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Testing and Evaluation of Eight Decontamination Chemicals

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ABSTRACT

This report details the testing and evaluation of eight decontamination chemicals. Tests were conducted with SIMCON (simulated contamination) coupons under controlled conditions to compare cleaning effectiveness, overall corrosion potential for plant equipment, interim waste generation and final waste generation.

CONTENTS

ACKNOWLEDGEMENTS	iii
ABSTRACT	v
ACRONYMS	x
1.0 INTRODUCTION	1
2.0 EXPERIMENTAL METHODS	2
3.0 DISCUSSION OF RESULTS	3
3.1 TURCO 4502	4
3.2 Nitric Permanganate	5
3.3 Ascorbic, Citric and Oxalic (Organic) Acids	7
3.4 Nitric Acid-Hydrofluoric Acid	8
3.5 Fluoroboric acid	9
3.6 Thermally Unstable Complexing Solutions (TUCS)	10
3.7 Aluminum Nitrate	11
4.0 RESULTS AND SUMMARY	11
5.0 RECOMMENDATION	12
6.0 REFERENCES	12

SUMMARY

This report covers experimental work comparing eight different decontamination chemicals. Seven of these chemicals have some novelty, or are not currently in use at the ICPP. The eighth is a common ICPP decontamination reagent used as a baseline for effective comparison. Decontamination factors, waste generation values, and corrosion rates are tabulated for these chemicals. Recommendations are given for effective methods of non-sodium or low-sodium decontamination chemicals.

The two most effective chemicals for decontamination found in these test were a dilute hydrofluoric and nitric acid (HF/HNO_3) mixture and a fluoroboric acid solution. The fluoroboric acid solution (1 molar) was by far the most effective decontamination reagent, but suffered the problem of generating significant final calcine volume. The HF/HNO_3 solution performed a very good decontamination of the SIMCON coupons while generating only small amounts of calcine volume. Concentration variables were also tested, and optimized for these two solutions.

Several oxidation/reduction decon chemical systems were also tested. These systems were similar to the TURCO 4502 and TURCO 4521 solutions used for general decontamination at the ICPP. A low sodium alternative, nitric acid/potassium permanganate, to the "high sodium" TURCO 4502 was tested extensively, optimized and recommended for general ICPP use. A reductive chemical solution, oxalic acid/nitric acid was also shown to have significant advantages.

ACRONYMS

DF (Df)	Decontamination Factor
ICPP	Idaho Chemical Processing Plant
NWCF	New Waste Calcine Facility
SIMCON	Simulated Contamination
WINCO	Westinghouse Idaho Nuclear Company
XRF	X-ray Fluorescence
mpy	mills per year (corrosion)

Testing and Evaluation of Eight Decontamination Chemicals

1.0 INTRODUCTION

The Idaho Chemical Processing Plant (ICPP) Decontamination Development Group has the task of evaluating and assisting in implementing new decontamination techniques for waste minimization and effectiveness. For the past two decades, the Decontamination Development Group has tested various methods of decontamination for the ICPP and supported decontamination projects in the many processes. Since process equipment was typically flushed to remove radioactive contamination prior to major repair work, evaluations were primarily focused on chemical decontamination methods. There has been extensive work at the ICPP during the '70s and '80s on chemical decontamination testing (including electropolishing) with an emphasis placed on effective decontamination of ICPP first cycle process equipment.¹

Current, emphasis is on minimization of chemical decontamination waste because of restrictions on the use of hazardous chemicals and ICPP waste handling issues. The use of chemical decon methods usually entails the generation of large quantities of secondary (in addition to the contaminant and substrate) waste. The increased regulation and concern about secondary waste has caused many nuclear facilities to abandon many of their former chemical decon methods. At the ICPP, this emphasis has taken the form of research and application of several nonchemical based decon methods (light ablation, CO₂ pellet blasting and liquid abrasive blasting) and also novel chemical flushing methods.

Another reason for investigating novel decontamination methods was the difficulty of processing typical decontamination liquid waste (containing sodium and potassium salts) through the waste calcining facility. "Sodium waste" requires large amounts of chemical additives to produce a satisfactory calciner product. Past decontamination and solvent recovery activities at the ICPP have resulted in the accumulation of 1.5 million gallons of radioactively contaminated liquid waste (referred to as sodium-bearing waste due to the high sodium or potassium content of the waste). Future decontamination activities at the ICPP could result in the production of over 1 million gallons of sodium-bearing waste using the current decon techniques of chemical/water flushes and steam jet cleaning. This waste requires a large amount of cold chemical additive to process because the low melting temperature of sodium and potassium salts cause calciner bed agglomeration.

The ICPP Decontamination Development Group is working to evaluate and introduce new methods of decontamination to solve the problems of sodium and secondary waste generation. The Decontamination Development Group has performed on-site demonstration/ evaluation activities at the ICPP on CO₂ pellet blasting, liquid abrasive grit blasting and novel chemical flushing. Additional off-site testing sponsored by, or in cooperation with, the ICPP have resulted in the testing/evaluation of light ablation decontamination, liquid nitrogen blasting, CO₂ snowflake blasting and improved concrete scabbling equipment. The first portions of this report are devoted to the reporting of non-chemical decon methods and novel chemical testing is reported in the final section.

2.0 EXPERIMENTAL METHODS

An experimental method was chosen to allow a reasonable comparison of the effectiveness of the decon chemicals while limiting variables. To facilitate this, a test involving SIMCON 2, a simulated contaminant matrix on stainless steel coupons², was devised to compare these criteria. All chemicals were tested under the following conditions:

- Approximately 50 ml of solution was used.
- Solutions were stirred continuously during the tests.
- Solutions were heated to 55° C for a duration of 3 hours.

Two SIMCON 2 coupons were used in each test of a given chemical. Coupons were placed in a 150 ml beaker on a hotplate in the solutions under the above conditions for the full duration of the test, then rinsed with distilled water, and left to air dry. The chemicals tested included: TURCO 4502, organic acids, nitric acid/oxalic acid, nitric permanganate, hydrofluoric acid/nitric acid, TUCS, fluoroboric acid and aluminum nitrate.

The coupons were analyzed for removal of the SIMCON contaminants using X-ray fluorescence (XRF). Results on the amount of SIMCON remaining after preparation (prior to chemical decontamination) were compared to the results after cleaning. A decontamination factor (Df) was calculated from the ratio of the before analysis to the after analysis. The two analytes used on the SIMCON 2 were removed at differed rates. In general, cesium was easier to remove than the zirconium; therefore, Df results are reported by element.

Additional tests to those described above were conducted to evaluate the temperature and solution concentration dependence effects. Temperatures were varied from 20°C to 80°C for the nitric acid/potassium permanganate solution. The concentrations of acid, permanganate, aluminum nitrate and organic acids were

also varied.

Corrosion tests were performed using Polarization Admittance Inductive Resistivity (PAIR) instrumentation. The PAIR instrumentation is standard metal corrosion rate instrumentation which uses three metal probes of the same composition as equipment of interest. Particular metals of interest at the ICPP were 304 Stainless Steel and Nitronic 50. Waste generation data is calculated using ASPEN process modeling software. Modeling software generated relative calcine volumes for the individual chemicals that were tested.^{3,4}

3.0 DISCUSSION OF RESULTS

This section is divided into discussions of the various chemicals used in the tests. Table 1 describes the overall results of the comparison of decontamination factors, waste generation and corrosivity. This information allows the projection of not only the benefits of using a particular reagent, but also the deterrents in terms of corrosion and waste. Corrosion can be a major factor for selecting decon reagents. Uncontrollable corrosion could cause significant harm to process equipment; not only the equipment to be decontaminated, but equipment downstream (holding vessels, evaporators, etc). Waste generation is not as straightforward as it initially seems. For example, the use of sodium and potassium salt based decon reagents (typically alkaline KMnO_4) yields excessive calcine. Also, many chemicals (hydrofluoric acid) should be complexed with other reagents (aluminum nitrate) after their use. This complexation could result in significant volume increases.

Table 1, Laboratory Chemical Decontamination Studies.

Chemical Reagent	Df S2-Cs	Df S2-Zr	Corrosion (mpy)	evaporated volume(m ³)	calcine volume(m ³)
Turco 4502	5.5	(0)	90	0.45	0.34
nitric permanganate	2.8	1.1	100	0.0125	0.019
aluminum nitrate	1.8	1.7	*	0.273	0.23
organic acids	3.1	1.6	0.1	0.26	0.07
nitric/oxalic	4.5	1.3	0.1	0.67	0.18
TUCS	4.8	3.3	*	1	0.27
nitric 800ppm hydrofluoric	6.1	13.8	0.08	0.0525	0.0058
fluoroboric acid	12.6	37.2	0.1	2	0.26

* This data is not available.

3.1 TURCO 4502

TURCO 4502 is a commercially available decon chemical that is marketed by the TURCO Company of Cincinnati, Ohio. It is one of a suite of chemicals marketed under the "TURCO DECON" umbrella. These products were specifically tailored to the cleaning and decontamination needs of the nuclear industry. TURCO has since dropped their TURCO DECON line, but specially mixed chemicals (like TURCO 4502) are still available upon request. Its inclusion in this report is as a baseline, or a historical benchmark to compare with new techniques.

This chemical is in a broad family termed "AP", for alkaline potassium permanganate, that are used extensively in nuclear decontamination. "AP" chemicals contain sodium hydroxide or potassium hydroxide (5 to 20%) and potassium permanganate (1 to 5%), are typically strongly basic and oxidizing.⁵ This allows them to disrupt and dissolve metal surface films according to the equation:

The HCrO_4^- is more soluble than Cr_2O_3 . Disrupting the metal surface film releases the trapped radionuclide particles and decontaminates the metal.⁶ This is often the first step of the two step alkaline permanganate/oxalic acid process that uses oxalic acid ($\text{C}_2\text{H}_2\text{O}_4$) (or a suitable reductant) to reduce iron oxide in the second step, disrupting the strongly held oxide according to the reaction:

Cycling the surface film chemistry in this fashion, through oxidation/reduction steps, has been demonstrated to be very effective in decontamination.⁷ Additional discussion of reducing organic acids is given in section 3.3.

A test of TURCO 4502 was conducted in the ICPP laboratories to determine the effectiveness at cleaning SIMCON 2 test coupons. The TURCO Company recommends use of TURCO 4502 at a concentration of 2 lb. per gal.⁸ ICPP usage has indicated that a quarter this concentration has performed satisfactorily.⁹ The solutions were therefore mixed to a concentration of 1/2 lb. per gal. for bench top testing. The results indicated that while the TURCO 4502 was moderately successful in removing cesium from the SIMCON 2 coupon, it was unsuccessful in removing zirconium. This probably stems from TURCO's oxidizing nature, while reducing chemistries and complexing chemistry (fluoride, ZrF_3^+) were typically much better at removing zirconium. TURCO 4502 had the lowest Df for zirconium that was encountered in the testing.

Corrosion factors and waste generation were also generally unfavorable for TURCO 4502. The corrosion factor of 90 mpy (short term corrosion, long term the metal is passivated and less corrosion occurs) is the second highest of those chemicals tested. This could potentially cause corrosion problems downstream of the intended vessel, but, in practice, the reducing solution is mixed with the oxidizer after the decon, so the TURCO 4502 is essentially neutralized.

As mentioned earlier, using concentrated sodium and potassium chemicals causes waste generation problems in the ICPP calcination process. Nonradioactive additives (predominantly aluminum nitrate) must be added to prevent sodium/potassium agglomeration of the calcine bed. A volume ratio of approximately 1 volume of AP to 4 volumes of aluminum nitrate must be used. This addition results in the high volumes of waste for to this reagent.

3.2 Nitric Permanganate

Traditional alkaline permanganate (AP) decon solutions are generally preferred to acidic permanganate because of their lower corrosion of metals (due to their basic nature). Corrosion remains fairly well controlled with AP solutions even when used at high concentrations. However, strongly alkaline solutions were inherently high in sodium and potassium hydroxide, resulting in high waste volumes. For this reason, adapting the oxidizing chemistry of the alkaline permanganate reagents to a nitric acid system could offer large benefits.

Nitric acid-potassium permanganate (NP) decon solutions have been tried in the nuclear industry with notable success. Decontamination factors for the NP system at the Ringhals 2 (Sweden) nuclear reactor system were 6.4 to 7.3, while AP Df stood at 1.9 to 2.3.⁶ Decon solutions of AP and NP are also often used with reductive solutions such as low oxidation metal ion (LOMI) processes. LOMI decontamination campaigns show little difference in well managed NP and AP decontaminations.

Laboratory tests were undertaken to compare the effectiveness of the NP chemistry. Table 1 documents the decontamination factors, corrosion and waste volumes for the 0.5 M HNO_3 · 0.005 M KmnO_4 system. Generally good results were achieved for this decon system, with over 60% of the cesium and about 12% of the zirconium removed. The cesium values, while not as high as TURCO 4502, compare well, and far exceed that system in zirconium.

Using NP instead of AP results in a significant waste savings. The potassium/sodium content of the NP is 99.44 % less than that of AP. Table one shows the relative volumes of calcine waste generated by AP (0.34) compared to

NP (0.019); both would be subject to accounting for the additional amount of waste from a reductant (oxalic acid, 0.18).

Optimization of the nitric acid concentration and permanganate concentration was undertaken for the NP system. Due to its ready acceptability in the process waste systems, this system is of particular interest to process support personnel. A primary concern, though, was its harsh, corrosive nature. At the original concentrations, 0.5 M HNO_3 · 0.05 M KMnO_4 , the PAIR instantaneous corrosion measurement was about 500 mpy. Subsequent tests showed that a potassium permanganate concentration of 0.005 M should be sufficient for the oxidative decon step. At that concentration, the measurement for NP fell to about 100 mpy, which is close to that of the AP system. Figure 1 shows the effect of varying the nitric acid concentration. A relatively stable increase of the decontamination factor is noted for the higher nitric acid concentrations, with a small increase for cesium at 0.25 M (which may have been spurious).

Figure 1, Effect of Acid Concentration on NP/oxalic acid Decontamination System.

Additional testing was performed to check the temperature effect on the efficiency of the NP system. Temperature was varied between 20 C and 80 C. These are typical temperatures used at the ICPP. Figure 2 shows the results of these tests. Generally the trend is as expected, with an increase in decontamination with temperature. However, this increase is not large (particularly for zirconium), and could be compensated by longer residence times in the plant.

Figure 2, Effect of Temperature on NP/oxalic acid Decontamination System.

3.3 Ascorbic, Citric and Oxalic (Organic) Acids

The use of organic acids is a novel, though not unknown, approach to reductive chemical decontamination at the ICPP. Plant decontamination records document the use of tartaric, oxalic, citric and ascorbic acids. Ammonium citrate and oxalate are also commercially available for decontamination (TURCO 4521). Commercial nuclear decontamination vendors are available to apply the CITROX, CANDECON and CANDEREM processes that have been developed and patented by the AECL of New London, Ontario, Canada. These processes make use of citric and oxalic acids in low (<1%) concentrations for nuclear plant cooling system decontamination.¹⁰ In general, these acids (particularly oxalic and ascorbic acids) are primarily reductive in nature. Their effect on the metal surface film is to reduce ferrous oxide making it more soluble. They also partially complex the contaminants, making them more soluble in solution.

A solution of Ethylenediamine tetraacetic acid (EDTA), oxalic, citric and ascorbic acids at 1% concentration of each component was developed to be used in these tests. Full stability of the solutions only lasts for about 2 weeks. This is largely due to the instability of ascorbic acid, which reacts with air to become oxidized. However, this would allow for adequate storage and use. Tests were conducted with the 1% solution to determine the effectiveness. This solution was informally termed "RIXBRU".

A second organic acid system was also tested with this series. A solution of 3.5 M HNO₃ · 0.5 M oxalic acid was developed by Steve Zohner during the 1980s.¹¹ This chemical was tested in plant use at that time. It shows very good results, both in plant and in the SIMCON tests. It appears to have a general reductive property, along with a strongly acidic dissolution and complexation capability.

Table 1 shows the Df of the organic acids in removing the SIMCON 2 contaminants. The solution was slightly less effective at removing cesium, but more effective at removing zirconium than TURCO 4502. As part of a two step oxidative/reductive process, the Df of the organic acids has been found to be somewhat higher. A comparison of decontamination capability of the NP/"RIXBRU" vs NP/nitric/oxalic cleaning systems is presented on Table 2. Similar Df can be observed for these two step systems and both are a significant improvement over the effectiveness of using a single solution.

Table 2, Comparison of NP/RIXBRU vs NP/nitric-oxalic

Concentration of solutions	Cesium Df	Zirconium Df
0.55 M HNO ₃ 0.05 M KMnO ₄ , 0.5 M HNO ₃ 0.5 M oxalic acid	10.25	2.14
0.55 M HNO ₃ 0.05 M KMnO ₄ , 1% RIXBRU	5.64	2.12

3.4 Nitric Acid-Hydrofluoric Acid

The nitric acid-hydrofluoric acid solution has been examined to varying degrees for several years. Solutions of 3.5 M HNO₃ and 0.04 M HF concentrations have been used as cleaning/etching solutions on stainless steel.¹² To this end, this HNO₃-HF solution was posed as a candidate for examination as a nonsodium chemical decon reagent.

Tests using SIMCON 2 produced comparatively high decontamination factors and good overall results. A solution of 3.55 M HNO₃ · 0.04 M HF was tested in the manner described in section 2.0. Relative decontamination values can be seen in Table 1. Cesium decontamination factors were the third highest of those chemicals tested. Zirconium decon factors were almost ten times higher than the next higher chemical; second only to fluoroboric acid. Zirconium decontamination values that are higher (in this case twice as high) than those of cesium is unique to the hydrofluoric and fluoroboric acid systems. This makes sense due to the ZrF³⁺ complexing of zirconium in fluoride solutions.

While the efficacy of the HNO₃-HF solution is viewed as compelling, the waste and corrosion values are especially attractive. This solution produces significantly lower waste volume than the baseline solution (TURCO 4502). As the decontamination factor is quite a bit higher, less solution would be used and even less waste produced. Another attractive feature of this solution is that it may chemically simulate Fluorinel dissolver Process (FDP) product. This would allow higher blends of sodium waste to be processed in the calciner. Prior to the cessation of fuel reprocessing, this method of blending relieved sodium waste inventory. FDP product had a complexed (aluminum nitrate added) concentration of 0.04 M free HF. This solution actually starts with that concentration of free HF, and will likely contain aluminum nitrate to fully complex the HF. The corrosion value of the solution is low (<0.1 mpy), significantly less than that of TURCO 4502.

Unfortunately, adoption of this solution as a general/all purpose decon solution is slow in developing. Concerns over the health effects of HNO₃-HF have imposed a great barrier to it's adoption. A healthy "fear" of hydrofluoric acid has been developed in operators, engineers and support personnel at the ICPP. This stems from years of working with concentrated hydrofluoric acid in the ROVER and FAST process. While it is good that such a respect has been generated for hydrofluoric acid, it is certainly not a grave concern using low (0.04 M) concentrations of the reagent. Equally, concerns have been raised as to excessive corrosion of process equipment. Excessive corrosion has occurred in ICPP process systems using hydrofluoric acid. Laboratory tests support the claim that this is a low corrosion solution, giving a 0.08 mpy corrosion rate. This is far less than currently used decon chemicals (TURCO 4502), which can have corrosion rates measuring up to 100 mpy.

3.5 Fluoroboric acid

Fluoroboric acid (HBF₄) is an excellent decontamination reagent with extremely high decontamination factors. The HBF₄ was tested at a significantly high concentration of 1 M, which makes a potential fluoride concentration of the

solution 4 molar (equilibrium transfer of up to four fluorides per acid). The SIMCON 2 coupons showed a consistently high decontamination factor, cesium Df 12.6 and zirconium Df of 37.2 using this solution. These were the highest decontamination factors found in these series of cold chemical tests. A commercial vendor, ALARON Co., reports decontamination factors of 50 to 100 using the fluoroboric acid process.¹³ The likely cause of these high factors, particularly of zirconium which responds very well to fluoride, is the high fluoride concentration. The fluoride is mainly complexed in the HBF_4 by the boron molecule. The equilibrium of free fluoride in solution is displaced as ions on the metal surface are dissolved. The HBF_4 may then release more fluoride to dissolve ions as it is depleted in solution. The corrosion rate remained low at the 0.1 mpy level. The reagent is not a particularly strong acid as the boron inhibits the high fluoride corrosion one might expect.

The one main disadvantage to the fluoroboric acid is the high amount of waste that is generated by its use. Basically, the overwhelmingly high amount of waste produced in using this decon reagent is in complexing the high amounts of fluoride present. Under current ICPP philosophy, each fluoride molecule must be accompanied by two aluminum molecules. Not only does this produce excessive waste due to the use of eight moles of aluminum per mole of fluoroboric acid, but it doesn't take into account the presence of the boron, which of itself is an adequate complexing agent. Others argue that a complexing rate of one aluminum to three fluoride (the exact opposite of that given in ICPP manuals) is adequate. That change alone would reduce the ratio from 4 moles of aluminum to 1 mole of fluoroboric acid to 4:3. Yet to be examined is the beneficial use of this reagent for blending with sodium feed. That has a potential to decrease the overall waste production well below current levels and substantially reduce cold aluminum nitrate input into the calcine process.

A process for recycling and regenerating fluoroboric acid exists that could even decrease the amount of fluoroboric acid that goes to waste. A Swiss company, Recytec, has patented a process called "DECOHA" that electrochemically restores the acid and removes the metal and contaminants from the fluoroboric acid. This DECOHA technology has been proven in laboratory tests¹⁴ and has been placed in use at the damaged reactor at Chernobyl. While not a perfect "closed loop" system, DECOHA offers capabilities that would provide significant recycling benefits.

3.6 Thermally Unstable Complexing Solutions (TUCS)

TUCS chemical reagents offer the potential of effective decontamination solutions. Ionquest 201 is a strong complexing reagent marketed by Albright and Wilson chemical company. This is a proprietary chemical that is an organic,

phosphonic acid. It was developed to extract chemical species, notably uranium and some actinides, from nuclear processes. Used with a catalyst, it has the capability to remove contaminants from the surface of metal.

Waste handling concerns could limit the adoption of TUCS decon systems. The organic reagent waste is reduced to carbon dioxide, water, and phosphoric acid through another catalyzed reaction. Phosphoric acid detrimentally effects the complexation of fluoride. Phosphate binds the boron and aluminum causing precipitation and release of free fluoride.¹⁵

Preliminary results show that lonquest 201 has been moderately successful at removing SIMCON 2 type contaminants. Four SIMCON 2 coupons were sent to Albright and Wilson's laboratory to be tested using lonquest 201. These were then returned to the ICPP for analysis. These results, which were of a scoping nature, demonstrated average removals of 76% of the cesium and 61% of the zirconium. Albright and Wilson engineers report that 1 M lonquest 201 solutions were used in these tests.

3.7 Aluminum Nitrate

Using aluminum nitrate as a decon reagent has some advantages. In the calcination process, nonradioactive aluminum nitrate is often added to prevent agglomeration of the fluidized bed. Currently, this accounts for a significant amount of the waste generated in the calcination process. By using this reagent for some decontamination, a benefit could be derived where otherwise none exists. Tank farm projects personnel first suggested that aluminum nitrate be used to flush out the waste tank, since it then could be put into the calciner for beneficial use.

Tests were performed to verify the effectiveness of aluminum nitrate on SIMCON 2. Results of these tests demonstrated that on average, about 47% of cesium and 42% of zirconium were removed from the SIMCON 2; and that a concentration of 0.5 M aluminum nitrate was about optimum. These results show some merit, particularly from a chemical not expected to be of service in this area. As a result, it is suggested that chemical additives to the various processes, particularly calcination, be tested and used for decontamination.

4.0 RESULTS AND SUMMARY

According to the SIMCON 2 tests, novel chemical decon will be able to significantly improve decontamination efforts at the ICPP. Current practices using alkaline potassium permanganate are among the least effective, yet most wasteful of these those tested. Higher decontamination factors and significantly lower final

waste volumes are two notable improvements. Process equipment corrosion could be lower using some of the novel chemicals than that of the APAC.

The best decon reagent likely to be a compromise of several factors. Highest decontamination factors were found for the 1 M HBF_4 solution. This is likely to generate larger volumes of waste than even the TURCO 4502. Waste volume concerns roughly eliminate this solution from consideration. However, technology exists to recycle, regenerate and reuse the HBF_4 reagent, making this an attractive option if the technology could be implemented at the ICPP. Second to the HBF_4 was the HF/HNO_3 solution in decontamination factor. This is actually a dilute solution, allowing evaporation, concentration and virtually the lowest final waste volumes.

5.0 RECOMMENDATION

Novel decontamination methods, particularly HF/HNO_3 and NP type solutions should be rapidly adopted for ICPP decontamination. These solutions provided good decontamination results of the SIMCON 2 coupons, generally as well or better than TURCO 4502. In the case of the HF/HNO_3 solution, it has the additional potential effect of allowing a beneficial blend with current sodium waste. These two chemicals are also allowed under current waste processing regulations, though fluoride solutions even as dilute as 0.04 M still come under scrutiny.

Further test of these chemicals on actual contaminated metal is greatly desired. Simulated contamination tests have shown that improvements in decon flushing can be easily obtained. Initial steps to realizing these improvements has been taken, through the use of the HF/HNO_3 solution on an NWCF drain line. Another important phase is expected in October 1994 when joint tests by the NWCF decon facility and the Decontamination Development Group further the adoption of these novel chemicals

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