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**Bench Scale Testing - Phase I
Task 4**

**Topical Report
September 1994 - January 1995**

July 1995

Work Performed Under Contract No.: DE-AC21-93MC30168

U.S. Department of Energy
Office of Environmental Management
Office of Technology Development
Washington, DC

For

U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Babcock and Wilcox Company
Contract Research Division
Alliance, OH 44601

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TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
EXECUTIVE SUMMARY	1
1.0 INTRODUCTION	4
2.0 BACKGROUND/OBJECTIVES	5
2.1 Overall Program Background/Goals	5
2.2 Results - Solvent Screening/Refinement Tests	5
2.3 Specific Objectives - Bench Scale Testing	6
3.0 TEST PROGRAM	7
3.1 Approach	7
3.2 Test Apparatus	8
3.3 Test PRocedure	8
4.0 RESULTS AND DISCUSSION - PLUG DEVELOPMENT TESTS	12
5.0 RESULTS AND DISCUSSION - BENCH SCALE TESTS	13
5.1 Test #1 - EDTA/Carbonate Solvent - Initial Plug Formulation	13
5.2 Test #2 - Carbonate-Only Solvent - Initial Plug Formulation	16
5.3 Test #3 - EDTA/Carbonate Solvent - Modified Plug Formulation	19
5.4 Test #4 - Carbonate-Only Solvent - Modified Plug Formulation	22
5.5 Corrosion Results - Bench Scale Testing	25
6.0 CONCLUSIONS	26
7.0 RECOMMENDATIONS	27
8.0 REFERENCES	28

APPENDIX A - TEST PROCEDURES AND SIGN-OFF SHEETS FOR BENCH SCALE TESTS

APPENDIX B - RAW DATA FROM BENCH SCALE TESTS &
CORROSION AND MASS BALANCE CALCULATIONS

LIST OF TABLES

<u>TITLE</u>	<u>PAGE</u>
1 Dissolved Metal Analysis - Bench Scale Test #1	14
2 Dissolved Metal Analysis - Bench Scale Test #2	17
3 Dissolved Metal Analysis - Bench Scale Test #3	20
4 Dissolved Metal Analysis - Bench Scale Test #4	23
5 Corrosion Results - Bench Scale Testing	25

LIST OF FIGURES

<u>TITLE</u>	<u>PAGE</u>
1 Photograph of Bench Scale Test Loop	10
2 Schematic of Bench Scale Test Loop	11
3 Total Uranium Dissolved - DOE Bench Scale Test #1	15
4 Total Uranium Dissolved - DOE Bench Scale Test #2	18
5 Total Uranium Dissolved - DOE Bench Scale Test #3	21
6 Total Uranium Dissolved - DOE Bench Scale Test #4	24

EXECUTIVE SUMMARY

Under contract to the U. S. Department of Energy, the Babcock & Wilcox Company is developing a chemical decontamination process using chelating agents to remove uranium compounds and other actinide species from process equipment. The overall goal of the program is to develop a solvent capable of selectively chelating uranium; an additional requirement of the process is the ability to control corrosion of the base metal during decontamination. In cases where reuse of the equipment is a possibility, corrosion must be controlled at a low rate. However, if contamination has migrated into the grain boundaries of the base metal, a more aggressive solvent is required to corrode away base metal and expose radioactive species for removal. Another objective of the program is to recycle the chelating solvent, thereby minimizing the generation of secondary waste streams.

The project is divided into Phase I: "Lab Scale/Small Pilot Proof of Concept," and Phase II: "Field Demonstration." Phase I encompasses Task 1: "NEPA Study"; Task 2: "Chelate Chemistry/Process Optimization," the goal of which is to identify promising chelating solvents and optimize application parameters for maximum effectiveness; Task 3: "Solvent Recycle/Contaminant Reduction," the objective of which is to develop a process to break the chelant-metal bond and make chelant available for reuse; Task 4: "Bench Scale Tests," to evaluate the developed solvent system under dynamic test conditions; and Task 5: "Pilot Plant Study," involving application of the process to equipment actually contaminated in service. This report describes bench scale testing performed under Task 4 of the program.

The specific objective of this task of the test program was to evaluate the solvent system developed in Task 2 under dynamic conditions that more closely simulated the conditions of actual field application. Secondly, the procedures developed during this bench scale testing were to be used as the basis for procedures for the pilot plant testing planned under Task 5. Both objectives of the task were successfully accomplished.

BACKGROUND

Based on the results of the screening/refinement test program, two solvents were selected for bench scale application. The first solvent contained 20 grams per liter ammonium carbonate, pH adjusted to 9.0 with ammonium hydroxide, with hydrogen peroxide added. The second solvent was similar to the first, except it also contained the chelating agent ethylenediamine-tetraacetic acid (EDTA). Because it increased solvent corrosivity, the addition of EDTA improved the solvent's effectiveness in removal of contamination embedded in base metal grain boundaries. Thus, the chelant made the solvent more versatile, and this formulation would likely be the solvent of choice for field use.

Based on Task 2 process development testing, the application scenario chosen for bench scale testing was:

1. Two-hour exposure at room temperature (either solvent)
2. Heatup to 93°C (200°F) and hold for four hours
3. Cooldown to room temperature and inject hydrogen peroxide
4. Hold at room temperature for two hours.

The rationale for this application sequence was that readily available uranium will be dissolved during the initial low temperature exposure. The four-hour 93°C exposure will result in corrosion of base metal, thereby releasing contamination trapped in grain boundaries. The released contamination will be dissolved during the final low temperature exposure.

TEST PLUG DEVELOPMENT

The first phase of the task was development of a suitable binder for making a UO_2 test plug that would release uranium slowly throughout the dynamic exposure. Plugs consisting of various combinations of calcium carbonate, trisodium phosphate, silicon dioxide, and mortar were evaluated with both solvents. A formulation of 10 grams calcium carbonate, 10 grams silica, and 2.5 grams mortar appeared to produce a plug that deteriorated slowly throughout the test exposure. Plugs for the first portion of bench scale testing were made by adding uranium dioxide to this formulation.

Results of initial testing indicated that the plugs were more robust than testing without UO_2 in the formulation had indicated, and decomposed at a significantly slower rate than anticipated. Additional testing was performed using smaller plugs made from a formulation with an increased ratio of uranium dioxide to binder.

BENCH SCALE TESTS

The dynamic tests were performed in B&W's low temperature chemical cleaning velocity test apparatus. Initial testing consisted of two tests using the original plug formulation; one test with each of the two candidate solvents. The uranium dioxide test plugs used in these two tests did not disintegrate to the extent predicted by plug formulation testing. For this reason, some of the encapsulated uranium did not become accessible to the solvents, and, hence, was not dissolved. The EDTA/carbonate/peroxide solvent dissolved 25% of the added uranium; the solvent containing carbonate and peroxide dissolved about 18%.

As mentioned above, the plugs were then reformulated for additional bench scale testing. Two tests were run with the smaller, modified-formulation plugs; one test with each of the two candidate solvents. The EDTA/carbonate/peroxide solvent attacked the reformulated plug more aggressively than the initial formulation; 75% of the added uranium was dissolved. The carbonate/peroxide solvent dissolved only 10% of added uranium. Based on the low dissolved calcium values observed, the revised plug formulation appeared to be very resistant to this solvent.

Corrosion losses observed on test specimens in all four tests were uniform, with no localized attack evident. The application of the carbonate/peroxide solvent resulted in slight corrosion of a Monel coupon in one of the tests, but not the other; no measurable corrosion occurred for carbon steel or nickel during use of this solvent. Application of the EDTA-based solvent resulted in corrosion of all three materials, ranging from an average of 0.2 mils for Monel 400, 0.3 mils for Nickel 200, and 0.4 mils for carbon steel. It was shown that the addition of chelant will be required to effect consistent base metal corrosion for release of contamination below the surface of process equipment being decontaminated.

CONCLUSIONS

The conclusions drawn from Task 4 bench scale testing were as follows:

- (1) The configuration of the test loop was adequate to apply and control the process. It is important to have on-line cooling capabilities during low-temperature portions of the application, especially during the final H_2O_2 spike, when dissolved metal ions present will catalyze exothermic peroxide decomposition reactions.
- (2) The test plugs used in the study shielded some of the uranium dioxide from the applied solvent. It appeared that the UO_2 that was accessible to the solvent did dissolve, mainly during the initial low-temperature step.
- (3) Essentially no corrosion occurred without EDTA being present in the solvent formulation; thus, chelant will be required to effect base metal corrosion for release of embedded contamination. The EDTA also appeared to stabilize the uranium dissolved in the process, preventing it from dropping out of solution during the cooldown phase of the exposure.
- (3) Hydrogen peroxide concentration was identified as an important control parameter during Task 2 testing. The necessity for having an adequate analytical technique for performing real-time peroxide analysis was further demonstrated during bench scale testing.
- (4) The procedures developed for bench scale testing were suitable to be used as the basis for the pilot plant testing planned under Task 5.

RECOMMENDATIONS

It is recommended that both the EDTA/carbonate/peroxide and carbonate/peroxide solvents continue to be evaluated in the pilot plant phase of this program. Development of an analytical method for hydrogen peroxide that can provide real-time results is also recommended; this should be accomplished prior to field demonstration of the process.

1.0 INTRODUCTION

The Department of Energy (DOE) is now faced with the task of meeting decontamination and decommissioning obligations at numerous facilities by the year 2019. Due to the tremendous volume of material involved, innovative decontamination technologies are being sought that can reduce the volumes of contaminated waste materials and secondary wastes requiring disposal. With sufficient decontamination, some of the material from DOE facilities could be released as scrap into the commercial sector for recycle, thereby reducing the volume of radioactive waste requiring disposal. Although recycling may initially prove to be more costly than current disposal practices, rapidly increasing disposal costs are expected to make recycling more and more cost effective (Reference 1). Additionally, recycling is now perceived as the ethical choice in a world where the consequences of replacing resources and throwing away reusable materials are impacting the well-being of the environment.

Current approaches to the decontamination of metals most often involve one of four basic process types: (1) chemical, (2) manual and mechanical, (3) electrochemical, and (4) ultrasonic (Reference 2). "Hard" chemical decontamination solutions, capable of achieving decontamination factors (Df's) of 50 to 100, generally involve reagent concentrations in excess of 5%, tend to physically degrade the surface treated, and generate relatively large volumes of secondary waste. "Soft" chemical decontamination solutions, capable of achieving Df's of 5 to 10, normally consist of reagents at concentrations of 0.1 to 1%, generally leave treated surfaces in a usable condition, and generate relatively low secondary waste volumes. Under contract to the Department of Energy, the Babcock & Wilcox Company is developing a chemical decontamination process using chelating agents to remove uranium compounds and other actinide species from process equipment.

The project is divided into Phase I: Lab Scale/Small Pilot Proof of Concept, and Phase II: Field Demonstration. Phase I activity under Task 1: "NEPA Study," Task 2: "Chelate Chemistry/Process Optimization," the goal of which is to identify promising chelating solvents and optimize application parameters for maximum effectiveness, and Task 4: "Bench Scale Tests," to evaluate the developed solvent system under dynamic test conditions, has been completed. Remaining Phase I activity includes Task 3: "Solvent Recycle/Contaminant Reduction," the objective of which is to develop a process to break the chelant-metal bond and make chelant available for reuse, and Task 5: "Pilot Plant Study," involving application of the process to equipment actually contaminated in service. This topical report describes the technical approach being taken to achieve project objectives, and presents the results of bench scale testing performed under Task 4 of the program.

2.0 BACKGROUND/OBJECTIVES

2.1 Overall Program Background/Goals

This work is being performed in response to DOE's request for research on methodologies for the decontamination of process equipment. At the time of project initiation, DOE offered equipment from the gaseous diffusion plant at Oak Ridge, Tennessee, for use in the full scale demonstration of the developed technology. For this reason, the selected technical approach was to develop a process suitable for application to process equipment contaminated in this manner.

Internal components of equipment utilized in the uranium enrichment process were in contact with uranium hexafluoride (UF_6) gas at elevated temperatures over many years of operation. It is suspected that inleakage of air during operations and after plant shutdown have resulted in deposits of other uranium compounds, possibly UO_2F_6 and UF_4 . Additionally, occasional outleakage of process gas has left small radioactive deposits on external surfaces.

The compressor internals are expected to not only have surface contamination, but, due to long exposures at high temperatures, it is anticipated that the radioactive contaminants have penetrated the grain boundaries of the base metal. The Allis Chalmers centrifugal compressors in the K-27 building typically have housings constructed of carbon steel with less than 0.001 inch of internal nickel plating. The rotors are typically made of Monel; the shafts, of carbon steel.

DOE's preliminary evaluation of the potential for recycling the compressor materials was that some equipment parts, such as the compressor base and rotor shaft, might be surface contaminated only, and, as such, releasable for unrestricted use under existing guidelines (eg. NRC Regulatory Guide 1.86, DOE Order 5400.5). Other parts, such as the compressor housing, rotor, and piping, were expected to be bulk contaminated with radionuclides, preventing unrestricted release and making the equipment attractive for reuse within the DOE complex. Requirements of the decontamination technology applied to the compressors were: (1) removal of as much contamination as possible in a cost effective manner, (2) minimization of the volume of secondary waste generated, and (3) minimization of worker exposure. The technical approach and test plan for this program were developed in accordance with this information.

2.2 Results - Solvent Screening/Refinement Tests

During Task 2 of this program, a large number of candidate solvents were evaluated for uranium dioxide dissolution and base metal corrosion response (Reference 3). The combination of chelant, ammonium carbonate, and hydrogen peroxide at a pH of 9.0 (adjusted with ammonium hydroxide) was found to effect virtually 100% dissolution of the uranium dioxide placed in the test flask, within the first two hours of exposure. The solvent was effective in uranium dissolution at room temperature. Based on the results of the screening and refinement test program, two solvent systems were concluded to provide optimum uranium dioxide dissolution:

- (1) EDTA, ammonium carbonate, and hydrogen peroxide: pH 9.0 with NH_4OH
- (2) ammonium carbonate and hydrogen peroxide: pH 9.0 with NH_4OH .

For the remainder of this report, these two formulations will be referred to as the (1) EDTA/carbonate solvent, and (2) carbonate-only solvent, respectively.

Testing during Task 2 of this program indicated that the basic solvent systems were effective over a wide range of concentrations. This can allow for tailoring the solvent for differing levels of contamination and also for minimization of chemical usage.

Based on the Task 2 testing, the recommended application scenario for the process is as follows:

1. 2 Hours at room temperature with either solvent
2. Heatup to 93°C and hold for approximately 4-hours
3. Cooldown to room temperature
4. Inject peroxide to remove additional uranium contamination uncovered by the high temperature exposure
5. 2 Hours at room temperature.

By starting with an initial low temperature application, all readily available uranium contamination will be easily removed. Heatup to 93°C appears to dissolve the remaining readily available uranium contamination. The 93°C application results in higher corrosion rates than a 66°C application. This is necessary to expose contamination buried more deeply into the component. Even at 93°C , the corrosion is uniform in nature.

2.3 Specific Objectives - Bench Scale Testing

The purpose of bench scale tests was to evaluate the solvent system developed in the solvent refinement test under dynamic test conditions that more closely simulate the actual field application. This was the first set of tests required for scale up of the candidate processes. The test procedures developed from this testing were to be used as the models for the pilot plant testing planned for the follow-on Task 5 (which is covered in a separate topical report). Corrosion coupons and mortar plugs containing uranium dioxide were loaded into a sealed reaction chamber. Solvent was circulated through the reaction chamber at a velocity approaching one foot per second. Samples were taken periodically and analyzed for dissolved uranium, iron, copper, nickel, and available chelant.

3.0 TEST PROGRAM

3.1 Approach

The first phase of this task was to develop a suitable binder for making UO_2 test plugs. Results of Task 2 static testing showed that powdered uranium dioxide dissolved almost immediately in both of the solvents tested. In order to more closely simulate the removal of uranium contamination embedded in the surface of process equipment, or even penetrating base metal grain boundaries to a depth of several mils, a method of encasing the powder was required. The use of UO_2 baked onto coupons was attempted during the foam testing in Task 2 of this program (Reference 3), but with little success because the baked on sludge did not adhere well to the coupon surfaces. A slower release of the UO_2 was desired to ensure the solvent remained active over a longer period under the dynamic test conditions. For this reason, it was decided to formulate mortar plugs containing uranium dioxide; as the plugs dissolved during exposure to solvent (simulating the corroding away of surface layers on contaminated process equipment) the encapsulated uranium would be released for dissolution.

Plugs consisting of calcium carbonate, trisodium phosphate, silicon dioxide and mortar were evaluated. The results of this evaluation will be presented in this report.

Two tests with each of the two candidate solvents were performed:

Solvent 1:

50 g/L Ethylenediaminetetraacetic Acid (EDTA)
20 g/L Ammonium Carbonate $(\text{NH}_4)_2\text{CO}_3$
5 g/L Hydrogen Peroxide (H_2O_2)
pH Adjusted to 9.0 with Ammonium Hydroxide (NH_4OH)

Solvent 2:

20 g/L Ammonium Carbonate $(\text{NH}_4)_2\text{CO}_3$
5 g/L Hydrogen Peroxide (H_2O_2)
pH Adjusted to 9.0 with Ammonium Hydroxide (NH_4OH)

The UO_2 plugs were exposed to the solvents under dynamic flowing conditions. Corrosion coupons of carbon steel, Nickel 200, and Monel 400 were also placed in the test vessel, for corrosion monitoring by weight loss determination. These materials were selected because all three were represented in the materials of construction of the compressors from the gaseous diffusion plant at Oak Ridge, Tennessee, which were being used as the preliminary basis for evaluation of the solvent systems (Reference 3). The tests were performed over a temperature range from ambient to 93°C (200°F). The solvent was periodically sampled and analyzed. The results of these four dynamic tests are included in this report.

3.2 Test Apparatus

The dynamic tests were performed in B&W's low temperature chemical cleaning velocity test rig. A photograph of the test rig is shown in Figure 1. A schematic of the rig is provided as Figure 2. The rig includes a solvent reservoir. The solvent volume used for testing was seven (7) liters. Immersion heaters and a cooling coil are contained in the reservoir for rapid heating and cooling of the solvent as required. The solvent was sampled via a sample line contained in the reservoir. The solvent reservoir was fabricated from stainless steel.

The other major components of the rig were the centrifugal pump, the magnetic flowrator and the test vessel. The stainless steel test vessel housed the UO_2 plugs and the corrosion coupons. The entire loop was contained in a stainless steel tray which, in turn, was located within a chemical fume hood. In addition, a supplemental stainless steel tray was placed under the test vessel.

3.3 Test Procedure

A detailed test procedure with step by step sign-offs was used to control all operations of these tests. A copy of this procedure is included as Appendix A to this report. The following is an outline detailing the major steps in the procedure.

1. Prepare the solvent according to the appropriate procedure. Sample and analyze the solvent to verify that it is within specification.
2. Dimension and weigh the corrosion coupons.
3. Assemble the test loop and the test vessel. Place appropriate plug and corrosion coupons in vessel.
4. Fill the solvent reservoir and begin recirculation at 3 gallons per minute (gpm).
5. Use the cooling capability of the reservoir, if necessary, to maintain the solvent temperature less than 29.5°C (85°F) for two hours.
6. After the initial two hours of exposure, heat the solvent to 93°C (200°F). Maintain recirculation during heat up.
7. After reaching 93°C , maintain temperature and flow conditions for an additional four hours. Sample the solvent when it reaches 93°C and after one hour and two hours at 93°C .
8. After the four hour 93°C application, begin cooling to 24°C (75°F).
9. After reaching 24°C , sample the solvent.

10. Inject enough H_2O_2 to bring the H_2O_2 concentration back to 5 g/L assuming that all of the H_2O_2 originally added has decomposed. Make this addition into the flowing stream just prior to the test vessel.
11. Use the cooling capability of the reservoir, if necessary, to maintain the solvent temperature less than 29.5°C (85°F) for two hours.
12. Sample the solvent after one hour and two hours of exposure under these low temperature conditions.
13. After this final two hour exposure, terminate the test, drain the solvent, and retrieve all specimens for post test examination.

All samples were analyzed for dissolved metals (uranium, iron, copper, and nickel). For the tests that included EDTA, free chelant was also determined. The pH of all samples was also determined.

Figure 1
Photograph of Bench Scale Test Loop

Figure 2
Schematic of Bench Scale Test Loop

4.0 RESULTS AND DISCUSSION - PLUG DEVELOPMENT TESTS

As previously stated, a series of tests were performed to determine a suitable binder with which to fabricate plugs of UO_2 . This plug testing was initially performed using only the binding materials. The candidate plugs were exposed to both of the test solvents in the planned application sequence (Room Temperature to 93°C to Room Temperature). Peroxide injection was used for both of the low temperature phases.

The initial test used the carbonate-only solvent. During this test three test plugs were evaluated:

1. Calcium carbonate (CaCO_3) and trisodium phosphate (TSP) in equal amounts.
2. CaCO_3 , TSP, and silicon dioxide (SiO_2) in equal amounts.
3. Industrial grade mortar

Plugs 1 and 2 began to deteriorate as soon as they contacted the test solution. After the entire exposure, plug number 1 had lost about 66% of its original material while plug number 2 lost about 50%. Plug number 3 was essentially unaffected by the solvent exposure.

The second test used the carbonate/EDTA solvent. During this test four test plugs were evaluated:

1. 11 grams CaCO_3 , 11 grams SiO_2 , and 5.5 grams mortar
2. 10 grams CaCO_3 , 10 grams SiO_2 , and 2.5 grams mortar
3. CaCO_3 and TSP in equal amounts
4. CaCO_3 , TSP, and SiO_2 in equal amounts

After the exposure, plugs 3 and 4 were completely dissolved. Only small remnants of plug number 2 could be recovered after the test. Plug number 1 was about 50% dissolved.

The next step was to perform a series of static beaker tests with the EDTA/carbonate solvent to determine the rates of dissolution of the different plugs in the different stages of the process. In this testing, plug number 1 showed very little deterioration throughout the test, losing only about 20% of its original material.

Plug number 2 lost about 5% of its material during the initial room temperature application. An additional 55% was lost during the high temperature (93°C) exposure. It then lost an additional 15% (for a total of about 75%) during the final room temperature exposure.

Both plugs 3 and 4 were 100% destroyed in the first 2 hours at room temperature. Based on these results, it was decided to fabricate the UO_2 test plugs according to the plug number 2 formulation. This material seemed to meet the requirement of slow deterioration over the entire exposure time.

5.0 RESULTS AND DISCUSSION - BENCH SCALE TESTS

A total of four (4) bench scale tests were performed. Two different plug combinations were used in these tests. Plug 2 as defined in the preceding section was used for the first two tests. It was found that the addition of the UO_2 to the plug tended to stabilize the plug further. A modified plug was then used in the final two tests. The results of each of these tests will be presented in this section.

5.1 Test #1: EDTA/Carbonate Solvent - Initial Plug Formulation

The initial two tests used the following plug formulation:

56 grams CaCO_3
56 grams SiO_2 ,
14 grams mortar
28 grams UO_2

The first test used the EDTA/Carbonate solvent system. A compilation of all of the raw data from this test, along with a material balance calculation, is included in Appendix B to this report. The initial EDTA concentration, prior to addition of 116 mL of 30% H_2O_2 , was 51.9 g/L. The pH just prior to the H_2O_2 addition was 9.05. A total of 20 g/L ammonium carbonate was added to this solvent mixture.

Table 1 contains a summary of the dissolved metals analysis performed during the test. Note that dissolved calcium is included in Table 1. The source of the calcium is the plug material (CaCO_3 and the mortar). The calcium was readily chelated by the EDTA. The dissolved calcium values indicate slow dissolution of the plug throughout the test exposure. From Table 1 it is evident that the majority of the iron, nickel, and copper went into solution during the 93°C exposure period. This indicates that the majority of the corrosion occurred during the high temperature exposure, as expected.

As seen from the data in Appendix B, 16.8 g/L free EDTA remained at the end of the test. This is an indication that the solvent still had capacity to dissolve additional uranium. The reduction in free EDTA would be a result of chelation by the various metals, and destruction by the H_2O_2 . Note from Appendix B that very few H_2O_2 results were obtained. Intermittent difficulties were encountered with the spectrophotometer used in the colorimetric peroxide method (it periodically became non-operational) during this entire test sequence. This made it difficult to obtain reliable H_2O_2 values, since H_2O_2 samples cannot be preserved for a long period of time (the H_2O_2 tends to decompose with time in this solvent system, even if refrigerated).

Table 1
Dissolved Metal Analysis - Bench Scale Test #1

Sample I.D	[U] (g/L)	[Fe] (g/L)	[Cu] (g/L)	[Ni] (g/L)	[Ca] (g/L)
1 hr @ R.T. ¹	0.355	0.010	<0.001	0.001	0.455
2 hr @ R.T.	0.604	0.012	0.001	0.001	0.778
1 hr @ 93°C	0.740	0.029	0.002	0.008	1.190
2 hr @ 93°C	0.810	0.075	0.005	0.028	1.536
0 hr @ R.T. ²	0.840	0.115	0.007	0.047	1.765
1 hr @ R.T.	0.877	0.115	0.008	0.047	1.863
2 Hr @ R.T.	0.885	0.116	0.008	0.047	1.911

1 - R.T. = Room Temperature Exposure
2 - Upon Return to R.T. Prior to H₂O₂ Spike

From the Task 2 testing (Reference 3), it was known that control of H₂O₂ concentration is important to the success of the process application, since a certain level of peroxide was found to be required to promote uranium dissolution. The on-going difficulties with H₂O₂ analysis during this phase of testing, as well as the length of time normally required to obtain spectrophotometric peroxide results (approximately one hour) made it evident that improvements in the H₂O₂ analytical technique would be required prior to taking this process to the field. For this reason, an effort was made to identify an analytical method that could provide real-time (within fifteen minutes of sampling) peroxide results. The results of this investigation will be discussed later in the report.

Figure 3 shows the dissolved uranium versus time for this test. The Figure 3 values are based on the Appendix B materials balance calculations. Also included in Figure 3 is a curve of the test temperature versus time. Uranium dissolved throughout the entire exposure period. The majority of the dissolution occurred at the lower temperatures. Based on the fact that dissolved uranium values were still increasing slightly at the end of the test period, it appeared that limited dissolution was still occurring at that time. However, since exposures were all limited to eight hours in order to facilitate comparisons between tests, it was not determined if the remaining uranium could have been dissolved by extending the exposure time.

Figure 3
Total Uranium Dissolved
DOE Bench Scale Test Number 1

At the end of the test, approximately 25% of the uranium added to the system was dissolved. A substantial portion of the plug was retrieved at the end of testing. The plug served the purpose of slow dissolution during the test exposure, but proved more robust than originally expected. It appears that the solvent was actively dissolving uranium as it became accessible to the solvent. The uranium not accessible to the solvent was retained in the plug matrix.

It was necessary to use the cooling capability of the loop for both the initial low temperature exposure and also during the final low temperature exposure. Without this capability, the temperature would have exceeded the 29.4°C (85°F) specification for the room temperature phases of the exposure. The purpose of keeping the temperature below 85°F is to control the decomposition of hydrogen peroxide. A low solvent temperature allows the peroxide to remain active as a uranium oxidizer, which is required for dissolution, rather than decomposing through reaction with EDTA. Additionally, if the temperature of the solution becomes too high after addition of fresh peroxide, exothermic decomposition reactions between peroxide and EDTA, catalyzed by dissolved metal ions, can become violently uncontrolled.

The corrosion results from this test are presented in Appendix B. Uniform corrosion occurred on all specimens. The corrosion results will be discussed in more detail in Section 5.5 of this report.

5.2 Test #2: Carbonate-Only Solvent - Initial Plug Formulation

As previously stated, the initial two tests used the following plug formulation:

56 grams CaCO_3
56 grams SiO_2 ,
14 grams mortar
28 grams UO_2

The second test used the carbonate-only (20 g/L ammonium carbonate) solvent system. A compilation of all of the raw data from this test, along with a material balance calculation, is included in Appendix B to this report. The pH just prior to the addition of 116 mL of 30% H_2O_2 addition was 9.05. As with Test 1, very few H_2O_2 results were obtained.

Table 2 contains a summary of the dissolved metals analysis taken during the test. As with Table 1, dissolved calcium is included in Table 2. The source of the calcium is the plug material (CaCO_3 and the mortar). Without the presence of EDTA, the dissolved calcium was much less in Test 2 than in Test 1 (compare the Table 1 and Table 2 dissolved calcium values). The dissolved calcium values indicate slow dissolution of the plug throughout the first two hours of the 93°C exposure with little plug dissolution during the later stages of the test.

The Table 2 dissolved iron, nickel, and copper values were substantially lower than the comparable Table 1 values. This indicates much lower corrosion in the system without the EDTA present. This was verified by the coupon weight loss values.

Table 2
Dissolved Metal Analysis - Bench Scale Test #2

Sample I.D	[U] (g/L)	[Fe] (g/L)	[Cu] (g/L)	[Ni] (g/L)	[Ca] (g/L)
1 hr @ R.T. ¹	0.441	0.017	0.002	0.006	0.297
2 hr @ R.T.	0.510	0.016	0.001	0.006	0.308
0 hr @ R.T.	0.554	0.017	0.002	0.005	0.296
1 hr @ 93°C	0.620	0.018	0.003	0.007	0.312
2 hr @ 93°C	0.638	0.019	0.004	0.009	0.340
0 hr @ R.T. ²	0.631	0.020	0.005	0.010	0.336
1 hr @ R.T.	0.639	0.020	0.005	0.011	0.323
2 Hr @ R.T.	0.641	0.020	0.006	0.011	0.310

1 - R.T. = Room Temperature Exposure

2 - Upon Return to R.T. Prior to H₂O₂ Spike

Figure 4 shows the dissolved uranium versus time for this test. The Figure 4 values are based on the Appendix B materials balance calculations. Also included in Figure 4 is a curve of the test temperature versus time. As with Test 1, the majority of uranium dissolution occurred at the lower temperatures. It appears that dissolution stopped during the 93°C portion of the exposure and never restarted, even after the reduction in temperature and the spike of additional H₂O₂. Uranium dissolution stopped at the same time that dissolved calcium levels stopped increasing, indicating that when plug dissolution ceased, the remaining uranium did not become accessible to the solvent. As with Test 1, it appears that the uranium accessible to the solvent was dissolved. The uranium stabilized in the plug did not dissolve.

At the end of the test, approximately 18% of the uranium added to the system was dissolved. A substantial portion of the plug was retrieved at the end of testing. The plug served the purpose of slow dissolution during the test exposure, but proved more robust than originally expected. The EDTA/carbonate solvent was more effective than the carbonate-only solvent. This was due to the better dissolution of the plug, presumably due to the chelation of the calcium by the EDTA.

Figure 4
Total Uranium Dissolved
DOE Bench Scale Test Number 2

It was necessary to use the cooling capability of the loop for both the initial low temperature exposure and also during the final low temperature exposure. The cooling coil was turned off during the final hour of the test to see if the reactions could be restarted with the heat generated by the pump and the hydrogen peroxide reactions. The maximum temperature achieved was 32.2°C (90°F). As previously described, the dissolution reactions did not appear to restart during this exposure period.

The corrosion results from this test are presented in Appendix B. Low corrosion occurred on all specimens. Thus, the carbonate-only solvent will not be suitable for applications requiring removal of base metal to expose embedded contamination. The corrosion results will be discussed in more detail in Section 5.5 of this report.

5.3 Test #3: EDTA/Carbonate Solvent - Modified Plug Formulation

The lower than expected plug dissolution rate in the first two tests prompted a reformulation of the uranium dioxide plug. The final two tests used the following combination of ingredients:

28 grams CaCO_3
28 grams SiO_2 ,
6 grams mortar
28 grams UO_2

The third test used the EDTA/Carbonate solvent system. A compilation of all of the raw data from this test along with a material balance calculation is included in Appendix B to this report. The initial EDTA concentration, prior to addition of 116 mL of 30% H_2O_2 was 51.2 g/L. The pH just prior to the H_2O_2 addition was 9.06. A total of 20 g/L ammonium carbonate was added to this solvent mixture.

Table 3 contains a summary of the dissolved metals analysis taken during the test. As with the previous tests, dissolved calcium is included in Table 3. The source of the calcium is the plug material (CaCO_3 and the mortar). As with Test 1, the calcium was readily chelated by the EDTA. The dissolved calcium values indicate slow dissolution of the plug through the initial stages of the test exposure. During the 93°C exposure, dissolution of the plug apparently ceased.

From Table 3 it is evident that the majority of the iron, nickel, and copper went into solution during the 93°C exposure period. This indicates that the majority of the corrosion occurred during the high temperature exposure, as expected and consistent with Test 1.

Table 3
Dissolved Metal Analysis - Bench Scale Test #3

Sample I.D.	[U] (g/L)	[Fe] (g/L)	[Cu] (g/L)	[Ni] (g/L)	[Ca] (g/L)
1 hr @ R.T. ¹	1.392	0.011	0.002	0.003	0.830
2 hr @ R.T.	1.915	0.013	0.002	0.003	1.022
0 hr @ R.T.	2.484	0.021	0.004	0.006	1.401
1 hr @ 93°C	2.484	0.044	0.004	0.016	1.492
2 hr @ 93°C	2.663	0.068	0.008	0.051	1.656
0 hr @ R.T. ²	2.664	0.073	0.011	0.070	1.712
1 hr @ R.T.	2.591	0.071	0.011	0.068	1.717
2 Hr @ R.T.	2.600	0.070	0.010	0.066	1.712

1 - R.T. = Room Temperature Exposure

2 - Upon Return to R.T. Prior to H₂O₂ Spike

An increased number of H₂O₂ results were obtained during this test. The performance of the spectrophotometer was verified by running a standard curve with each set of samples analyzed. For most samples, there was a time lag between sampling and analysis. As previously noted, H₂O₂ samples cannot be preserved for a long period of time (the H₂O₂ tends to decompose with time in this solvent system, even if refrigerated). It is likely that the actual H₂O₂ values were higher than reported in Appendix B. However, the values do indicate that there was a level of peroxide present throughout the entire exposure period.

Figure 5 shows the dissolved uranium versus time for this test. The Figure 5 values are based on the Appendix B materials balance calculations. Also included in Figure 5 is a curve of the test temperature versus time. As with Test 1, the majority of the dissolution occurred at the lower temperatures. It appears that dissolution stopped during the 93°C portion of the exposure and never restarted, even after the reduction in temperature and the spike of additional H₂O₂. Uranium dissolution stopped at the same time that dissolved calcium levels stopped increasing, indicating that when plug dissolution ceased, the remaining uranium did not become accessible to the solvent. As with Test 1, it appears that the uranium accessible to the solvent was dissolved. The uranium stabilized in the plug did not dissolve.

Figure 5
Total Uranium Dissolved
DOE Bench Scale Test Number 3

At the end of the test, approximately 75% of the uranium added to the system was dissolved. A portion of the plug was retrieved at the end of testing. The revised plug provided better performance than the initial plug; however, a portion of the plug was insoluble in the solvent mixture. The failure of the solvent to degrade the plug sufficiently to release all uranium dioxide included does not indicate that the solvent will exhibit the same deficiency when applied to process equipment. It has been demonstrated (Reference 3) that the EDTA/carbonate solvent system is sufficiently corrosive to carbon steel to remove 1.5 mils of material in a four-hour 93°C exposure. This will release embedded contamination for subsequent dissolution.

It was necessary to use the cooling capability of the loop for the final low temperature exposure. The dissolved metals generated by corrosion during the high temperature application apparently catalyzed the exothermic H_2O_2 decomposition reactions.

The corrosion results from this test are presented in Appendix B. Again uniform corrosion occurred on all specimens. The corrosion results will be discussed in more detail in Section 5.5 of this report.

5.4 Test #4: Carbonate-Only Solvent - Modified Plug Formulation

As previously stated, the final two tests used the following plug formulation:

28 grams $CaCO_3$
28 grams SiO_2 ,
6 grams mortar
28 grams UO_2

The fourth test used the carbonate-only (20 g/L ammonium carbonate) solvent system. A compilation of all of the raw data from this test along with a material balance calculation is included in Appendix B to this report. The pH just prior to the addition of 116 mL of 30% H_2O_2 addition was 9.00.

Table 4 contains a summary of the dissolved metals analysis taken during the test. The dissolved calcium and uranium peaked during the 93°C exposure period, then fell after cooling and the H_2O_2 spike. The low calcium levels in the solvent indicate that the revised plug was very stable in the carbonate-only solvent system.

The Table 4 dissolved iron, nickel, and copper values were substantially lower than the results from the previous tests. This indicates a very low corrosion rate. This was verified by the coupon weight loss values.

Several H_2O_2 results were obtained during this test. Timely results were obtained on the initial sample and the samples after the cool down and H_2O_2 spike. Substantial peroxide remained at the end of test.

Table 4
Dissolved Metal Analysis - Bench Scale Test #4

Sample I.D	[U] (g/L)	[Fe] (g/L)	[Cu] (g/L)	[Ni] (g/L)	[Ca] (g/L)
1 hr @ R.T. ¹	0.209	0.002	<0.001	0.002	0.040
2 hr @ R.T.	0.61	0.002	<0.001	0.002	0.040
0 hr @ R.T.	0.356	0.003	<0.001	0.001	0.032
1 hr @ 93°C	0.450	0.004	<0.001	0.002	0.041
3 hr @ 93°C	0.450	0.004	<0.001	0.002	0.053
0 hr @ R.T. ²	0.400	0.004	<0.001	0.002	0.056
1 hr @ R.T.	0.330	0.003	<0.001	0.001	0.041
2 Hr @ R.T.	0.330	0.003	<0.001	0.001	0.040

1 - R.T. = Room Temperature Exposure

2 - Upon Return to R.T. Prior to H₂O₂ Spike

Figure 6 shows the dissolved uranium versus time for this test. The Figure 6 values are based on the Appendix B materials balance calculations. Also included in Figure 6 is a curve of the test temperature versus time. Uranium dissolution apparently stopped during the 93°C exposure period. Further, uranium appears to be dropping out of solution during the cool down and peroxide spike phases. As previously stated, this was accompanied by an apparent dropping out of dissolved calcium from solution. The reasons for this are unknown. Based on the results of the four tests, the addition of the EDTA provides more consistent results and helps to stabilize the dissolved uranium.

At the end of the test, only about 10% of the uranium added to the system was dissolved. A substantial portion of the plug was retrieved at the end of testing. The revised plug worked very well with the EDTA/carbonate system, but was resistant to the carbonate-only solvent system.

The corrosion results from this test are presented in Appendix B. Essentially no corrosion occurred on the specimens. The corrosion results will be discussed in more detail in Section 5.5 of this report.

Figure 6
Total Uranium Dissolved
DOE Bench Scale Test Number 4

5.5 Corrosion Results - Bench Scale Testing

Table 5 summarizes the corrosion results from all four tests performed during this phase of the test program. By exposing the three coupon materials simultaneously, the conditions expected to be encountered in cleaning centrifugal compressors from the Oak Ridge gaseous diffusion plant were simulated. In all cases, corrosion loss was uniform with no localized attack evident. Overall, the corrosion results with the EDTA/carbonate solvent were more consistent than those from the carbonate-only solvent.

The final test with the carbonate-only solvent (Test 4) resulted in essentially no corrosion of any of the coupons. In the other test with carbonate-only (Test 2) the Monel 400 suffered 0.044 mils corrosion while the carbon steel and the Nickel 200 experienced essentially no corrosion. These inconsistent corrosion results were also observed during the Task 2 screening and development testing (Reference 3).

Table 5
Corrosion Results - Bench Scale Testing

Material	Mils Corrosion*			
	Test 1	Test 3	Test 2	Test 4
AISI-1018	0.531	0.406	0.000	0.000
AISI-1018	0.536	0.254	0.000	0.001
Nickel-200	0.225	0.351	0.001	0.004
Nickel-200	0.224	0.402	0.000	0.002
Monel-400	0.116	0.203	0.044	0.001
Monel-400	0.121	0.166	0.044	0.001

* - Tests 1 and 3 used the EDTA/Carbonate solvent while Tests 2 and 4 used the carbonate-only solvent

It is obvious that the addition of chelant is required to effect consistent base metal corrosion for release of contamination below the surface of the equipment being decontaminated. This is consistent with the earlier testing in this program (Reference 3).

6.0 CONCLUSIONS

The following is a summary of the significant conclusions from the data generated during Task 4 of the DOE Program DE-AC21-83MC30168, "Chemical Decontamination of Process Equipment Using Recyclable Chelating Agents."

The basic test loop was adequate to apply and control the process. Hydrogen peroxide could easily be spiked and temperature control could be maintained. It is important to have on-line cooling capabilities during the low temperature phases of the process. This capability was utilized several times to keep solvent temperature below the specified temperature for low temperature applications. The purpose of keeping the temperature control is to limit peroxide decomposition. A low solvent temperature allows the peroxide to remain in solution longer without decomposing, and helps to prevent uncontrolled exothermic decomposition reactions. This is especially true during the final H_2O_2 spike when dissolved metal ions may be present to catalyze the various exothermic reactions.

The results of bench scale testing in Task 4 were consistent with those of process application testing in Task 2. In both cases, readily available uranium dioxide dissolved completely during the initial low temperature exposure to either solvent. The plug system utilized in the bench scale program shielded some of the uranium dioxide from the solvent system; none of the plugs used dissolved completely during the eight hour exposures. As predicted by Task 2 testing, corrosion took place with the EDTA/carbonate solvent only, the majority occurring during the 93°C exposure. These results indicated that the application scenario developed in the Task 2 process application testing would be suitable for use in the pilot scale testing utilizing contaminated process equipment.

There is essentially no corrosion without EDTA present in the solvent system. The EDTA is required to effect consistent base metal corrosion for release of contamination below the surface of the equipment being decontaminated. The EDTA also appears to stabilize the dissolved uranium, preventing it from dropping out of solution during the cooldown phase of the process.

Hydrogen peroxide is an important variable that needs to be monitored and controlled during the process application. Task 2 testing (Reference 3) showed that uranium dissolution does not occur in either solvent system without a certain level of peroxide being present. The colorimetric method utilized during this testing required approximately one hour after sampling to produce results. During field application, a method capable of producing real-time (within 15 minutes of sampling) results will be required, since peroxide levels must be maintained in the solvent at all times during low-temperature application in order for complete uranium dissolution to occur.

The basic test procedures developed from this testing are suitable as a model for the pilot plant testing to be performed in the follow-on Task 5.

7.0 RECOMMENDATIONS

It is recommended to continue to test both of the basic solvent systems in the pilot plant phase of the program:

- (1) EDTA, ammonium carbonate, and hydrogen peroxide: pH 9.0 with NH_4OH
- (2) ammonium carbonate and hydrogen peroxide: pH 9.0 with NH_4OH

The bench scale study corroborated the conclusions drawn after Task 2 testing, and indicated that the solvents could successfully be applied to pilot scale decontamination of process equipment. The carbonate-only solvent efficiently dissolved available uranium, and as such should be effective for removal of surface uranium contamination. The addition of EDTA appeared necessary to stabilize the dissolved uranium during long exposures and process temperature cycles. In addition, the chelant was required for corrosion of the base metal, and hence would be required to release contamination below metal surfaces. For these reasons, the EDTA/carbonate system would probably be the solvent of choice for field application. The carbonate-only solvent should continue to be tested as it currently offers some advantages for solvent recycle (not covered in this specific report, but is a part of the overall program - Reference 4).

It is also recommended to develop an analytical method for H_2O_2 analysis that can provide more real-time data. The colorimetric method currently in use requires at least one hour after sampling to produce results. A method should be sought that is capable of determining peroxide concentration within fifteen minutes of sampling, since peroxide is an important process control parameter. One promising method is a potentiometric titration that can be automated using appropriate laboratory instrumentation. If possible, such a method should be used for the pilot plant test. However, an improved H_2O_2 method is definitely required prior to the Phase II field demonstration.

8.0 REFERENCES

- (1) "Decontamination and Decommissioning Integrated Demonstration Strategy," Draft for Review, November 1991, a product of the DOE Environmental Management D&D Workshop, August 12-16, 1991
- (2) W. J. Manion and T. S. LaGuardia, "Decommissioning Handbook," prepared for U.S. DOE by Nuclear Energy Services, Inc., November, 1980
- (3) Task 2 Topical Report, DOE Contract DE-AC21-93MC30168.
- (4) Task 3 Topical Report, DOE Contract DE-AC21-93MC30168.

APPENDIX A

TASK 4 TOPICAL REPORT

DOE CONTRACT DE-AC21-93MC30168

TEST PROCEDURES AND SIGN-OFF SHEETS

FOR BENCH SCALE TESTS

TEST PROCEDURE CTP NO. 1.1.9:9317A-00
PREPARATION OF SOLUTION CONTAINING HYDROGEN PEROXIDE AND AMMONIUM
CARBONATE

PREPARED BY: S.P. Ulbricht _____
APPROVED BY: J.M. Jevic _____

Description of the Procedure

This Test Procedure describes the formulation of 3.0 liters of the solvent containing 5.0 grams of hydrogen peroxide and 20 grams of ammonium carbonate per liter. Other quantities may be made by simple proportioning all of the constituents. The composition of the solvent is specified to be:

H₂O₂ - 5.0 g/L (calculated for addition)
(NH₄)₂CO₃ - 20 ± 1 grams per liter
pH - 9.0 ± 0.1, adjusted with ammonium hydroxide

This test procedure was used to formulate the solvent used in
_____ (fill in test name and number) _____.

Procedure

Initial/Date

- 10) Add a stirring bar to a clean, dry, 4-liter beaker. Weigh the beaker containing the stirring bar to the nearest 0.1 gram on a top loading balance and record the weight in the laboratory notebook. _____
- 20) Add about 1.5 liters of deionized water to the weighed 4-liter beaker. _____
- 30) Add sufficient ammonium hydroxide to the beaker to bring the pH to at least 8.0. Then place the beaker on a magnetic stirrer. _____
- 100) Add 60.0 grams of ammonium carbonate to the solution. Stir until the ammonium carbonate is completely dissolved. _____
- 120) Stir the solvent well and determine the pH. Adjust the pH to 9.0 ± 0.1 with ammonium hydroxide. Then adjust the volume to exactly 2.950 liters with deionized water, using a graduated cylinder. _____
- 130) Determine the pH of the adjusted solution. Record the final pH in the laboratory note book. _____

TEST PROCEDURE CTP NO. 1.1.9:9317A-00
PREPARATION OF SOLUTION CONTAINING HYDROGEN PEROXIDE AND AMMONIUM
CARBONATE

Initial/Date

- 170) If the temperature of the solvent is below 85°F, add 50 mL of fresh 30% hydrogen peroxide to the solvent. Stir the solvent to make it homogeneous, but do not over-stir, as this will promote solvent decomposition.

TEST PROCEDURE CTP NO. 1.1.9:9317B-00
PREPARATION OF CHELATING SOLVENT CONTAINING 50 GRAMS PER LITER EDTA

PREPARED BY: S.P. Ulbricht _____
APPROVED BY: J.M. Jevac _____

Description of the Procedure

This Test Procedure describes the formulation of 3.0 liters of the solvent containing 50 grams of EDTA, 20 grams of ammonium carbonate, and 5.0 grams of hydrogen peroxide per liter. Other quantities may be made by simple proportioning all of the constituents. The composition of the solvent is specified to be:

EDTA	-	50 ± 5 grams per liter
(NH ₄) ₂ CO ₃	-	20 ± 1 grams per liter
pH	-	9.0 ± 0.1, adjusted with ammonium hydroxide
H ₂ O ₂	-	5.0 g/L (calculated for addition)

This test procedure was used to formulate the solvent used in
(fill in test name and number).

Procedure

Initial/Date

- 10) Add a stirring bar to a clean, dry, 4-liter beaker. Weigh the beaker containing the stirring bar to the nearest 0.1 gram on a top loading balance and record the weight in the laboratory notebook. _____
- 20) Add about 1.5 liters of deionized water to the weighed 4-liter beaker. _____
- 30) Add 80 ml of ammonium hydroxide to the beaker and place the beaker on a magnetic stirrer. _____
- 70) Weigh 150.0 grams of EDTA on the top loading balance. _____
- 80) Start the magnetic stirrer and slowly add the EDTA to the water-ammonia solution in the 4-liter beaker. Do not add the EDTA too rapidly or the stirrer will become overloaded and then it will stop. _____

TEST PROCEDURE CTP NO. 1.1.9:9317B-00
PREPARATION OF CHELATING SOLVENT CONTAINING 50 GRAMS PER LITER EDTA

Initial/Date

- 90) Stir until all of the EDTA has been dissolved. If the EDTA does not dissolve in a reasonable period of time, check the pH of the solution. If the solution pH is below 4.5, insufficient ammonia is present. Ammonia must be added to the solution if the pH is low. The pH may be raised to a value as high as 7.0. Continue stirring until all of the EDTA has been dissolved. Adjust the pH of the solution to at least 7.0, to avoid loss of carbonate in the next step. _____
- 100) Add 60.0 grams of ammonium carbonate to the solution. Stir until the ammonium carbonate is completely dissolved. _____
- 120) Stir the solvent well and determine the pH. Adjust the pH to 9.0 ± 0.1 with ammonium hydroxide. Then adjust the volume to exactly 2.950 liters with deionized water, using a graduated cylinder. _____
- 130) Determine the pH of the adjusted solution. Record the final pH in the laboratory note book. _____
- 140) Analyze the solution for EDTA according to ARC-TP-868, Rev 1, "Method for Determining of Unchelated EDTA in Chemical Cleaning Solutions". Record all measurements, dilutions, and calculations in the laboratory note book. _____
- 160) If the solution is within the specifications as shown in Section 1.1 above or as described in an applicable test specification which will supersede Section 1.1, determine the density of the solution with a hydrometer. Record this density in the laboratory notebook and set the solution aside for use in the test. If the concentration of the constituents in the solvent is not within specifications, discard the solution and make a new one. _____
- 170) If the temperature of the solvent is below 85°F, add 50 mL of fresh 30% hydrogen peroxide to the solvent. Stir the solvent to make it homogeneous, but do not over-stir, as this will promote solvent decomposition. _____

Velocity Test Procedure and Sign-Off Sheet Uranium Dioxide Dissolution Testing

Prepared By: _____ C.R.Turner

Approved By: _____ J.M.Jevic

Test Description

Uranium dioxide crystals will be encapsulated in a medium containing a combination of calcium carbonate and mortar. These capsules will be exposed to chelating solvents to evaluate the performance of the developed decontamination process under dynamic conditions.

Several metal coupons will also be exposed to the solvents to characterize the corrosion behavior of the solvents to carbon steel, Nickel 200 alloy, and Monel 400 alloy under velocity conditions.

Initially the test specimens and the coupons will be exposed to the solvents at room temperature with peroxide added and some velocity for two (2) hours. Subsequently the solvent will be heated to 200°F (93°C) and held for four (4) hours at the same velocity. After this heated exposure, the solvent will be quick cooled to 75°F (24°C) and hydrogen peroxide will be added to the solvent. The solvent will be held at this temperature and velocity for an additional two (2) hours.

At the conclusion of the test, the solvent will be drained from the test apparatus and filtered. The coupons will also be cleaned of corrosion deposit and weighed for corrosion calculations.

Test Apparatus

The test apparatus will consist of modified B&W equipment: **Low Temperature Velocity Test Apparatus** and the **Velocity Impingement Test Vessel**. The test apparatus has several modifications to allow quick cooling and sampling of the solvent from the reservoir. The minimum reservoir operation volume of the test apparatus is 4 liters (this is because of the immersion heaters and also to help prevent vortices from causing pump cavitation). Seven and one-half (7.5) liters of solvent will be formulated. Seven will be used in the test with one half (0.5) liters retained for ICP matrix. More solvent may be required depending on what velocity is chosen.

The volume of the test vessel is eight hundred and seventy mL (0.870 L) empty. The coupon and specimen holder occupy 105 ml. Therefore, the volume without specimens is 765 mL. The volume of the flowrator and tubing is about 2 liters.

The original design of the B&W reservoir cover used a water seal. However, the water seal could not be used because in the velocity testing, on cooling, the water would be sucked into the reservoir diluting the solvent. There will be no N₂ cover gas. Hydrogen peroxide is used in the cooled solvent; therefore, a nitrogen blanket would have little effect on the solvent. It may be necessary to prop the lid open during out-gassing of the solvent.

Velocity Test Procedure and Sign-Off Sheet

Uranium Dioxide Dissolution Testing

The cooling coil requires penetrations in the lid of the reservoir where the lines exit the reservoir. These lines will be connected to the existing water supply in the hood. The return to drain will be into the cup sink in the hood. The other lid penetration will accommodate a sample line and a thermocouple.

The test vessel will be placed in the smaller stainless tray within the larger tray in the hood. The open drain on the left side of the larger tray will be stoppered to prevent any spills from entering the left cup sink.

Sampling will be 1 and 2 hours after the start of the test; 0, 1, and 3 hours after the solvent reaches 200°F (93°C); just prior to H₂O₂ injection, and 1 and 2 hours after the injection of peroxide @ 75°F (24°C).

All samples will be analyzed for H₂O₂, U, Fe, Cu, and Ni. If an analytical method for the chelant is available, the sample will be analyzed for that chelant.

Because this test will be conducted in an all steel environment, no video camera will be used and no visual observations can be made. It is, therefore, important to record any unusual occurrences in the lab notebook (loss of flow, loss of temperature, etc.).

These two parameters (flow rate and temperature) will be recorded in the log book every half hour. If the temperature and flow reading are taken more often than each half hour, those readings will also be recorded.

All samples will be taken in the same manner. Dry disposable syringes will be used to extract the sample from the reservoir. These samples will be filtered and analytical samples withdrawn using disposable pipettes. All syringes and pipettes will be disposed of after one use. 15 mL (1/2oz) plastic bottles will be used to store any sample remnants as well as the samples for peroxide analysis, which will be refrigerated until the analysis is performed.

The following personnel involved with conducting this test have read and understood the above test description. This sign-off is to be completed before the individual performs any steps.

NAME	SIGNATURE	DATE
S. Bozeka	_____	_____
R. Dye	_____	_____
J.M. Jevic	_____	_____
T.S. Pressley	_____	_____
C.R. Turner	_____	_____
S.P. Ulbricht	_____	_____

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

Detailed Test Procedure

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
10	Calibrate the loop in gpm. The reservoir contains 1.64 liters of solvent per inch of height. The minimum volume of solvent without pump cavitation is 4 liters or about 2.5 inches. The test vessel volume is 0.870 liters without any specimens or teflon holder.	
20	Prepare 7.5 liters of test solvent per the appropriate procedure: CTP:1.1.9:9317A-00 (Carbonate Only) CTP:1.1.9:9317B-00 (Carbonate/EDTA)	
25	Note the procedure used for solvent preparation in the laboratory notebook. Record all weights and measurements including density in the lab notebook. Save 0.5 liters of solvent as matrix solution.	
30	Obtain dimensions and weights of all corrosion specimens to be placed in the test. All weights and dimensions shall be checked by a second operator. The two weights must agree within 0.3 mg and the dimensions must agree within 0.5 mils. If necessary reweigh to obtain the appropriate agreement. Both operators must sign-off this step.	
40	Dry the test vessel so that no remaining rinse water or other solvent will dilute the test solvent. Place the test specimens behind the teflon specimen holder and suspend the coupons from the coupon holder.	
50	Place the lid on the vessel and fasten with quarter inch machine screws. Eight are required.	
60	Make sure the drain, throttle, and recirculation valves are closed.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
70	Fill the reservoir with seven liters of solvent. Note that the H ₂ O ₂ should be added to the solvent just prior to adding it to the reservoir.	
80	Place the lid on the reservoir and connect the sample line and place the thermometer (or thermal couple) in the lid.	
90	Start circulating the solvent through the by-pass line. Check the system for leaks. If any leaks are apparent at this time, stop the pump and repair. Note times and repairs, if required, in the laboratory notebook.	
100	Open the return valve (outlet of the vessel) then open the throttle valve. Adjust the throttle valve to obtain 3 gpm.	
110	The magnetic flowrator reads total flow. Be sure the by-pass valve is closed tight to ensure accurate readings.	
120	The pump will generate some heat and the solvent reactions are exothermic. Carefully watch solvent temperature and use the cooling coil, if required to maintain solvent temperature less than 85°F.	
130	Record the start time in the lab book. Fill in the clock time, based on this start time, for all sequences where a blank space has been left for the insertion of time.	
140	Note any unusual observations in the lab book.	
150	One half hour after the start of the test record the temperature and the velocity in the lab book. Note any unusual observations.	
160	The time is _____ one hour after the start of the test. Take three (3) samples of the solvent for analyses via the following procedure.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

Charge # 43177-004-001	Project/Test DOE/Decon Bench Scale Test	Date
Sequence	Procedure	Initials
160-A	Identify and tare weigh three clean, dry 12 mL syringes. Record this information in the lab notebook.	
160-B	Insert the first syringe tip into the rubber tube on the sample line on the lid of the reservoir. Pull a syringe full and push back into the reservoir. Do this three times to flush the line. Then draw a 12 mL sample.	
160-C	Take a second 12 mL sample with a separate tared syringe.	
160-D	Take a third 12 mL sample with a separate tared syringe for H ₂ O ₂ analysis. Transfer this sample to a 15 mL sample bottle and refrigerate for the analyst. Tighten the screw clamp on the tubing.	
160-E	Monitor and record the flow and temperature to ensure that they have not been disturbed. Adjust as necessary. Record any adjustments in the lab notebook.	
165	Handle and analyze the remaining samples as follows:	
165-A	Note that step 165 can be performed as time permits. Ensure that the timed steps of this procedure, such as Step 170, are performed as scheduled.	
165-B	Weigh and record the weights of the syringe samples, clock time, and volumes in the lab notebook.	
165-C	If sampled hot, cool the syringes in the cooling fixture, to room temperature, using stoppers on the syringe tips.	
165-D	Filter two samples through a tared 0.45 micron filter. Collect the filtrate in a clean and dry suction flask.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
165-E	Use a clean, dry, disposable pipette to withdraw a five (5) mL sample for ICP analysis. Place in a 50 mL volumetric flask and notify the ICP operator.	
165-F	Withdraw another five (5) mL sample for EDTA analysis, if applicable. Analyze per ARC-TP-868.	
165-G	Obtain the pH value on the remaining solution and record in the lab notebook.	
170	The time is _____ 1.5-hours after the start of the test, it is time to monitor and record the test conditions as follows:	
170-A	Record the temperature in the lab notebook. Cool as required to maintain temperature less than 85°F.	
170-B	Record the flow rate in the lab notebook. Adjust as necessary.	
170-C	Record any observations of the test in the lab notebook.	
180	The time is _____ two hours after the start of the test. Take three (3) samples of the solvent for analyses via the following procedure.	
180-A	Identify and tare weigh three clean, dry 12 mL syringes. Record this information in the lab notebook.	
180-B	Insert the first syringe tip into the rubber tube on the sample line on the lid of the reservoir. Pull a syringe full and push back into the reservoir. Do this three times to flush the line. Then draw a 12 mL sample.	
180-C	Take a second 12 mL sample with a separate tared syringe.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
180-D	Take a third 12 mL sample with a separate tared syringe for H ₂ O ₂ analysis. Transfer this sample to a 15 mL sample bottle and refrigerate for the analyst. Tighten the screw clamp on the tubing.	
180-E	Monitor and record the flow and temperature to ensure that they have not been disturbed. Adjust as necessary. Record any adjustments in the lab notebook.	
185	Handle and analyze the remaining samples as follows:	
185-A	Note that step 185 can be performed as time permits. Ensure that the timed steps of this procedure, such as Step 190, are performed as scheduled.	
185-B	Weigh and record the weights of the syringe samples, clock time, and volumes in the lab notebook.	
185-C	If sampled hot, cool the syringes in the cooling fixture, to room temperature, using stoppers on the syringe tips.	
185-D	Filter two samples through a tared 0.45 micron filter. Collect the filtrate in a clean and dry suction flask.	
185-E	Use a clean, dry, disposable pipette to withdraw a five (5) mL sample for ICP analysis. Place in a 50 mL volumetric flask and notify the ICP operator.	
185-F	Withdraw another five (5) mL sample for EDTA analysis, if applicable. Analyze per ARC-TP-868.	
185-G	Obtain the pH value on the remaining solution and record in the lab notebook.	
190	The time is _____ approximately two hours after the start of the test. The three samples of the solvent have been taken. Begin heating the solvent to 200°F.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

Charge # 43177-004-001	Project/Test DOE/Decon Bench Scale Test	Date
Sequence	Procedure	Initials
195	The time is _____ 2.5-hours after the start of the test, it is time to monitor and record the test conditions as follows:	
195-A	Record the temperature in the lab notebook. Continue heating to 200°F.	
195-B	Record the flow rate in the lab notebook. Adjust as necessary.	
195-C	Record any observations of the test in the lab notebook.	
200	The time is _____ 3-hours after the start of the test, it is time to monitor and record the test conditions as follows:	
200-A	Record the temperature in the lab notebook. Continue heating to 200°F.	
200-B	Record the flow rate in the lab notebook. Adjust as necessary.	
200-C	Record any observations of the test in the lab notebook.	
200-D	If the temperature has reached 200°F note the time in the lab notebook and proceed to step 210.	
200-E	If the temperature has not reached 200°F, repeat step 200 each half hour until the 200°F temperature is obtained.	
210	The time is _____. The solvent has reached 200°F. Take three (3) samples of the solvent for analyses via the following procedure.	
210-A	Identify and tare weigh three clean, dry 12 mL syringes. Record this information in the lab notebook.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
210-B	Insert the first syringe tip into the rubber tube on the sample line on the lid of the reservoir. Pull a syringe full and push back into the reservoir. Do this three times to flush the line. Then draw a 12 mL sample.	
210-C	Take a second 12 mL sample with a separate tared syringe.	
210-D	Take a third 12 mL sample with a separate tared syringe for H ₂ O ₂ analysis. Transfer this sample to a 15 mL sample bottle and refrigerate for the analyst. Tighten the screw clamp on the tubing.	
210-E	Monitor and record the flow and temperature to ensure that they have not been disturbed. Adjust as necessary. Record any adjustments in the lab notebook.	
215	Handle and analyze the remaining samples as follows:	
215-A	Note that step 215 can be performed as time permits. Ensure that the timed steps of this procedure, such as Step 220, are performed as scheduled.	
215-B	Weigh and record the weights of the syringe samples, clock time, and volumes in the lab notebook.	
215-C	If sampled hot, cool the syringes in the cooling fixture, to room temperature, using stoppers on the syringe tips.	
215-D	Filter two samples through a tared 0.45 micron filter. Collect the filtrate in a clean and dry suction flask.	
215-E	Use a clean, dry, disposable pipette to withdraw a five (5) mL sample for ICP analysis. Place in a 50 mL volumetric flask and notify the ICP operator.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
215-F	Withdraw another five (5) mL sample for EDTA analysis, if applicable. Analyze per ARC-TP-868.	
215-G	Obtain the pH value on the remaining solution and record in the lab notebook.	
220	The time is _____ 1-hour after reaching 200°F, it is time to take three (3) samples of the solvent for analyses via the following procedure.	
220-A	Identify and tare weigh three clean, dry 12 mL syringes. Record this information in the lab notebook.	
220-B	Insert the first syringe tip into the rubber tube on the sample line on the lid of the reservoir. Pull a syringe full and push back into the reservoir. Do this three times to flush the line. Then draw a 12 mL sample.	
220-C	Take a second 12 mL sample with a separate tared syringe.	
220-D	Take a third 12 mL sample with a separate tared syringe for H ₂ O ₂ analysis. Transfer this sample to a 15 mL sample bottle and refrigerate for the analyst. Tighten the screw clamp on the tubing.	
220-E	Monitor and record the flow and temperature to ensure that they have not been disturbed. Adjust as necessary. Record any adjustments in the lab notebook.	
225	Handle and analyze the remaining samples as follows:	
225-A	Note that step 225 can be performed as time permits. Ensure that the timed steps of this procedure, such as Step 230, are performed as scheduled.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
225-B	Weigh and record the weights of the syringe samples, clock time, and volumes in the lab notebook.	
225-C	If sampled hot, cool the syringes in the cooling fixture, to room temperature, using stoppers on the syringe tips.	
225-D	Filter two samples through a tared 0.45 micron filter. Collect the filtrate in a clean and dry suction flask.	
225-E	Use a clean, dry, disposable pipette to withdraw a five (5) mL sample for ICP analysis. Place in a 50 mL volumetric flask and notify the ICP operator.	
225-F	Withdraw another five (5) mL sample for EDTA analysis, if applicable. Analyze per ARC-TP-868.	
225-G	Obtain the pH value on the remaining solution and record in the lab notebook.	
230	The time is _____ 1.5-hours after reaching 200°F, it is time to monitor and record the test conditions as follows:	
230-A	Record the temperature in the lab notebook. Adjust as necessary to maintain 200°F $\pm$ 5°F.	
230-B	Record the flow rate in the lab notebook. Adjust as necessary.	
230-C	Record any observations of the test in the lab notebook.	
240	The time is _____ 2-hours after reaching 200°F, it is time to monitor and record the test conditions as follows:	
240-A	Record the temperature in the lab notebook. Adjust as necessary to maintain 200°F $\pm$ 5°F.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
240-B	Record the flow rate in the lab notebook. Adjust as necessary.	
240-C	Record any observations of the test in the lab notebook.	
250	The time is _____ 3-hours after reaching 200°F, it is time to take three (3) samples of the solvent for analyses via the following procedure.	
250-A	Identify and tare weigh three clean, dry 12 mL syringes. Record this information in the lab notebook.	
250-B	Insert the first syringe tip into the rubber tube on the sample line on the lid of the reservoir. Pull a syringe full and push back into the reservoir. Do this three times to flush the line. Then draw a 12 mL sample.	
250-C	Take a second 12 mL sample with a separate tared syringe.	
250-D	Take a third 12 mL sample with a separate tared syringe for H ₂ O ₂ analysis. Transfer this sample to a 15 mL sample bottle and refrigerate for the analyst. Tighten the screw clamp on the tubing.	
250-E	Monitor and record the flow and temperature to ensure that they have not been disturbed. Adjust as necessary. Record any adjustments in the lab notebook.	
255	Handle and analyze the remaining samples as follows:	
255-A	Note that step 255 can be performed as time permits. Ensure that the timed steps of this procedure, such as Step 260, are performed as scheduled.	
255-B	Weigh and record the weights of the syringe samples, clock time, and volumes in the lab notebook.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
255-C	If sampled hot, cool the syringes in the cooling fixture, to room temperature, using stoppers on the syringe tips.	
255-D	Filter two samples through a tared 0.45 micron filter. Collect the filtrate in a clean and dry suction flask.	
255-E	Use a clean, dry, disposable pipette to withdraw a five (5) mL sample for ICP analysis. Place in a 50 mL volumetric flask and notify the ICP operator.	
255-F	Withdraw another five (5) mL sample for EDTA analysis, if applicable. Analyze per ARC-TP-868.	
255-G	Obtain the pH value on the remaining solution and record in the lab notebook.	
260	The time is _____ 3.5-hours after reaching 200°F, it is time to monitor and record the test conditions as follows:	
260-A	Record the temperature in the lab notebook. Adjust as necessary to maintain 200°F ± 5°F.	
260-B	Record the flow rate in the lab notebook. Adjust as necessary.	
260-C	Record any observations of the test in the lab notebook.	
270	The time is _____ 4-hours after reaching 200°F, it is time to monitor and record the test conditions as follows:	
270-A	Record the temperature in the lab notebook.	
270-B	Record the flow rate in the lab notebook. Adjust as necessary.	
270-C	Record any observations of the test in the lab notebook.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

Charge # 43177-004-001	Project/Test DOE/Decon Bench Scale Test	Date
Sequence	Procedure	Initials
270-D	It is now time to start cooling the solvent, with the aid of the cooling coil, to less than 75°F.	
280	Record the temperature and velocity each half-hour until the 75°F target is reached. Record the time that 75°F was reached in the lab notebook and in step 300.	
290	During cooldown calculate the amount of 30 hydrogen peroxide that will be required for the H ₂ O ₂ injection.	
295	Independent check of the calculation performed in Step 290.	
300	The time is _____ and the target temperature of 75°F has been achieved, it is time to take three (3) samples of the solvent for analyses via the following procedure.	
300-A	Identify and tare weigh three clean, dry 12 mL syringes. Record this information in the lab notebook.	
300-B	Insert the first syringe tip into the rubber tube on the sample line on the lid of the reservoir. Pull a syringe full and push back into the reservoir. Do this three times to flush the line. Then draw a 12 mL sample.	
300-C	Take a second 12 mL sample with a separate tared syringe.	
300-D	Take a third 12 mL sample with a separate tared syringe for H ₂ O ₂ analysis. Transfer this sample to a 15 mL sample bottle and refrigerate for the analyst. Tighten the screw clamp on the tubing.	
300-E	Monitor and record the flow and temperature to ensure that they have not been disturbed. Adjust as necessary. Record any adjustments in the lab notebook.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
305	Handle and analyze the remaining samples as follows:	
305-A	Note that step 305 can be performed as time permits. Ensure that the timed steps of this procedure, such as Step 310, are performed as scheduled. Step 310 shall be performed as soon as possible after the Step 300 samples are taken.	
305-B	Weigh and record the weights of the syringe samples, clock time, and volumes in the lab notebook.	
305-C	If sampled hot, cool the syringes in the cooling fixture, to room temperature, using stoppers on the syringe tips.	
305-D	Filter two samples through a tared 0.45 micron filter. Collect the filtrate in a clean and dry suction flask.	
305-E	Use a clean, dry, disposable pipette to withdraw a five (5) mL sample for ICP analysis. Place in a 50 mL volumetric flask and notify the ICP operator.	
305-F	Withdraw another five (5) mL sample for EDTA analysis, if applicable. Analyze per ARC-TP-868.	
305-G	Obtain the pH value on the remaining solution and record in the lab notebook.	
310	Add the 30% peroxide calculated in Step 290 to the system via the in-line injection with the peristaltic pump.	
320	The time is _____ 0.5-hours after reaching 75°F, it is time to monitor and record the test conditions as follows:	
320-A	Record the temperature in the lab notebook.	
320-B	Record the flow rate in the lab notebook. Adjust as necessary.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

Charge # 43177-004-001	Project/Test DOE/Decon Bench Scale Test	Date
Sequence	Procedure	Initials
320-C	Record any observations of the test in the lab notebook.	
330	The pump will generate some heat and the solvent reactions are exothermic. Carefully watch solvent temperature and use the cooling coil, if required to maintain solvent temperature less than 85°F.	
340	The time is _____ 1-hour after reaching 75°F, it is time to take three (3) samples of the solvent for analyses via the following procedure.	
340-A	Identify and tare weigh three clean, dry 12 mL syringes. Record this information in the lab notebook.	
340-B	Insert the first syringe tip into the rubber tube on the sample line on the lid of the reservoir. Pull a syringe full and push back into the reservoir. Do this three times to flush the line. Then draw a 12 mL sample.	
340-C	Take a second 12 mL sample with a separate tared syringe.	
340-D	Take a third 12 mL sample with a separate tared syringe for H ₂ O ₂ analysis. Transfer this sample to a 15 mL sample bottle and refrigerate for the analyst. Tighten the screw clamp on the tubing.	
340-E	Monitor and record the flow and temperature to ensure that they have not been disturbed. Adjust as necessary. Record any adjustments in the lab notebook.	
345	Handle and analyze the remaining samples as follows:	
345-A	Note that step 345 can be performed as time permits. Ensure that the timed steps of this procedure, such as Step 350, are performed as scheduled.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Séquence</u>	<u>Procedure</u>	<u>Initials</u>
345-B	Weigh and record the weights of the syringe samples, clock time, and volumes in the lab notebook.	
345-C	If sampled hot, cool the syringes in the cooling fixture, to room temperature, using stoppers on the syringe tips.	
345-D	Filter two samples through a tared 0.45 micron filter. Collect the filtrate in a clean and dry suction flask.	
345-E	Use a clean, dry, disposable pipette to withdraw a five (5) mL sample for ICP analysis. Place in a 50 mL volumetric flask and notify the ICP operator.	
345-F	Withdraw another five (5) mL sample for EDTA analysis, if applicable. Analyze per ARC-TP-868.	
345-G	Obtain the pH value on the remaining solution and record in the lab notebook.	
350	The time is _____ 1.5-hours after reaching 75°F, it is time to monitor and record the test conditions as follows:	
350-A	Record the temperature in the lab notebook. Adjust as necessary to maintain the temperature less than 85°F.	
350-B	Record the flow rate in the lab notebook. Adjust as necessary.	
350-C	Record any observations of the test in the lab notebook.	
360	The time is _____ 2-hours after reaching 75°F, it is time to take three (3) samples of the solvent for analyses via the following procedure.	
360-A	Identify and tare weigh three clean, dry 12 mL syringes. Record this information in the lab notebook.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

Charge # 43177-004-001	Project/Test DOE/Decon Bench Scale Test	Date
Sequence	Procedure	Initials
360-B	Insert the first syringe tip into the rubber tube on the sample line on the lid of the reservoir. Pull a syringe full and push back into the reservoir. Do this three times to flush the line. Then draw a 12 mL sample.	
360-C	Take a second 12 mL sample with a separate tared syringe.	
360-D	Take a third 12 mL sample with a separate tared syringe for H ₂ O ₂ analysis. Transfer this sample to a 15 mL sample bottle and refrigerate for the analyst. Tighten the screw clamp on the tubing.	
360-E	Monitor and record the flow and temperature to ensure that they have not been disturbed. Adjust as necessary. Record any adjustments in the lab notebook.	
365	Handle and analyze the remaining samples as follows:	
365-A	Note that step 365 can be performed as time permits. Ensure that the timed steps of this procedure, such as Step 370, are performed as scheduled. Step 370 shall be performed as soon as possible after taking the Step 360 samples.	
365-B	Weigh and record the weights of the syringe samples, clock time, and volumes in the lab notebook.	
365-C	If sampled hot, cool the syringes in the cooling fixture, to room temperature, using stoppers on the syringe tips.	
365-D	Filter two samples through a tared 0.45 micron filter. Collect the filtrate in a clean and dry suction flask.	
365-E	Use a clean, dry, disposable pipette to withdraw a five (5) mL sample for ICP analysis. Place in a 50 mL volumetric flask and notify the ICP operator.	

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

Charge # 43177-004-001	Project/Test DOE/Decon Bench Scale Test	Date
Sequence	Procedure	Initials
365-F	Withdraw another five (5) mL sample for EDTA analysis, if applicable. Analyze per ARC-TP-868.	
365-G	Obtain the pH value on the remaining solution and record in the lab notebook.	
370	The time is _____ approximately 2-hours after reaching 75°F and the samples have been taken. It is time to drain the remaining solvent into a seven liter stainless steel beaker.	
375	Set the solvent aside for filtering when time permits.	
380	Retrieve and clean all corrosion specimens	
390	Rinse and dry the specimens in a drying oven for 15 minutes. Transfer specimens to a desiccator after drying to cool.	
400	Retrieve the remains of the uranium dioxide plug specimen.	
410	Weigh the coupons to the nearest 0.1 milligram. Record the weights in the lab notebook.	
420	Weigh the coupons by a second operator. Record the weights in the lab notebook.	
430	Compare the weights obtained by the two operators. The two weights must agree within 0.3 milligrams.	
430	Both operators must continue weighing, as necessary, until agreement is reached. If agreement cannot be reached, reclean and dry the specimen and repeat the weighing of the specimen until agreement is reached. Both operators must sign this step.	_____

TEST PROCEDURE AND SIGN-OFF SHEET
BENCH SCALE URANIUM DIOXIDE DISSOLUTION TESTING UNDER DYNAMIC
CONDITIONS

<u>Charge #</u> 43177-004-001	<u>Project/Test</u> DOE/Decon Bench Scale Test	<u>Date</u>
<u>Sequence</u>	<u>Procedure</u>	<u>Initials</u>
440	Clean the reservoir and the vessel of any residual deposit.	
450	Quantify the residual deposit obtained in Step 440.	
460	The test is over. Clean-up the lab. Good Job.	

APPENDIX B

TASK 4 TOPICAL REPORT

DOE CONTRACT DE-AC21-93MC30168

**RAW DATA FROM BENCH SCALE TESTS &
CORROSION AND MASS BALANCE CALCULATIONS**

DOE Uranium Dioxide Dissolution Testing

Date of test: 12/6/94

Solvent composition:

50 g/L EDTA + 20g/L AMMONIUM CARBONATE + 5.0g/L H₂O₂

ROOM TEMP - 200°F - ROOM TEMP @pH 9.0 (1)

FLOW 3GPM

Coupon #	Pre-Weight	Post-Weight	Weight Loss	S.A.(in ²)	Calculated General Corrosion (mils)
C1018-1	22.5231	22.2666	0.2565	3.75	0.531
C1018-2	23.0244	22.7649	0.2595	3.75	0.536
N200-1	24.5014	24.3939	0.1075	3.71	0.225
N200-2	24.5719	24.4645	0.1074	3.72	0.224
M400-1	24.3151	24.2596	0.0555	3.70	0.116
M400-2	24.5579	24.5004	0.0575	3.70	0.121

Volume Solvent Transferred

7000

Initial Density (g/L)

1.0330

Final Density (g/L)

1.0300

ORIGINAL SOL. EDTA 51.9g/L, pH 9.05

INJECT 116mL OF H₂O₂ AT ZERO HOURINJECT 116 mL H₂O₂ AFTER 7.0HR SAMPLE

ANALYTICAL RESULTS

Sample Time (Hours)	pH	g/L Chelant	g/L H ₂ O ₂	g/L Uranium	g/L Iron	g/L Copper	g/L Nickel	g/L Calcium	Sple Wt (g)	Sple Vol (ml)
0	8.93	44.3	6.8	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1 RT	8.87	37.5	-	0.355	0.010	<0.001	0.001	0.455	38.511	37.302
2 RT	8.83	33.8	-	0.604	0.012	0.001	0.001	0.778	39.004	37.791
1hr 200F	8.92	22.3	-	0.740	0.029	0.002	0.008	1.190	38.875	37.677
2hr 200F	8.32	21.4	-	0.810	0.075	0.005	0.028	1.536	38.543	37.366
0 hr RT	8.25	22.5	-	0.840	0.115	0.007	0.047	1.765	37.597	36.459
1 hr RT	7.69	18.4	-	0.877	0.115	0.008	0.047	1.863	37.114	36.002
2hr RT	7.67	16.8	0.2	0.885	0.116	0.008	0.047	1.911	37.564	36.449

GRAMS DISSOLVED

Elapsed Time (Hours)	Uranium Sample	Uranium Flask	Uranium Total	Iron Sample	Iron Flask	Iron Total
0	0.0000	0.00	0.00	0.00	0.00	0.00
1 RT	0.0132	2.47	2.49	0.00	0.07	0.07
2 RT	0.0228	4.18	4.22	0.00	0.09	0.09
1hr 200F	0.0279	5.10	5.16	0.00	0.20	0.20
2hr 200F	0.0303	5.55	5.64	0.00	0.51	0.52
0 hr RT	0.0306	5.72	5.85	0.00	0.78	0.79
1 hr RT	0.0316	6.05	6.20	0.00	0.79	0.81
2hr RT	0.0323	6.07	6.26	0.00	0.80	0.81

Elapsed Time<-----Copper----->			GRAMS DISSOLVED			12/6/94			-----Calcium-----		
(Hours)	Sample	Flask	Total	Sample	Flask	Total	Sample	Flask	Total		
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
1 RT	0.00	0.00	0.00	0.00	0.01	0.01	0.02	3.07	3.08		
2 RT	0.00	0.01	0.01	0.00	0.01	0.01	0.03	5.24	5.29		
1hr 200F	0.00	0.02	0.02	0.00	0.05	0.05	0.04	8.02	8.11		
2hr 200F	0.00	0.03	0.03	0.00	0.19	0.19	0.06	10.35	10.50		
0 hr RT	0.00	0.05	0.05	0.00	0.32	0.32	0.06	11.90	12.11		
1 hr RT	0.00	0.05	0.05	0.00	0.32	0.33	0.07	12.77	13.05		
2hr RT	0.00	0.05	0.05	0.00	0.32	0.33	0.07	13.10	13.45		

Syringe #	Sple Time	Tare Wt.	After Wt.	Sple Wt.	Sample Vol.		
1	1 RT	8.6413	21.1313	12.4900	12.095		
2	1 RT	8.5916	21.7984	13.2068	12.792		
3	1 RT	8.6359	21.4498	12.8139	12.415		
4	2 RT	8.6934	21.6769	12.9835	12.583		
5	2 RT	8.6192	21.6965	13.0773	12.678		
6	2 RT	8.6444	21.1265	12.4821	12.104		
7	1hr 200F	8.6249	20.6626	12.0377	11.677	Total Removed (g)	261.94
8	1hr 200F	8.7797	21.3741	12.5944	12.220		
9	1hr 200F	8.6811	21.6133	12.9322	12.552	Total Removed (mL)	254.38
10	2hr 200F	8.6540	21.3661	12.7121	12.342		
11	2hr 200F	8.7450	20.2653	11.5203	11.188		
12	2hr 200F	8.7097	21.1942	12.4845	12.128		
13	0 hr RT	8.6971	20.5152	11.8181	11.484		
14	0 hr RT	8.7020	21.2508	12.5488	12.198		
15	0 hr RT	8.6684	20.5366	11.8682	11.539		
16	1 hr RT	8.5401	21.5641	13.0240	12.667		
17	1 hr RT	8.7722	21.6656	12.8934	12.543		
18	1 hr RT	8.6778	19.6878	11.0100	10.714		
19	2hr RT	8.7380	20.9275	12.1895	11.866		
20	2hr RT	8.7444	21.3149	12.5705	12.240		
21	2hr RT	8.5859	21.2720	12.6861	12.356		

Coupon #	Length (in)	Width(in)	Height (in)	Hole D (in)	S.A. (in2)
C1018-1	2.0090	0.7625	0.1190	0.1235	3.75
C1018-2	2.0100	0.7600	0.1215	0.1235	3.75
N200-1	1.9995	0.7525	0.1235	0.1290	3.71
N200-2	1.9990	0.7540	0.1245	0.1290	3.72
M400-1	2.0045	0.7580	0.1160	0.1235	3.70
M400-2	2.0075	0.7555	0.1165	0.1245	3.70

Grams Uranium Dioxide Added to Flask 28 (PART OF PLUG 53g CaCO3 +53g SiO2 + 10g MORTAR)

Grams Uranium Dioxide Added to Flask 28 (PART OF PLUG 53g CaCO₃ +53g SiO₂ + 10g MORTAR)

Millipore #	Spile Time	Tare Wt.	After Wt.	Deposit Wt.
1	1 RT	0.0211	0.0256	0.0045
2	2 RT	0.0213	0.0275	0.0062
3	1hr 200F	0.0211	0.0278	0.0067
4	2hr 200F	0.0211	0.0261	0.0050
5	0hr RT	0.0211	0.0282	0.0071
6	1 hr RT	0.0211	0.0281	0.0070
7	2hr RT	0.0211	0.0305	0.0094

Total on Millipores (g) 0.0295

12/6/94

DOE Uranium Dioxide Dissolution Testing

Date of test: 12/7/94

Solvent composition:

20g/L AMMONIUM CARBONATE + 5.0g/L H2O2

ROOM TEMP - 200°F - ROOM TEMP @pH 9.0 (2)

FLOW 3GPM

Coupon #	Pre-Weight	Post-Weight	Weight Loss	S.A.(in2)	Calculated General Corrosion (mils)
C1018-3	22.7229	22.7230	-0.0001	3.74	-0.000
C1018-4	22.7648	22.7650	-0.0002	3.73	-0.000
N200-3	24.5266	24.5261	0.0005	3.67	0.001
N200-4	24.6800	24.6798	0.0002	3.69	0.000
M400-3	24.4791	24.4581	0.0210	3.70	0.044
M400-4	24.4301	24.4089	0.0212	3.70	0.044

Volume Solvent Transferred

7000

Initial Density (g/L)

1.0100

Final Density (g/L)

1.0300

ORIGINAL SOL. pH 9.07

INJECT 116mL OF H2O2 AT ZERO HOUR

INJECT 116 mL H2O2 AFTER 7.0HR SAMPLE

ANALYTICAL RESULTS

Sample Time (Hours)	pH	g/L Chelant	g/L H2O2	g/L Uranium	g/L Iron	g/L Copper	g/L Nickel	g/L Calcium	Sple Wt (g)	Sple Vol (ml)
0	9.08	NA	6.80	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1 RT	8.98	NA	-	0.441	0.017	0.002	0.006	0.297	21.765	21.478
2 RT	8.98	NA	-	0.510	0.016	0.001	0.006	0.308	25.049	24.612
0hr 200F	8.99	NA	-	0.554	0.017	0.002	0.005	0.296	23.683	23.168
1hr 200F	9.17	NA	-	0.620	0.018	0.003	0.007	0.312	24.191	23.562
2hr 200F	9.34	NA	-	0.638	0.019	0.004	0.009	0.340	24.329	23.595
0hr RT	9.56	NA	-	0.631	0.020	0.005	0.010	0.336	23.825	23.007
1 hr RT	9.52	NA	-	0.639	0.020	0.005	0.011	0.323	24.973	24.013
2hr RT	9.51	NA	0.03	0.641	0.020	0.006	0.011	0.310	24.155	23.128

GRAMS DISSOLVED

Elapsed Time (Hours)	Sample	Flask	Total	Sample	Flask	Total
0	0.0000	0.00	0.00	0.00	0.00	0.00
1 RT	0.0095	3.08	3.09	0.00	0.12	0.12
2 RT	0.0126	3.55	3.57	0.00	0.11	0.11
0hr 200F	0.0128	3.84	3.87	0.00	0.12	0.12
1hr 200F	0.0146	4.28	4.33	0.00	0.13	0.13
2hr 200F	0.0151	4.39	4.46	0.00	0.13	0.13
0hr RT	0.0145	4.33	4.41	0.00	0.14	0.14
1 hr RT	0.0153	4.44	4.54	0.00	0.14	0.14
2hr RT	0.0148	4.44	4.55	0.00	0.14	0.14

GRAMS DISSOLVED									
12/7/94									
Elapsed Time<	Copper----->			Nickel----->			Calcium----->		
(Hours)	Sample	Flask	Total	Sample	Flask	Total	Sample	Flask	Total
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1 RT	0.00	0.01	0.01	0.00	0.04	0.04	0.01	2.02	2.03
2 RT	0.00	0.01	0.01	0.00	0.04	0.04	0.01	2.10	2.11
0hr 200F	0.00	0.01	0.01	0.00	0.04	0.04	0.01	2.02	2.04
1hr 200F	0.00	0.02	0.02	0.00	0.05	0.05	0.01	2.13	2.15
2hr 200F	0.00	0.02	0.02	0.00	0.06	0.06	0.01	2.32	2.35
0hr RT	0.00	0.03	0.03	0.00	0.07	0.07	0.01	2.29	2.33
1 hr RT	0.00	0.04	0.04	0.00	0.08	0.08	0.01	2.24	2.29
2hr RT	0.00	0.04	0.04	0.00	0.08	0.08	0.01	2.15	2.21

Syringe #	Sple Time	Tare Wt.	After Wt.	Sple Wt.	Sample Vol.		
1	1 RT	8.6370	19.4140	10.7770	10.647		
2	1 RT	8.6287	19.6166	10.9879	10.831		
3	2 RT	8.5758	21.4128	12.8370	12.627		
4	2 RT	8.7129	20.9249	12.2120	11.986		
5	0hr 200F	8.6192	20.4823	11.8631	11.618		
6	0hr 200F	8.7202	20.5402	11.8200	11.550		
7	1hr 200F	8.6501	20.3570	11.7069	11.415	Total Removed (g)	191.97
8	1hr 200F	8.6491	21.1335	12.4844	12.147		
9	2hr 200F	8.6106	20.4696	11.8590	11.514	Total Removed (mL)	186.56
10	2hr 200F	8.6388	21.1090	12.4702	12.081		
11	0hr RT	8.6828	20.5581	11.8753	11.480		
12	0hr RT	8.6186	20.5680	11.9494	11.527		
13	1 hr RT	8.7186	21.0346	12.3160	11.855		
14	1 hr RT	8.6646	21.3220	12.6574	12.158		
15	2hr RT	8.6376	21.0500	12.4124	11.897		
16	2hr RT	8.5589	20.3017	11.7428	11.231		

Coupon #	Length (in)	Width(in)	Height (in)	Hole D (in)	S.A. (in2)
C1018-3	2.0090	0.7600	0.1195	0.1235	3.74
C1018-4	2.0080	0.7595	0.1195	0.1240	3.73
N200-3	2.0005	0.7520	0.1170	0.1255	3.67
N200-4	2.0020	0.7540	0.1170	0.1265	3.69
M400-3	2.0060	0.7590	0.1155	0.1245	3.70
M400-4	2.0095	0.7570	0.1150	0.1240	3.70

Grams Uranium Dioxide Added to Flask 28 (PART OF PLUG 53g CaCO₃ +53g SiO₂ +10g MORTAR)

Millipore #	Spile Time	Tare Wt.	After Wt.	Deposit Wt.
1	1 RT	0.0210	0.0227	0.0017
2	2 RT	0.0210	0.0220	0.0010
3	2:45 0hr 200F	0.0210	0.0220	0.0010
4	3:45 1hr 200F	0.0210	0.0217	0.0007
5	5:45 2hr 200F	0.0211	0.0213	0.0002
6	7:25 0hr RT	0.0210	0.0213	0.0003
7	8:25 1 hr RT	0.0210	0.0214	0.0004
8	9:25 2hr RT	0.0222	0.0222	0.0000

Total on Millipores (g) 0.0046

12/7/94

DOE Uranium Dioxide Dissolution Testing

Date of test: 12/14/94

Solvent composition:

50 g/L EDTA + 20g/L AMMONIUM CARBONATE + 5.0g/L H₂O₂

ROOM TEMP - 200°F - ROOM TEMP @pH 9.0 (3)

FLOW 3GPM

Coupon #	Pre-Weight	Post-Weight	Weight Loss	S.A.(in ²)	Calculated General Corrosion (mils)
C1018-5	22.5786	22.3832	0.1954	3.73	0.406
C1018-6	22.5760	22.4536	0.1224	3.74	0.254
N200-5	24.8513	24.6844	0.1669	3.68	0.351
N200-6	24.6266	24.4352	0.1914	3.69	0.402
M400-5	23.8404	23.7439	0.0965	3.68	0.203
M400-6	24.3713	24.2920	0.0793	3.70	0.166

Volume Solvent Transferred

7000

Initial Density (g/L)

1.0350

Final Density (g/L)

1.0300

ORIGINAL SOL. EDTA 51.2g/L, pH 9.06

INJECT 116mL OF H₂O₂ AT ZERO HOURINJECT 116 mL H₂O₂ AFTER 7.0HR SAMPLE

ANALYTICAL RESULTS

Sample Time (Hours)	pH	g/L Chelant	g/L H ₂ O ₂	g/L Uranium	g/L Iron	g/L Copper	g/L Nickel	g/L Calcium	Sple Wt (g)	Sple Vol (ml)
0	9.13	47.7	1.70	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1 RT	9.05	34.3	-	1.392	0.011	0.002	0.003	0.830	37.712	36.464
2 RT	9.05	29.5	0.05	1.915	0.013	0.002	0.003	1.022	38.462	37.203
0hr 200F	9.12	21.1	0.05	2.484	0.021	0.004	0.006	1.401	38.462	37.203
1hr 200F	9.08	17.8	0.15	2.484	0.044	0.004	0.016	1.492	36.832	35.638
2hr 200F	9.36	17.7	0.18	2.663	0.068	0.008	0.051	1.656	34.097	33.005
0hr RT	9.37	17.7	0.18	2.664	0.073	0.011	0.070	1.712	32.294	31.284
1 hr RT	9.23	15.9	1.00	2.591	0.071	0.011	0.068	1.717	33.204	32.191
2 hr RT	9.21	15.3	0.47	2.600	0.070	0.010	0.066	1.712	34.560	33.529

GRAMS DISSOLVED

Elapsed Time<-----Uranium----->				<-----Iron----->			
(Hours)	Sample	Flask	Total	Sample	Flask	Total	
0	0.0000	0.00	0.00	0.00	0.00	0.00	
1 RT	0.0508	9.69	9.74	0.00	0.07	0.07	
2 RT	0.0712	13.26	13.39	0.00	0.09	0.09	
0hr 200F	0.0924	17.11	17.33	0.00	0.15	0.15	
1hr 200F	0.0885	17.02	17.33	0.00	0.30	0.31	
2hr 200F	0.0879	18.16	18.55	0.00	0.46	0.47	
0hr RT	0.0833	18.09	18.56	0.00	0.50	0.50	
1 hr RT	0.0834	17.81	18.37	0.00	0.49	0.50	
2 hr RT	0.0872	17.78	18.43	0.00	0.48	0.49	

Elapsed Time<-----Copper----->			GRAMS DISSOLVED			Nickel----->			12/14/94-----Calcium----->		
(Hours)	Sample	Flask	Total	Sample	Flask	Total	Sample	Flask	Total		
0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00		
1 RT	0.00	0.01	0.01	0.00	0.02	0.02	0.03	5.58	5.61		
2 RT	0.00	0.02	0.02	0.00	0.02	0.02	0.04	6.87	6.94		
0hr 200F	0.00	0.02	0.03	0.00	0.04	0.04	0.05	9.42	9.54		
1hr 200F	0.00	0.03	0.03	0.00	0.11	0.11	0.05	10.03	10.21		
2hr 200F	0.00	0.05	0.05	0.00	0.34	0.35	0.05	11.13	11.36		
0hr RT	0.00	0.07	0.07	0.00	0.47	0.47	0.05	11.51	11.79		
1 hr RT	0.00	0.07	0.07	0.00	0.46	0.47	0.06	11.74	12.08		
2 hr RT	0.00	0.07	0.07	0.00	0.45	0.46	0.06	11.71	12.10		

Syringe #	Sple Time	Tare Wt.	After Wt.	Sple Wt.	Sample Vol.		
1	1 RT	8.5497	20.8927	12.3430	11.930		
2	1 RT	8.7150	21.2490	12.5340	12.119		
3	1 RT	8.5736	21.4084	12.8348	12.415		
4	2 RT	8.7348	21.8277	13.0929	12.669		
5	2 RT	8.6675	19.5713	10.9038	10.555		
6	2 RT	8.6595	18.7601	10.1006	9.781		
7	0hr 200F	8.7965	21.2995	12.5030	12.112		
8	0hr 200F	8.5644	20.6535	12.0891	11.715		
9	0hr 200F	8.7304	20.7978	12.0674	11.698		
10	1hr 200F	8.6212	19.9108	11.2896	10.949	Total Removed (g)	291.23
11	1hr 200F	8.5577	20.3715	11.8138	11.461		
12	1hr 200F	8.6294	20.0861	11.4567	11.119	Total Removed (mL)	282.71
13	2hr 200F	8.6925	20.7260	12.0335	11.683		
14	2hr 200F	8.6220	20.6169	11.9949	11.650		
15	2hr 200F	8.7276	20.9309	12.2033	11.857		
16	0hr RT	8.6371	21.3571	12.7200	12.363		
17	0hr RT	8.6510	20.9073	12.2563	11.917		
18	0hr RT	8.6856	20.7617	12.0761	11.746		
19	1 hr RT	8.7256	21.1217	12.3961	12.062		
20	1 hr RT	8.7374	22.1526	13.4152	13.059		
21	1 hr RT	8.7841	21.1937	12.4096	12.084		
22	2hr RT	8.6792	21.5368	12.8576	12.525		
23	2hr RT	8.5416	20.6904	12.1488	11.839		
24	2hr RT	8.6151	20.3093	11.6942	11.400		

Coupon #	Length (in)	Width(in)	Height (in)	Hole D (in)	S.A. (in2)
C1018-5	2.0015	0.7610	0.1200	0.1235	3.73
C1018-6	2.0100	0.7600	0.1195	0.1240	3.74
N200-5	2.0010	0.7535	0.1175	0.1240	3.68
N200-6	2.0025	0.7545	0.1170	0.1235	3.69

Grams Uranium Dioxide Added to Flask 28 (PART OF PLUG 28g CaCO₃ + 28g SiO₂ + 6g MORTAR)

Millipore #	Spile Time	Tare Wt.	After Wt.	Deposit Wt.
1	1 RT	0.0220	0.0309	0.0089
2	2 RT	0.0220	0.0290	0.0070
3	0hr 200F	0.0208	0.0271	0.0063
4	1hr 200F	0.0202	0.0266	0.0064
5	2hr 200F	0.0205	0.0250	0.0045
6	0hr RT	0.0206	0.0270	0.0064
7	1 hr RT	0.0205	0.0293	0.0088
8	2hr RT	0.0207	0.0321	0.0114

Total on Millipores (g) 0.0331

12/14/94

DOE Uranium Dioxide Dissolution Testing

Date of test: 1/6/95

Solvent composition:

20g/L AMMONIUM CARBONATE + 5.0g/L H₂O₂

ROOM TEMP - 200°F - ROOM TEMP @pH 9.0 (4)

FLOW 3GPM

Coupon #	Pre-Weight	Post-Weight	Weight Loss	S.A.(in ²)	Calculated General Corrosion (mils)
C1018-7	22.6660	22.6659	0.0001	3.76	0.000
C1018-8	22.5153	22.5149	0.0004	3.73	0.001
N200-7	24.4526	24.4507	0.0019	3.69	0.004
N200-8	24.4038	24.4027	0.0011	3.67	0.002
M400-7	24.4118	24.4115	0.0003	3.69	0.001
M400-8	24.4025	24.4021	0.0004	3.70	0.001

Volume Solvent Transferred

7000

Initial Density (g/L)

1.0100

Final Density (g/L)

1.0300

ORIGINAL SOL. pH 9.00

INJECT 116mL OF H₂O₂ AT ZERO HOURINJECT 116 mL H₂O₂ AFTER 7.0HR SAMPLE

ANALYTICAL RESULTS

Sample Time (Hours)	pH	g/L Chelant	g/L H ₂ O ₂	g/L Uranium	g/L Iron	g/L Copper	g/L Nickel	g/L Calcium	Sple Wt (g)	Sple Vol (ml)
0	9.17	NA	7.72	0.000	0.000	0.000	0.000	0.000	0.000	0.000
1 RT	9.15	NA	-	0.209	0.002	<0.001	0.002	0.040	24.296	23.976
2 RT	9.12	NA	-	0.261	0.002	<0.001	0.002	0.040	24.637	24.206
0hr 200F	9.34	NA	-	0.356	0.003	<0.001	0.001	0.032	24.397	23.867
1hr 200F	9.54	NA	-	0.450	0.004	<0.001	0.002	0.041	24.407	23.773
3hr 200F	9.51	NA	-	0.450	0.004	<0.001	0.002	0.053	23.706	22.992
0hr RT	9.64	NA	-	0.400	0.004	<0.001	0.002	0.056	21.635	20.891
1 hr RT	9.41	NA	3.47	0.330	0.003	<0.001	0.001	0.041	24.984	24.023
2hr RT	9.39	NA	2.72	0.330	0.003	<0.001	0.001	0.040	24.614	23.567

GRAMS DISSOLVED

Elapsed Time (Hours)	Uranium			Iron		
	Sample	Flask	Total	Sample	Flask	Total
0	0.0000	0.00	0.00	0.00	0.00	0.00
1 RT	0.0050	1.46	1.46	0.00	0.02	0.02
2 RT	0.0063	1.81	1.83	0.00	0.02	0.02
0hr 200F	0.0085	2.47	2.49	0.00	0.02	0.02
1hr 200F	0.0107	3.11	3.14	0.00	0.03	0.03
3hr 200F	0.0103	3.10	3.14	0.00	0.03	0.03
0hr RT	0.0084	2.74	2.79	0.00	0.03	0.03
1 hr RT	0.0079	2.29	2.35	0.00	0.02	0.02
2hr RT	0.0078	2.29	2.35	0.00	0.02	0.02

GRAMS DISSOLVED									
1/6/95									
Elapsed Time<	Copper			>	Nickel			<	Calcium
(Hours)	Sample	Flask	Total		Sample	Flask	Total	Sample	Flask
0	0.00	0.00	0.00		0.00	0.00	0.00	0.00	0.00
1 RT	0.00	0.00	0.00		0.00	0.01	0.01	0.00	0.27
2 RT	0.00	0.00	0.00		0.00	0.01	0.01	0.00	0.27
0hr 200F	0.00	0.00	0.00		0.00	0.01	0.01	0.00	0.22
1hr 200F	0.00	0.00	0.00		0.00	0.01	0.01	0.00	0.28
3hr 200F	0.00	0.00	0.00		0.00	0.01	0.01	0.00	0.36
0hr RT	0.00	0.00	0.00		0.00	0.01	0.01	0.00	0.38
1 hr RT	0.00	0.00	0.00		0.00	0.01	0.01	0.00	0.28
2hr RT	0.00	0.00	0.00		0.00	0.01	0.01	0.00	0.28

Syringe #	Sple Time	Tare Wt.	After Wt.	Sple Wt.	Sample Vol.	
1	1 RT	8.6903	20.6674	11.9771	11.832	
2	1 RT	8.7111	21.0298	12.3187	12.143	
3	2 RT	8.7751	20.9903	12.2152	12.015	
4	2 RT	8.6363	21.0581	12.4218	12.192	
5	0hr 200F	8.6501	21.0479	12.3978	12.141	
6	0hr 200F	8.7345	20.7335	11.9990	11.725	
7	1hr 200F	8.5750	20.5795	12.0045	11.705	Total Removed (g)
8	1hr 200F	8.6469	21.0494	12.4025	12.067	192.68
9	3hr 200F	8.6713	21.1446	12.4733	12.110	Total Removed (mL)
10	3hr 200F	8.6121	19.8443	11.2322	10.882	187.29
11	0hr RT	8.6718	19.2115	10.5397	10.189	
12	0hr RT	8.6868	19.7819	11.0951	10.703	
13	1 hr RT	8.5883	20.6836	12.0953	11.643	
14	1 hr RT	8.5505	21.4396	12.8891	12.380	
15	2hr RT	8.7724	21.0058	12.2334	11.725	
16	2hr RT	8.7159	21.0966	12.3807	11.841	

Coupon #	Length (in)	Width(in)	Height (in)	Hole D (in)	S.A. (in2)
C10186-7	2.0120	0.7630	0.1200	0.1240	3.76
C1018-8	2.0050	0.7600	0.1195	0.1235	3.73
N200-7	2.0025	0.7565	0.1160	0.1240	3.69
N200-8	1.9990	0.7540	0.1160	0.1240	3.67
M400-7	2.0065	0.7565	0.1155	0.1255	3.69
M400-8	2.0030	0.7590	0.1160	0.1255	3.70

Grams Uranium Dioxide Added to Flask 28 (PART OF PLUG 53g CaCO₃ +53g SiO₂ +10g MORTAR)

Millipore #	Spile Time	Tare Wt.	After Wt.	Deposit Wt.
1	1 RT	0.0207	0.0211	0.0004
2	2 RT	0.0205	0.0211	0.0006
3	0hr 200F	0.0204	0.0205	0.0001
4	1hr 200F	0.0208	0.0208	0.0000
5	3hr 200F	0.0207	0.0212	0.0005
6	0hr RT	0.0205	0.0216	0.0011
7	1 hr RT	0.0207	0.0207	0.0000
8	2hr RT	0.0207	0.0221	0.0014

1/6/95

Total on Millipores (g) 0.0016