Decontamination of Reactor Systems and Contaminated Components for Disposal or Refurbishment

Developments and Experience with the EPRI DFD Chemical Decontamination Process

Technical Report

Decontamination of Reactor Systems and Contaminated Components for Disposal or Refurbishment

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REPORT SUMMARY

This report describes recent developments using the EPRI DFD (Decontamination for Decommissioning) dilute chemical decontamination process on reactor coolant systems and components. The nuclear industry has now used the process for a variety of applications, from cleaning small components to full system decontamination of retired plants. The report documents substantial savings in radiation exposures during decommissioning work and discusses the benefits of using the process to reduce radioactive waste disposal requirements.

Background
Utilities have used chemical decontamination processes for many years to reduce radiation doses at operating nuclear plants. EPRI developed the DFD Process specifically for decontamination of reactor systems and plant components at the end of life. In this application, the primary purpose is to aid waste management rather than dose reduction. Therefore, greater decontamination effectiveness is required to allow recycling of the treated materials.

Previous EPRI reports (TR-106386, TR-107707, TR-109036 and TR-112877) describe the EPRI DFD process. This report updates and largely supercedes the previous EPRI reports by presenting additional data on materials corrosion and waste management. It also provides information on the subsequent benefits of the EPRI DFD applications at Big Rock Point (BWR) and Maine Yankee (PWR). The report also includes the results of work to prepare for use of the process in new applications, particularly cleaning various metallic waste components in the USDOE complex.

Objective
To review application of the EPRI DFD process for decontaminating reactor coolant systems and retired contaminated components for material release or recycling.

Approach
This report describes work carried out to increase the range of components that utilities could treat with the process. The project team modified the process chemistry to adapt the technology for full system decontamination of zircalloy pressure tube reactors. They also reviewed utility data on the benefits achieved by decontamination during the subsequent decommissioning work at the two plants treated by the process in 1998.

Results
The EPRI DFD Process involves circulating dilute fluoroboric acid and potassium permanganate though the system or component requiring decontamination. Addition of oxalic acid removes manganese dioxide, and is followed by permanganate to destroy any excess oxalate. Cation

exchange removes dissolved metal ions. The decontamination team repeats the cycle until the metal surfaces are clean. Mixed bed ion exchange achieves final clean up of the process solution

Since the previous reports, a considerable amount of decommissioning work has taken place at the two plants, Big Rock Point and Maine Yankee, where technical staff applied the EPRI DFD Process to the full plant systems. The plants report substantial savings of radiation dose, time, effort, and therefore cost, due to application of the process before decommissioning. Because of its environmental and dose saving benefits, stakeholders and regulators view the applications of the process positively.

New ex-situ applications of the EPRI DFD Process continue to be developed. The project team has demonstrated that the process is suitable for cleaning aluminum USDOE enrichment plant compressor blades and steam generator material release. It is also cost effective in allowing refurbishment and recycling of nuclear plant pumps.

EPRI Perspective

Previous EPRI reports documented the benefits of the EPRI DFD Process in reducing radiation exposures during decommissioning work. This report substantiates these benefits and provides more recent results from two nuclear power plants. In the future, the main impact of the process will be in the area of radioactive waste processing. Radioactive waste disposal remains a major issue, for both nuclear power plants and USDOE facilities. This issue has a negative effect on public perception of nuclear power, which becomes more important as the industry moves towards ordering new nuclear power plants. The EPRI DFD Process achieves complete cleaning of contaminated metals, which permits beneficial reuse of the material rather than necessitating burial in a disposal site. This is an environmentally sound policy, as organizations can recover costly metals, thereby avoiding new material production, and preventing disposal sites from filling with large volumes of retired components. The industry can use the recovered material economically in controlled applications in nuclear facilities.

The DFD process in its existing form collects the radioactive contamination on ion exchange resin, which has a volume of less than 10% of the original material. However, disposal of IX resin is expensive, and suitable disposal sites are not always available. A forthcoming development of the DFD process, already demonstrated in laboratory tests, will significantly reduce the volume of residual waste, and eliminate the need to dispose of ion exchange resin. EPRI will report on this development early in 2002.

Key Words

Decommissioning
Low level waste disposal
Decontamination

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INTRODUCTION

Chemical Decontamination of sub-systems of operational LWR plants has now become relatively common, and full system decontamination has now been performed at a number of plants. This decontamination involves exposing parts of the reactor circuit to chemical solutions which dissolve the radioactive deposits which have accumulated on the process equipment including piping. The spent decontamination solutions are then treated by ion exchange to retain all the chemical and radioactive burden of the decontamination solution on the resin, while clean water is returned to the system. An example of such a process is the LOMI process [1].

In decontamination processes used at operating plants the purpose is to remove the radioactive deposit to reduce the radiation exposure of plant workers. Because the processes are applied to items of plant which are returned to service, they are designed to avoid any damage to the materials exposed to the process. Such damage could occur as a result of corrosion during application of the process, or as a result of exposure of plant items to the operating conditions of the nuclear plant subsequent to the decontamination. The restriction of avoiding damage has resulted in processes which do not attack base metal, but that operate by dissolving the overlying layer of corrosion product metal oxides. Because of this the effectiveness of such processes is limited – typically 80-95% of the radioactivity is removed – but this is nevertheless sufficient for the purpose of reducing radiation dose to workers.

The effectiveness of such processes is not, however, sufficient for the purpose of removing all the radioactivity from the surfaces, thereby allowing the items to be treated as non-radioactive waste. In order to achieve this it is necessary to remove a thin layer of base metal, so as to release radioactivity trapped in fissures in the metal (occurring, for example, as a result of mild intergranular attack of the metal surface). For decommissioning the restriction concerning plant damage is not so severe, because the plant items are not required for further operational duty. The only requirements with regard to damage are that the plant items must maintain their integrity against leakage during the operation of the process and must remain structurally sound. Removal of a thin layer of base metal is consistent with these requirements, although if too much metal is removed there will be a problem concerning the amount of radioactive waste generated. Minimization of secondary radioactive waste is an extremely important objective in decontamination for decommissioning, since the purpose of applying the process is usually to achieve overall waste volume reduction.

Recognizing that existing technology was not ideal for retired components and plant systems, EPRI initiated work with Bradtec in 1995 to develop a new process. The intention was to combine the operational advantages of dilute decontamination processes used on operating plant (such as LOMI) with the high decontamination factors achievable with concentrated, aggressive processes. Such processes result in radioactive waste that presents difficult management challenges.

The result called EPRI DFD (decontamination for decommissioning) is a dilute chemical decontamination process for unrestricted release of stainless steel and alloy 600 components. The process can also be applied to other metals. Contamination levels can be reduced to background, with decontamination factors (DFs) exceeding 1000. The process uses standard equipment and conventional ion exchange technology for waste processing.

The EPRI DFD Process is designed to be as similar as possible to existing decontamination processes which are routinely used on subsystems of operating nuclear plants. In this way the new process can utilize and build on the extensive experience which has already been gained with the latter type of process. The process relies upon circulation of very dilute chemicals to remove progressively a thin layer of base metal to release radioactivity trapped in fissures. During application the solution is treated by ion exchange to recover released radioactivity and metals. After completion the solution is finally purified by ion exchange to leave the system full of clean water. The final waste form is conventional ion exchange, though further processing could be performed to reduce volumes and convert to different types of waste form if desired.

The initial development work on EPRI DFD was completed in 1996; the process has been patented and licensed to qualified vendors. These licenses have undertaken a number of projects relating to nuclear power plants, including treatment of heat exchangers, shroud head bolts, control rod drives and pumps. Much of the treated material has been cleaned to clearance levels allowing unrestricted recycle. The process has also been applied to full system or full loop decontamination of retired plants – e.g. Big Rock Point BWR, Maine Yankee PWR and Trojan. Successful decontamination at these plants has reduced costs, reduced radiation exposure and opened new options for the management of redundant plant components (e.g. unrestricted release and recycle). This experience with the EPRI DFD process has also confirmed its potential as a flexible means of achieving a wide variety of decontamination tasks in the management of retired components and facilities.

Following the initial success of applying the process to retired nuclear plants as described above, the long term development objective is to make the process technically suitable and economically viable for carrying out decontamination of as many different types of metal system and components as possible. The world nuclear industry has a very wide variety of such systems and components surface-contaminated with radioactivity. In the absence of a process such as EPRI DFD all this material would ultimately need to be disposed of as radioactive waste. If EPRI DFD can be used to treat these items there is the possibility that a significant proportion of the material could ultimately be recycled in a non-radioactive condition, which would have very significant economic and environmental benefits. For this to be a practical option both regulators and the public must have complete confidence in material being properly cleaned before it is released for recycle. Concerns of this nature are at the moment severely restricting the commercial application of this type of strategy. However, there is no doubt that if confidence can be established in this type of operation, it would have economic and environmental benefits. Development of and experience with the EPRI DFD process can provide assurance that the required standards of cleanliness for recycle can routinely and consistently be met, and that the volume reduction achieved by the process constitutes a significant economic and environmental gain.

Work in support of the above objectives has been taking place and is described in this report. For example, the process has been adapted for use with Zircaloy pressure tube reactors. In this case there is a challenge because of the aggressiveness of fluoroboric acid towards the Zircaloy material. It has been possible to produce a process variant which can be used for this particular type of application, and this work is described in the report. Also, the USDOE complex is probably the country's largest source of contaminated metal waste. Many of the potential applications of EPRI DFD to USDOE materials involve cleaning metals such as aluminum, nickel and carbon steel rather than the stainless steel and alloy 600 traditionally encountered in the commercial nuclear industry. Also contaminating radionuclides encountered (such as Technetium-99) are often different from the commercial industry. Adaptation of the process to solve a number of USDOE problems is also described in this report. Finally, the reduction of secondary waste volume is essential to gain the maximum economic value from the process. A forthcoming development of the EPRI DFD process, which has already been demonstrated in laboratory tests, will significantly reduce the volume of residual waste, and eliminate the need to dispose of ion exchange resin. Research on this is underway, and will be reported early in 2002.

This report updates earlier EPRI reports issued [2, 3, 4]. It discusses the chemistry of the process, reports on the large scale projects undertaken with it, and provides information on the radiological and economic benefits achieved through these applications. The report describes the adaptation of the process for use with Zircaloy pressure tube reactors, and similar work relating to a number of potential USDOE applications. Some of this information is also available in technical papers [5, 6, 7].

2

PROCESS CHEMISTRY

General Theoretical Considerations

The overall objective of the EPRI DFD Process is to achieve conditions in which base metals are dissolved slowly and uniformly (together with their overlying contaminated oxide film) while at the same time the dissolved radioactivity and metals are removed on ion exchange resin.

The normal mechanism for cleaning metals is to employ acid dissolution by hydrogen (H^+) ions. The overall reaction is:

$$x(\text{Metal}) + y\,\left(H^{+}{}_{x} - \text{Anion}^{x-}\right) \text{-->} \text{Metal}^{y+}{}_{x}\left(\text{Anion}^{x-}\right)_{y} + \left(y^{*}x/2\right)H_2 \qquad \text{(Eq. 2-1)}$$

In many decontamination applications where avoidance of base metal corrosion is required, a relatively high pH is used coupled with the presence of chelants to keep dissolved metals in solution. These applications often take advantage of sophisticated chemical mechanisms such as reductive dissolution. In the case of decontamination for decommissioning the strategy is simply to use as low a pH as possible, to encourage uniform corrosion of the base metal. Chelants are then not necessary (an advantage from waste management considerations). Although dissolution of base metal can be retarded by the formation of protective films on the surface of the metals, the particular acid used and the conditions of application can be chosen to minimize this.

A pH of about 2 is the minimum which can be used for an acid cleaning solution in decontamination of a large reactor system for three reasons:

- Even if the acid used is a “strong” (i.e. completely dissociated) acid, a pH of two requires the presence of 10 millimoles per liter of the acid. Much more than this concentration would require large quantities of chemical reagent. Any residual chemicals not converted to the gas phase will end up as part of the radioactive waste resulting from the process. It is paramount to minimize chemical usage.
- There is an absolute requirement to ensure the structural integrity of all the many different types of components wetted by the decontamination solution in a large system. Some minor materials are capable of corroding extremely rapidly in concentrated acid, so highly acidic pH’s should be avoided.
- Continuous clean-up of the solution by ion exchange removes the dissolved metals and radioactivity and regenerates the acid reagent. If concentrated acid is used in the formulation, the continuous circulation through the ion exchange column will drive the metals through the ion exchange resin leading to low capacity of the resin and hence excessive volumes of radioactive waste.

Many steels typically present in reactor systems are resistant to dissolution by typical acids at pH 2, even at the maximum temperature practically achievable in a non-pressurized system (ca 200 °F). The desirable properties for an acid used in the metal dissolution system are that it should be a strong acid, the acid anion should have a minimal tendency to complex metals. Complexation can cause binding of the anion to the metal surface with attendant problems in the metal dissolution mechanism such as localized attack. Also, the metal salts of the anion should be soluble in the prevailing pH of the solution. Excluding exotic chemicals (which would be costly for routine application in a decontamination process) the most superior acid according to these criteria is fluoroboric acid (HBF_4). However, it is not greatly superior to nitric acid on these criteria alone, and nitric acid is demonstrably unable to dissolve relevant metals at pH 2.

The benefits of fluoroboric acid as a decontaminant in concentrated small scale applications were first reported some years ago by Hanulik [8, 9]. This work demonstrated empirically that fluoroboric acid was very effective as a dissolvant for many metals typical in the nuclear industry. Although the mechanism was not discussed, the reagent was shown (as was well known) to dissolve silica based materials. This may play some role in its surprising efficiency (minor silica impurities within the steel structure could form protective layers which prevent or slow dissolution by other acids). Also it is known that free fluoride enhances the nitric acid dissolution of stainless steels. It is possible that free fluoride in equilibrium with the fluoroborate anion plays some role in enhancing the dissolution. This previous work also showed that, particularly where lead metal was concerned, the dissolution was enhanced by presence of an oxidizing agent We have found that use of fluoroboric acid is particularly useful when decontaminating Inconel alloys–conventional decontamination formulations have limited effectiveness on this alloy.

The concentrated fluoroboric acid process was developed to a sophisticated degree, including such features as manufacturing the acid *in situ* by reaction of free fluoride with boric acid present at the nuclear plant site and novel techniques for waste management including the recovery of the fluoroboric acid from waste solutions. The focus of all this work, however, was a "once-through" system in which the workpiece was contacted with the solution followed by the solution being treated for waste management when ion exchange purification was being employed.

This "once-through" concentrated methodology is acceptable for small components, but is not convenient for large scale systems. It is often thought that concentrated reagents are necessary in order to give adequate dissolution rates, but this question is often masked by the requirement of such systems to have enough acid capacity to complete the decontamination. The solution needs to be concentrated for the latter reason, which is why the validity or otherwise of the former reason is never investigated. In the early history of "oxide dissolving" operational reactor decontamination processes, highly concentrated solutions such as "APAC" (alkaline permanganate followed by acid citrate) were applied to reactor circuits to dissolve deposits, and when the decontamination was complete the solutions were removed and waste-managed. In the late 1970's workers in Canada and elsewhere demonstrated that the established concentrated dissolution chemistries could be adapted for use with dilute recirculating ion exchange systems, in which the required reagent capacity was supplied by continuous ion exchange regeneration. They then showed that with the aid of certain technical improvements the dilute solution could still be made to yield an oxide dissolution rate similar to the original concentrated process.

The EPRI DFD Process represents an exactly parallel development for fluoroboric acid metal dissolving chemistry to that described in the previous paragraph for oxide dissolving chemistries.

The Role of Oxidation

Early in the development program of the EPRI DFD Process it was found that, particularly for 300-series stainless steels, dissolution in dilute fluoroboric acid would not take place. It was also found that increasing the oxidation potential of the solution would then allow dissolution to take place. It is known that these steels are protected by a rapidly formed chromium rich film, in which the chromium is present as Cr (III). It was therefore suggested that when the oxidation potential of the solution is raised this film would not be stable, (chromium is then stable as soluble chromium (VI)). Accordingly the EPRI DFD Process employs an oxidizing reagent where necessary to allow dissolution of steels. Ozone can be employed, but it was found that although ozone can raise the bulk oxidation potential of solution to the required degree, nevertheless the oxidation potential at the steel surface (where it matters) can in certain circumstances be depressed. It was also reported to us that decontamination vendors consider the employment of ozone generators inconvenient. Potassium permanganate is a familiar oxidant employed in decontamination processes, and is very effective as the oxidant for this process. Quite small concentrations are employed and indeed 100 ppm or less is effective. Stainless steel corrosion rates increase linearly with permanganate concentration up to about 100 ppm, but higher concentrations give little further benefit.

Application in Cycles

The well established methodology of applying permanganate and then periodically removing the product manganese dioxide with an oxalate rinse has proved practical. Ion exchange can be discontinued during the permanganate phase to avoid oxidative degradation of the ion exchange resins.

When manganese dioxide is dissolved by addition of oxalic acid, one product is manganese (II) ions:

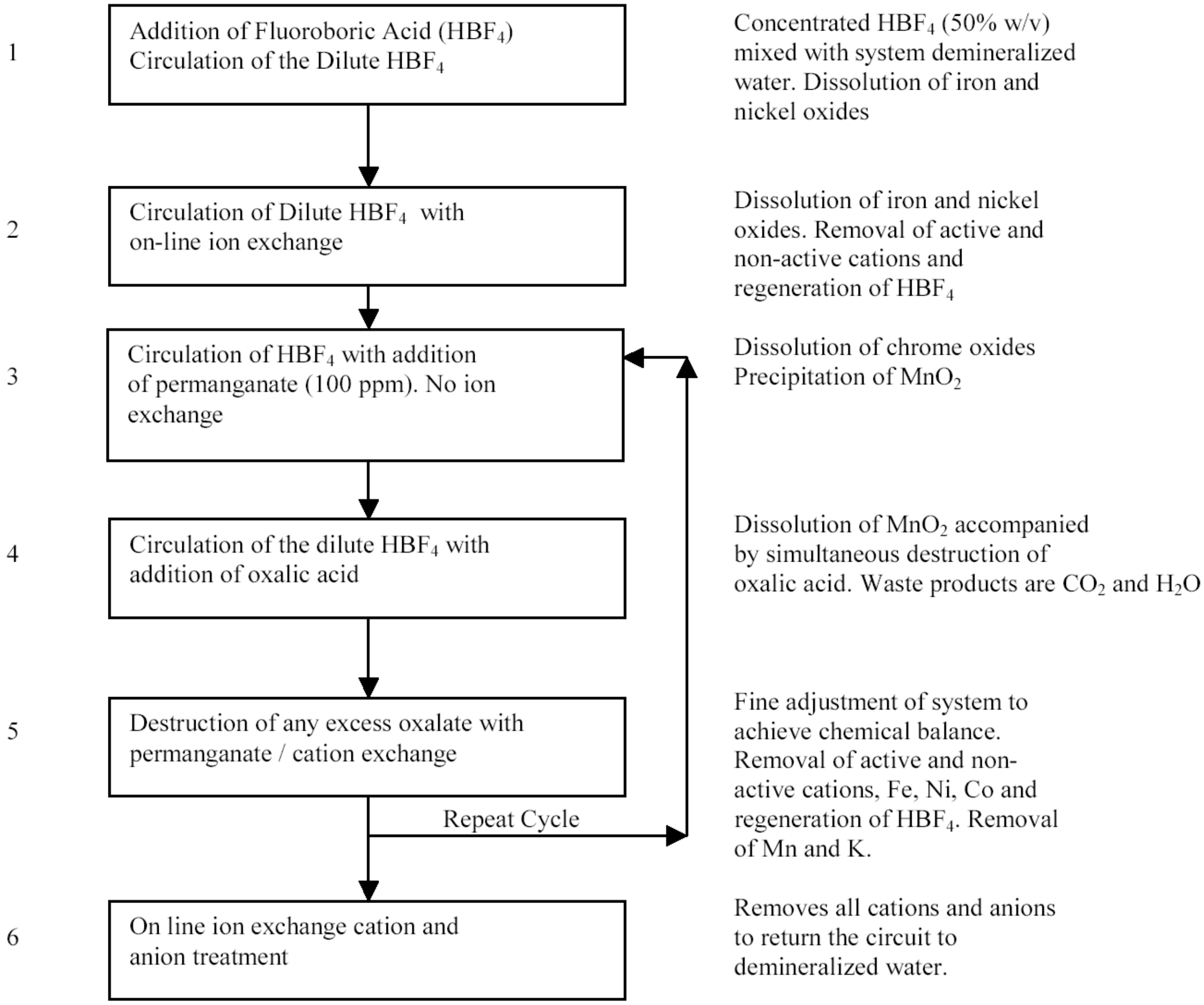
$$MnO_2 + (COOH)_2 + 2H^+ \text{ - -- > } Mn^{2+} + 2H_2O + 2CO_2 \qquad \text{(Eq. 2-2)}$$

This manganese (II) must be removed by ion exchange before the addition of further potassium permanganate to avoid the reaction of manganese (II) with manganese (VII) to produce further manganese dioxide

$$2MnO_4^- + 3Mn^{2+} + 2H_2O \text{ - -- > } 5MnO_2 + 4H^+ \qquad \text{(Eq. 2-3)}$$

When potassium permanganate is used as the oxidant in the process it is therefore applied in “cycles”. Initially the potassium permanganate is added to achieve approximately 100 ppm concentration (more can be added to make up for decomposition). When all the permanganate has decayed to manganese dioxide, oxalic acid is added to convert the manganese dioxide to manganous ions (see Eq. 2-2 above). The manganous ions are then removed by ion exchange during what is called the "transition phase" before adding more potassium permanganate to commence the next cycle. The potassium added with the permanganate is also removed during the ion exchange.

The overall process is shown in Figure 2-1



1 Addition of Fluoroboric Acid (HBF_4) Circulation of the Dilute HBF_4

Concentrated HBF_4 (50% w/v) mixed with system demineralized water. Dissolution of iron and nickel oxides

2 Circulation of Dilute HBF_4 with on-line ion exchange

Dissolution of iron and nickel oxides. Removal of active and non-active cations and regeneration of HBF_4

3 Circulation of HBF_4 with addition of permanganate (100 ppm). No ion exchange

Dissolution of chrome oxides Precipitation of MnO_2

4 Circulation of the dilute HBF_4 with addition of oxalic acid

Dissolution of MnO_2 accompanied by simultaneous destruction of oxalic acid. Waste products are CO_2 and H_2O

5 Destruction of any excess oxalate with permanganate / cation exchange

Fine adjustment of system to achieve chemical balance. Removal of active and non-active cations, Fe, Ni, Co and regeneration of HBF_4. Removal of Mn and K.

6 On line ion exchange cation and anion treatment

Removes all cations and anions to return the circuit to demineralized water.

Figure 2-1
Schematic of the EPRI DFD Process

The Oxalate "Spike"

When oxalic acid is added to the system during the decontamination, the amount added is usually just sufficient to dissolve the manganese dioxide according to Eq. 2-2 above. If excess oxalic acid is added to the system a residual concentration of oxalate will be present after the manganese dioxide has all dissolved. It is preferable to avoid ion-exchanging the solution with an excess of oxalate present, because the oxalate can form complexes with the metals which can make them more weakly held on the ion exchange column or even cause them to be eluted from the column.

When the decontamination solution is circulated without ion exchange and with an excess of oxalate, this is called an “oxalate spike”. It has been found that an oxalate spike is effective at improving decontamination performance, particularly in the final cycles of application. For example decontamination of UK PWR steam generator tube shows an acceleration of the decontamination when oxalate spiking was used in the final cycles. An oxalate spike does not have to be used in each cycle.

After circulation of the excess oxalate, the oxalate is destroyed by the addition of the appropriate amount of potassium permanganate, and clean up then takes place during the transition phase as above.

Process Temperature

The required system temperature is dependent on the metals to be cleaned. Decontamination of 300 series stainless steels or Inconel requires the highest temperature practical within an atmospheric pressure system. If the temperature is lowered, the metal loss rates will be reduced and the decontamination will take place more slowly. There does not appear to be any adverse effect of lowering temperature, other than increasing the process time, and it is desirable to retain the flexibility in procedures to lower temperature. Decontamination of carbon steel or 400 series stainless steel can take place at ambient temperature (due to the higher inherent corrosion rates of these materials). The use of the oxidation cycle may not be strictly necessary for decontamination of these materials. However, it has been found that where oxidation is not used, an insoluble surface film can sometimes be formed arising from minor components in the steel (such as sulfide inclusions). This surface film incorporates radioactivity and limits the decontamination factor achievable, however much metal is removed. Oxidative conditions can help to avoid the formation of this film.

Process Sequence

This section gives a typical outline of the sequence of the EPRI DFD Process. The following procedure is typical for decontaminating 300 stainless steels.

Initially the system is filled with demineralized water and circulation through the chosen flowpath is established. The system is heated to 200^{o}F.

1. When temperature and flow have been established fluoroboric acid is added to the system to achieve 10 mM concentration (0.88 grams per liter). The pH should be maintained in the region 2.0–2.4 from now until final clean-up. The solution is circulated.

2. Flow to the cation exchange column is opened up to remove any initial burst of metals or radioactivity released by the first chemical addition.

3. Once this phase is complete the ion exchange column is valved out and potassium permanganate is added to the system. Typically 300 ppm or less total addition is weighed out and added to the system gradually at a rate suitable to maintain a standing concentration of 100 ppm. This step continues until permanganate is no longer present. Permanganate often lasts longer in the system in later cycles as the metal surface becomes clean.

4. Oxalic acid is added to the system and the cation exchange flow re-established. The amount of oxalic acid should be equivalent to the amount required to dissolve manganese dioxide resulting from the reduction of added potassium permanganate. It is preferable to apply this step even if an oxalate spike is to be performed, because it provides the opportunity to remove metals and radionuclides before the oxalate spike commences.

5. If an oxalate spike is to be performed, ion exchange is placed off-line and oxalic acid is added to achieve a concentration of about 50 ppm.

6. Any residual oxalate is destroyed by adding the amount of potassium permanganate necessary to convert it to carbon dioxide. Ion exchange flow is continued (or re-established) to remove residual potassium, manganese, metals and radioactivity. This continues until the pH and radioactivity measurements indicate an equilibrium level has been reached.

The steps 3, 4, 5 and 6 are now repeated for the required number of “cycles”. A typical number of cycles required would be eight to achieve free release. The time required for each cycle depends on system parameters, but a typical value would be six hours per cycle (more on a large complex system). Completion is determined from a combination of direct dose rate readings on system components, radioactivity in solution or examination of system surfaces (or a removable artifact).

7. Fluoroboric acid is removed and water purity is re-established with anion or mixed bed ion exchange.

For other metals (such as carbon steel) decontamination is achieved with milder conditions, in particular the temperature of application can be lowered to ambient (see above).

The above scheme is summarized in Figure 2-1.

Anion Exchange During EPRI DFD

For some applications the radioactivity present is anionic in character, rather than cationic, and cannot be removed during the cyclic phases of the process as described above (although such contamination will be removed during the final clean-up). A particular example is technetium-99, which is a frequently encountered contaminant when cleaning material from uranium enrichment plants. If no action is taken to control these anionic contaminants during the decontamination, the concentration of these contaminants can build up and cause problems of re-deposition on the surfaces being cleaned. To avoid this a small anion exchange column can be used during the process. Optimally this anion exchange column is in the fluoroborate form, to avoid altering the concentration of fluoroborate during the decontamination. This strategy is also very effective for removing anionic chemical impurities, which might otherwise build up during the decontamination. Use of the anion column does not have to be continuous – it can just be used occasionally as required.

Replacement of Some of the Fluoroboric with Nitric Acid

It has been found that for some applications the presence of 10 mM fluoroboric acid can cause excessive corrosion of certain materials, particularly Zircaloy, if the highest temperatures are used. Although this corrosion is not a concern for structural integrity, the loss of metals involved can cause an excessive volume of secondary waste to be generated. The process cannot be used conveniently as described above for Zircaloy pressure tube systems. Another separate problem encountered with 400 series stainless steels is that application of the process can cause a black slime to appear on the surface during the reductive phases of the process (this, it is thought, is due to sulfide inclusions within the metal structure). In both cases it is possible to substitute part of the fluoroboric acid concentration with nitric acid. The combined concentration of both acids is usually equal to 10 mM for this procedure. In the Zircaloy pressure tube case, this action reduces the problems of Zircaloy corrosion, without seriously compromising the benefits of using fluoroboric acid provided that some residual concentration of this acid is retained. In the 400 series application the nitric acid acid helps to maintain slightly oxidizing conditions throughout the process, which helps to prevent slime formation. Oxidizing conditions also maintain technetium in the anionic “pertechnetate” form, which is important for achieving anionic removal of this species for enrichment plant decontamination operations.

Effect of Boric Acid

PWR systems sometimes contain residual boric acid prior to decontamination. The boric acid may potentially interfere with the operation of the process. Removal of boric acid prior to the decontamination may require both time and cause the generation of extra waste, and therefore should be avoided if possible.

Work by PN Services [16] shows that the process can tolerate the presence of 700 ppm boric acid with minimal detrimental effect. Further, more detailed information is available in the Appendix of that reference.

EPRI DFD “Lite”

For applications such as cleaning 400 series stainless steel pump impellers, aluminum, carbon steel etc., ambient temperature application of the process is normally preferable as described above. The maintenance of oxidizing conditions can also be accomplished through the replacement of some of the fluoroboric acid with nitric acid (also as described above). Under these conditions there is no need for permanganate cycles, and the process can be applied as one continuous step. This combination is sometimes referred to as “EPRI DFD Lite”, and has been used very effectively by ALARON Corporation [10] and for the cleaning of aluminum contaminated with technetium as described later.

3

PROCESS ENGINEERING

Introduction

The EPRI DFD Process can be applied in one of two ways, depending on the type of object or system to be cleaned. Either the solution is circulated through the internals of an existing enclosed system or the object to be cleaned is immersed in a tank through which the process solution flows. Both alternatives can be serviced with the same equipment for flow, ion exchange, chemical injection etc.

The technology for application of chemical decontamination was originally developed for (temporarily shut down) operational nuclear plants. The constraints of an operational plant are such that very little space is available to locate decontamination equipment, and the equipment has to enter and exit through narrow and confined access routes. The equipment must be located in such a way that operators and other plant workers can be shielded from temporary radiation fields arising from the decontamination operation. Waste resin from the decontamination has to be processed or stored at an appropriate location which may be some distance from the decontamination equipment. All these factors have led to equipment based on small and modular skids, which can be interconnected once in position. This approach leads to a high degree of flexibility, so that the same process equipment can be used for a variety of different tasks at different locations. Although fixed equipment might be justified for some production-line tasks, mobile equipment of the type described can be used for any size of task, up to an including full commercial reactor coolant system decontamination.

Equipment

The equipment will typically consist of chemical injection equipment, a particulate filter, temporary ion exchange columns with shielding and a process heater/cooler.

An example follows of parameters for equipment to clean the largest system (e.g. a reactor coolant system). A diagram of the equipment is shown in Figure 3-1.

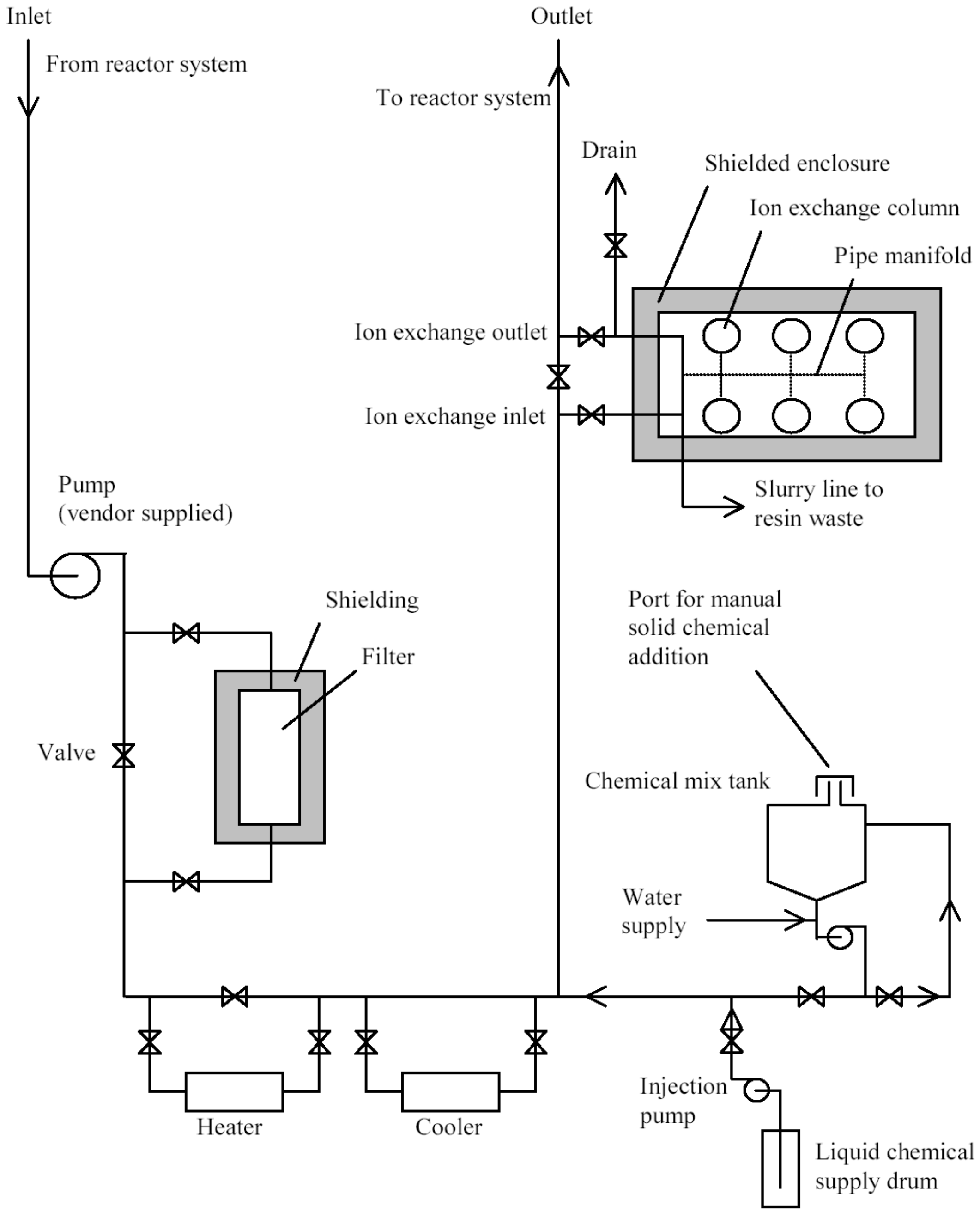


Figure 3-1
Typical EPRI DFD Equipment Diagram

Approximate three dimensional representations of the individual skids are given in Figures 3-2 thru 3-7 and the approximate size and weight of each skid is given in Table 3-1.

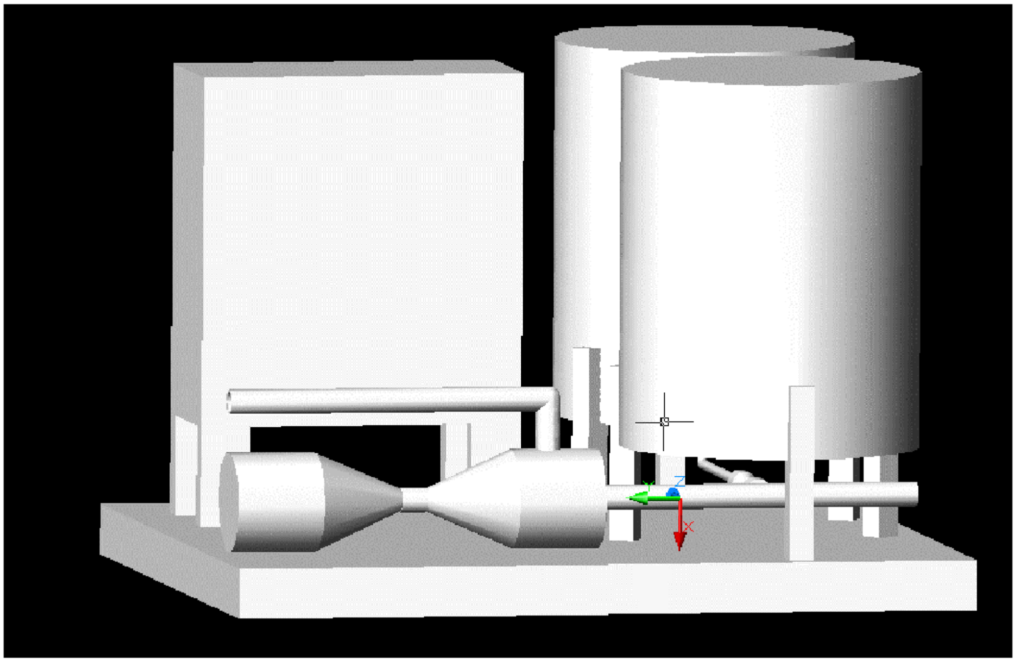


Figure 3-2
Diagram of Pump Skid

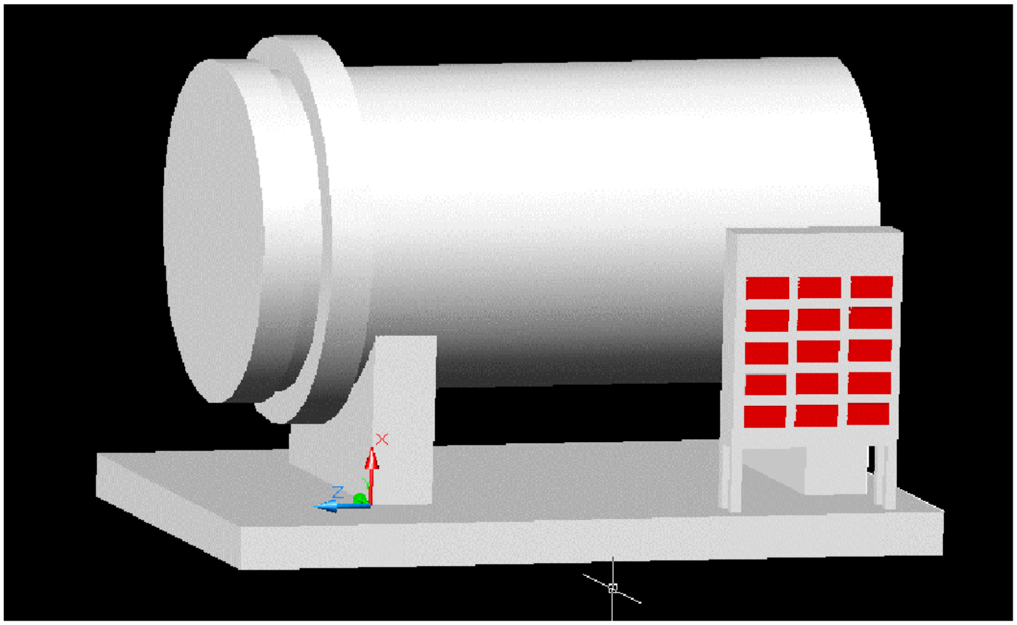


Figure 3-3
Diagram of Heater Skid

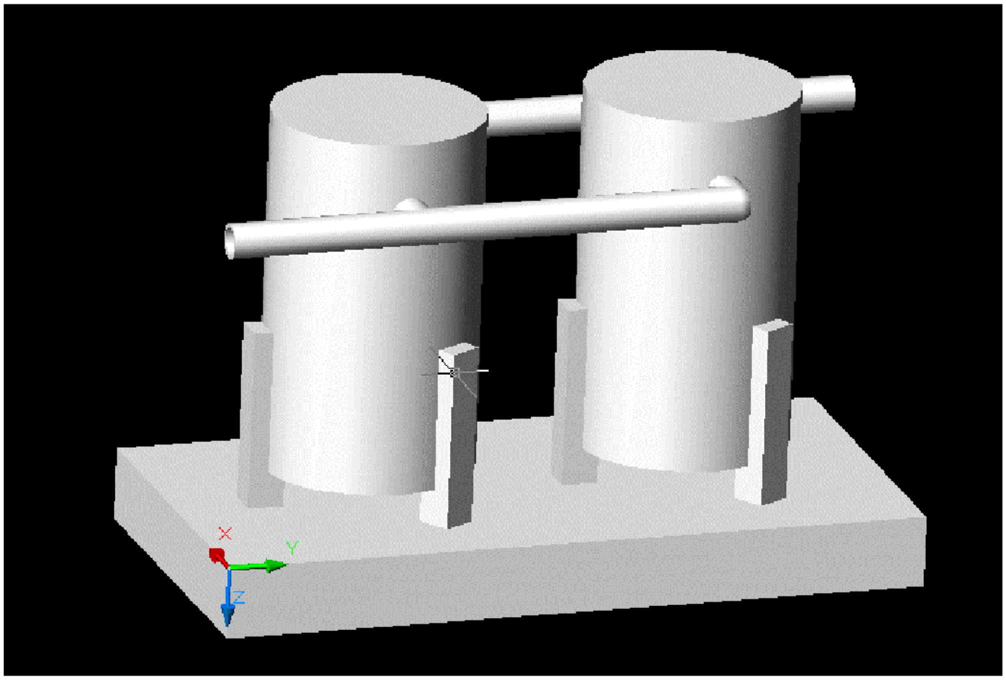


Figure 3-4
Diagram of Filter Skid

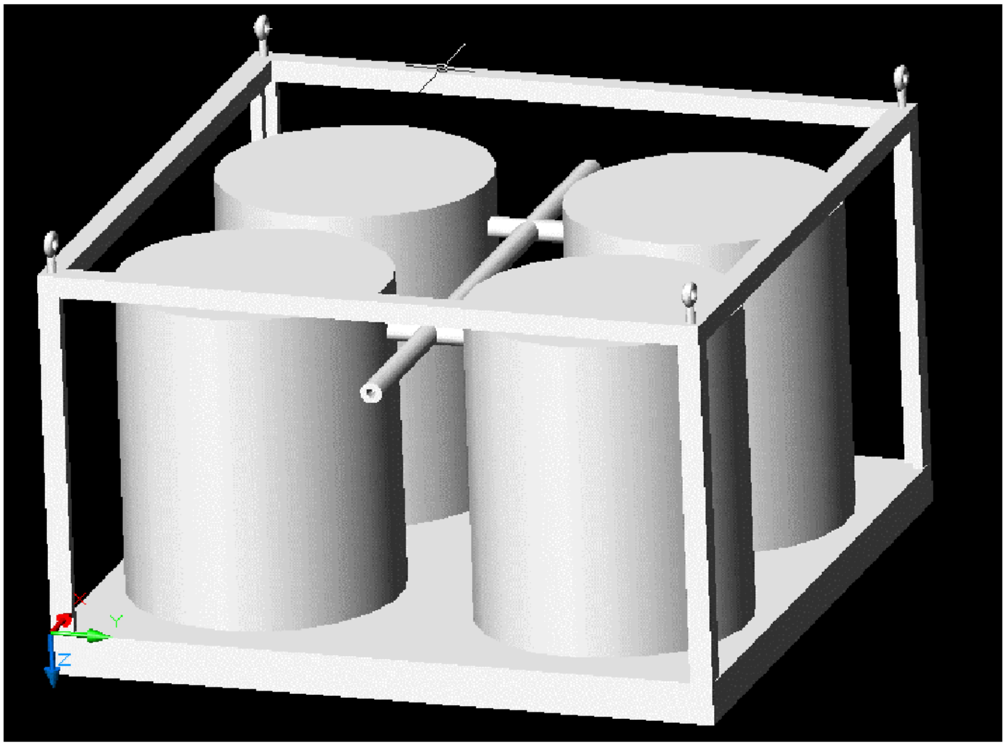


Figure 3-5
Diagram of Cation Exchange Skid

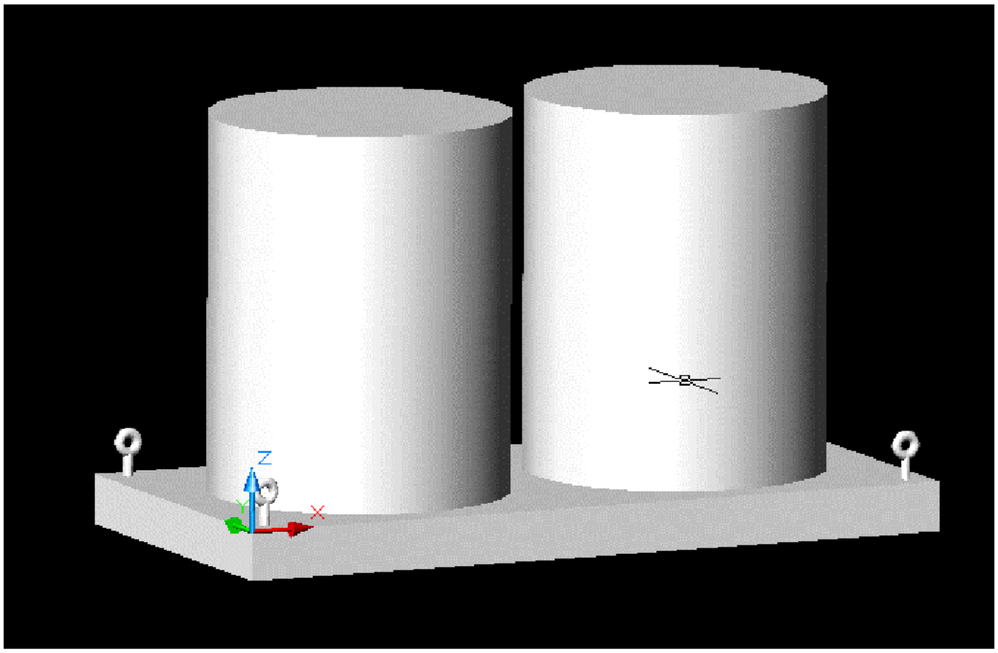


Figure 3-6
Diagram of Anion Exchange Skid

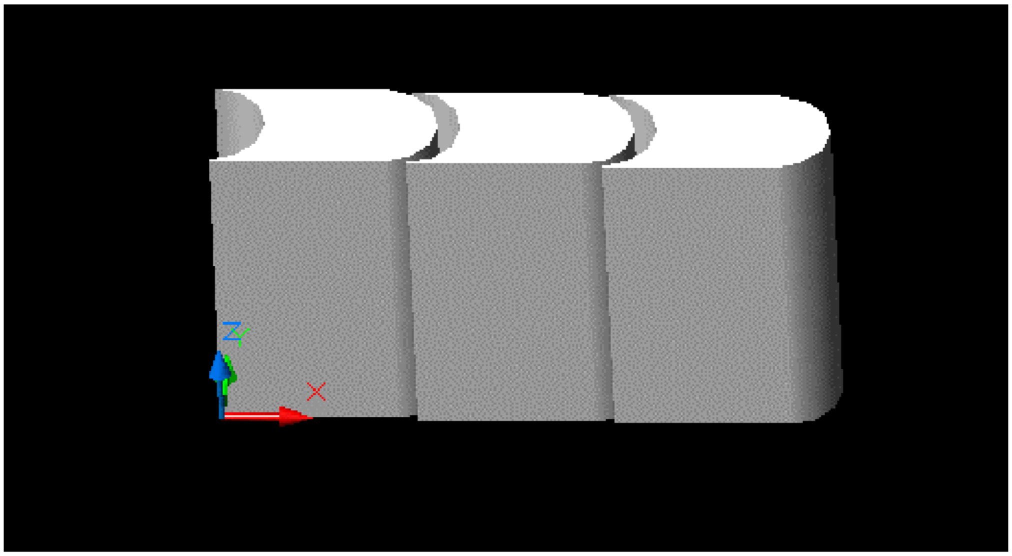


Figure 3-7
Diagram of Water Wall Shielding

Table 3-1
Approximate Dimensions and Weights of EPRI DFD Skids

Skid description	Length	Width	Height	Empty Weight	Full Weight
	(m)	(m)	(m)	(kg)	(kg)
Pump Skid	2.8	2.9	2	3,400	5,400
Filter skid	2	1	1.5	1,000	2,000
Cation Exchange skid	3	2.5	2	5,300	13,700
Anion Exchange Skid	3	1.5	2	2,350	6,600
Heater skid	3	2	1.8	3,000	6,000
Shielding tank	1	0.6	2	(300	1,500)
Assume12 tanks				3,600	18,000
			Totals	18,650	51,700

Mix Tank

The chemical injection equipment will consist of a mix-tank of approximately 1 m^3 capacity with a pump for recirculation and injection. The mix tank is filled with process solution (or demin water) and solid chemicals are added manually. The mixed solution is injected to the system. Fluoroboric and nitric acid is directly injected to the system from drums using a flexible hose and injection pump.

Pump

The pump must be of sufficient capacity to give an adequate rate of circulation and ion exchange clean-up (about 40 $dm^3 s^{-1}$, 630 gpm), and to fulfil the requirements of process temperature control. Materials for the pump must be carefully selected for compatibility with the EPRI DFD – e.g. 300 and not 400 series stainless steel must be used for the impeller.

Ion Exchange Columns

Ion exchange columns are provided as part of the process (i.e. it is unlikely to be appropriate to use the reactor water clean-up columns at a nuclear plant). Reactor columns are not of suitable design.

Ion exchange columns are based on a standard design (depending on space limitations, between six and twelve columns of about 1000 dm^3 , 30 cu ft, capacity each). Attention is needed to the material for the resin screens. A suitable grade of corrosion resistant material such as Hastelloy must be used. Ion exchange resin is standard H and OH form strong acid and strong base bead resin, as used typically in nuclear plants.

Unless local conditions prevent this strategy ion exchange columns are shielded by temporary shield blocks or “water walls”, i.e. temporary tanks which are interlocking and are put in position in an empty condition. The tanks are then filled with water to provide radiation shielding. After the decontamination when the ion exchange resin has been discharged to waste the water can be emptied and the tanks removed. Where water must be drained from the system for volume control, the water can be drained through the cation and anion columns (i.e. demin quality water is what is drained) for recycle.

Ion exchange columns are filled by vacuuming resin beads into the columns from the supply drums. The columns are emptied by standard water slurry techniques. Motive water used to slurry the used resin is recovered from the receipt vessel and recycled.

Filter

Nearly all applications of the EPRI DFD process include filtration. During the application of the process some of the contamination is spalled off the surfaces as particulate material. Although this material would ultimately dissolve in the EPRI DFD chemistry, the removal of this material by filtration is beneficial because:

- Circulating particulate material can settle out in low flow areas where it may be impossible to re-suspend it.
- Removal of contamination as particulate represents an efficient form of waste minimization
- To ensure efficient operation ion exchange columns should be protected from the ingress of particulate material.

Typically a full flow filter is used in the process line. Ideally this filter is back-washable, to reduce waste volumes and avoid difficult cartridge-changing operations.

The filter is a physically a small item, usually of a multiple cartridge design. It must be capable of handling the full flow of the pump. In the US the filter elements are usually changed by manual disassembly, but this procedure can cause significant operator dose unless the crew is very experienced, and it may be that a design with an automated filter element change is more appropriate.

Piping

Piping of the equipment should be “hard piping” (flanged stainless steel) design as far as possible for the main system. Good quality flexible hoses can be used for any piping which does not experience potassium permanganate (e.g. the ion exchange connections). Where flexible hoses are necessary in the main system, PTFE lined hoses should be used, unless the lengths are very short. Polymer hoses are not a safety or integrity concern, but can cause degradation of the permanganate chemical if significant surface area is present.

Required Power Source and Ancillary Services

Electric Power is required during the decontamination for running the pump and the heater (if this is required). The requirements for electric power are typically about 500kw. If no heating is required it may be considerably less than this.

A modest supply of compressed air is usually needed to operate valves etc.

All vessel vent points are usually led to a common outlet which is tied in to a ventilation extract system which discharges to atmosphere through a HEPA system.

Demineralized water is needed for the decontamination, but the amounts required for all purposes (e.g. resin transfer, chemical make-up) can be minimized by recycling water wherever possible.

Laboratory facilities are needed for the control of the decontamination. It is usually very expensive and inconvenient to supply these services on a nuclear power plant site, and the plant usually has a chemical laboratory with equipment which can provide the necessary analytical parameters. Solution samples withdrawn from the system for analysis are all returned to the system and there is no accumulation or requirement to manage liquid radioactive waste of samples.

Smaller Scale Decontamination

The equipment for small scale decontamination will be similar to the equipment described above but much smaller. Shielding may not be necessary, and all the process items can be probably be located on a single skid. Also, depending on the materials to be cleaned, and hence the process temperature requirements, the connections and piping can be made from uPVC or similar.

Special tanks can be designed and used to cause appropriate process solution flow around and through individual types of component. Internal inlet feed nozzles can also be used to generate high flow in specific places.

The system will probably not require a liquid chemical drum addition system. The requirements for liquid chemicals are so small that they can be added manually to the mix tank.

Operational Pressure and Flow

Pressure is not an important parameter for the operation of the EPRI DFD Process. In some cases decontamination systems are open to atmosphere, which effectively determines the operating pressure of the system. When reactor coolant pumps are used to circulate the solution the pumps have a minimum NPSH requirement for correct operation. This, with an appropriate margin of safety, will determine the minimum pressure at which the decontamination can take place. There is no need to increase pressurization beyond this.

The flow rate used to apply the EPRI DFD process should be the maximum reasonably and safely achievable. Experience shows that the results with the EPRI DFD Process are improved with increasing linear flow rate (or Reynolds Number is a probably a better parameter to correlate). The factors are less time required for each cycle, less cycles being required to achieve a given decontamination factor, less residual loose contamination after the decontamination and better control of the chemistry during the process application. The performance and limitations of the pumps are likely to determine the maximum flow achievable through individual systems. Improvements in decontamination performance may sometimes be achieved in specific parts of the plant by diverting extra flow to those parts by valve manipulations. This is an important technique to be applied during the decontamination in conjunction with radiological survey data, to identify any areas of poor performance and remedy the situation.

The ion exchange flow rate is determined by the requirements of the ion exchange resin for efficient operation. This topic should be addressed in the detail design stage and operational procedures. As a general rule decontamination ion exchange columns operate at a flow rate of 10-20 column volumes per hour (column volume refers to the volume of ion exchange resin in the ion exchange column). This may rise to 60 column volumes per hour when chemical concentrations are low.

Ion Exchange Slurry and Refill and Filter Change

The process of “slurrying” the resin refers to fluidizing the ion exchange resin beads in water and pumping them with the aid of (e.g.) a diaphragm pump through temporary pipework to another location. This is a standard procedure for moving ion exchange resin and is well established. Despite the fact that the resin with a high radioactivity content is moving through exposed pipework, this process does not cause significant radiation exposure, provided that remotely actuated equipment is used.

When the ion exchange resin in a column is expended the column must be slurried to waste and refilled. It is an essential function of the chemist to ensure that the resin filling plan allows for slurry and refilling of the columns at convenient times when the ion exchange columns are not required in service. The ion exchange columns are normally slurried to a plant waste resin receipt tank, or to a shielded transportation container for removal from site.

Filter elements must be changed when the pressure drop across the filter shows that the filter elements are fully loaded. Filters can either be changed manually by disassembling the housing and placing the elements in a shielded waste container, or can be “back-washed” if the design allows it. Hand-changing of filters can be achieved with little radiation exposure when the operators are experienced, but reliable back-washable filter designs now exist, and would probably be a better choice for new equipment.

4

IN SITU APPLICATIONS

Full System Decontamination

Full System Decontamination at Big Rock Point

Big Rock Point (BRP) is a 70 MWe BWR (Boiling Water Reactor) run by Consumers Energy in Michigan, USA. The plant shutdown for the final time in August 1997 after thirty five years of successful operation. The policy of the BRP management was to initiate a seven year restoration plan that would clear the site to "green field" conditions by the end of the year 2004.

An important first step after defuelling was a chemical decontamination of the system to reduce the radiation fields around the plant. This was a necessary part in meeting the USNRC (Nuclear Regulatory Commission) restrictions on total personnel dose allowed for the decommissioning of the plant.

The preparations for the decontamination were begun in late November/early December 1997, and the application started on December 28. The decontamination project was managed and applied by PN Services Inc. of Richland, Washington.

The total system volume was $121m^3$ (32,000 US gal) with an internal surface area of $1000m^2$ (10,764 sq ft) and comprised the reactor vessel (with internals removed), the circulation piping and circulation pumps, the steam drum and the chemical treatment plant. A small part of the circuit (Reactor Clean Up and Shutdown Cooling System) was fabricated from carbon steel, the remainder being type 304 stainless steel. During the decontamination the reactor coolant pumps were operated, giving a system turnover time of approximately 1 minute.

The mobile treatment plant, for process applications, provided a flow rate to and from the reactor system of $2.3m^3\ min^{-1}$ (600 US gal per minute). The rapid treatment and circulation rates employed ensured rapid mixing and treatment of chemicals used during the process cycles. In addition to rapid mixing, a high velocity of the decontamination reagent through the system pipework aids the removal of the oxide coating and ensures all parts of the internal surfaces are exposed to fresh solutions during chemical cycles. The application started on December 28, 1997 and finished on January 14, 1998 with the circuit chemistry being returned to demineralized quality water chemistry. Two separate applications were involved - a hot (95^oC, 203^o F) run on the primary system stainless steel lasting nine days and a cooler (30^o C, 86^o F) run through the carbon steel of the chemical treatment plant lasting three days. One day was used to clean all the circuits to demineralized water.

A total of 15 T Bq (409 Ci) of gamma emitting isotopes were removed from the BRP system. This was accompanied by 470 kg of iron, 62.7 kg of chromium and 41.8 kg of nickel. Radiation measurements around the plant dropped significantly with an average dose reduction factor of 15 being achieved. High radiation points of up to 80 mSv hr^{-1} (8 R/hr) were reduced to 200 µSv hr^{-1} (20 mR/hr). The average contact radiation field was reduced to 120 µSv hr^{-1} (12 mR/hr) with some points being as low as one tenth of that number.

Waste was in the form of dewatered ion exchange resin. 15.2 m^3 (540 cu ft) of resin was used for the absorption of the cationic metals and radioactivity. About 80% of the resin's theoretical capacity was occupied by metals removed from the system, the remaining capacity being used for process chemicals added. The total resin waste produced can be accommodated in a cube of sides 2.6 m (9 ft). The waste was tested and passed the US EPA's TCLP (Toxic Characteristic Leaching Procedure) leach test thereby avoiding a "mixed waste" classification . The resin was dewatered and placed in a HIC and sent to Barnwell for burial.

The decontamination application was achieved below budget and with two days to spare as allowed by the plant schedule. The project manager for Consumer Energy, Stanley Kupka, stated "EPRI's DFD Process was found to be safe, effective and economical, providing us with the results we were seeking".

Full Loop Decontamination at Maine Yankee

Maine Yankee is an 810 MWe PWR situated near Bath on the coast of Maine, USA. It shut down permanently in August 1997.

The decontamination took place as a two system application [11, 12]. The first system comprised a single coolant loop plus the chemical volume control system. The second system comprised all three coolant loops and the residual heat removal system. The steam generators were adapted by using an engineered connection from the cold leg to the hot leg to avoid flow through the steam generator tubes. No station pumps were used on this occasion, the circulation being achieved by a 2.3 m^3 min^{-1} (600 gpm) pump on the mobile decontamination skid. The reactor vessel was not included in either of the two decontamination applications. This was achieved by the installation of a flow through nozzle dam assembly, called a spider, at the interface of the reactor coolant loops and the reactor pressure vessel.

The process was begun on February 10, 1998 and was completed by March 7. This included two days to change over systems and two days for system clean-up at the end of decontamination.

A total of 3.7 T Bq (100 Ci) of gamma emitting isotopes were removed from the Maine Yankee system. This was accompanied by 120 kg iron, 115 kg nickel and 50 kg chrome. The higher proportions of nickel in the waste are due to the inclusion of Inconel in the circuit. The metals and radioactivity removed were contained on 15m^3 of resin (535 cu ft)(3) which was dewatered and placed in a HIC for burial at Barnwell.

The reduction of radiation dose rates was measured on 48 contact points and 43 general area meters throughout the plant. The decontamination factor (DF) achieved by order of radiological significance are given in Table 4-1.

Table 4-1
DF in Order of Radiological Significance

Initial Contact Dose Rate mSv h^{-1}	Rem h^{-1}	DF
>10	>1	107
5 to 10	0.5 to 1.0	170
1 to 5	0.1 to 0.5	25
<1	<0.1	5

Application At Trojan PWR

PN Services applied the EPRI DFD Process to a number of stainless steel tanks and heat exchangers at the Trojan PWR plant in April and May 1998. Good results were achieved, particularly with the heat exchangers, where an overall decontamination factor of 66 and dose reduction factor of 33 was achieved with 8 cycles of the process. This application demonstrates the viability of on-site decontamination of components prior to disposal.

Benefits of Full System Decontamination with the EPRI DFD Process

It is now some time since the two full system decontamination operations described above were performed. During the subsequent time significant progress has been made in decommissioning both the plants, and it has been possible to make an assessment of the benefits achieved by the process applications.

The benefits at Big Rock Point and Maine Yankee were summarized at an EPRI workshop in Wiscasset, Maine in June 2000 by Palaggi [13] and Plante and Collins [14]. The referenced presentations are summarized below.

Big Rock Point

The principal factors, which drove the decision to undertake full system decontamination prior to decommissioning of Big Rock Point were dose reduction and alpha contamination control. In both respects the plant's expectations of the decontamination itself have been achieved, though some additional actions were identified and undertaken which have allowed enhancement of the benefits obtained.

A good example of this latter point is that decontamination does not yield uniform reduction of dose rate in all plant areas. In some cases individual components may be out of the decontamination flowpath, or poorly decontaminated due to low flow. However, while these items blended into the radiological map before the decontamination, they are easily identified after the decontamination as "hot spots" and can be simply and quickly removed. The removal of these hot spots significantly enhances the subsequent dose reduction benefits of the decontamination itself. Figure 4-1 relating to the Recirc Pump Room illustrates this point.

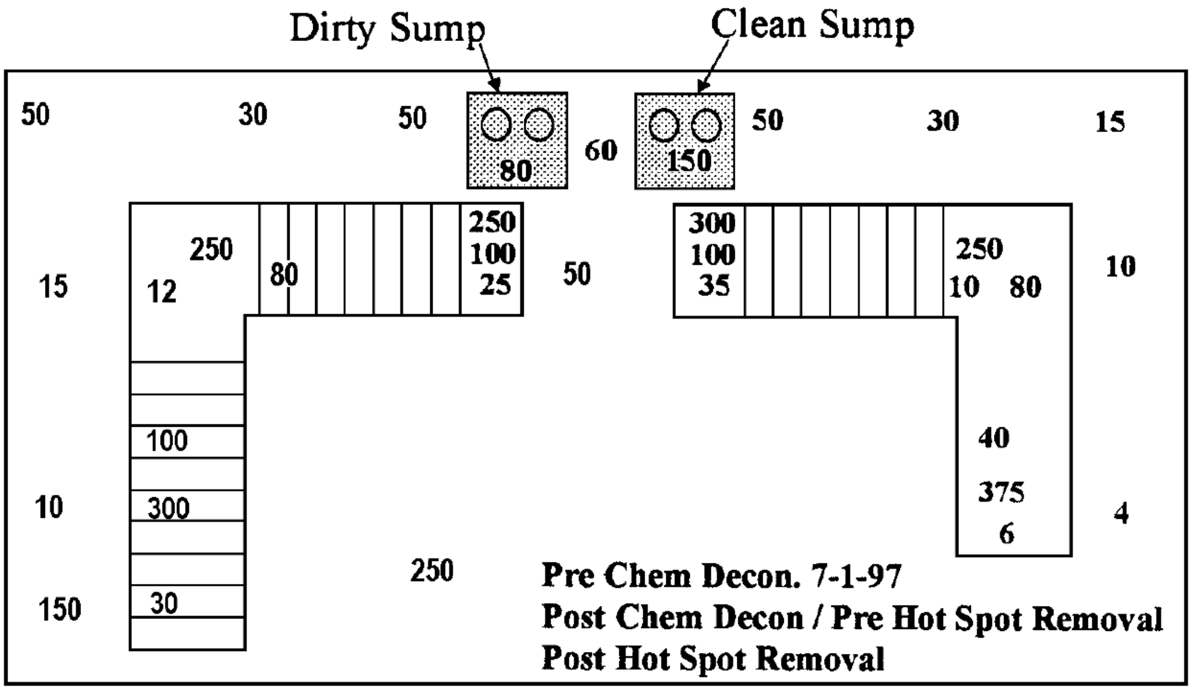


Figure 4-1
Big Rock Point Recirc Pump Room Dose Rate Reduction

In the recirc pump room itself it has been possible to estimate the difference in average dose rate with and without the decontamination. Clearly radiation fields reduce with time both due to the effects of radioactive decay, and because the decommissioning process removes hot items. However the decontamination has significantly steepened the downward gradient, as shown in figure 4-2, and from this it has been possible to estimate the savings of dose due to the decontamination which are given in Table 4-2.

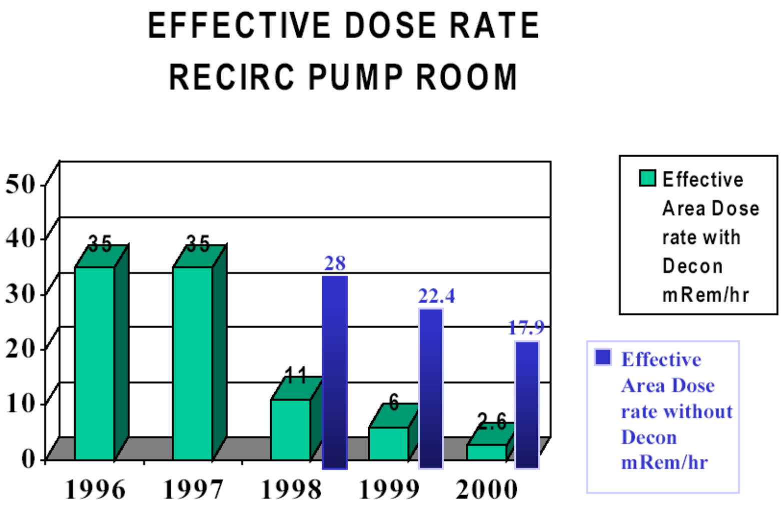


Figure 4-2
Big Rock Point Effective Dose Rate Recirc Pump Room

Table 4-2
Big Rock Point Estimated Dose Savings–Recirc Pump Room

Year	Estimated Radiation Dose (Man Rem)		Estimated Saving (Man Rem)
	With Decon	Without Decon	
1998	67	115	48
1999	34	163	129
to 6/2000	13	87	74

The EPRI DFD Process proved to be extremely effective at removing alpha activity. Due to fuel failures in early operation it was known that significant alpha contamination would be present and that it could create problems in the decommissioning operations. A post decontamination survey of the reactor vessel head showed that alpha contamination levels were negligible on this component. Subsequent survey work has confirmed that alpha contamination levels in the plant are now almost universally low, and there have been no problems with airborne alpha, uptakes or ingestions or dose assessments during the subsequent decommissioning work.

In addition to the dose reduction benefits the health physics resources for decommissioning have been significantly reduced due to the decontamination. It has been possible to relax job coverage requirements such that two technicians can cover the work which would otherwise have required four to six. Minimal shielding has been needed and there has been minimum interference with process work. Respirator work has been required for first time evaluation only. The ventilation system has been found to be adequate for the purpose with no additional engineering controls required. The containment building has remained clean, which has avoided interference with other work. There have been less than 20 personnel contaminations since the decontamination and no pre-work site-specific decontamination has been necessary. There have been no extremity dose monitoring issues.

With regard to waste management, it has been possible to process the reactor vessel head separately from the reactor vessel and apart from two “Hot Spot” high integrity containers of waste, it has been possible to send all the other waste to a contractor for processing.

All the above factors have meant that the decontamination has caused a significant net saving of time, radiation dose and money in the decommissioning operations.

Maine Yankee

The original reasons for proceeding with the decontamination were similar to Big Rock Point, although alpha contamination as not such an issue. The Maine Yankee presentation repeated many of the points mentioned by Big Rock Point.

It has been determined that 3.3 Man Sievert (330 Man Rem) has been saved as the a result of the decontamination, which compares favorably with the original estimate of 2.56-2.71 Man Sievert (256-271 Man Rem).

The decontamination was stated to have achieved additional benefits. There have been savings in packaging radioactive material from containment, and the decontamination has reduced off-site doses to the public (e.g. during transport operations) and at the waste processing contractors. The decontamination has also enabled cost effective alternate waste reprocessing options.

The other point mentioned was that the decontamination created a positive impression created with stakeholders and regulators.

The continuing benefits of decontamination will diminish as decommissioning progresses and components are removed, and it was pointed out that the greatest value can be obtained by decontamination as early as possible after final shutdown.

Decontamination of Nuclear Submarines

Information released recently in the UK by the Ministry of Defence [15] indicates that after a study by them, they are considering the possibility of land storage for redundant submarine reactors in the UK. In a separate move Babcock Rosyth Defence Limited has completed extensive feasibility and planning work on a proposal they have put forward to dismantle the reactor compartment from one of the decommissioned nuclear submarines, HMS Renown. In this proposal, all radioactive material would be removed from the submarine for land storage, and the remainder of the submarine structure would be broken up and recycled. Full system decontamination with EPRI DFD forms an integral part of this plan.

After decontamination one possible option for the metal components is that they could be sent to the Studsvik facility in Sweden where metal is melted to be released for recycling. Melting provides a further layer of assurance when measuring materials for unrestricted release. Internal surfaces and complex components are homogenized in the melt and processed into metal ingots. This process affords the opportunity for the precise determination of radioactivity and the production of archive samples. Melting also reduces the need for expensive space in waste repositories because only the slag has to be disposed of.

5

EX-SITU APPLICATIONS

Applications at Alaron Corporation

Alaron Corporation has decontaminated of a total of twelve stainless steel heat exchangers using the EPRI DFD Process. The heat exchangers weighed about 3,500kg (7,717lbs) each. Initial radiation levels were in the range 5-10mSv h^{-1} (0.5-1.0 rem/hr).

For the first four heat exchangers, the EPRI DFD Process was applied with a sufficient number of cycles to achieve levels close to unrestricted release. After dismantling and minor secondary decontamination (e.g. surface wiping or light blasting with aluminum oxide grit) 90% by weight of the heat exchanger was cleared for non-nuclear recycle. A different strategy was employed for the last eight heat exchangers. The number of chemical cycles was reduced to a point where the heat exchanger shell could be released, while the much thinner tubes were crushed and sent for low level waste disposal by Envirocare. This method was described as “meeting the profit expectations of ALARON for the process work”, and achieved the processing of the last eight heat exchangers in twenty two days.

The secondary waste produced in these applications was a small volume of ion exchange resin and particulate filters.

The EPRI DFD process has significant potential to achieve decontamination of components to unrestricted release levels so that those components can be refurbished in a non-radioactive shop. The low temperature EPRI DFD process was utilized by Alaron to allow contaminated pump elements (i.e., shaft, impellers, diffusers, pressure reducing sleeve, etc.) to be free-released such that the components could be re-conditioned in a non-radioactive workshop, incorporating the upgrades and performance improvements requested by the utility. Development of this alternative has created a competitive market, improved pump element quality, and opened up other opportunities for contaminated pump repair. Figure 5-1 illustrates an ex-situ apparatus.

The DFD Process has also been applied to shroud head bolts and control rod drives. These have been successfully decontaminated, although in the case of the shroud head bolts, approximately one third of the bolt is neutron-activated. This portion is cut and removed for burial.

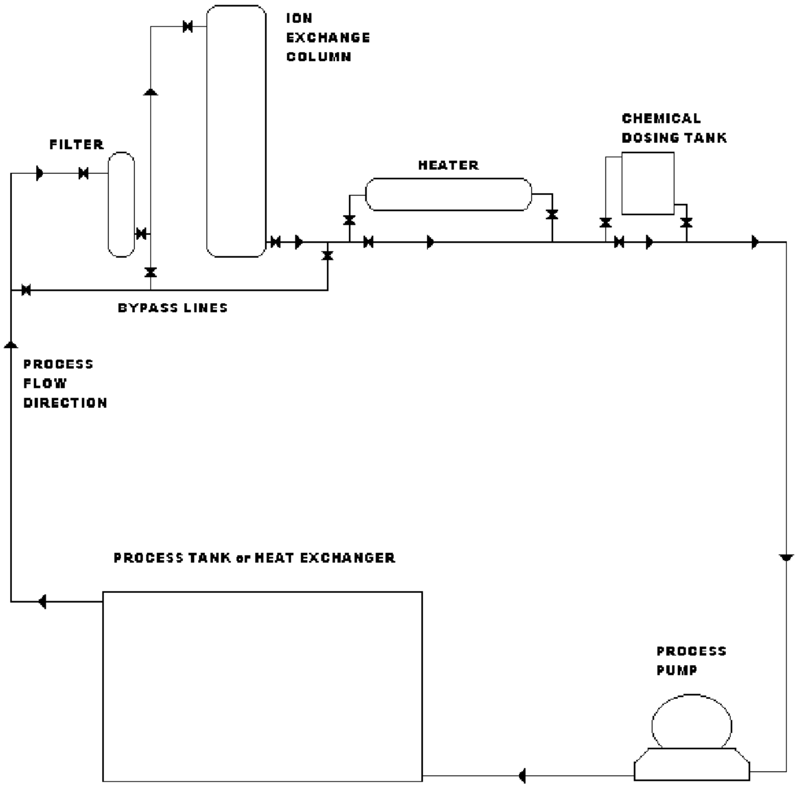


Figure 5-1
Ex-Situ Process Flow Diagram

US Department of Energy Compressor Blades

Trials have taken place at the Oak Ridge Reservation near Knoxville, Tenn. Part of the accumulated retired plant waste is the discarded aluminum compressor blades that have failed in service and subsequently been removed during maintenance outages.

Decontamination Recovery Services (DRS) received several drums of blades to be used in a decontamination trial at their Oak Ridge Facility. DRS supplied a secure facility and project support such as health physics. A combined team from Practical Machine Engineering and Bradtec Decon Technologies applied and engineered the EPRI DFD Process.

The blades were chosen at random from each of the four drums provided, but care was taken to ensure that a mixture of sizes was included. The isotopes of primary concern were uranium and technetium, technetium being the more abundant of the two. More than 70% of the blades were able to be released, with some of the remainder being volumetrically contaminated due to recasting in previous recycling campaigns. After the process trials, both cation and anion resins were successfully regenerated and a neutralized sludge produced. The sludge was successfully incorporated into a cement matrix. Samples of resin taken before regeneration were subject to a TCLP test by an independent laboratory. The resin successfully passed the test. Security considerations did not allow the blades to be released from the USDOE compound.

6

MATERIALS CORROSION DATA

The corrosion of materials during application of the EPRI DFD process is a key issue from point of view of safety, waste generation and process effectiveness. A significant amount of data has now been gathered from laboratory testing and field application of the EPRI DFD Process. The majority of this data is reported below, but some additional data may be found elsewhere [16].

General Corrosion Data

The corrosion data gathered to date is as a result of coupon exposure in Bradtec's laboratory test loop, results from field tests at ALARON's Northwest facility, artifact testing by PN Services in their test loop at the Richland facility and in a corrosion chamber at the HAKE Field test. This HAKE test itself was described in an earlier reports [4, 16].

Metals such as carbon steel, 400 series stainless steel and aluminum all ehibit high corrosion rates at higher temperatures, 60 C–94 C (140^o–200^o F), but can successfully be decontaminated at lower temperatures with a concomitant reduction of corrosion rate. A number of non-metallic materials have been exposed to the EPRI DFD solution, these have comprized seals, gaskets, valve seats, valve packing and valve diaphragms. Visual examination of the materials detected no obvious deterioration. Parameters such as elasticity or surface finish all appeared to be unaffected by the exposure. The materials are listed in Table 6-1. Tables 6-2, 6-3 and 6-4 summarize the metal corrosion from the field tests at ALARON and HAKE, and the material testing by PN Services.

Table 6-1
Non-Metallic Materials Tested

RCP Seal 9.4"	**RCP O Ring 7.7"**	**Asbestos Seal 7.2"**
RHRP Seal Plate O Ring 5"	Charging Pump Gasket 4.7"	Charging Pump O Ring 2.9"
1" Ball valve Seat	Valve Diaphragm EPDM	Valve Packing
Titanium Carbide	Carbon	Ethylene/Propylene Rubber
Ag coated 718 Inconel	Ag Coated 304 Stainless	

Table 6-2
Results from an Initial Materials Test Program at ALARON

Sample ID	1 Cycle g/m^2	5 Cycles g/m^2	1 Cycle Metal Loss (µm)	5 Cycle Metal Loss (µm)	Material Description
C61-43 C29-42	754.82 796.90	ND ND	97.40 102.83	ND ND	Stainless 420
CO8-83 CO8-62	1190.59 1148.14	ND ND	146.99 141.75	ND ND	Low alloy carbon steel arrived badly corroded
CO2-21 CO2-22 VO2-1	674.19 646.67 653.00	4236.38 4128.30 4080.83	86.99 83.44 84.26	523.01 509.67 503.81	Stainless 410
CO6-2 SO6-1 SO6-2	23.13 25.40 31.81	678.88 667.68 716.65	2.86 3.14 3.93	83.81 82.43 88.48	17-4 PH
D1 SO3-D6 SO3-D8	5.51 5.37 5.41	52.78 145.96 94.88	0.68 0.66 0.67	6.52 18.02 11.71	CF8 w/w weld
O3X2 O3X5 O3X6	6.63 5.28 4.90	71.47 47.44 54.00	0.82 0.65 0.61	8.82 5.86 6.67	CF8 w/o weld
SO1-1 VO1-3 VO1-5	7.61 10.91 13.91	227.55 289.99 ND	0.94 1.35 1.72	28.09 35.80 ND	Sensitized 304 Stainless
C56-42 C56-43 V56-42	0.11 0.15 0.11	9.74 10.83 10.25	0.01 0.02 0.01	1.06 1.18 1.12	HAYNES 25
C10-42 V26-4	6.41 16.18	131.69 177.11	0.79 2.00	16.26 21.87	Stellite 156
V26-64	0.62	23.46	0.08	2.9	Stellite 6
AL1100 6061/143	619.76 491.21	2819.56 2774.51	228.69 181.26	1040.43 1023.8	Aluminum
CDA110	8.53	206.68	1.05	23.25	Copper
CDA443	10.89	223.36	1.28	26.22	Cu 72%, Zn 27%, Sn 1%
CDA706	29.45	282.36	3.29	31.58	Cu 90%, Ni 10%
LEAD9	188.21	382.5	16.6	33.73	Lead
PTFE 0 PTFE1 PTFE2	-0.37 0.37 0.44	-0.37 0.37 0.44	-0.05 0.05 0.05	-0.05 0.05 0.05	PTFE (Teflon)
I-26 I-27	ND ND	254.6 258.95	ND ND	30.20 30.70	Inconel 600 (pre-ox) TOTAL 8 CYCLES
S-127 S-128	ND ND	69.87 62.62	ND ND	8.63 7.73	Stainless Steel 304L (pre-ox) TOTAL 8 CYCLES

Table 6-3
Artifact Test Results

Sample ID	3 Cycle Metal Loss (µm)	Material Description
HC-04-1 HC-28-1	1.1	Hastelloy C20004
HC-28-1 HC-28-2	0.8	Hastelloy C22-28
HC-28-21 HC-28-24	12.9	Inconel 600
04	2.5	Stainless 316
06	2.3	Stainless 316L
09	2.5	Stainless 304
10	3,3	Stainless 304L
5	3.9	Stainless 304 U-Bend
304-9 304-10	85	Sensitized 304 U-Bend

Table 6-4
Corrosion Data from the HAKE FIELD TEST

Material	Corrosion (microns)
304-308 (weld) - 304	1.88
I-182 weld metal	11.1 / 14.6
Inconel 600 (flat coupon)	5.8 / 5.4
Inconel 600 (SG Tube, mill annealed)	7.8 / 8.0
Inconel 600 (tubing)	3.1 / 3.4
Incoloy 800 (flat coupon)	2.7 / 2.0
Incoloy 800 (tubing)	3.6 / 3.5
321 Stainless (flat coupon)	2.8 / 1.2
321 Stainless (tubing)	6.8 / 6.7
70/30 Cu-Ni	31.4
CRM (weld)	0.8
CRM (tube)	0.5
Admiralty Brass	47.7
Zircaloy-4 (tube)	329
410 Stainless	489.2 / 527.2

These specimens were exposed throughout the decontamination of RWCU heat exchangers from "as-received" condition to the point where much of the material could be free released.

Laboratory Materials Testing

The initial development of the EPRI DFD Process was achieved using inactive oxidized coupons of Stainless 304 and Inconel 600 in a corrosion loop. Coupons of the type used in the test program were also exposed in the field test programs and the full system decontaminations at Big Rock Point and Maine Yankee. This comparison showed the laboratory results to be consistent with what is experienced on commercial applications (See Table 6-5)

Table 6-5
Comparison of Corrosion Rates for Oxidized 304 Stainless Steel Obtained in Laboratory Tests and Commercial Applications

Corrosion Rate Lab µm/cycle	Corrosion Rate Field Tests µm/cycle
0.24	0.27
0.27	0.25
0.26	0.33

A new corrosion test program was performed to include the 400 series stainless steels which are prevalent in ancillary equipment, e.g. pumps. Also included were samples of copper, silver and gold, which are used in various types of seals. The coupons used in the program were treated in three separate ways. The first set of coupon samples was exposed in "as received" condition. The second set was heat treated in an inert atmosphere to produce a sensitized coupon. The third set was heat treated in air to produce a sensitized and oxidized coupon. The conditions for heat treatment and oxidation were in accordance with the procedures of Allen et al [17, 18], which produces surface oxidation representative of that during reactor operation over a number of years.

Samples were exposed in a test loop at temperatures of 90^{o} C and a flow rate of 0.15 m s^{-1} (0.5 ft s^{-1}). The coupons were measured for weight loss after the third cycle and the sixth cycle. Table 6-6 summarizes the weight loss data and the depth of corrosion for each of the coupons tested.

Effective decontamination is a balance between requiring some corrosion and dissolution in order to get the materials clean, but not too much so that integrity is compromised or excessive waste is produced. Metals can be broadly divided into three groups according to their rate of dissolution in EPRI DFD. At the top end is aluminum which had to be removed from the hot tests due to loss of control over the process chemistry. The corrosion rate in the first three cycles was about 55 µm per cycle although previous experience in field tests has shown that aluminum dissolution can be as high as 200 µm per cycle. Although not part of this test program, previous work has shown carbon steel has similar behavior.

Table 6-6
Corrosion Data from Laboratory Test Program

Alloy Type	Coupon Ident	Heat Treatment	Initial Weight	3 Cycle Weight	3 Cycle Loss (g)	6 Cycle Weight	6 Cycle Loss (g)	Corrosion Loss (µm)	
								3 Cycle	6 Cycle
304	119	ox	14.5253	14.4956	0.0297	14.4853	0.0400	1.2802	1.7241
304	118	ox	14.5604	14.5335	0.0269	14.5225	0.0379	1.1595	1.6336
304	117	ox	14.5583	14.5310	0.0273	14.5211	0.0372	1.1767	1.6034
304	26	n/a	14.4062	14.4012	0.0050	14.3966	0.0096	0.2155	0.4138
304	25	n/a	14.3495	14.3451	0.0044	14.3421	0.0074	0.1897	0.3190
304	1	ox	14.9088	14.8837	0.0251	14.8717	0.0371	1.0819	1.5991
304	2	ox	14.8264	14.8037	0.0227	14.7931	0.0333	0.9784	1.4353
304	5	ht	14.7640	14.7538	0.0102	14.7322	0.0318	0.4397	1.3707
304	4	ht	14.6669	14.6635	0.0034	14.6441	0.0228	0.1466	0.9828
304	7	n/a	14.6445	14.6361	0.0084	14.6276	0.0169	0.3621	0.7284
304	8	n/a	14.9225	14.9182	0.0043	14.9094	0.0131	0.1853	0.5647
304	9	n/a	15.0543	15.0490	0.0053	15.0402	0.0141	0.2284	0.6078
316	1	ox	14.1971	14.1699	0.0272	14.1673	0.0298	1.1724	1.2845
316	2	ox	14.4211	14.3968	0.0243	14.3945	0.0266	1.0474	1.1466
316	4	ht	14.5054	14.4880	0.0174	14.4849	0.0205	0.7500	0.8836
316	5	ht	14.4804	14.4633	0.0171	14.4579	0.0225	0.7371	0.9698
316	9	n/a	14.3543	14.3526	0.0017	14.3520	0.0023	0.0733	0.0991
316	8	n/a	14.5001	14.4984	0.0017	14.4977	0.0024	0.0733	0.1034
316	7	n/a	14.3562	14.3548	0.0014	14.3538	0.0024	0.0603	0.1034
410	1	ox	15.1478	15.0485	0.0993	14.3636	0.7842	4.2802	33.8017
410	2	ox	15.3377	15.2334	0.1043	14.4327	0.9050	4.4957	39.0086
410	5	ht	14.9973	14.5761	0.4212	13.6102	1.3871	18.155	59.7888
410	4	ht	15.2759	15.0146	0.2613	14.4133	0.8626	11.263	37.1810
410	7	n/a	15.4111	14.3760	1.0351	13.4838	1.9273	44.616	83.0730
410	8	n/a	15.3892	14.4746	0.9146	13.6885	1.7007	39.422	73.3060
410	9	n/a	15.145	14.4245	0.7205	13.8108	1.3342	31.056	57.5086
416	3	ox	17.8154	17.6075	0.2079	17.0604	0.7550	8.9612	32.5431
416	1	ox	17.6425	17.4134	0.2291	16.9485	0.6940	9.8750	29.9138
416	4	ht	17.8307	17.4507	0.3800	16.5503	1.2804	16.3790	55.1897
416	5	ht	18.1146	17.8865	0.2281	17.3920	0.7226	9.8319	31.1466
416	8	n/a	17.8448	16.9590	0.8858	16.2194	1.6254	38.1810	70.0603
416	9	n/a	17.5984	16.8269	0.7715	16.2813	1.3171	33.2543	56.7716
416	7	n/a	17.9071	17.2773	0.6298	16.7142	1.1929	27.1466	51.4181
420	1	ox	17.5063	17.0902	4.4161	16.4230	1.0833	17.9353	46.6940
420	2	ox	17.4023	16.9670	0.4353	16.2900	1.1123	18.7629	47.9440
420	5	ht	17.3793	16.8395	0.5398	15.7845	1.5948	23.2672	68.7414
420	4	ht	17.1604	16.7671	0.3933	15.9852	1.1752	16.9526	50.6552
420	7	n/a	17.1636	16.5975	0.5661	16.0729	1.0907	24.4009	47.0129
420	8	n/a	17.4300	16.8370	0.5930	16.3383	1.0917	25.5603	47.0560
420	9	n/a	17.0153	16.5609	0.4544	16.1351	0.8802	19.5862	37.9397
430	1	ox	14.3601	14.2860	0.0741	14.1395	0.2206	3.1940	9.5086

Table 6-6
Corrosion Data from Laboratory Test Program (Continued)

Alloy Type	Coupon Ident	Heat Treatment	Initial Weight	3 Cycle Weight	3 Cycle Loss (g)	6 Cycle Weight	6 Cycle Loss (g)	Corrosion Loss (µm)	
								3 Cycle	6 Cycle
430	2	ox	14.4970	14.4424	0.0546	14.3582	0.1388	2.3534	5.9828
430	4	ht	14.5113	14.4263	0.0850	14.3028	0.2085	3.6638	8.9871
430	5	ht	14.3278	14.2562	0.0716	14.0669	0.2609	3.0862	11.2457
430	7	n/a	14.6579	14.4001	0.2578	14.2977	0.3602	11.1121	15.5259
430	8	n/a	14.5185	14.3201	0.1984	14.2268	0.2917	8.5517	12.5733
430	9	n/a	14.3493	14.1783	0.1710	14.0806	0.2687	7.3707	11.582
440	2	ox	15.9535	15.6925	0.261	15.0358	0.9177	11.2500	39.556
440	1	ox	16.5191	16.3642	0.1549	15.9934	0.5257	6.6767	22.6595
440	5	ht	16.0897	15.8247	0.2650	15.3803	0.7094	11.4224	30.5776
440	4	ht	15.9831	15.7751	0.2080	15.3549	0.6282	8.9655	27.0776
440	9	n/a	16.4499	15.4052	1.0447	14.6916	1.7583	45.0302	75.7888
440	7	n/a	16.3260	15.5131	0.8129	14.9686	1.3574	35.0388	58.5086
440	8	n/a	16.4209	15.7432	0.6777	15.2474	1.1735	29.2112	50.5819
I-600	1124	ox	16.0413	15.8903	0.1510	15.8314	0.2099	6.5086	9.0474
I-600	1123	ox	16.4084	16.3095	0.0989	16.1856	0.2228	4.2629	9.6034
I-600	1121	ox	16.3526	16.2546	0.0980	16.1307	0.2219	4.2241	9.5647
CF8	1	ox	15.0102	14.9882	0.0220	14.9762	0.0340	0.9483	1.4655
CF8	2	ox	15.1431	15.1249	0.0182	15.1166	0.0265	0.7845	1.1422
CF8	3	ox	14.9836	14.9649	0.0187	14.9578	0.0258	0.8060	1.1121
CF8	6	ht	11.3241	11.3115	0.0126	11.3013	0.0228	0.5431	0.9828
CF8	4	ht	13.5789	13.5679	0.0110	13.5604	0.0185	0.4741	0.7974
CF8	5	ht	12.3133	12.2991	0.0142	12.2945	0.0188	0.6121	0.8103
CF8	7	n/a	14.4717	14.4628	0.0089	14.4554	0.0163	0.3836	0.7026
CF8	8	n/a	15.1005	15.0938	0.0067	15.0904	0.0101	0.2888	0.4353
CF8	9	n/a	15.1668	15.1596	0.0072	15.1555	0.0113	0.3103	0.4871
17.4PH	2	ox	14.7423	14.6950	0.0473	14.6576	0.0847	2.0388	3.6509
17.4PH	4	ht	15.1387	15.0789	0.0598	0.0294	0.1093	2.5776	4.7112
17.4PH	5	ht	14.9981	14.9363	0.0618	14.8846	0.1135	2.6638	4.8922
17.4PH	1	ox	14.7623	14.7069	0.0554	14.6615	0.1008	2.3879	4.3448
17.4PH	6	ht	14.8499	14.7850	0.0649	14.7282	0.1217	2.7974	5.2457
17.4PH	7	n/a	14.9058	14.8914	0.0144	14.8746	0.0312	0.6207	1.3448
17.4PH	9	n/a	14.9764	14.9624	0.0140	14.9457	0.0307	0.6034	1.3233
17.4PH	8	n/a	15.2732	15.2597	0.0135	15.2429	0.0303	0.5819	1.3060
Al	1	n/a	4.6066	2.9474	1.6592	4.6066	0.0000	211.902	0.0000
Al	3	n/a	4.8317	3.5192	1.3125	4.8317	0.0000	167.624	0.0000
Al	2	n/a	4.5466	3.6025	0.9441	4.5466	0.0000	120.574	0.0000
Cu	2	n/a	17.0639	16.9977	0.0662	15.9058	1.1554	2.5589	44.6618
Cu	3	n/a	17.0594	17.0243	0.0351	16.2580	0.8014	1.3568	30.9789
Cu	1	n/a	16.9535	16.9219	0.0316	16.2053	0.7482	1.2215	28.9215
Ag	1	n/a	21.5102	21.4623	0.0479	20.9958	0.5144	1.5757	16.9211
Ag	2	n/a	21.1105	21.0744	0.0361	20.6777	0.4328	1.1875	14.2362
Au	1	n/a	9.3194	9.3195	-0.0001	9.3197	-0.003	-0.0018	-0.0054

The middle group includes 400 series stainless steels which exhibit medium to slow dissolution depending on the alloy being tested. This ranges from 8 µm per cycle to 1.5 µm per cycle (alloy 430). Type 410 and 420 are martensitic alloys and have low nickel content with 12 to 20 % chromium. They are generally only used in mildly corrosive environments such as fresh water and atmospheric conditions. Type 430 is a ferritic stainless and contains 15-30 % chromium. The higher chromium content improves corrosion resistance and although it is not good in non-oxidizing acids, such as hydrochloric acid, it is resistant to mildly acidic and oxidizing media.

Also in this group are copper and silver, with recorded corrosion rates of 6 µm per cycle and 3 µm per cycle respectively.

The corrosion control required to process metals in the top two groups is easily obtained by a reduction in the process temperature. For cleaning aluminum, carbon steel and the 400 series stainless steels, ambient temperature is used for processing. However, where these materials are unavoidably present in high temperature decontaminations, this can be tolerated as long as there is adequate thickness for integrity not to be compromised and their surface area represents only a small fraction of the total surface area being cleaned.

Inconel 600 has a similar dissolution rate to that of stainless 430, i.e. 1.5 µm per cycle, on the borderline between the middle and bottom group. This behavior is very different from its corrosion resistance in other decontamination formulations which do not contain fluoroboric acid. *Fluoroboric acid is an essential ingredient for decontaminating Inconel-600.*

The final group of corrosion resistant materials includes 300 series stainless steels which are austenitic. The tests demonstrate a corrosion rate of approximately 0.3 µm per cycle. This group of low corrosion materials includes CF8, which is a cast form of stainless steel similar to type 304, and 17.4 PH (precipitation hardened), which is a heat treated stainless steel exhibiting slightly higher corrosion rate (1µm per cycle) than the 300 series. Some materials show very low or no corrosion at all. These materials (such as Hastelloy in Table 6-3) can be used for critical components such as resin screens in EPRI DFD Processing equipment.

Materials Corrosion in EPRI DFD Lite

Corrosion test work was undertaken by PN Services [16] in ambient temperature conditions. A summary of results is given in Table 6-7

Table 6-7
Ambient Temperature Corrosion of Key Materials

Material	Corrosion (µm)
Cast Iron	54.6
A 285 GrC	20.8
A 106 GrB	26.7
Admiralty Brass	1.0
70/30 Cu-Ni	1.0

A series of corrosion tests were performed at ALARON on 400 series stainless steel in relation to the use of EPRI DFD Lite to decontaminate pump impellers for release to a non-radioactive workshop environment. The tests were designed to show that it was practical to limit corrosion to

50 microns loss from the metal surfaces in normal and fault conditions. The two out of specification conditions tested were high temperature (116°F, 47°C) and low pH (1.3).

The test coupons, 410 series stainless steel, were subjected to a variety of pre-decontamination treatments including all or none of the following:

- Heat, 446°F (230°C) for 30 minutes in an oven open to the atmosphere.
- Degreasing chemical - Five minutes of exposure to the commercially available product "Goo-Gone" followed by rinsing with demineralized water and drying with a paper towel
- Releasing Oil - Five minutes of exposure to the commercially available product WD40 followed by rinsing with demineralized water and drying with a paper towel.

The pre-treatment conditions were a direct copy of the conditions undergone during preparation of the pump parts prior to a EPRI DFD Lite application.

The pre-treatment conditions had no significant effect on the overall performance of the corrosion behaviour with the three conditions tested as compared with untreated. The difference between the normal and fault conditions were however noticeable. The low pH run was limited to one cycle as the corrosion rate was sufficiently high to have exceeded the 50 micron limit on the second cycle. The other two runs were performed for three cycles as follows:

The samples were loaded in a sample chamber and exposed to the EPRI DFD Lite solution for two hours. The flow rate was 0.25 ft sec^{-1} (7.6 cm sec^{-1}) to represent flow rates within a treatment tank. The samples were then removed from the solution and placed in an ultrasonic bath to remove chrome oxide precipitate from the surfaces. Any residual surface precipitate was removed by wiping with a paper towel before replacing the coupons for another cycle.

A comparison of the results is given in Figure 6-1 which displays the average corrosion rate for each of the three conditions.

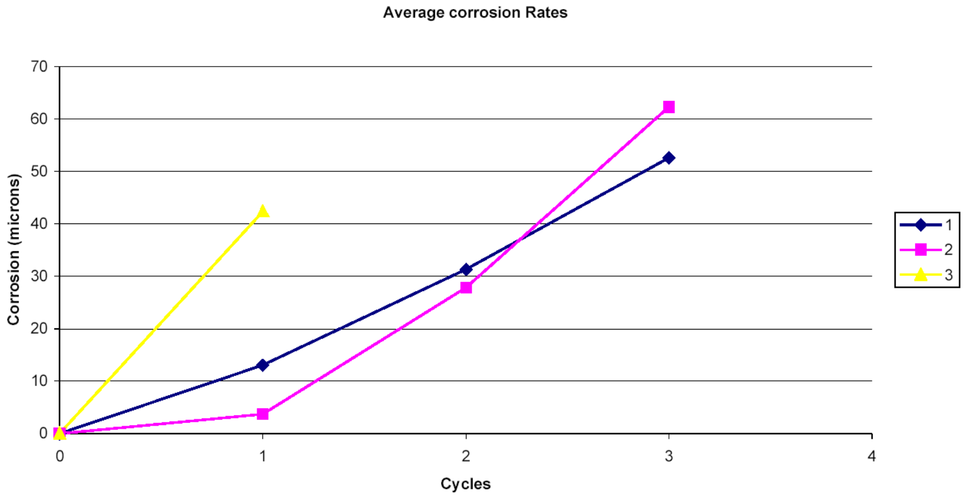


Figure 6-1
Comparison of Corrosion Rates in Tested Conditions

Series 1 = Ambient temperature (86°F, 30°C) at pH 2
Series 2 = Raised temperature (116°F, 47°C) at pH 2
Series 3 = Ambient temperature (86°F, 30°C) at pH 1.3

7

WASTE MANAGEMENT AND PROCESS SAFETY

The EPRI DFD Process has now been applied to many different tasks and at many different facilities. As part of the work done, exhaustive safety analyses have been performed, particularly where the process has been applied to full reactor system decontamination. This work has proved that, with the appropriate precautions, the EPRI DFD Process could in all cases be applied to the chosen task without significant risk to people, property or the environment.

The discussion below summarizes some of the points frequently raised in these analyses.

Chemical and Radiation Safety Issues

Material Safety Data Sheets for the chemicals used are provided in Appendix B.

Significant problems have been encountered in applying *concentrated* fluoroboric acid processes due to the corrosive nature of fluoroboric acid and its volatility. Extensive personnel and equipment protection is necessary, particularly where raised temperatures are involved. The EPRI DFD Process, however, uses fluoroboric acid in a very *dilute* concentration, once the stock chemical has been diluted into the process system as the first stage of the process. Respiratory protection is not normally required, even when the process solution is open to atmosphere.

Dilution is therefore an important factor assisting the maintenance of chemical safety during the decontamination. All chemicals are used in the plant system in very dilute concentration, such that the procedures and requirements of radiological protection are usually sufficient to protect workers and the general public from the adverse effects of leaks or spills. The dilute concentration means that spilled solutions do not have the molar capacity to cause serious structural corrosion of plant components or to build up large explosive or toxic gas concentrations.

Special precautions are needed by the operators handling the concentrated chemicals for dispensing into the chemical mix tank (or direct injection into the system in the case of fluoroboric acid). Even for a large system only quite small quantities of concentrated chemicals are needed, and it is good safety practice to keep the inventory of chemicals in the operational area to the minimum necessary. The operators should be properly briefed and trained and should wear special chemical-impervious protective clothing. Bunded areas should be provided as part of the chemical injection equipment design and commercial spill kits should be available for the collection and disposal of any spilled concentrated chemicals. No chemical disposal of any kind is permitted except bound to the decontamination ion exchange waste. Even samples taken for chemical analysis are processed through the system ion exchange resins. Unused chemicals are removed from site by the vendor.

Oxalic acid and potassium permanganate are used in most decontamination processes, and their properties are familiar. Oxalic acid dihydrate is supplied as a free flowing powder which can be added to the chemical mix tank and dissolves readily in water. Potassium permanganate is also a free flowing powder, and is a strong oxidizing agent (the powder or strong solution can cause fire if it is spilled on organic material).

Fluoroboric acid is provided in drums (usually about 100 litres of 50% solution). The concentrated acid is volatile, highly corrosive and has a harmful vapor. The first and only operation with the concentrated acid is to inject it through an injection "wand" (which dips directly into the supply drum) by means of an injection pump directly into the system. The injection system must have a properly operating check valve in the line to prevent any backflow. Once diluted in the system the chemical hazard characteristics are as referred to above. Concern has been raised previously that fluoroboric acid is corrosive towards Zircaloy fuel cladding. However, the quantity of fluoroboric acid required for a system decontamination is not sufficient to penetrate fuel cladding, even if dumped directly into the fuel pool.

The chemicals, including fluoroboric acid have been used safely in this manner as part of the EPRI DFD Process at many different plants and facilities, and this methodology has been accepted by independent plant safety reviews.

Other chemicals used in the EPRI DFD Process are familiar and are regularly used in commercial decontamination projects.

Concentrated fluoroboric acid is highly corrosive towards Zircaloy. Corrosion of Zircaloy proceeds at a slightly higher rate than carbon steel in the hot EPRI DFD solution, but much less than in concentrated fluoroboric acid. In recent times it has been shown that if the process chemistry is adjusted by reducing the fluoroboric acid concentration (as described in the Process Chemistry section) the process is compatible with being used in systems which contain Zircaloy components. The total inventory of fluoroboric acid is extremely small (e.g. one or two 55 gallon drums for a full system decontamination). When the process is used at a power plant the inventory of this chemical on site should be kept to a minimum. By doing this and taking other reasonable precautions, it is possible to avoid concerns about the inadvertant expsoure of Zircaloy to the chemical.

Radioactivity is removed from the circulating solution by ion exchange on a continuous basis. The circulating concentration of radioactivity during the decontamination is unlikely ever to exceed 10^{-1} microcurie per ml. During system heat up or towards the end of the decontamination, (which are the most likely times that a system leak would occur) the circulating radioactivity is likely to be two orders of magnitude below this level. Radiation levels in contact with process equipment during the decontamination are well within the experience typical of commercial decontaminations.

Mechanical Safety

The similarity of the EPRI DFD Process to other decontamination processes means that the mechanical safety issues are familiar.

Materials of construction for equipment have been addressed by process vendors. Although some changes to traditional equipment materials have proved necessary, no serious problems have been encountered.

Leakage

Leakage of the decontamination solution is an issue which must be addressed in the planning and execution of the decontamination. The use of an oxide and metal dissolving decontamination solution can be expected to open up existing leaks in the plant system, and particular care must be made to exclude small carbon steel threaded fittings from the decontamination flow path. The loss of material from the carbon steel threads can cause the fitting to be ejected creating a leak situation. Galvanic couples of carbon steel to stainless steel (e.g. welds) in small diameter pipes should be excluded from the hot decontamination system.

Having said that, problems with leakage can be largely avoided with proper planning and preparation. During the decontamination the operators must be constantly vigilant for signs of leaks. The chemical and radiological nature of the circulating decontamination solution is such that the consequence of leakage is not much worse than leaks of normal reactor coolant, and can be dealt with by normal procedures.

If a leak occurs the decontamination may have to be put into a “holding pattern” while the leak situation is addressed. The best way to do this is to hold the chemistry at the end of a cycle at the point before potassium permanganate is added, and to cool the reactor system to the minimum achievable temperature to reduce materials corrosion.

Countermeasures for leaks will include the use of clean-up by wipes, rags and mops for small leaks. Larger leaks will need to be collected in secondary containment and processed by the system ion exchange resins or sent for radwaste processing. Although the processing of decontamination solution by radwaste ion exchange systems is not permitted in routine circumstances, the systems can usually respond to an emergency and manage to process any leaked solution without difficulty. This is an advantage of using a very chemically dilute decontamination solution such as the EPRI DFD Process.

Waste Volumes

The volume of ion exchange resin waste generated by any particular application has to be calculated by the process vendor. The theoretical guide to the expected waste volume is that 1 liter of ion exchange resin waste will be generated per square meter of surface area decontaminated (1 cu ft per 300 sq ft surface area decontaminated). However, there are many reasons why the resin waste generated will be more than this minimum. Most notably there may

be significant thick deposits of metal oxides in old reactor systems or components which are being cleaned by the process. All this material will need to be removed by dissolution in addition to a thin layer of base metal to get the system clean. The dissolved deposit will create additional resin waste. Also, certain parts of a system or component may corrode preferentially. While measures can be taken to minimize this effect, the process cannot be terminated until the slowest-corroding part of the system is clean. For these reasons the waste volumes may in practice be about 10 times the theoretical minimum. Despite this the application of the process usually leads to secondary waste which has only a small fraction of the volume of the original component.
A small fraction of the total resin waste will be anion exchange resin, the volume of which is dependent on the volume of the system being decontaminated.

Where a production line situation exists, the same fluoroboric acid solution can be used for many decontaminations, all but eliminating the need for anion exchange resin. The water can, of course, be recycled as well.

Ion exchange resin will normally be buried as the final waste form from the process. However, chemical regeneration and reuse of the ion exchange resin is feasible. In this case the regeneration solution obtained can be treated by other methods (e.g. neutralization, precipitation and stabilization). This type of processing can be expected to lead to significant volume reduction.

A forthcoming development of the DFD process, which has already been demonstrated in laboratory tests, will significantly reduce the volume of residual waste, and eliminate the need to dispose of ion exchange resin. The use of this new development in combination with the EPRI DFD Process is likely to lead to a *volume reduction factor of between 10 and 20* compared with the conventional ion exchange waste alternative.

However, we have found that the practical ion exchange waste volumes generated for some full system decontaminations with the EPRI DFD process match quite closely the maximum curie loading which can be tolerated on ion exchange resin due to radiolysis and “Greater than Class C” limitations. Reducing further the volumes of ion exchange resin waste would not therefore be beneficial.

Mixed Waste Issues

The major concern with regard to the potential classification of the waste as “hazardous” is the presence of chromium initially present in the oxide films and metal, and subsequently removed by ion exchange resin.

Toxic Characteristic Leaching Procedure (TCLP) testing has been completed on resin waste arising from a number of different applications of the EPRI DFD process. In all cases anion resin shows less chromium leached than the 5 ppm RCRA limit defined in 40 CFR 261. In the case of cation resin, however, the leachable chromium was sometimes less and sometimes marginally greater than the 5 ppm limit.

The RCRA limit is intended to apply to hexavalent chromium, and on theoretical grounds it can be stated that hexavalent chromium is extremely unlikely to be present in the cation resin, since the form of any hexavalent chromium would be anionic in the prevailing chemical conditions.

Work by PN Services has confirmed this, showing, for example, that chromium leached from the cation resin is not hexavalent according to the diphenylcarbohydrazide analysis method. Good evidence now exists that leachable chromium is in the form of chromium mono-oxalate complex, formed due to traces of oxalate running through the cation exchange column during the process application. This complex is weakly held by the cation resin.

The extent of leachable chromium is dependent on the control of process parameters. The more care taken to exclude oxalate from the cation exchange column, the less leachable chromium there will be. As is typical of all chromium chemistry complexes form and are broken very slowly. If chromium is removed promptly by cation exchange and the blue-colored chromium complexes are not allowed to build up, there will be reduced problems with chromium leachability and, incidentally, greater stability of the permanganate stage and hence greater decontamination effectiveness. If resin is not fully loaded and there is spare capacity this will also reduce the chromium leachability. Use of these parameters has been successful, for example at Big Rock Point, in ensuring that waste passes a TCLP test.

Another potential solution to the mixed waste issue is to process the resin waste. The use of “Molten Metal” technology effectively renders the chromium non-leachable, and other techniques which destroy the resin waste and provide volume reduction (e.g. pyrolysis of the resin) will usually reduce any leachable chromium to a level well below the RCRA limit.

8

DISCUSSION

Introduction

The use of the EPRI DFD Process is potentially a major contributor both to the waste management of retired components from operating plants and to the decommissioning of nuclear plants. Surface-contaminated metallic waste forms a major part of the total radioactive waste arising from decommissioning of nearly all retired radioactive facilities such as power plants, uranium enrichment plants, fuel reprocessing plants and research facilities. There is an obvious need to manage this material in an optimally safe, environmentally acceptable and cost effective way. The alternatives (which are to delay dealing with the problem or dispose of all the material as waste) are not in accordance with environmental and sustainable objectives, and are unlikely to be acceptable in the long term. The EPRI DFD process provides a safe and cost effective means to remove surface contamination and convert it to a suitable waste form for disposal, while allowing the base metal to be recovered for recycle. The contamination is removed remotely, avoiding the need for "hands-on" radiation dose. The process effectiveness has been demonstrated as described in this report.

An essential advantage of the EPRI DFD Process is its flexibility. It can be applied to a wide range of different components for a wide variety of objectives. It uses simple and inexpensive equipment which requires little facility investment. This is an excellent advantage, because market conditions (such as number of plants shutting down, disposal site pricing or availability of disposal sites) can change suddenly, and a processor who has invested in expensive fixed plant may find the purpose of the original investment no longer applicable. As market conditions change the EPRI DFD Process can be quickly converted to different business lines according to what is economically attractive at the time.

There have been considerable benefits to the nuclear industry in the applications to date of the EPRI DFD Process. The benefits of the full system decontaminations have been discussed in Section 4. Significant cost savings have been achieved also by the ex-situ applications.

Despite these benefits, the process has only had limited application so far. The same is true for other competing technologies of similar type. The problem lies not so much in the technology, which is well advanced and is continuing to develop, but in the current circumstances in the nuclear industry. Some of the general and specific factors are discussed below, together with possible actions which could be taken to remove the "road-blocks".

General Factors

- Despite the considerable success of EPRI DFD at the shutdown plants at Big Rock Point and Maine Yankee (as described in this report), further plants are not coming off-line and into the decommissioning phase.

  While the turn-around in nuclear power performance and economics (which has led to plant life extension rather than shutdown) is good for the industry as a whole, there is likely to be a delay before further applications of the process take place. When final shutdown of the nuclear plants eventually comes the process or its successor will be needed and the experience gained in these initial decontamination applications will be valuable.

- It is hard to offer a cheaper solution when the alternative is to do nothing.

  For many potential applications of the EPRI DFD Process the alternative is prolonged storage. It is difficult to offer an economically competitive alternative to this, although storage only defers rather than solves the problem. For a number of reasons, including regulatory pressure, there seems to be an increasing international move towards speeding up decommissioning schedules. As in the previous point, the opportunities for the process have not gone, they have merely been deferred.

- The concept of free-release and recycle of cleaned material has particular problems of public perception, which at least in the short term are preventing widespread application of this type of operation. There is currently a moratorium on recycling material from the USDOE complex.

  In a way this is an example of a much wider issue about nuclear power - a technology with large potential benefit to mankind and the environment, which is held back by lack of public confidence. Free release of cleaned material into the public domain cannot be forced upon an unwilling public, however technically sensible or justifiable it is. The best approach is likely to be to find innovative ways to maximize the recycling benefit while minimizing any public risks. Recycling back into the nuclear industry is one such example. The key to successful free release is to gradually build public confidence in the quality of the operation, particularly the survey of material for clearance. This must be accompanied by clearly explaining the benefits. One of these benefits is that a decontamination and free release operation can accelerate the management and resolution of redundant facilities. Progress can also be assisted by standardizing practices as much as possible, both nationally and internationally.

- Chemical decontamination is unfamiliar and is often seen as unpredictable. There are concerns over the safety of handling chemicals from these types of operation.

  This type of concern, (together with extreme caution about the potential effect of chemical decontamination on reactor materials) was a familiar feature of operational decontamination when it was emerging in the 1980’s. It was eventually overcome by a combination of discussion and analysis, test work and operational experience. The chemical safety implications of the EPRI DFD Process are not onerous, as discussed in Section 7.

Specific Factors

- Many plants and waste processors prefer an "engineering" solution to decontamination problems.

  Simple mechanical methods (such as shot blasting) are often the right choice for accomplishing decontamination, but these methods cannot conveniently deal with components or systems which are complex, convoluted and contaminated internally. Chemical decontamination is appropriate for these tasks. Most mechanical decontamination alternatives require components to be dealt with in small pieces, which is more laborious and dose intensive than treating whole systems, as is possible with the EPRI DFD Process.

- Ion exchange waste is often not an ideal final waste form, and the overall volume reduction gained is not enough.

  For many applications ion exchange resin actually is the best final waste form, and as discussed earlier, there may be "Greater than Class C" implications which make further volume reduction of the waste undesirable.

The Way Forward

The process is at this point technically mature, and the next phase of work will be to seek additional applications for it. Some potential applications currently being addressed are as follows:

PWR Steam Generators

The EPRI DFD Process is uniquely suitable for PWR Steam Generator Decontamination. Steam Generator replacement has become a well-established operation to enable the use of upgraded materials and design. This type of operation is likely to become more prevalent with plant life extension. The options for direct management of the retired PWR steam generators are either long-term storage on site in a mausoleum, or direct burial. These options are quite expensive due to the large size of the components. The decontamination and size reduction of PWR Steam Generators has for some time been regarded as a possible alternative [19], allowing burial of the radioactivity in a small volume and recycle of much of the metal, particularly from the secondary side parts. There has also been a proposal to recycle the high-value Inconel-600 tube material from old steam generators to generate Inconel-690 for new steam generator tubes [20].

The principal material to be cleaned in PWR Steam Generators is usually Inconel, and the fluoroboric acid ingredient of EPRI DFD is essential for this purpose. Previous reports [3, 4] described this. Other processes, which rely on cycling between permanganate and oxalic acid are unlikely to accomplish efficient decontamination of Inconel. Experience with steam generator decontamination does not appear to have been very successful yet, and it will be important in the next stage of work to demonstrate the unique advantages of the process for this application.

USDOE Applications

The next phase of work will concentrate on demonstrating a number of applications to the USDOE complex. The following, in particular, are being targeted.

- Uranium Enrichment Waste (aluminum, nickel etc.)
- UF_6 Cylinders
- USDOE Fuel Casks

New Technical Developments

The development of a new technology to work with the EPRI DFD Process is an important objective of the next stage of work. The overall process, which has already been demonstrated on lab-scale, will enable the collection of radioactive contamination from a thin layer of the surface of components and systems and its conversion into a waste form ten to twenty times smaller than ion exchange resin. No other wastes are generated, and thus the new development will represent almost theoretical efficiency of decontamination.

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CONCLUSIONS

- The EPRI DFD Process has been developed to reduce dose and assist the waste management of nuclear power plant decommissioning operations. It has now been in use for several years and has been used successfully for a wide variety of applications.
- Full system application of the process at two plants (Big Rock Point and Maine Yankee) took place some time ago, and since then a considerable amount of decommissioning work has been done at these plants. The plants report substantial savings of radiation dose, time and effort and hence cost due to applying the process before decommissioning work. Because of its environmental and dose saving benefits, application of the process is viewed positively by stakeholders and regulators.
- Adjusting the process chemistry has led to new variants of the process. These new variants make it suitable for new applications such as Zircaloy pressure tube reactors, and 400 series stainless steel components.
- Issues such as Safety, Waste Management and Materials Corrosion are discussed in the report, which updates the information on these topics given in previous reports.
- Ion exchange resin remains the waste form of choice for most applications, although other alternatives are available.
- The EPRI DFD Process provides the technology for the waste management of a large proportion of the worldwide inventory of radioactive scrap metal.
- The development of a new technology to work with the EPRI DFD Process is an important objective of the next stage of work. The overall process, which has already been demonstrated on lab-scale, will enable the collection of radioactive contamination from a thin layer of the surface of components and systems and its conversion into a waste form ten to twenty times smaller than ion exchange resin. No other wastes are generated, and thus the new development will represent almost theoretical efficiency of decontamination.

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A

ALUMINUM TESTING AT OAK RIDGE

Introduction

The Oak Ridge Reservation is situated 25 miles west of Knoxville Tennessee and was built in 1943 as the first part of the Manhattan Project for the production of nuclear weapons material. Part of the accumulated redundant plant waste is the discarded aluminium compressor blades which have been removed during maintenance outages.

Several drums of blades were received by Decontamination Recovery Services (DRS) to be used in a decontamination trial at their Oak Ridge facility. DRS supplied a secure facility and project support such as health physics. The DFD process was applied and engineered by a combined team from Practical Machine Engineering (PMe) and Bradtec Decon Technologies.

The blades tested were chosen at random from each of four drums provided but care was taken to ensure that a mixture of sizes were included. The blades were of varying length (4”-12”) and width (2” to 4”) with lenticular cross-sections (1/8” – 1/2” thick) and a threaded boss on one end about 1” long by 1” diameter. Some of the blades used were believed to have undergone a nitric acid rinse when first removed from service. Others clearly had yellow deposit on their surfaces. The primary isotope of concern was initially believed to be ^{238}U however, the isotope of importance for attaining the recycle criteria was in fact ^{99}Tc. The compressor blades were controlled as classified material (UCNI) with appropriate security concerns being addressed by performing all blade handling activities within the visual containment area established in the north east end of the K-1420 building.

Pilot Plant Equipment

The equipment was designed by Bradtec and constructed by PMe Inc. The individual items were skid mounted (see Figure A-1) with the exception of the process tank, which was positioned in a remote location for security reasons.

The process tank was an upright open topped cylinder with a volume of approximately 260 US gallons (1.1m^3). The tank was contained within a bunded area to provide for secondary containment. The cation exchange column held 4 cu ft (140 l) of resin and removed the cationic contamination such as ^{238}U and the aluminum ions from the process solution. The anion exchange column held 2 cu ft (70 l) of resin and removed anionic contamination such as ^{99}Tc and other extraneous anions from the system. The resin was pre-treated with HBF_4/HNO_3 to ensure that any anions removed were replaced by BF_4^-/NO_3^- (see process chemistry).

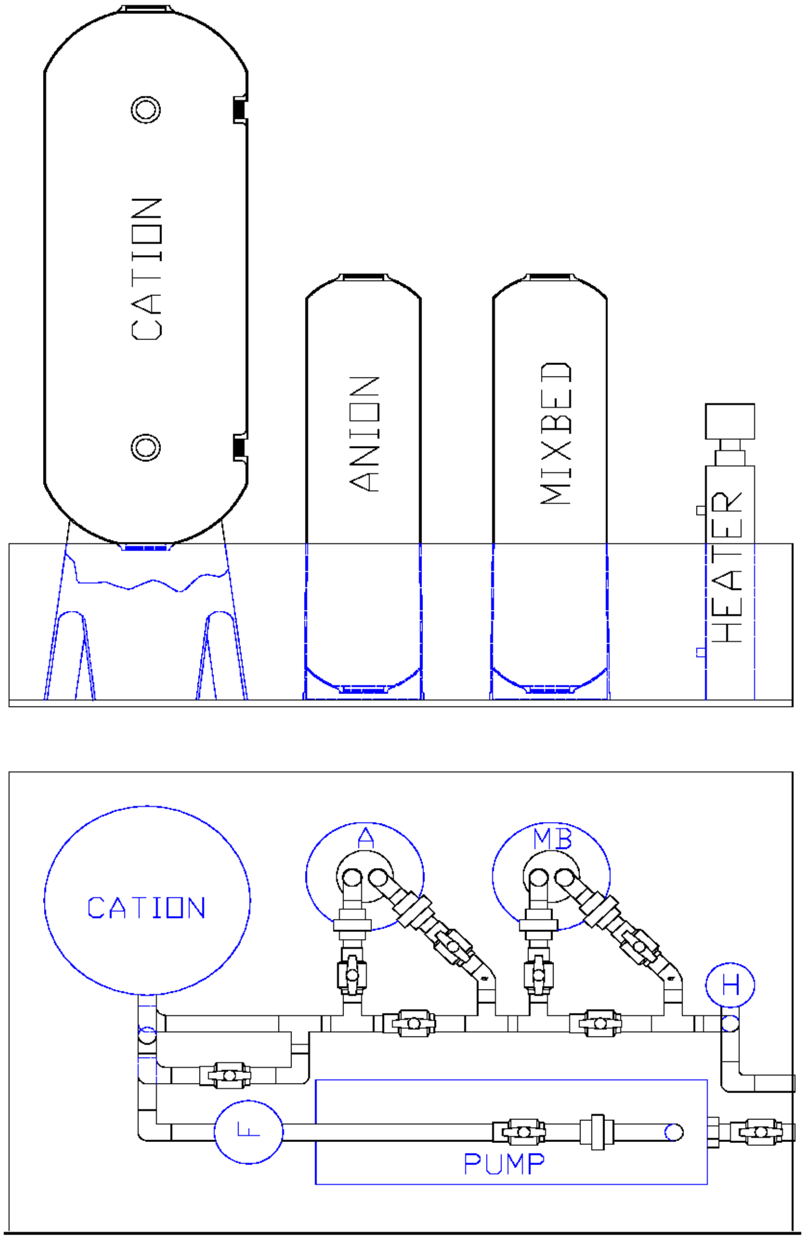

Figure A-1
Process Skid Layout

The mixed bed ion exchange column contained a mixture of anion (hydroxide form) and cation (hydrogen form) resin in a ratio of 2:1 anion:cation. The total capacity was 2 cu ft (70 l). It was used prior to testing to de-ionize the raw water, in-situ, before the addition of the processing reagent. It was used again at the end of the decontamination tests to remove the fluoroboric acid reagent and replace it with demineralized water.

With the tank, process piping and treatment columns, the volume of the process equipment was approximately 400 gallons (1.5 m^3). The system design and ion exchange capacity were calculated on the ability to process the system volume 3 times per hour.

Figure A-2 shows the basic flow diagram for the process. All items were manufactured from stainless steel or polypropylene. Carbon steel is not a suitable construction material for EPRI DFD application equipment.

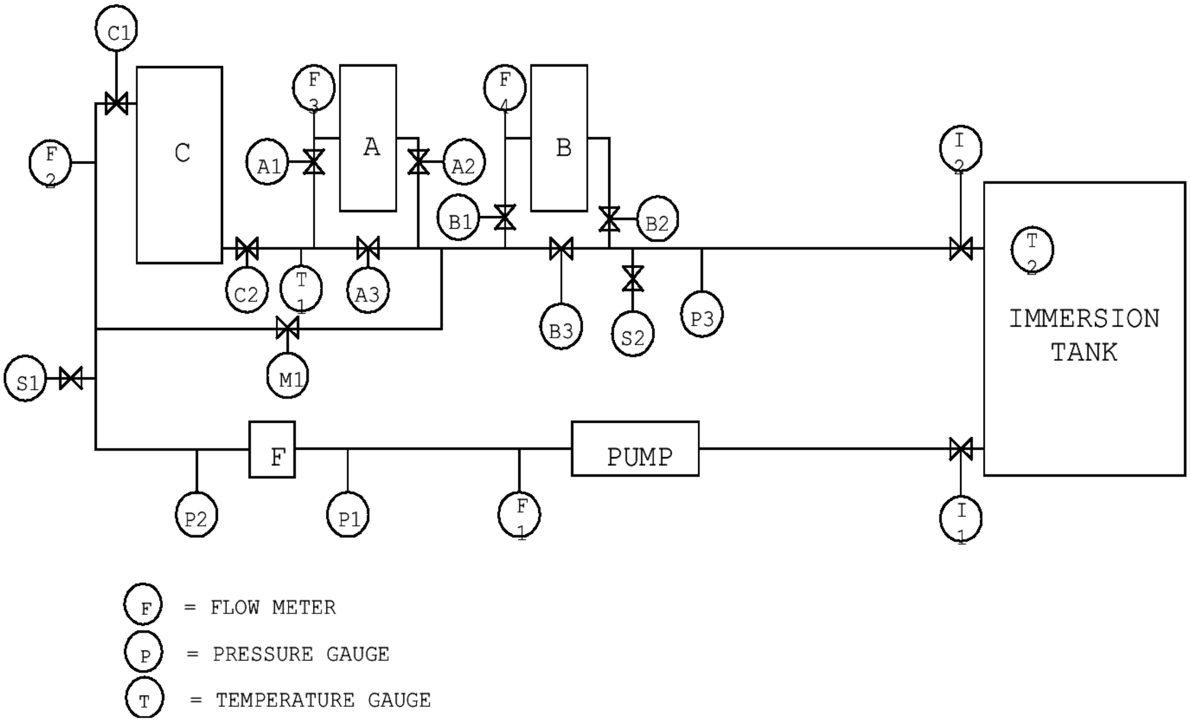

Figure A-2
Flow Diagram of EPRI DFD Process Loop

Experimental

Laboratory work predicted that a reasonable dissolution rate would be achieved at 43C, the temperature used.

In each batch of blades five blades were marked and weighed so as they could be used markers with which to follow the progress of the decontamination. For the first two pilot scale runs the 5 blades from each batch of 40 were carefully characterized pre-decontamination.

- *Appearance*: visual inspection notes made on type and location of any surface coloration or textures
- *Weight*: the blade was weighed to an accuracy of 0.1g if possible.
- *Activity*: surface activity was measured using a contamination meter, Ludlum 2224-1 Scalar/Rate meter with a 4368 gas proportional probe. General activity and any high spots were recorded.

The blades were evenly distributed about the process rack for the decontamination run. Contact between the solution and the blades in the immersion tank was improved by a proprietary contacting system.

The marked blades were removed at the end of each hour and measured for weight loss and activity loss. Any changes in surface appearance were noted. All the blades were measured for total weight before decontamination. At the end of the run, (as indicated by the results for the 5 blades), the bulk of the blades were measured for weight loss.

The surface area to weight ratios was measured for each size of blade. At the end of decontamination all blades were measured for activity. Any above free release limits were put aside and a percentage success rate calculated.

Solution samples were taken from the process skid before and after the treatment columns. The solution samples were tested for:

- gross activity [selected samples measured by gamma spec]
- pH
- aluminum
- visual clarity

Results and Discussion

Initial information indicated that uranium was the radionuclide of most concern for the purposes of recycling. Results from previous test work suggested that removal of surface metal would reduce the contamination levels to below free release. In the event there was some deviation from this, as described below.

The release limiting contamination was identified as ^{99}Tc. The contamination levels varied between blades with some blades being volumetrically contaminated. No indication could be gained by prescreening to identify volumetric contamination prior to treatment. The differences in contamination are thought to be a result of service history and post removal treatments. The volumetrically contaminated blades resulted from previous re-smelting of the aluminum to produces new replacement blades.

Uranium was present in small amounts on the surfaces of the majority of blades and was removed quickly, as was demonstrated by gamma and alpha measurements.

Once the ^{99}Tc is removed from the blade surface it is present in solution in the anionic pertechnetate form TcO_4^-.The process parameters were re-configured to concentrate on removal of anionic species as well as the expected cationic species. To achieve a 100% treatment rate when using only the smaller anion column, the total volume of DFD solution was reduced by lowering the level in the process tank by approximately 50%. This in consequence meant a reduction in the number of blades treated in each batch from 40 to 20.

The process chemistry used was EPRI DFD Lite (HBF4:HNO3, 70%:30%) the corrosion rates were as predicted and the radioactive contamination was removed efficiently to allow free release levels to be achieved.

The behavior of ^{99}Tc can be unpredictable in systems with newly exposed metal surfaces. However, control of the solution oxidation potential maintained the technetium in a soluble anionic form easily removed on anion exchange resin. The EPRI DFD solution remained very low in contaminant concentrations throughout the test period - an important condition to avoid recontamination. The importance of the recontamination was demonstrated on two occasions, once when there was no anion treatment and in an incident of inadvertent rinsing of clean blades with contaminated wash water. In both cases the metal surfaces became recontaminated above free release levels.

Surface smear tests demonstrated that at any one time only a small proportion of the activity was in the hydrated oxide layer on the metal surface. This indicated that the active species were being removed, into solution, simultaneously with the metal corrosion product. At ambient or raised temperatures (34C) there was no selective enrichment of the surface with radioactive contaminants. These conditions lead to efficient decontamination with only minimal metal removal required from the surface.

In the first batch of blades only 20% were released. This was because of the initial system configuration which had only a small anion treatment rate. The low success rate was attributed to the recontamination of the blade surfaces by ^{99}Tc. The system was reconfigured for the remaining batch trials as described above. The release rate improved to 72% in the final runs when the temperature, treatment rate and depth of metal removed had been optimized. The parameters used were 110^{0}F, 17microns per hour removal rate and 50 to 80 microns removed.

Metal removal was only 1-2% by weight for free release levels to be achieved. The percentage weight of metal removed is dependent on the surface area to volume ratio of the object. The results obtained were despite the blades representing a worst case for this ratio. Average surface area to volume ratio for the blades cleaned was 1.2 cm^{2} per gram. The situation is very much more favorable for objects such as the rotors or stator casings of the compressor units.

Waste Issues

The DFD process generates active ion exchange resin. In some instances this is the final waste form, the resin being de-watered and placed in a high integrity container for disposal. Alternatively the radioactivity can be regenerated from the resin to produce a low volume waste which can be solidified. In this case the ^{99}Tc regenerated from the anion resin was mixed with the aluminum regenerated from the cation resin. The regenerant mixture was then neutralized to produce an aluminum hydroxide precipitate, which carried the ^{99}Tc with it. The precipitate was de-watered and solidified.

Laboratory Trials on Aluminium Blade Coupons.

A number of core samples were removed from blades by DRS and sent to the Bradtec laboratory for decontamination.

The cores were 1 3/8” (3.3cm) in diameter with a central hole of ¼”(0.6cm) diameter. Figure A-3 shows the samples before and after decontamination. As can be seen from the figure the visible surface contamination is variable.

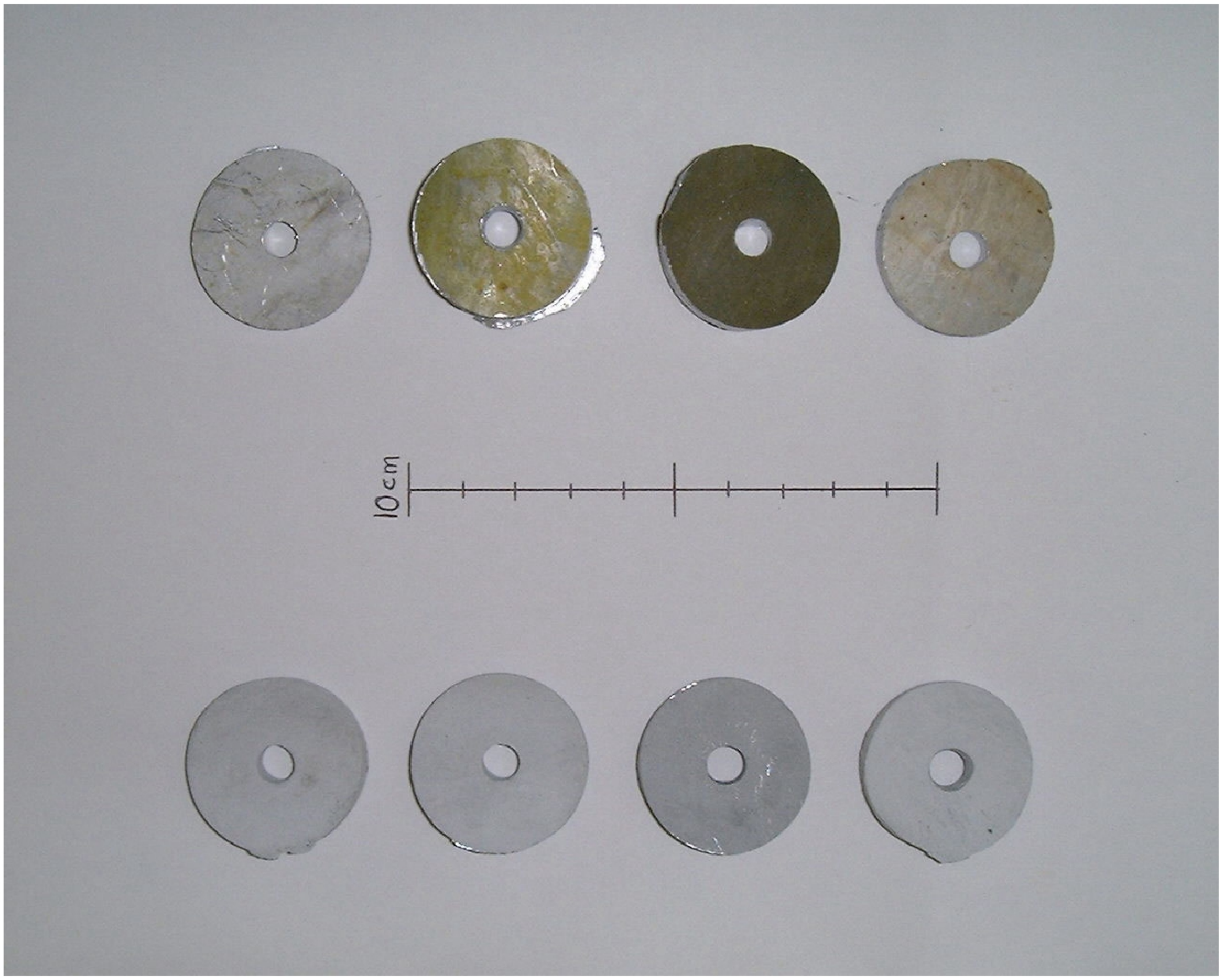

Figure A-3
Aluminium Coupons before and after Decontamination.

A selection of coupons were decontaminated for one hour as an initial screening trial. The temperature of the system rose to 115°F (46°C) and had a linear flow velocity of 1 ft sec^{-1} (0.3m sec^{-1}). Table A-1 gives the results for the initial 60 minute decontamination. The contamination on both sides of the blade has been recorded as “concave” and “convex” sides. The recycle limit is less than 5000 DPM $100cm^{-2}$ (Bkg=background).

Table A-1
Initial 60 minute Decontamination

Sample ID	START DPM 100cm^{-2}		FINISH DPM 100cm^{-2}		microns Removed
	Concave	Convex	Concave	Convex	
A1.3	188,328	247,800	Bkg	Bkg	112
A2.2	297,360	272,580	Bkg	Bkg	98
A2.3	140,420	348,128	16,520	16,520	117
A3.1	18,172	23,128	Bkg	Bkg	119
A3.2	396,480	495,600	8,000	33,000	96
A4.3	23,128	21,476	Bkg	Bkg	89
R1	109,000	90,860	Bkg	Bkg	75
R2	247,800	231,280	29,736	24,780	90

Further testing was performed removing samples after 20-minute intervals for measurement and replacing them if they were above recycling limits. Table A-2 summarizes the results.

Table A-2
Decontamination for 20 minute Intervals

Sample ID	DPM 100cm^{-2} START		DPM 100cm^{-2} FINISH		microns Removed	Duration Minutes
	Concave	Convex	Concave	Convex		
A1.1	41,300	4,300	Bkg	Bkg	20	20
A2.1	36,346	21,476	Bkg	Bkg	25	20
A4.1	8,260	8,260	Bkg	Bkg	23	20
A1.2	57,820	13,216	Bkg	Bkg	39	40
A4.2	99,920	9,912	Bkg	Bkg	42	40
A3.3	330,400	289,100	3,304	16,500	125	80

One sample suspected of volumetric contamination was given a longer exposure. The results are in Table A-3.

Table A-3
Suspected Volumetrically Contaminated Blade.

DPM 100cm^{-2} Concave	DPM 100cm^{-2} Convex	Microns Removed	Duration, Minutes
19,824	31,388	0	0
18,998	14,040	104	60
16,520	16,520	197	120
8,260	8,260	283	180
8,260	8,260	405	280

Conclusions

- The main contaminant controlling the blade release for recycle is ^{99}Tc.
- ^{99}Tc is removed as the anionic form TcO_4^- and requires 100% process flow through the anion column to prevent recontamination on the aluminium surfaces.
- Surface contaminated blades were successfully decontaminated using the EPRI DFD Lite reagent.
- The contamination levels of the blades tested was very variable.
- Over 70% of the blades processed were able to be recycled.

B
MATERIAL SAFETY DATA SHEETS

Material Safety Data Sheet

Product: **OXALIC ACID**

Hazard Class: **NR UN No CAS No 6153-56-6**

Physical Data

Description: Colorless crystals

M Pt(deg C) 101 **B Pt(deg C)** n/a **Specific Gravity** n/a

Solubility in water soluble

Vapor pressure n/a **mmHg at Deg C**

Vapor density n/a **(air=1)**

Fire and Explosion Hazard Not applicable

Flash point (deg C) n/a

Explosive limits (%): **lower** n/a, **upper**

Auto-ignition temperature (deg C) n/a

Firefighting measures Not applicable

Health Hazard

Harmful by ingestion and if inhaled as dust, irritating to skin and eyes. May cause burns and dermatitis if contact is prolonged. If ingested causes severe internal pain followed by collapse.

Toxicity data LD50 375 mg/kg oral, rat

Carcinogenicity No evidence of carcinogenic properties

Mutagenicity/Teratogenicity Evidence of reproductive effects

Exposure limits OES,mg/m3 1 **(Long-term, 8 hour TWA)**

First Aid

Eyes:	Irrigate thoroughly with water for at least 10 minutes. Obtain Medical Attention.
Lungs:	Remove from exposure, rest and keep warm. In severe cases Obtain Medical Attention.
Skin:	Wash off skin thoroughly with water. Remove contaminated clothing and wash before re-use. In severe cases Obtain Medical Attention.
Mouth:	Wash out mouth thoroughly with water and give plenty of water to drink. Obtain Medical Attention.

Reactive Hazards

Stability stable

Reaction with water none

Other known hazards Mixtures with sodium chlorite can explode on the addition of water.

Avoid contact with :	**Water** (no)	**Acids** (no)	**Bases** (no)
	Oxidizers (yes)		**Combustibles** (no)

Spillage Disposal

Precautions Wear appropriate protective clothing

If local regulations permit, mop up with plenty of water and run to waste, diluting greatly with running water. Otherwise transfer to container and arrange removal by disposal company. Wash site of spillage thoroughly with detergent and water.

For large spillages liquids should be contained with sand or earth and both liquids and solids transferred to salvage containers. Any residues should be treated as small spillages.

If material has entered surface drains it may be necessary to inform local authorities, including fire services if flammable.

Protective Measures as appropriate to quantity handled

Respirator	Dust respirator
Ventilation	Extraction hood
Gloves	Rubber or plastic
Eye protection	Goggles or face shield
Other measures	Plastic apron, sleeves, boots - if handling large quantities

Storage and Handling

Special requirements none

Material Safety Data Sheet

Product: **Tetra-Fluoroboric Acid**
(about 50%)

Hazard Class: 8 **UN No** 1775 **CAS No** 16872-11-0

Physical Data

Description: Colorless liquid

M Pt(deg C) n/a **B Pt(deg C)** n/a **Specific Gravity** 1.31

Solubility in water miscible in all proportions

Vapor pressure n/a **mmHg at Deg C**

Vapor density n/a **(air=1)**

Fire and Explosion Hazard May evolve toxic fumes in fire

Flash point (deg C) n/a

Explosive limits (%): **lower** n/a, **upper**

Auto-ignition temperature (deg C) n/a

Firefighting measures Not applicable

Health Hazard

Causes burns to eyes and skin. Extremely irritating to respiratory system. If ingested causes severe internal irritation and damage.

Toxicity data no data

Carcinogenicity No evidence of carcinogenic properties

Mutagenicity/Teratogenicity No evidence of mutagenic or teratogenic effects

Exposure limits OES,mg/m3 2.5 F **(Long-term, 8 hour TWA)**

First Aid

Eyes:	Irrigate thoroughly with water for at least 10 minutes. Obtain Medical Attention.
Lungs:	Remove from exposure, rest and keep warm. In severe cases or if exposure has been great, Obtain Medical Attention.
Skin:	Drench the skin thoroughly with water. Remove contaminated clothing and wash before re-use. Unless contact has been slight, Obtain Medical Attention.
Mouth:	Wash out mouth thoroughly with water and give plenty of water to drink. Obtain Medical Attention.

Reactive Hazards

Stability stable

Reaction with water none

Other known hazards Can react violently or explosively with acetic anhydride.

Avoid contact with :	**Water** (no)	**Acids** (no)	**Bases** (yes)
	Oxidizers (no)		**Combustibles** (no)

Spillage Disposal

Precautions Wear appropriate protective clothing

Spread soda ash liberally over the spillage. If local regulations permit, mop up cautiously with plenty of water and run to waste, diluting greatly with running water. Otherwise transfer to container and arrange removal by disposal company. Wash site of spillage thoroughly with water.

For large spillages liquids should be contained with sand or earth and both liquids and solids transferred to salvage containers. Any residues should be treated as small spillages.

If material has entered surface drains it may be necessary to inform local authorities, including fire services if flammable.

Protective Measures as appropriate to quantity handled

Respirator	Self-contained breathing apparatus
Ventilation	Fume-cupboard
Gloves	Rubber or plastic
Eye protection	Goggles or face shield
Other measures	Plastic apron, sleeves, boots - if handling large quantities

Storage and Handling

Special requirements none

Material Safety Data Sheet

Product: **Potassium Permanganate**

Hazard Class: 5.1 **UN No** 1490 **CAS No** 7722-64-7

Physical Data

Description: Dark purple crystals

M Pt(deg C) 240d **B Pt(deg C)** n/a **Specific Gravity** 2.70

Solubility in water soluble

Vapor pressure n/a **mmHg at Deg C**

Vapor density n/a **(air=1)**

Fire and Explosion Hazard May ignite combustible material

Flash point (deg C) n/a

Explosive limits (%): **lower** n/a, **upper**

Auto-ignition temperature (deg C) n/a

Firefighting measures Water spray

Health Hazard

Harmful by ingestion and if inhaled as dust, causing nausea, vomiting and kidney damage. Extremely irritating to eyes and irritating to skin. Solutions have a caustic effect.

Toxicity data LD50 1090 mg/kg oral, rat

Carcinogenicity No evidence of carcinogenic properties

Mutagenicity/Teratogenicity No evidence of mutagenic or teratogenic effects

Exposure limits OES,mg/m3 5 Mn **(Long-term, 8 hour TWA)**

First Aid

Eyes:	Irrigate thoroughly with water for at least 10 minutes. Obtain Medical Attention.
Lungs:	Remove from exposure, rest and keep warm. In severe cases Obtain Medical Attention.
Skin:	Wash off skin thoroughly with water. Remove contaminated clothing and wash before re-use. In severe cases Obtain Medical Attention.
Mouth:	Wash out mouth thoroughly with water and give plenty of water to drink. Obtain Medical Attention.

Reactive Hazards

Stability stable if kept away from combustible materials

Reaction with water none

Other known hazards

Spontaneously flammable in contact with glycerol and with ethanediol. Can react vigorously or explosively with acetic acid, acetic anhydride, ammonia/sulphuric acid mixtures, ammonium nitrate, antimony, arsenic, hydrogen peroxide, hydroxylamine, phosphorus, sulphur or titanium powder.

Avoid contact with : **Water** (no) **Acids** (no) **Bases** (no)

Oxidizers (no) **Combustibles** (yes)

Spillage Disposal

Precautions Wear appropriate protective clothing

If local regulations permit, transfer spillage into containers of water, stir to dissolve or suspend and run to waste, diluting greatly with running water. Otherwise mix with wet sand, transfer to container and arrange removal by disposal company. As contact with any oxidant can render organic matter (paper, wood, textiles) dangerously combustible, wash area of spillage and contaminated clothing thoroughly with water.

For large spillages liquids should be contained with sand or earth and both liquids and solids transferred to salvage containers. Any residues should be treated as for small spillages.

If material has entered surface drains it may be necessary to inform local authorities, including fire services if flammable.

Protective Measures as appropriate to quantity handled

Respirator	Dust respirator
Ventilation	Extraction hood
Gloves	Rubber or plastic
Eye protection	Goggles or face shield
Other measures	Plastic apron, sleeves, boots - if handling large quantities

Storage and Handling

Special requirements

In accordance with health and safety guidance notes. Store away from combustible materials.

Target:
Nuclear Power

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