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DEFENSE NUCLEAR FACILITIES SAFETY BOARD

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August 1, 2003

The Honorable Everet H. Beckner
Deputy Administrator for Defense Programs
National Nuclear Security Administration
U.S. Department of Energy
1000 Independence Avenue, SW
Washington, DC 20585-0104

Dear Dr. Beckner:

The Defense Nuclear Facilities Safety Board (Board) has been following closely the development of safety basis controls and startup activities for the new aqueous recovery line for plutonium-238 (Pu-238) scrap at Los Alamos National Laboratory (LANL).

Pu-238 is the dominant radioactive source term in glovebox operations in the LANL Technical Area-55 (TA-55) Plutonium Facility. A release of Pu-238 in TA-55 was the cause of multiple room contaminations and significant personnel intakes in March 2000. The accident resulted in a Department of Energy Type A investigation and subsequent corrective actions by the laboratory. Any future upset or accident involving Pu-238 in this unique facility could adversely affect the health and safety of the public, workers, and the environment, as well as national security.

LANL has been pursuing startup of the new Pu-238 aqueous recovery line for several years. In an April 23, 2002, letter to the National Nuclear Security Administration (NNSA), the Board observed that the potential hazards of this new line had not been adequately addressed. The Board requested that NNSA report on the resolution of deficiencies in hazard identification; hazard analysis; and selection of controls, including engineered controls and Technical Safety Requirements. NNSA responded on July 1, 2002; however, that response did little to address the cited deficiencies or improve the safety of this operation. As a result of subsequent comments provided by the Board, NNSA and LANL have since pursued improvements. In January 2003, LANL submitted to NNSA an updated process hazard analysis (PrHA) and new proposed controls. The Board reviewed this updated PrHA and proposed controls, and again provided comments to help resolve the remaining weaknesses in the safety basis. NNSA also provided LANL with comments on the updated PrHA. LANL issued another revised PrHA in May 2003, and it was immediately approved by NNSA.

The Board's evaluation of the revised PrHA has revealed weaknesses similar to those previously identified by the Board. The enclosed report provides a detailed discussion of the identified deficiencies and identifies measures for improving the safety of the recovery line. The actions that could improve safety include:

- designating safety-significant engineered controls to prevent the accumulation of flammable gases in the dissolver and filtrate storage vessels,
- implementing a Technical Safety Requirement control to track dose to the ion exchange resin to ensure it does not exceed safe levels of radiation exposure,
- designating safety-significant engineered controls to prevent the ion exchange resin from drying out,
- evaluating the safety impacts of the reformulation of the ion exchange resin,
- and designating the controls that prevent violent reactions involving hydroxylamine nitrate as Technical Safety Requirements.

Given the potential hazards of this new recovery line, the Board requests a briefing regarding resolution of the remaining issues before NNSA conducts its readiness review for startup of the recovery line.

Sincerely,

John T. Conway

John T. Conway
Chairman

c: Mr. Ralph E. Erickson
Mr. Mark B. Whitaker, Jr.

Enclosure

DEFENSE NUCLEAR FACILITIES SAFETY BOARD

Staff Issue Report

June 26, 2003

MEMORANDUM FOR: J. K. Fortenberry, Technical Director

COPIES: Board Members

FROM: J. Contardi

SUBJECT: Aqueous Processing of Scrap Plutonium-238 Oxide at Los Alamos National Laboratory

The staff of the Defense Nuclear Facilities Safety Board (Board) has been following closely the development of safety controls and startup activities for the new recovery line. This report documents the findings of the Board's staff concerning the adequacy of the hazards analysis and controls for the recovery line.

Background. Los Alamos National Laboratory (LANL) is preparing to start up an aqueous processing line for the purification of scrap plutonium-238 (Pu-238). The new recovery line will be in Technical Area-55 (TA-55). Currently, the Department of Energy (DOE) lacks the ability to produce high-quality Pu-238 oxide. Historically, Pu-238 was produced in the reactors at the Savannah River Site (SRS) by neutron capture in neptunium-237 (Np-237) targets. Following irradiation in the reactors, the targets were processed to separate the plutonium from other fission and activation products. Once the plutonium had been separated out, it was oxidized and processed into fuel pellets. The pellets were then shipped off site to be packaged into radioisotope thermoelectric generators (RTGs) or general-purpose heat sources. The production reactors at SRS have been shut down for more than a decade, and DOE does not have the means to produce new Pu-238 feed material, Np-237.

Pu-238 is used in RTGs for space exploration and has been employed in Department of Defense applications. Pu-238 has a 87.74-year half-life, corresponding to a specific activity of 17.1 curies/gram, and decays via alpha emission to uranium-234. Because of its short half-life and high alpha energy, Pu-238 produces 0.56 watts/gram from radioactive decay. A typical general-purpose heat source contains approximately 150 grams of Pu-238 oxide.

The new recovery line at LANL will purify scrap Pu-238, providing DOE with a source of Pu-238 oxide. The processing flowsheet is similar to current operations in HB-Line at SRS and various other chemical processing lines in TA-55 at LANL. However, these other operations process Pu-239, which has a much longer half-life (24,100 years) and a correspondingly smaller specific activity (0.0621 curies/gram). The expected throughput of the new aqueous recovery line is 5 kg of Pu-238 oxide per year, with a sprint capacity of 8 kg Pu-238 oxide per year. Although the throughput of the recovery line is small in comparison with HB-Line and other aqueous operations in TA-55, the radioactive source term of the Pu-238 recovery line is comparable to or greater than that of Pu-239 processes because of the high specific activity of Pu-238.

LANL has been operating a bench-scale aqueous purification process, but considers the worker doses too high to allow its use for routine production operations. Under optimal conditions, the bench-scale process can produce about 440 grams of purified Pu-238 per month (Los Alamos National Laboratory, 2003a), but the actual throughput is lower because of material-at-risk limits in the safety basis (Los Alamos National Laboratory, 2002a).

Because of its high specific activity, accidents involving Pu-238 can result in significant worker exposure. This is illustrated by the March 2000 accident at LANL involving multiple intakes of Pu-238. This accident resulted in one worker receiving approximately 100 rem committed effective dose equivalent (CEDE) and generated a DOE Type A investigation (U.S. Department of Energy, 2000a). If the accident had occurred with Pu-239, the worker dose would have been significantly less (e.g., less than 1 rem CEDE).

In preparation for startup of the recovery line, the Board's staff reviewed the project's safety basis. The findings of the review were that the March 27, 2000, Process Hazards Analysis (PrHA) prepared by LANL (Los Alamos National Laboratory, 2000) and the subsequent December 1, 2000, Safety Evaluation Report (SER) issued by the National Nuclear Security Administration (NNSA) (U.S. Department of Energy, 2000b), failed to analyze several plausible accident scenarios, relied overly upon administrative controls, and implemented controls inappropriately.

On April 23, 2002, the Board sent a letter to NNSA documenting these findings (Defense Nuclear Facilities Safety Board, 2002). The Board's letter requested a report documenting how the identified deficiencies would be resolved. In July 2002, NNSA responded to the Board's letter (National Nuclear Security Administration, 2002), but failed to address many issues adequately. In August 2002, following further discussions involving NNSA, LANL, and the Board and its staff, LANL decided to reconsider the use of engineered controls in place of administrative controls and to delay declaration of readiness for an NNSA readiness assessment. LANL subsequently prepared a revised PrHA (Los Alamos National Laboratory, 2003b), which was submitted to NNSA in January 2003. In May 2003, LANL prepared another revised PrHA (Los Alamos National Laboratory, 2003c) that incorporated comments from NNSA. On May 29, 2003, NNSA issued a revised SER approving the May 2003 PrHA (U.S. Department of Energy, 2003).

During this time, the Board continued to analyze the safety of the aqueous recovery line and to provide feedback to NNSA. Appendix A lists the key interactions among the Board, NNSA, and LANL relative to these issues. On two occasions, the staff informally provided questions on the safety basis and controls to NNSA and LANL (see Appendices B and C). The current status of the principal issues is summarized in Appendix D and discussed in detail below.

The purpose of the recovery line is to remove impurities and produce Pu-238 oxide that meets the quality requirements of the customer. This purification is accomplished using the following processes: comminution, dissolution, ion exchange, oxalate precipitation, calcination and oxygen-16 exchange, and various solution transfer activities (see Figure 1).

Comminution. To increase dissolution efficiency, the scrap Pu-238 oxide is ground to a fine powder. The final step of this process uses a ball mill to reduce the Pu-238 oxide to a mean particle size of 5 µm. The ball mill is a mechanical device that rapidly shakes jars filled with scrap oxide and metal balls. The original design of the ball mill included features to prevent the ejection of a jar, but none of these features were functionally classified as safety controls to protect facility workers. Instead, the integrity of the Pu-238 oxide containers was functionally classified as the safety-significant control to prevent the release of material should a jar be ejected. The Board's April 2002 letter questioned this safety control strategy. Even if the storage containers maintained their integrity, the ejected jar could damage the glovebox and result in significant worker doses. The Board observed that a more robust safety strategy would prevent ejection of the jar, rather than mitigate the consequences of an ejection.

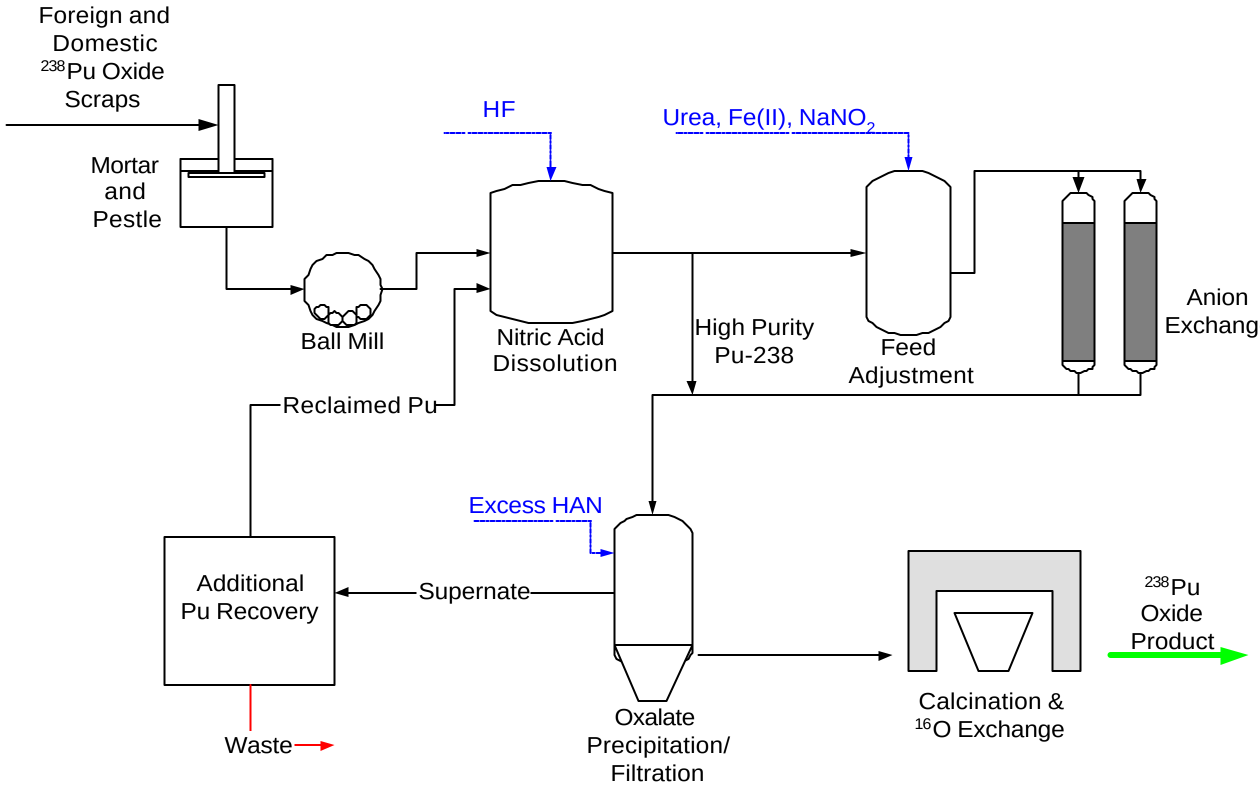


Figure 1. Simplified Process Flow Diagram (Los Alamos National Laboratory, 2003c)

NNSA's July 2002 response to the Board argued that an ejected ball mill jar lacks the energy necessary to break the glovebox windows and walls, but did not address the effect of an ejected jar on glovebox gloves, penetrations, or gloveport covers. In subsequent interactions, the Board provided additional comments on this safety strategy.

Subsequently, LANL decided to replace the plastic lid on the ball mill with a stainless steel lid to prevent the ejection of the ball mill jar. The May 2003 SER credits this as a safety-significant design feature.

The staff agrees that the new stainless steel lid provides an adequate safety control to address the potential for an ejection of a ball mill jar.

Dissolution. Following comminution, the Pu-238 oxide is dissolved for subsequent purification using ion exchange. Up to 300 g of oxide is dissolved in refluxed concentrated nitric acid. Although dissolved Pu-238 will generate hydrogen via radiolysis, a hydrogen deflagration in the dissolver or in a solution storage vessel was not analyzed in the March 2000 PrHA.

In its April 2002 letter, the Board raised the issue that hydrogen could be generated at a rate that would result in exceeding the lower flammability limit (LFL) in the dissolver headspace. NNSA's July 2002 response to the Board provided a calculation of the hydrogen concentration in the dissolver headspace. This calculation indicated that the hydrogen concentration would reach 95 percent of the LFL, at which point the hydrogen pressure would exceed the hydraulic head of the dissolver scrubber, allowing hydrogen to vent from the dissolver.

In subsequent interactions, the Board provided additional comments on this response. As a result, the January 2003 revision of the PrHA provides a revised analysis of the hydrogen concentration and addressed the hydrogen deflagration accident scenario.

The hydrogen generation analysis performed for the January 2003 PrHA calculates a maximum hydrogen generation rate of 0.056 liter/hour. Assuming a reasonably conservative 2 liter charge of acid and Pu-238 oxide in the dissolver, the minimum dissolver headspace will be 1 liter. The LFL for hydrogen/air mixtures is approximately 4 percent. Therefore, assuming no venting or purging, the dissolver headspace will reach the LFL within 1 hour and will reach the stoichiometric concentration in less than 8 hours. A typical dissolver operation is expected to require as long as 10 hours to complete.

In addition, solutions transferred from the dissolver to the filtrate storage vessels will continue to generate hydrogen at the same rate. The LFL will be reached in the filtrate storage vessels within 6 hours, assuming an unvented 7 liter headspace. The stoichiometric concentration for hydrogen and oxygen will be reached in the storage vessel in less than 2.5 days.

Dissolver Safety Controls—To protect facility workers, the January 2003 PrHA proposes safety-significant Technical Safety Requirement (TSR) administrative controls to verify the presence of an argon gas purge and to verify the operation of a purge/vent interlock within 24 hours of the start of dissolution. The argon gas purge will prevent the accumulation of flammable gases in the dissolver. In the event of a loss of argon purge, the purge/vent interlock will cause a valve to open and allow the dissolver to vent directly to the glovebox. Neither the argon purge nor the purge/vent interlock is classified as safety-significant. Although the TSR administrative control ensures that these features will be functional within 24 hours of starting the dissolution, it does not ensure the reliability of the argon purge or the purge/vent interlock during operation. The staff believes it would be appropriate to classify the purge/vent interlock as safety-significant to ensure prevention of a dissolver hydrogen deflagration.

The May 2003 SER credits the dissolver vessel as a safety-significant engineered passive control. This designation was mandated by NNSA because the gloveboxes have not been qualified to protect workers from missiles resulting from a hydrogen deflagration in the vessel. The SER references a September 2002 analysis performed by LANL to model the behavior of the dissolver vessel during a hydrogen deflagration. The results indicate that the stresses generated during a deflagration would be within 1–2 percent of the acceptable stress for a one-time-use pressure vessel under the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code. However, the ASME code does not envision the peak dynamic pressure effects of deflagrations. The differences in the static and dynamic pressure could be significant. In addition, the ASME code is not meant to be applied to teflon components such as the dissolver lid, and the analysis does not consider radiolytic degradation of the teflon lid. Also, the teflon lid of the dissolver has several penetrations for equipment (e.g., an electric mixer). Even if the dissolver maintained its integrity, equipment ejected through the lid could damage the glovebox. Based on these factors, the staff concludes that the dissolver vessel's ability to contain the force of a hydrogen deflagration has not been adequately demonstrated. The staff believes it would be more beneficial to upgrade the purge/flow interlock to safety-significant than to expend further effort analyzing the response of the vessel to a deflagration.

Filtrate Storage Vessel Safety Controls—Neither the PrHA nor the SER analyzes a deflagration in a filtrate storage vessel or identifies functionally classified safety controls to prevent such an event. In response to comments from NNSA on hydrogen generation, LANL states that small amounts of hydrogen will be generated in the storage vessel (Los Alamos National Laboratory, 2003d); as stated above, the staff's analysis shows that the LFL could be reached in less than 6 hours. There are two vessels, and each has a level sensor and pressure relief valve. The pressure relief valve is not functionally classified as a safety control and should not be relied upon to preserve the vessel's integrity in the event of a hydrogen deflagration. The storage vessels do have vent lines, but there are no safety controls identified to ensure that the vent lines are open. Also, the U-shaped configuration of the vent lines will hinder the buoyant migration of hydrogen (see Figure 2). Diffusion of hydrogen will occur through the line, but no analysis has been performed to show that the equilibrium hydrogen concentration in the vessel will remain below the LFL. Since filtrate may be stored for extended periods of time, the possibility exists for flammable amounts of hydrogen to accumulate in the filtrate storage vessel. The opportunity for an ignition source in the filtrate storage vessel is less than that in the dissolver vessel, but a conservative hazard analysis should always assume the presence of an ignition source. A safety control to either vent or purge the vessel would address this accident scenario.

Ion Exchange. Following dissolution, certain batches of the filtrate will be pretreated and purified via anion exchange. Nitrated ion exchange resins can undergo exothermic reactions or fires if they become degraded from excessive exposure to ionizing radiation and concentrated acid at elevated temperatures. The Reillex HPQ resin to be used in the recovery line has been shown to be more stable than previously used resins in the presence of concentrated acid. However, Reillex is still susceptible to runaway reactions if exposed to concentrated acid following 700 megarads of alpha radiation exposure or if allowed to dry out. The 700 megarad limit is merely a best estimate, and appropriate safety factors need to be considered in the safety analysis. Marsh (1990) has published data demonstrating that Reillex has superior stability for low radiation doses, but exhibited the worst thermal stability of the resins tested at high radiation doses. The experimental parameters (i.e., high temperatures) used to obtain these findings are not typical of conditions during normal aqueous processing, but the report nevertheless establishes a safe dose limit of 700 megarads alpha radiation for the resin.

The consequences of an unstable resin are illustrated by an accident that occurred at the Hanford Site on August 30, 1976, after a cation exchange column had been left loaded with americium for approximately 5 months (Science Applications International Corporation, 1985). Assuming similar loading, this is equivalent to a Pu-238 column left loaded for 1 month. When operations resumed, 7 M nitric acid was added to the column. The acid and degraded resin reacted exothermically, and the column exploded. The force of the explosion shattered the glovebox windows, injured the operator, and contaminated the room so severely that it was never used again. Although the type of resin and size of the column at Hanford (6 inches in diameter by 37 inches long) differ from the LANL recovery line (3 inches in diameter by 18 inches long), the accident illustrates the fact that resin columns can be left in a loaded condition and that reactions between degraded resin and acid can generate significant forces.

The March 2000 version of the PrHA did not analyze this accident scenario. The December 2000 SER recognized this deficiency and directed LANL to implement the following safety-class controls: (1) prevent resin dryout, (2) use only the Reillex HPQ resin, and (3) replace the resin every 5 years. NNSA also directed LANL to credit the stainless steel mesh around the glass ion exchange columns as safety-significant. In its April 2002 letter, the Board identified weaknesses in the controls preventing resin dryout and observed that crediting the steel mesh around the column was less desirable than crediting engineered controls that would prevent over pressurization (e.g., rupture discs on the columns). Furthermore, the December 2000 SER provided no justification or evaluation to ensure the adequacy of the stainless steel mesh to perform the desired safety function.

NNSA's July 2002 response to the Board's letter stated that a resin accident was impossible unless the resin had been in service for more than 10 years, and that a 5-year replacement interval offered a safety factor of two. The Board's staff evaluated this assertion, and informed NNSA that its conclusion was based incorrectly on dose estimates from historical processing of weapons-grade plutonium in TA-55. Both NNSA and LANL failed to consider that operations involving Pu-238 would subject the resin to a much higher dose rate, which would drastically reduce the safe service life.

Subsequently, LANL proposed a new control whereby visual observation of the resin would be used to determine whether it was discolored in a manner indicative of excessive radiation exposure. NNSA and LANL presented the Board with photographs showing the discoloration of the resin as a function of exposure to gamma radiation. Evaluation of this information by the Board's staff revealed that the resin did not exhibit equivalent behavior for alpha radiation, which is the principal mode of resin exposure from Pu-238. Photographs provided by LANL (see Figure 3) demonstrated that Reillex HPQ does not change color significantly between 500 and 800 megarads of alpha radiation exposure, rendering visual observation of resin discoloration ineffective as a means of surveillance. These observations were provided to NNSA and LANL.

The January and May 2003 versions of the PrHA identify new controls to limit resin degradation, to preclude resin dryout, and to minimize the impact of adverse resin reactions. LANL installed a safety-significant rupture disk on each column, which addresses the Board's concern regarding the need to prevent pressurization of the ion exchange columns. In the PrHA, LANL reestimates the dose rate to the resin and concludes that it would take only about 460 days to reach a dose of 700 megarads, not 10 years. Based on this calculation, the PrHA proposes safety-significant administrative controls consisting of a 1-year service life for the resin, supplemented by surveillances to ensure that the dose to the resin remains below 700 megarads and to check for discoloration of the resin. A monthly visual check of the liquid level in the column to prevent resin dryout is also identified.

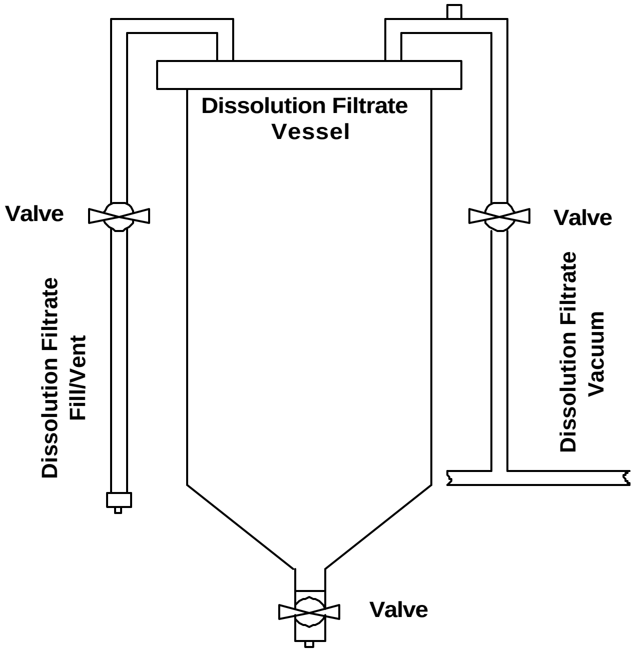


Figure 2. Dissolution Filtrate Storage Vessel (Los Alamos National Laboratory, 2002b)

The revised PrHA and the identified controls represent a major improvement over the previous treatment of resin accident scenarios; however, weaknesses remain in the analysis and identification of safety controls. The staff's evaluation is summarized below.

Resin Exposure Calculations and Controls—The dose calculation performed by LANL to support the proposed 1-year resin replacement interval assumes that the residual Pu-238 left on the column following elution would be the only source of radiation exposure to the resin.

However, the dose rate for a column loaded with one batch of Pu-238 (i.e., 75 grams Pu-238) is about 200 times greater than the dose rate for an eluted column. Depending on the how the ion exchange process is operated, the dose absorbed while fully loaded could dominate. For example, a column left loaded with Pu-

238 would exceed the 700 megarad limit in just over 2 days. The resin could become unstable simply by being left loaded over a weekend or for the duration of a maintenance evolution. The staff believes a control to track the time a column is loaded and the associated resin dose may be warranted.

The revised PrHA proposes surveillance of resin exposure in addition to annual resin replacement, but the PrHA and the SER do not explain how this surveillance will be performed. Furthermore, the December 2000 SER imposed what was intended to be a safety factor of two for the resin lifetime. The newly proposed controls accept a reduced safety factor. This reduction in the safety factor, combined with a potentially nonconservative estimation of resin dose, could result in an unsafe resin condition.

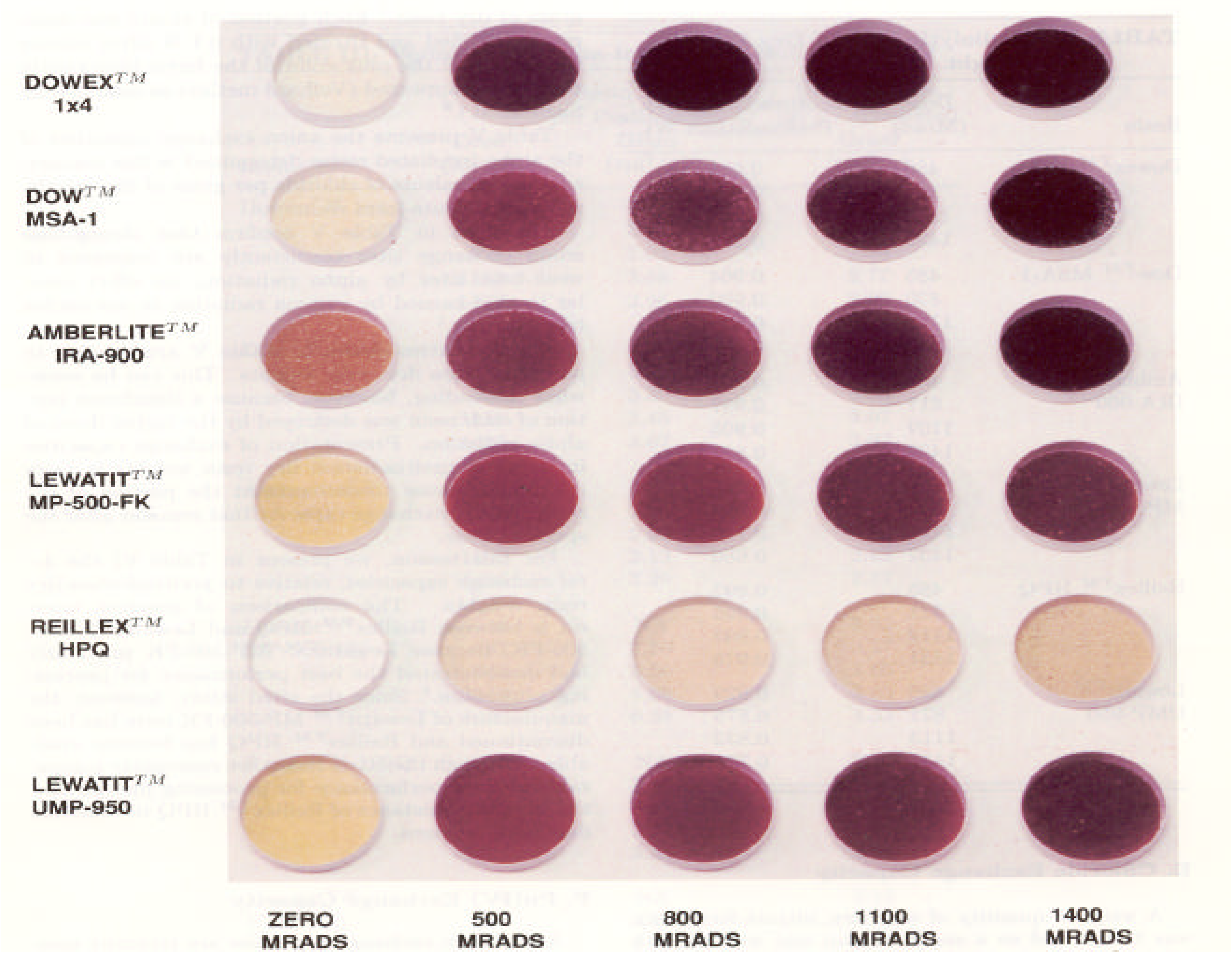


Figure 3. Resin Color as a Function of Alpha Dose (Marsh, 1991)

Resin Dryout—The newly installed safety-significant rupture disks may prevent rupture of the columns, but the columns are still susceptible to fire if the resin is allowed to dry out. LANL performed an analysis to determine the rate at which water will evaporate from the ion exchange column (Los Alamos National Laboratory, 2002c). The analysis modeled the transport of the water vapor from the column through a 5 ft vent line or 2 ft fill line, each with a 3/8 inch inner diameter. The analysis showed that, under normal operating conditions, 56 days would be necessary before 2 inches of solution above the resin would be evaporated. LANL used this finding to justify crediting a monthly visual surveillance of liquid level as the safety-significant control preventing resin dryout.

The staff believes that the implemented controls do not adequately consider off-normal conditions. For example, 2 inches of solution above the resin could evaporate within approximately 8 hours if the top of a loaded column were opened to the glovebox environment. Moreover, LANL did not evaluate leaks or improper valve alignments that could allow the entire column to drain and dry out in considerably less time. The staff believes engineered controls may be warranted to preclude inadvertent resin dryout. An example of such a control would be the implementation of a functionally classified liquid level monitor that would alarm in the TA-55 operations center.

Thermal Analysis—The May 2003 PrHA states that the resin temperature can reach a maximum of 50˚C. A thermal analysis was performed by Westinghouse Savannah River Company to determine the maximum temperatures of the ion exchange column (Laurinat and Panson-Hjevik, 1999). The analysis demonstrated that the column temperature would remain below 50˚C under normal operating conditions. However, the calculation concluded that the column could reach approximately 84˚C under abnormal conditions (see Table 1 and Figure 4). This scenario assumes that the resin has been loaded with twice the normal quantity of Pu-238 and that there is no flow of solution through the column. High column temperatures would degrade the resin, leading to further heat evolution. The column could eventually dry out or pressurize from evaporated water. Moreover, if the column heated to such a temperature, self-heating of the resin could initiate, as shown by the bomb calorimeter testing discussed below.

The PrHA indicates that the resin remains safe for temperatures below 120˚C. However, Crooks (2001) has shown that the Reillex HPQ resin may initiate self-heating at temperatures as low as 81˚C in a calorimeter. The bomb calorimeter was used to allow external heating of the resin at a rate of 1˚C per minute to determine resin stability as a function of temperature. The external heating may also simulate the increase in temperature due to radioactive decay upon loss of flow. Figures 5 and 6 show that self-heating begins at approximately 81˚C, and temperatures proceed to climb quickly. Resins tested in 8 M and 12 M nitric acid reached a maximum temperature of approximately 250˚C in 1 to 2 hours.

The staff believes the abnormal conditions that could lead to self-heating of the resin are credible. The pumps that provide solution flow through the columns are not functionally classified, and their reliability was not considered in the hazard analysis. Furthermore, although loading two batches of Pu-238 on the resin columns is not allowed in the operating procedure (Los Alamos National Laboratory, 2002d), the resin is physically capable of holding significantly more than a double batch of Pu-238. Kyser (2000) has demonstrated that the 2 liter volume of the Reillex HPQ resin to be used in the column can adsorb 234 g of

Pu. LANL has provided the Board's staff with documentation indicating that the maximum weight percent of Pu-238 in the scrap Pu feed material is 85 percent. Therefore, a column could be loaded with up to 198.9 grams of Pu-238, whereas the double-batch scenario in the thermal analysis considered only 150 grams of Pu-238. The maximum temperature of a fully loaded column has not been analyzed, but would likely be substantially greater than 84˚C if there were no flow of solution through the column.

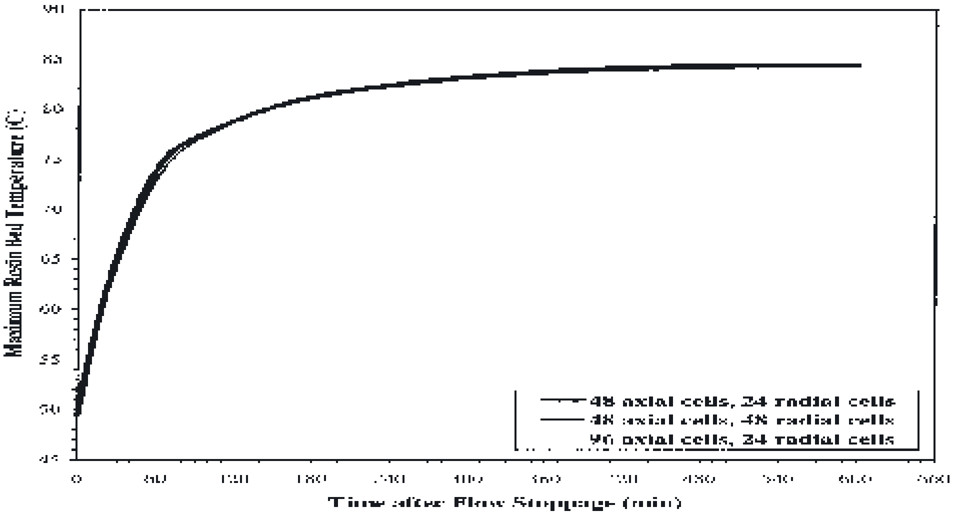
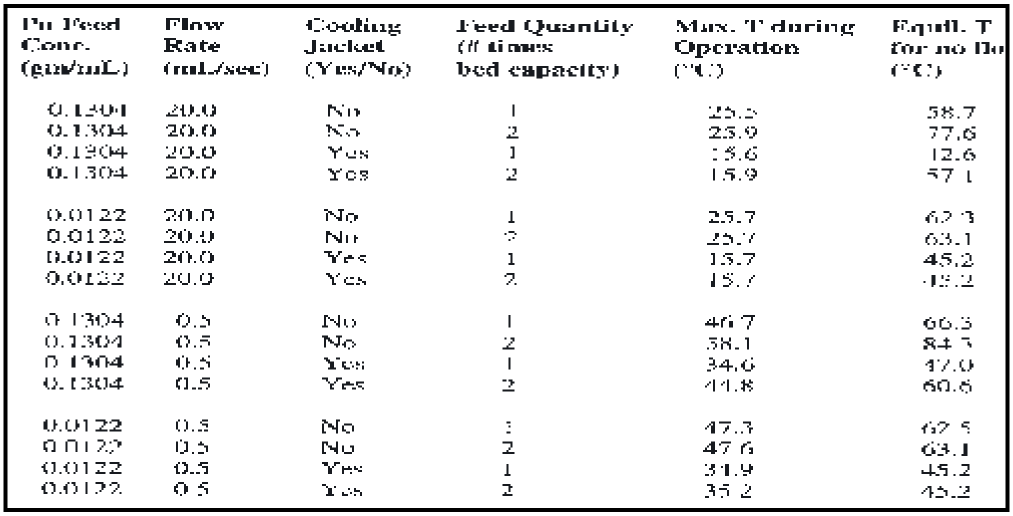


Figure 4. Maximum Resin Temperature for Loss of Flow, Maximum Concentration, and Double Loading (Laurinat, J. E. and M. E. Pansoy-Hjevik, 1999)

Table 1. Column Temperatures as a Function of Flowrate, Concentration, and Loading (Laurinat, J. E. and M. E. Pansoy-Hjevik, 1999)



Pu Feed Conc. (gm/mL)	Flow Rate (mL/sec)	Cooling Jacket (Yes/No)	Feed Quantity (# times bed capacity)	Max. T during Operation (°C)	Equil. T for no flo (°C)
0.1304	20.0	No	1	25.5	58.7
0.1304	20.0	No	2	25.9	77.6
0.1304	20.0	Yes	1	15.6	12.6
0.1304	20.0	Yes	2	15.9	57.1
0.0122	20.0	No	1	25.7	62.3
0.0122	20.0	No	2	25.7	63.1
0.0122	20.0	Yes	1	15.7	45.2
0.0122	20.0	Yes	2	15.7	45.2
0.1304	0.5	No	1	46.7	66.3
0.1304	0.5	No	2	58.1	84.3
0.1304	0.5	Yes	1	34.6	47.0
0.1304	0.5	Yes	2	44.8	60.6
0.0122	0.5	No	1	47.3	62.5
0.0122	0.5	No	2	47.6	63.1
0.0122	0.5	Yes	1	31.9	45.2
0.0122	0.5	Yes	2	35.2	45.2

Each ion exchange column is connected to an automatic elution system, classified as a defense-in-depth safety feature in the May 2003 PrHA. The system monitors the column temperature, pressure, and solution level and is controlled by a laptop computer. If any of these parameters are outside predetermined set points, the system will elute the column with 0.45 M nitric acid. The auto-elution system has an uninterruptible power supply (UPS) that allows the system to operate during loss-of-power conditions. This system is capable of preventing energetic reactions that may occur in the ion exchange columns by responding to precursor conditions (e.g., rising temperatures, low liquid level). However, neither the auto-elution system nor the UPS has been classified as safety-significant, and therefore they are not credited in the hazard analysis. As noted in the Board's April 2002 letter to NNSA, upgrading this system to allow it to be functionally classified as safety-significant would provide more certain protection against resin accidents.

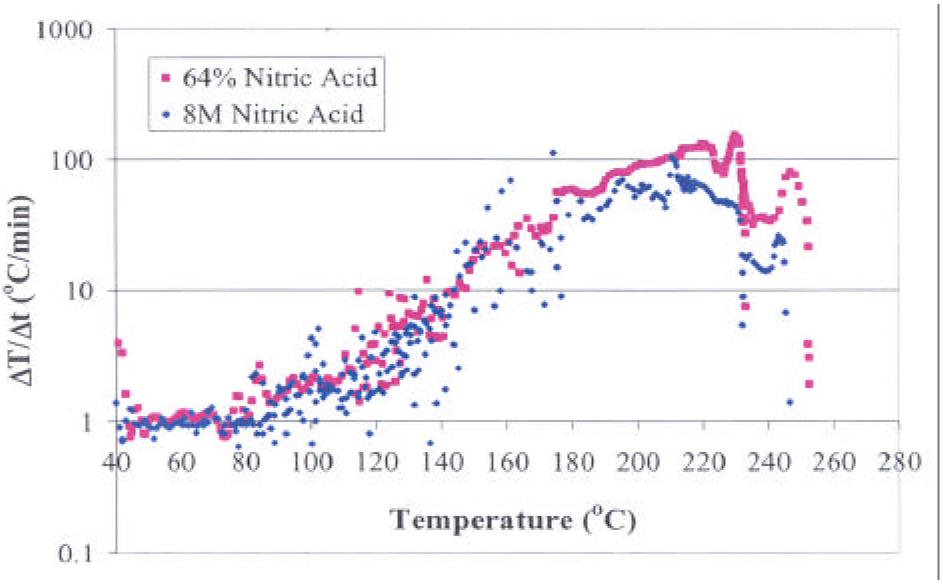


Figure 5. Rate of Thermal Increase as a Function of Resin Temperature (Crooks, W. J., 2001)

Modifications to the Resin—A literature review performed by the Board's staff revealed that the Reillex HPQ resin has been modified by its manufacturer to increase the Pu loading capacity (Kyser, 2000). This modification increases the number of "active" sites of the polymer and may decrease the stability of the resin by enhancing the degree to which it can become nitrated or degraded by radioactive decay. The stability testing that was used to develop safety limits for the resin predates the resin modification. To ensure that appropriate safety limits are implemented, it will be necessary either to impose procurement requirements to ensure that resin used in the recovery line is the same as that used to develop the PrHA, or to perform additional testing and analysis to characterize the stability of the modified resin. Another approach would be to increase the safety margin applied in the analysis and safety controls.

Oxalate Precipitation. From the ion exchange columns, the purified Pu-238 solution is pretreated, and the Pu-238 is precipitated using oxalic acid. Certain batches may be transferred directly from the dissolution process to pretreatment if ion exchange is not needed to meet product quality requirements. Part of the chemical pretreatment process involves the use of hydroxylamine nitrate (HAN) to reduce the tetravalent plutonium in solution to the trivalent state. When exposed to concentrated acid, HAN can rapidly decompose to nitrous oxide gases and pressurize storage or process vessels. In response to past accidents involving HAN, DOE published a technical report addressing the safety risks involved in the use of HAN (U.S. Department of Energy, 1998). This report presents an empirical formula for predicting the instability of HAN solutions as a function of temperature, nitric acid molarity, and iron molarity.

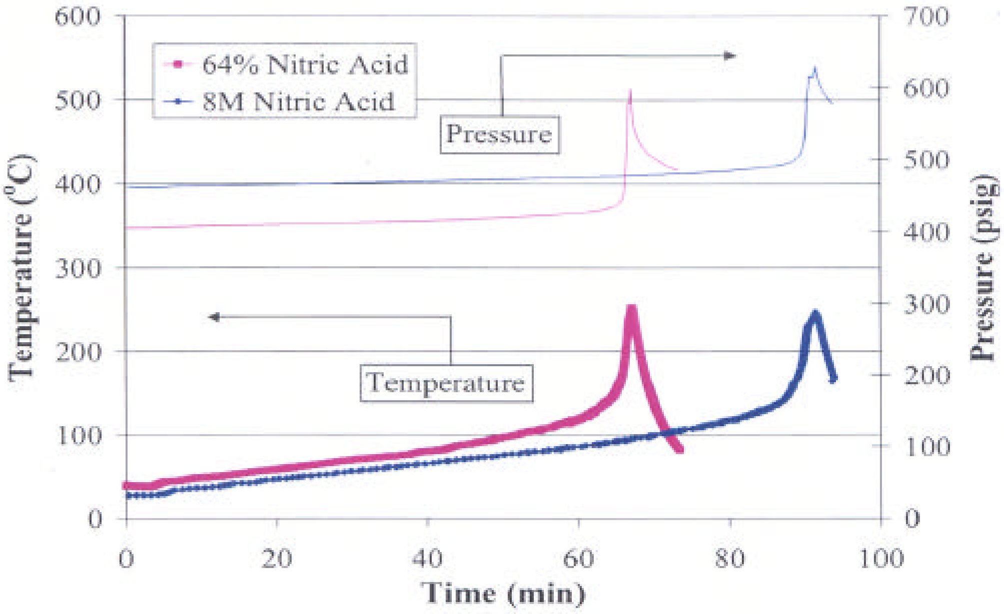


Figure 6. Maximum Resin Temperature and Pressure as a Function of Time (Crooks, W. J., 2001)

Under normal conditions, the recovery line's chemical pretreatment process is intended to operate in the stable range for HAN. However, several scenarios exist in which concentrated acid could be transferred to a vessel containing HAN. One such scenario is the direct transfer of dissolver filtrate (15.8 M nitric acid) to the pretreatment process. This scenario appears to be credible since dissolved Pu-238 scrap that does not need to be purified may be transferred from the dissolution glovebox to the precipitation pretreatment vessel. If procedures were followed properly, such a solution would be neutralized prior to the addition of HAN. However, the only preventive control is an administrative control to verify acid molarity prior to pretreatment processing. If dissolver filtrate were reacted directly with HAN, the instability index could be as high as 52, indicating that the mixture would become unstable at temperatures as low as 30˚C (see Figure 7). (This calculation assumes the filtrate contains 1500 ppm iron as stipulated in NNSA's July 1, 2002, report to the Board.)

The HAN used in the recovery line is 2.8 M. DOE's technical report suggests that HAN concentrations below 2–3 M lack the necessary energy density to generate high pressures or explosive reactions. Therefore, the 2.8 M HAN solution used by LANL appears to be in an intermediate range with respect to energy density. At a HAN concentration of 2.8 M, the heat of reaction may vaporize enough water to concentrate the HAN and thus increase its energy density to an unsafe regime.

The March 2000 PrHA did address reactivity hazards of HAN, but identified no functionally classified controls. The Board's April 2002 letter stated that LANL had yet to demonstrate how the recommendations provided in DOE's technical report on HAN had been implemented in the recovery line, as required by NNSA's December 2000 SER. The January 2003 and May 2003 PrHAs reanalyze the consequences of a HAN/nitric acid reaction and propose dual independent administrative verifications of acid concentration to prevent undesirable reactions. This control is not captured as a TSR. Given the numerous other administrative controls, it is not clear to the Board's staff that the verification of acid concentration will be implemented with a rigor commensurate with its importance to worker safety. The report of a LANL readiness review of the aqueous recovery line performed in July 2002 concluded that overuse of second-person verifications limits their effectiveness (Los Alamos National Laboratory, 2002e). The staff believes verification of acid molarity would be implemented with greater certainty if specified as a TSR.

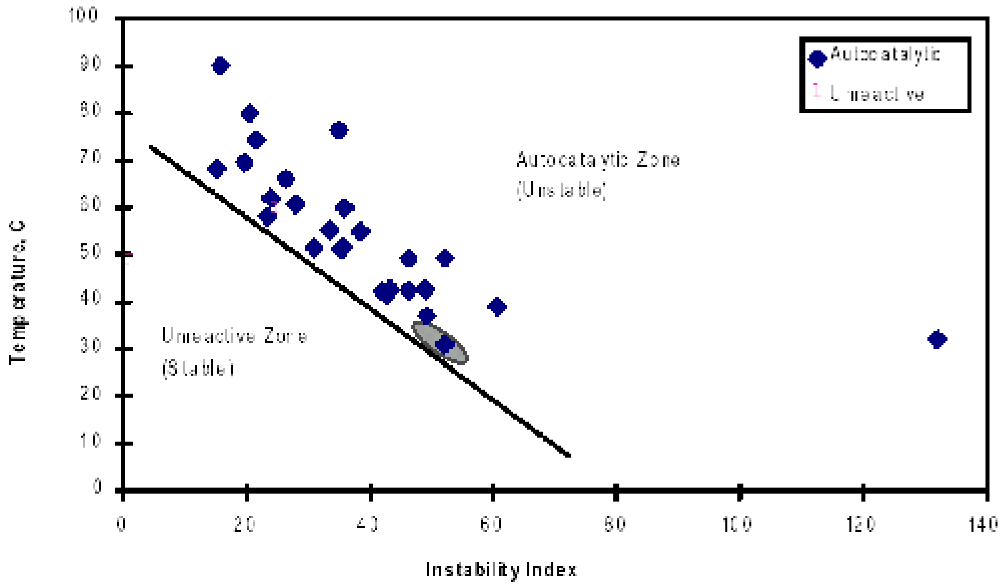


Figure 7. HAN/HNO$_3$ Instability as a Function of Temperature (U.S. Department of Energy, 1998)

Calcination and Oxygen-16 Exchange. Following oxalate precipitation, the plutonium oxalate is calcined to form plutonium oxide. Calcination involves the heating of the plutonium oxalate to a temperature of 800˚C in a furnace. After the calcination stage, the atmosphere in the furnace will be purged with oxygen-16 and argon to reduce the neutron emission rate of the oxide. The oxygen-16 reduces neutron emission rates by replacing other oxygen isotopes that have higher cross sections for alpha-n reactions. The hazard analysis and controls identified in the May 2003 PrHA for these operations appear to be thorough and appropriate.

Solution Transfers. In its April 2002 letter, the Board suggested that LANL might be better served by investigating a method for solution transfer that does not require repeated connection and disconnection of flexible transfer lines. The new PrHA contains a section that specifically addresses solution transfer methodologies and presents a qualitative comparison of the use of flexible tubing versus hard piping. This comparison states that flexible tubing offers superior operational flexibility and reduced likelihood of leakage outside the glovebox as compared with external runs of single-wall piping. In general, the comparison considers only historical hard piping in TA-55, which is single-wall pipe. Double-wall piping would, however, likely be required and would substantially reduce the likelihood of leakage outside the glovebox.

LANL estimates that several million dollars would be required for the design and implementation of hard piping in the existing gloveboxes. A significant delay in schedule would also be necessary, which may be unacceptable to the LANL customer.

Interglovebox transfers require opening the doors between gloveboxes and will result in temporary violations of the material-at-risk assumptions in the safety basis. For every door that is open, the potential material-at-risk increases by 500 g, but the resulting increase in consequences is not enough to warrant further safety controls. Despite this finding, the staff believes it would be prudent to implement preventive controls to minimize the possibility of an accident during such transfers (e.g., terminate unrelated operations).

Although the use of flexible tubing within the glovebox line does not require safety-significant controls, LANL is still obligated to ensure that operator doses are kept as low as possible. This should include such actions as limiting the service life of flexible tubing and applying good housekeeping procedures to keep connection points clean and to thoroughly clean up any drips or leaks in the gloveboxes.

The long-term production operation of the new Pu-238 recovery line would likely benefit from more robust hard pipe connections. Hard piping would eliminate the need for multiple manual connections and disconnections and the corresponding opportunities for connection errors, glove tears, and loss of solution to the glovebox. Hard piping would also minimize material-at-risk and reduce glovebox combustables.

Identification and Classification of Controls Relied Upon for Safety. The revised PrHA still lacks a methodical process for identification and implementation of functionally classified safety controls. This deficiency is highlighted by the fact that the neither the PrHA nor the TA-55 Documented Safety Analysis identifies which controls are functionally classified as safety-significant for the recovery line. Instead, LANL's forwarding memorandum for the May 2003 PrHA simply states which controls are safety-significant, safety-class, and TSR-level controls. The NNSA SER approved this control set with some modifications. If no basis for functional classification of controls is provided, the identification of a complete and effective control set cannot be assured.

Additionally, the NNSA approves treating the safety credited controls as TSRs until they are incorporated into the TA-55 TSRs at the next update. Consistent with the Board's April 2002 letter, the staff believes that the controls should be incorporated into the TSRs before startup of the recovery line.

Alarms and External Monitoring. Given the many administrative controls, it is conceivable that at some point during the life of the project, certain controls may be eliminated or overlooked. Properly implemented alarms (e.g., resin column level sensors and argon flow monitors) could provide additional assurance that certain administrative controls and procedures will be followed properly. The auto-elution system and associated computer hardware have alarm indications, but since they are not functionally classified, their reliability is not assured. Annunciation of the alarms in the TA-55 operations center could allow external monitoring of vital system parameters when the recovery line is unmanned.

The room in which the recovery line will be located does have continuous air monitors (CAMs). The CAMs alarm when airborne radioactivity levels exceed a predetermined set point. CAMs are useful for alerting occupants to adverse conditions, but should not be relied upon to protect facility workers from process upsets. The previously mentioned Type A event occurred in a room that had CAMs, but significant worker intakes still occurred.

Summary. The new PrHA is an improvement over the first submission, but some of the same weaknesses remain. These issues could have been identified and resolved much sooner if functional classification of controls had been considered earlier in the development of the process, and if LANL had performed an independent review of the safety controls before submitting the first PrHA to NNSA. This latter conclusion was also reached by the LANL readiness review team that evaluated the project in July 2002 (Los Alamos National Laboratory (2002e).

APPENDIX A

Timeline of the Board's Involvement in the Plutonium-238 Aqueous Scrap Recovery Line at LANL

December 14, 2001—Board's staff obtains authorization basis documents for the Pu-238 scrap recovery line.

December 28, 2001—Weekly report by the Board's LANL Site Representative reviews the status of the Pu-238 scrap recovery line.

March 6–7, 2002—Board's staff conducts an on-site review of the Pu-238 scrap recovery line.

March 28, 2002—Board's staff holds a follow-up conference call with Nuclear Materials Technology (NMT-9) staff from LANL. The staff discussed concerns related to the implementation of Technical Safety Requirements, use of flexible tubing/temporary connections for solution transfers, hydrogen generation in the dissolver, and resin accident scenarios.

April 23, 2002—Board issues a letter forwarding an issue paper to NNSA. The Board's letter describes the deficiencies in the Pu-238 scrap recovery line and requests a written response within 60 days.

July 1, 2002—NNSA issues a report in response to the Board's reporting requirement.

July 10–22, 2002—LANL conducts a readiness assessment of the Pu-238 scrap recovery line.

July 15, 2002—Board members discuss safety issues related to the Pu-238 scrap recovery line with NNSA.

July 17, 2002—Board members discuss safety issues related to the Pu-238 scrap recovery line with senior NNSA and laboratory personnel during a visit to LANL.

July 29, 2002—Board's staff conducts a conference call with LANL managers and NE-50 to discuss issues not addressed adequately in NNSA's response to the Board's letter.

August 7, 2002—Board's staff conducts a video conference with representatives of LANL and NNSA's Los Alamos Site Office to further discuss safety issues.

August 9, 2002—Weekly report by the Board's LANL Site Representative indicates LANL is considering the addition of engineered controls to address issues identified by the Board.

September 23, 2002—Board's staff visiting LANL meets with NMT-9 and LANL managers to discuss issues related to the scrap recovery line.

October 9, 2002—Board's staff provides informal questions on unresolved issues to NNSA's Los Alamos Site Office.

January 15, 2003—LANL submits a revised PrHA to NNSA.

March 18, 2003—Board's staff provides questions on the revised PrHA to NNSA's Los Alamos Site Office.

May 28, 2003—LANL submits a revised PrHA to NNSA.

May 29, 2003—NNSA approves the latest PrHA.

APPENDIX B

Open Questions on LANL's Plutonium-238 Scrap Recovery Operations

October 9, 2002

1. Process Hazard Analysis

A. What is the status of the PrHA submittal? How broad is the scope of the revised PrHA? Has the revised PrHA uncovered any hazards that were not analyzed previously?

2. Ion Exchange Column Resin Dose

A. What is the procedure/process used to estimate and control the resin dose? What margin will this provide relative to a 700 Mrad alpha dose? What will be the proposed TSR controls?

3. Ion Exchange Column Resin Dryout

A. What is the status of the proposed reservoir design? What are the proposed TSR controls?

B. Is the resin dryout calculation sensitive to operational perturbations? For example, will the liquid evaporation rate be different if the pressure relief valve lifts and remains open? Does the proposed surveillance frequency adequately bound off-normal scenarios such as this one?

C. What are the pros and cons of including an active component (e.g., solenoid valve) between the ion exchange column and the proposed reservoir that fails open upon loss of power, thereby changing the range of scenarios that the reservoir addresses?

D. What are the pros and cons of reservoir level indication?

E. In addition to the proposed quarterly TSR surveillance, is there a need for operations personnel to check the liquid level at a set frequency during operation of the ion exchange column? If so, how should the frequency be determined?

Some of the following questions stem from concerns that the dryout calculations for the ion exchange column resin do not address leaks, column damage, or inadvertent draining.

F. What scenarios, other than evaporation, have been identified as potentially leading to a resin dryout condition?

G. How has LANL engineered the ion exchange column/piping to minimize the potential for leakage, leading to dryout, and to increase the potential that any leakage would be detected and corrected prior to the resin being uncovered?

H. Has LANL considered a safety-significant go/no-go level indicator at the top of the column with an alarm system that annunciates locally and in the Operations Center?

I. What are the advantages of having the existing column low level alarm that triggers auto-elution annunciate locally and in the Operations Center?

J. How would the operator be expected to react to an evacuation announcement while a local alarm (e.g., a low level alarm) required his/her attention?

K. What are the pros and cons of a glovebox low point leak detector with local and/or remote alarm?

4. Dissolver Hydrogen Deflagration

A. What is the status of the design of the proposed vent/interlock?

B. When the argon sparge is lost, what is the time to LFL? Considering that LFL is reduced in Ar/air/H_2 atmosphere, what are the pertinent assumptions? What is the maximum expected H_2 concentration in the head space with the vent design currently under consideration? What will be the proposed TSR controls?

C. Does the proposed design include an alarm that activates when power/argon sparge is lost and the interlock opens the vent? If so, what are the pros and cons of having the alarm annunciate locally and in the operations center?

D. How would the operator be expected to react to an evacuation announcement while a local alarm (e.g., loss of argon purge) required his/her attention?

E. Has the Pu dissolution vessel stress analysis been reviewed by LANL’s Pressure Vessel Safety Committee? If so, what were the results of this review?

5. Acid/HAN Reactions

6. Solution Transfer Strategy

A. Does the 2.8 M HAN contain sufficient energy to cause adverse consequences in an accident?

B. What would happen if concentrated acid (e.g., > 7 M) came into contact with the 2.8 M HAN or ion exchange resin?

C. Will the proposed administrative controls with independent verification of acid concentrations have TSR implications, or are they strictly process control steps?

A. What are the impacts on the accident analyses and control sets if the door between individual gloveboxes is open? If all doors are open?

B. How would the operator be expected to react to an evacuation alarm if the doors between the gloveboxes are open?

C. What would be required to install permanent solution transfer lines from the top of one glovebox to the next, thereby eliminating the need to open glovebox doors for solution transfers? What are the pros and cons of this option?

D. What are the pros and cons of hard piping to address those solution transfers that involve higher source-term or acid concentration and are more frequent than others?

E. It appears that some operations will require the flexible tubing to pass through the doors of multiple gloveboxes. What is the longest length of flexible tubing, and how many doors will it pass through? How long will these glovebox doors stay open? What will be the frequency of these operations? What will be the proposed TSR controls?

F. What are the personal protective equipment (PPE) requirements for operators during solution transfers?

APPENDIX C

Questions on the LANL Plutonium-238 Scrap Recovery Line
March 18, 2003

General

- How were functional classifications of safety controls determined? What are the safety system boundaries? Where is it demonstrated that the list of safety systems and controls in the memorandum forwarding LANL PrHA represents a complete set?

- What alarms should be considered to indicate loss of a safety system's operability? Besides local alarms, should operations center alarms be considered?

- Considering the suite of facility alarms, what would be the required operator actions and the impact of an unrelated facility alarm, such as an evacuation alarm, during operations on the scrap recovery line?

Dissolver

- Why are the systems that make up the argon purge and interlock controls not functionally classified? How does TSR surveillance on a non-safety-credited component ensure the necessary reliability during operation?

- To what extent is the analysis of dissolver deflagration reliant on the assumption that there is no ignition source?

- Should purging of the dissolver continue during and after cooldown, given that the filtrate will continue to generate hydrogen after the power has been turned off? Should head-space purge also be considered for vessels storing solutions containing concentrated Pu-238 solution?

- Has LANL's Pressure Vessel Safety Committee reviewed the deflagration analysis for the dissolver, particularly given that this static analysis for a dynamic phenomenon predicts von Mises stresses from deflagration within 1–2 percent of the vessel's capacity?

- Is the conservatism in the calculation of time to reach the LFL (e.g., the assumed nitric acid molarity) appropriate ?

Ion Exchange

- How will cumulative resin dose be tracked and controlled? Does 700 Mrad alpha dose as a limit ensure adequate margin?

- Should the calculation of resin dose include the contribution when the column is fully loaded with Pu-238 as well as loaded with residual? Should a TSR control be considered to protect assumptions being made about the residence time of Pu-238 on each column?

- Has LANL evaluated the impact on resin stability of process modifications made by Reilly Industries in recent years that have increased the anion exchange site yield (ref: WSRC-TR-2000-00372)?

- What procurement requirements are invoked to ensure that the resin has the expected chemical and radiation stability?

- What does the operator look for to establish that a resin column has not dried out? Should this be done more frequently when a column is operational (i.e., loaded)?

- Should the impact of off-normal conditions or hardware defects also be considered in establishing the periodicity of liquid-level surveillance (e.g., column or tubing cracks and leaks, valve leaks, valve misconfiguration), given that the resin dryout calculation relies heavily on limited mass flow down long tubular pathways?

- Should more robust level detection and/or glovebox low-point leak detection and alarm be considered?

- Should features that minimize the probability of a column leak be functionally classified?

Chemical Safety

- Should explicit TSR controls be specified to prevent accidents involving contact of either HAN or ion exchange resin with concentrated acid? In procedures, is there a mechanism that would flag for the operator such explicitly defined TSR-level safety controls?

Material Transfers (Solids and Solutions)

- How is the material-at-risk (MAR) assumption protected during interglovebox transfers?

- How is glovebox Pu-238 holdup from legacy spills tracked and included in the MAR controls?

- Should additional controls be invoked or unrelated operations secured to minimize the possibility of an accident during interglovebox transfers?

- Should the lifetime of flexible tubing used for solution transfers be administratively controlled?

APPENDIX D

Plutonium-238 Aqueous Processing at Los Alamos National Laboratory

Hazard	Initial PrHA	Current Proposal/Actions	Staff Position
Comminution–ball mill jar ejection	Mitigative controls–container integrity	Safety significant steel lid covering ball mill	Acceptable
Dissolution–deflagration due to hydrogen generation	Did not analyze	9.7 rem (unmitigated), 0.15 rem (mitigated), off-site	
	No controls	Safety significant dissolver vessel	Analysis questionable; deflagrations should be prevented
		Argon purge during dissolution and direct vent during cooldown	Controls also needed for storage vessels
		TSR administrative surveillance on non-safety equipment	Does not ensure reliability of control during operation
Ion exchange–column pressurization or fire	Did not analyze	156 rem (unmitigated), 2.5 rem (mitigated), off-site	
	No resin dose tracking	Replace resin annually, based on estimated dose to resin from residual plutonium on the column	Dose calculation is non-conservative. Efficiency of resin has been changed and may affect resin stability
		Surveillance of the dose to resin and resin discoloration	Dose needs to be rigorously tracked, color is not an effective indicator of dose
	Limited controls to prevent dryout and pressurization (noncredited level sensors)	Safety-significant rupture disks installed	Acceptable
		No leak detection; hard piping at IX column	Dryout analysis discounts leaks and inadvertent draining Leak detection may be warranted

Hazard	Initial PrHA	Current Proposal/Actions	Staff Position
Energetic chemical reactions	Did not implement recommendations of DOE technical report	2.7 rem (unmitigated), 0.04 rem (mitigated), off-site	
	Administrative controls for acid concentrations	Same administrative controls	Chemical safety controls should be TSR
Material transfers–contamination and Materials-at-Risk	Flexible tubing	Flexible tubing; installed hard piping at interface with ion exchange column	Limit tube life Limit unrelated operations during transfers
TSRs	Did not plan to incorporate before startup	SC/SS/Admin controls "shall all be regarded as TSR level controls", incorporate in Safety Analysis Report and TSRs during next update	Need to be incorporated into authorization basis prior to start up
General	Identified safety-credited systems	Unclear how safety-credited systems were derived	Not clear that a complete suite of functionally classified controls has been identified
	No external alarms	No change	Alarms may help monitor and prevent dangerous conditions

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