of plutonium stored in any vessel of non-safe geometry shall not exceed 90 percent of the calculated minimum critical mass for that vessel based on a completely water-tamped, water-moderated, uniformly settled precipitate. The pertinent vessels and limits are as follows:

* When F-8 is used for plutonium rework, critical mass control is exercised by means of batch size control (Specification 1.04), not by always-safe ratio.

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(a) Tanks Normally Containing Plutonium Only

<u>Vessel</u>	<u>0 to 400 MWD/T</u>		<u>Over 400 MWD/T</u>	
	<u>Total Grams</u>	<u>Grams/Gal</u> (1)	<u>Total Grams</u>	<u>Grams/Gal</u> (1)
3AF Tank (E-1)	1710	8.10	1890	8.92
2BP Ozonator (E-2), 3BP Sampler (E-3), and 3BP Rework Tank (E-4)	1260	10.0	1440	11.5
2AF Tank (E-5)	2340	7.30	2520	7.85
1BP Receiver (E-6)	2340	7.62	2520	8.21
Crossover Oxidizer (E-7)	2980	5.90	3240	6.42
233-S Feed Tank (L-1)	1340	10.0	1530	11.5
233-S Recycle Addition Tank (L-16)(2)	700	23.0	790	23.0

(1) The volume to overflow is used as a basis for setting concentration limits for all tanks except the 233-S Recycle Addition Tank (L-16).

(2) The maximum quantity of plutonium allowed in L-16 is less than 47 percent of the minimum critical mass for this tank.

(b) Tanks Normally Containing Very Low Plutonium Concentrations Or a Mixture of Plutonium and Uranium

The large tanks (4 to 10 feet in diameter) which normally contain only uranium or uranium and plutonium in the always-safe ratio, may on occasion be needed to contain plutonium solution. The batch limits on these tanks are as follows:

<u>Vessel</u>	<u>0 to 400 MWD/T</u>	<u>Over 400 MWD/T</u>
	<u>Total Grams</u> (1)	<u>Total Grams</u> (1)
4 to 10-ft. diameter	2980	3240
2DW Concentrator (D-14)(2)		
Pot & Reboiler Section	2980	3240
Cooler Section	1710	1890
2DFS Concentrator (F-2)	600	600

(1) At no time should these plutonium solutions be allowed to exceed 23 grams per gallon (6 grams per liter).

(2) Since steam condensate from the Plutonium Concentrator (L-3) is routed to D-14, the plutonium inventory in L-3 above the lowest possible point of leakage to the steam chest must conform to the D-14 limits.

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(c) Tanks Having Safe Geometries

These tanks are geometrically safe based on plutonium solutions of 1892 grams per gallon (500 grams per liter) and air tamping. All of these tanks are located in the Plutonium Concentration Building (233-S) and are L-2, L-3, L-4, L-5, L-6, L-7, L-9, L-10, L-12, L-14, and L-17.

S 1.05 Avoiding Precipitation of Fissionable Materials

Agents which precipitate plutonium and uranium shall not be piped to the Metal Solution Storage Tanks (H-7, H-8, and H-9), to the Plutonium Cycle Tanks (E-1, E-2, E-3, E-4, E-5, E-6, and E-7), to the 2DW Concentrator (D-14), or to the 233-S Building except under carefully controlled conditions specified and monitored by Redox Technology Operation.

Hexone-bearing plutonium solutions such as 2AF, 3AF, or 3BP, shall not be stored for more than fifteen days unless constant monitoring is provided which detects any change in the concentration of plutonium in solution as a function of time. Solution temperatures during storage must be held below 50 C unless hexone is first stripped from the aqueous phase.

To prevent polymerization and precipitation of plutonium within the Plutonium Stripper (L-2) the minimum nitric acid concentration in the 3BP stream shall be 0.4 molar^(21, 22).

In order to detect any uranium and/or plutonium that has gradually accumulated, a 60 percent nitric acid flush of all uranium and plutonium processing equipment shall be performed once every six months. If, at any time, there is an apparent "loss" of plutonium between the 3BP Sampler Tank (E-3) and the Concentrated Plutonium Sampler Tank (L-6) greater than five (5) kilograms of plutonium (exclusive of normal heel holdups) the 233-S Building equipment shall be repeatedly flushed with 60 percent nitric acid until the plutonium accumulation has been removed or proved not to exist.

If there are any indications of uranium and/or plutonium deposition, the Manager, Redox Technology Operation, shall be notified at once and an immediate investigation of the situation shall be made.

S 1.06 Avoiding Two-Phase Systems

The 1B and 2B Columns must not be operated in a flooded condition. The volume velocities shall be held below 90 percent of the flooding values. These columns are provided with instruments which reveal flooding or instability and it is required that these instruments be read hourly.

Any indication of flooding in the 1B or 2B Columns or of two-phase systems in the 2AF or 3AF Tanks must be investigated immediately and action must be taken to control the plutonium inventories in the affected vessels.

The salt strength of the aqueous phase in vessels where two-phase systems may accumulate shall be limited so that the plutonium concentration in any organic phase will be no more than 90 percent of the minimum critical concentration.

S 1.07 Limitations on Column Stream Compositions and Flow Ratios

S 1.07.01 Plutonium Stability in Hexone

The hexone streams leaving the 1A and 2D Columns shall have a minimum nitric acid concentration of 0.015 M to prevent valence reduction and subsequent polymerization and deposition of the contained plutonium. Because of the difficulty of getting a valid HNO_3 analysis in these organic streams plus the even more difficult task of computing the theoretical acid concentration from basic flowsheet data, conformance to this specification shall be determined by laboratory test⁽²⁰⁾. The stability of plutonium in these organic streams shall be checked by this method on samples taken from the plant immediately following a change in the flowsheet, e.g., changes in the feed or scrub compositions or flow ratios, which might be expected to affect the acidity of the organic streams in question.

Flowsheets employing 1AA and 2DA streams containing less than 0.9 M equivalent aluminum nitrate concentration shall also be tested in the laboratory to ensure freedom from hazardous plutonium concentrations or precipitation before being put into practice in the plant.

Organic solutions containing plutonium (1AP and 2DU) shall not be held more than eight hours unstripped.

S 1.07.02 1BX Ferrous Ion Concentration

The ferrous ion concentration of the 1BX shall not exceed 0.12 M.

S 1.07.03 2AS(3AS) Compositions

The acidity of the 2AS(3AS) shall be greater than 0.02 M nitric acid-deficient, at aluminum nitrate concentrations above 1.3 M and scrub-to-extractant volume flow ratios less than unity.

S 1.07.04 IOX Flow

The minimum IOX flow ratio shall not allow the maximum permissible plutonium concentration in IOX to exceed 23 grams per gallon (based on complete loss of plutonium into the organic waste streams at a rate no greater than that represented by the 1AF flow). The flow of IOX to the IO Column shall be confirmed at a minimum frequency of once every 8 hours.

S 1.08 Plutonium Concentration Facility (233-S Building)

S 1.08.01 233-S Equipment and Personnel Approach Thereto

Changes to the 233-S Building equipment and the approach of personnel to this equipment are defined below.

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(a) 233-S Building Equipment

1. The as-built arrangements of 233-S vessels with respect to one another or with respect to any physical features of the Greenhouse shall not be changed without review by the Manager, Redox Technology Operation.
2. No temporary containers of any kind shall be used for plutonium solutions (regardless of location) without prior review of pertinent circumstances by Technology personnel and authorization by the Manager, Redox Technology Operation.
3. Foreign objects such as tools, ladders, pieces of pipe, failed equipment, etc., must not be brought into the Greenhouse when vessels contain plutonium solutions of greater than 23 grams per gallon concentration and must not be left in the Greenhouse during operation without the approval of the Manager, Redox Technology Operation.

(b) Personnel Entry

Entry of personnel into the Greenhouse during a time when the plutonium hold-up in any vessel may be greater than 500 grams shall be governed by the following provisions:

1. In the absence of other limitations, only one man at a time may approach any plutonium-containing vessel and his body must not be nearer than four feet.
2. Any number of persons may approach any cylindrical vessel whose inside diameter does not exceed 4.5 inches.
3. Any number of persons may approach any vessel in which the plutonium concentration does not exceed 23 grams per gallon.
4. Special situations not covered by the above will be handled on the basis of consultations with members of the Redox Technology Operation and the Senior Engineer for Nuclear Safety, Advanced Process Development Operation, Research and Engineering.

S 1.08.02 Feed Tank (L-1) and Recycle Addition Tank (L-16)

- (a) Batch size and concentration limits for the Feed Tank (L-1) are listed in Specification 1.04 (a).

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- (b) Batch size and concentration limits for the Recycle Addition Tank (L-16) are listed in Specification 1.04 (a).
- (c) Enough water shall be added to L-16 to allow the operation of the agitator prior to the addition of any plutonium solution (recycle, rework, etc.).
- (d) The agitator in L-16 shall be operated whenever the liquid level in the tank is sufficient to cover the blades.
- (e) The final acidity in L-16 shall be 0.3 M HNO_3 (0.15 lb. per gallon) or more.
- (f) The L-16 Tank shall be completely drained and flushed after every second batch of recycle.
- (g) The maximum single-batch addition of material to L-16 from the Greenhouse sump (IS-1) shall be 1.2 gallons. The specific gravity of each 1.2-gallon batch of material added to L-16 shall be determined in L-17 and the plutonium content thereof shall be calculated prior to its addition to L-16. If for any reason the validity of the specific gravity determination in L-17 is in doubt (and therefore the maximum plutonium content also) the material shall not be added to L-16 until the problem is adequately resolved.

S 1.08.03 Greenhouse Sump and Load-Out Hood Pan

Liquid in the Greenhouse floor pan shall not exceed 58 gallons, i.e., liquid shall not be allowed to touch the side of the floor pan at its upper edge.

The floor of the 233-S Loadout Hood pan shall not be covered with liquid to a depth greater than 1.8 inches.

S 1.08.04 Recycle Cans

- (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) Not more than one bare (i.e., without can carrier) loaded recycle can shall be kept in the Loadout Room at any time.
- (c) A bare, loaded recycle can shall be kept at a minimum distance of 3 feet from PR Cans, even though the other cans may be in can carriers.

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- (d) Loaded recycle cans shall not be removed from the can carriers except in the Loadout Room.
- (e) Loaded recycle cans (in can carriers) shall be moved and stored in a horizontal single-layer array of any number of cans. Loaded recycle cans shall not be stacked in more than one horizontal layer.

S 1.09 PR Cans

- (a) As used in these specifications, "PR Cans" are those vessels defined by Drawing H-2-56967, and "can carriers" are those carriers defined by Drawing H-2-52984.
- (b) The maximum allowable mass of plutonium in a PR Can is 4.5 kilograms; the maximum allowable plutonium concentration is 1703 grams per gallon (450 grams per liter).
- (c) Not more than one PR Can at a time shall be located in either the Loadout Hood or the Decontamination Hood.
- (d) Not more than one bare (i.e., without can carrier) loaded PR Can shall be kept in the Loadout Room at any time.
- (e) The center-to-center distance between a bare, loaded PR Can and a second loaded can shall not be less than 23.5 inches.
- (f) Loaded PR Cans shall be placed in can carriers before removal from the Loadout Room and shall be moved and stored in these carriers.
- (g) PR Cans shall be stored and transported only when they are in their can carriers. Arrays of PR Cans (in can carriers) shall be limited to a maximum of 60 cans in a single-layer array and to 30 cans in a two-layer array. Loaded cans shall not be stacked in more than two layers and if stacked in two layers, the second layer must be positioned above the first (can for can) on a structurally sound and continuous support-mechanism.

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FIGURE I

PLUTONIUM CONTENT OF IRRADIATED E-METAL

VERSUS

"ALWAYS-SAFE" RATIO OF PLUTONIUM TO URANIUM

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