Revision 1

# **APPENDIX I**

# SURFACE ANALYSIS OF CORROSION TEST SPECIMENS FOR DEALLOYING (RESPONSE TO CLST 1.02)

#### Note Regarding the Status of Supporting Technical Information

This document was prepared using the most current information available at the time of its development. This Technical Basis Document and its appendices providing Key Technical Issue Agreement responses that were prepared using preliminary or draft information reflect the status of the Yucca Mountain Project's scientific and design bases at the time of submittal. In some cases this involved the use of draft Analysis and Model Reports (AMRs) and other draft references whose contents may change with time. Information that evolves through subsequent revisions of the AMRs and other references will be reflected in the License Application (LA) as the approved analyses of record at the time of LA submittal. Consequently, the Project will not routinely update either this Technical Basis Document or its Key Technical Issue Agreement appendices to reflect changes in the supporting references prior to submittal of the LA.

### **APPENDIX I**

#### SURFACE ANALYSIS OF CORROSION TEST SPECIMENS FOR DEALLOYING (RESPONSE TO CLST 1.02)

This appendix provides a response to Key Technical Issue (KTI) agreement Container Life and Source Term (CLST) 1.02. This KTI agreement relates to surface analysis of welded specimens, particularly dissolution and dealloying potentially resulting from microbially influenced corrosion.

## I.1 KEY TECHNICAL ISSUE AGREEMENT

## I.1.1 CLST 1.02

Agreement CLST 1.02 was reached during the U.S. Nuclear Regulatory Commission (NRC)/U.S. Department of Energy (DOE) Technical Exchange and Management Meeting on Container Life and Source Term held September 12 and 13, 2000, in Las Vegas, Nevada. Subissues 1 (effects of corrosion processes on the lifetime of the containers), 2 (effects of phase instability and initial defects on the mechanical failure and lifetime of the containers), 3 (the rate at which radionuclides in spent nuclear fuel are released from the engineered barrier subsystem through the oxidation and dissolution of spent nuclear fuel), 4 (the rate at which radionuclides in high-level radioactive waste glass are released from the engineered barrier subsystem), and 6 (effects of alternate engineered barrier subsystem design features on container lifetime and radionuclide release from the engineered barrier subsystem) were discussed at the meeting (Schlueter 2000). There has been no previous submittal to the NRC related to this agreement.

Wording of the agreement is as follows:

## CLST 1.02

Provide the documentation for the path forward items listed on slide 12. DOE will provide the documentation in a revision to AMR "General and Localized Corrosion of Waste Package Outer Barrier" by LA.

Slide 12, as cited in the agreement, pertained to the subject of microbially influenced corrosion effects on Alloy 22 (i.e., surface elemental analysis of alloy test specimens for determination of selective dissolution, surface analysis of welded specimens for evidence of dealloying, and continued testing including simulated saturated repository environment to confirm the corrosion enhancement factor). Consequently, the response to this agreement is structured within this context.

Agreement GEN 1.01 was reached during the NRC/DOE Technical Exchange and Management Meeting on Range of Thermal Operating Temperatures held September 18 to 19, 2001. Associated with that meeting, the DOE provided initial responses to GEN 1.01 comments 21 and 64 that referred in part to CLST 1.02 (Reamer and Gil 2001). Comments 21 and 64, however, are related to postclosure criticality and will not be addressed here; they are more appropriately addressed in the response to agreement CLST 5.03.

# I.1.2 Related Key Technical Issue Agreements

CLST 1.02 is related to CLST 1.15 (Appendix D) where specimens for microbially influenced corrosion studies are discussed.

# **I.2 RELEVANCE TO REPOSITORY PERFORMANCE**

Microbially influenced corrosion could promote degradation of the waste package container materials, and hence degrade the integrity of the waste package, thereby impacting the performance of the repository over the regulatory period.

# I.3 RESPONSE

The Slide 12 path forward items were directed toward studying the microbially influenced corrosion effects by surface elemental analysis of alloy test specimens for determination of selective dissolution and by surface analysis of welded specimens for evidence of dealloying and continued testing in saturated repository environments to confirm the enhancement factor. The studies conducted to date have covered the items described in Slide 12.

Microbially influenced corrosion could affect materials degradation in several ways: (1) enhanced general dissolution due to general chemical environment change, (2) localized corrosion due to microenvironmental change, and (3) selective dissolution (dealloying) of one or more of the alloy components. Experimental evidence of the effect of Yucca Mountain microbes on Alloy 22 indicates a localized effect that takes the form of submicron-sized degradation in nutrient-enhanced aqueous solutions after almost 5 years of exposure.

Comparative analysis of solutions in which Alloy 22 base metal had been incubated for 5 months at room temperature with either a mixture of Yucca Mountain bacteria or left sterile indicated a higher level of nickel and chromium in microbe-containing solutions than in sterile solutions. That is, that nonstoichiometric dissolution of the alloy may occur in a microbially influenced environment. This was observed for nonwelded coupons, but the same may happen for welded coupons. Sputtering x-ray photoelectron spectroscopy of microbe-colonized Alloy 22 indicated the presence of alloying elements in the biofilm layer. Scanning electron microscopy analysis of Alloy 22 samples exposed to microbial environments showed submicron-sized degradation in nutrient-enhanced aqueous solutions after almost 5 years of exposure; no such degradation was seen in sterile controls after 43 months of exposure (Figure I-1).

Linear polarization studies indicate an approximately two-fold greater dissolution rate of nonwelded Alloy 22 in nutrient-enhanced nonsterile microbial environments relative to a sterile environment. In terms of general corrosion rate, the dissolution rate was  $0.022 \,\mu$ m/yr under nonsterile biotic conditions and  $0.011 \,\mu$ m/yr under sterile conditions. Initial results using Alloy 22 welded coupons show that the corrosion rate under biotically incubated conditions was on the same order as that observed for nonwelded coupons and in the range between 10 and 35 nm/yr (Figure I-2). However, the corrosion rates for the coupons exposed to the sterile conditions are, in general, lower than for the nonsterile conditions. A few corrosion rate data for the sterile conditions seem higher because of contamination and intermediate sterilization. The microbially influenced corrosion enhancement factor both for welded and nonwelded Alloy 22 coupons seems similar and approximately equal to 2. It should be noted, however, that

conditions between linear polarization experiments for base metal and weldments differed in that nutrient supply was significantly greater for base metal experiments conducted in batch mode with no aeration. In contrast, weldment specimens are being incubated with  $100 \times J$ -13 water supplemented with 0.1% glucose, a more representative but less nutrient-rich environment than used previously; these experiments are also continually aerated and supplied with simulated groundwater on a flow-through basis.





Sterile Control

Nonsterile



Source: DTN: LL040303612251.078.

- NOTE: Coupons were (a) unexposed to a microcosm environment, (b) exposed for 43 months in a sterile microcosm, and (c) exposed for 57 months in a nonsterile, biotic microcosm.
- Figure I-1. Comparison of Scanning Electron Microscopy–Imaged Alloy 22 Coupons (8,000× Magnification)

Most of the results presented here were obtained for nonwelded coupons; however, it is noted that similar corrosion rate results are being obtained for welded coupons.

The information in this report is responsive to agreement CLST 1.02 made between the DOE and NRC. The report contains the information that the DOE considers necessary for NRC review for closure of this agreement.



Source: DTN: LL040402912251.085.

Figure I-2. Polarization Resistance Testing of Welded Alloy 22 Coupons

## I.4 BASIS FOR THE RESPONSE

#### I.4.1 Introduction

Microorganisms, those native to Yucca Mountain and those that have been introduced, have metabolic activities that may cause corrosion of waste package materials. Generally, microbially influenced corrosion could directly induce localized attack through, for example, the production of acids, which would then influence dealloying, or indirectly influence bulk chemical conditions that damage waste package materials. These processes would only be expected to occur if a sufficient number of organisms were present (requiring growth), and the organisms remained active (rather than dormant or dead). Previous studies have shown that Yucca Mountain rock contains  $4.0 \times 10^4$  to  $6.9 \times 10^4$  microbial cells per gram of dry rock and that the lack of water is the primary limiting factor preventing microbial growth in Yucca Mountain. Both phosphate and carbon availability were also identified as growth-limiting factors, but they have a less dramatic effect on growth levels than water. It was determined that adding just unconcentrated simulated Yucca Mountain groundwater (without phosphate or carbon amendment) to nonsterilized Yucca

Mountain rock resulted in steady-state cell densities of at least  $1.3 \times 10^6$  cells per milliliter (Horn et al. 2003). Therefore, it appears that given sufficient groundwater availability within Yucca Mountain and the abatement of other inopportune conditions (e.g., radiation fields), microorganisms will eventually grow. However, it should be noted that the aqueous solutions used in the microbial corrosion tests described here were enhanced with a carbon source, glucose.

Approximately 65 species of microorganisms were identified through molecular analysis of DNA isolated from Yucca Mountain rock. Many of these species are able to withstand periods of desiccation and elevated temperatures, low nutrient concentrations, and can degrade complex organic compounds, produce organic acids and extracellular polymers, and consume nitrate (Horn et al. 2003). The results described here, as well as ongoing investigation, evaluate the effects of Yucca Mountain organisms on both material corrosion and groundwater chemistry.

# I.4.2 Corrosion Rate

Linear polarization studies were performed on Alloy 22 base metal coupons in batch mode over a period of 5 months in a nutrient-enhanced media. This level of nutrients, in particular the carbon source, exceeds that currently present in the unaltered repository horizon; hence, the testing is conservative in this respect. The organic carbon level used in the test media was 0.1 wt % glucose, whereas it is only present in trace amounts in groundwater at Yucca Mountain and in its vicinity (Kerrisk 1987, p. 45). The study was carried out on coupons inoculated with microorganisms isolated from Yucca Mountain rock that had been previously characterized as having activities associated with corrosion (i.e., sulfate reduction, production of extracellular polysaccharides, acid production, or iron oxidation) in a medium composed of nutrients and concentrated carbonate type groundwater as solvent at the concentration of 100× J-13 well water. The medium was not replenished nor were the vessels (Figure I-3) aerated over the 5-month duration of the experiment; vessels were also not deaerated before or during the experimental procedures. Experiments were carried out at room temperature. It should be noted that earlier studies indicated that there were no general corrosion effects at 60°C or at 90°C. Also, microbial activity was evident at 60°C and not at 90°C. In general, ambient temperature conditions are more conservative from the standpoint of microbial effects. As a control to account for abiotic corrosion rates under these experimental conditions at ambient temperature, sterile coupons were incubated in parallel under the identical conditions as biotic coupons. Linear polarization results indicated that after an initial period of instability, corrosion rates reached a steady state, averaging 0.022 µm/yr under nonsterile biotic conditions and 0.011 µm/yr under sterile conditions, indicating that the inoculated microorganisms increased the general corrosion rate of Alloy 22 by a factor of 2 (Table I-1). The average corrosion rate data on other materials in Table I-1 provide a comparison with the Alloy 22 data.



Figure I-3. Configuration of Experimental Vessels Used for Electrochemical and Dissolution Experiments

Table I-1.	Ecorr and Calculated Corrosion Rates Determined for Alloy 22 and Other Materials in
	Preliminary Linear Polarization Testing

Tested Sample Initial Condition	Average Corrosion Rate (um/vr)	Corrosion Potential <i>E<sub>corr</sub></i> (V vs. SCE)		
		Initial	Endpoint	
CS1020 + Yucca Mountain Microbes	8.8	-0.660	-0.685	
Sterile CS1020	1.4	-0.500	-0.550	
M400 + Yucca Mountain Microbes	1.02	-0.415	-0.315	
Sterile M400	0.005	-0.135	-0.070	
Alloy 22 + Yucca Mountain Microbes	0.022	-0.440	-0.252	
Sterile Alloy 22	0.011	-0.260	-0.200	
l625 + Yucca Mountain Microbes	0.013	-0.440	-0.285	
Sterile I625	0.003	-0.160	-0.130	
Stainless Steel Type 304 + Yucca Mountain Microbes	0.035	-0.540	-0.280	
Sterile Stainless Steel Type 0.003 304		-0.145	-0.065	

Source: Lian et al. 1999.

NOTE: V = potential in volts; SCE = saturated calomel electrode.

## I.4.3 Microbially Influenced Corrosion Studies of Welded Coupons

Corrosion testing of welded Alloy 22 coupons were conducted for more than five years in the Long Term Corrosion Test Facility (LTCTF) at Lawrence Livermore National Laboratory. Tests were carried out in concentrated simulated groundwaters. The environments were not initially sterilized and the test solutions were intermittently exposed to the laboratory air; hence; ambient microbes could have become established. Indeed, characterization tests showed the presence of heat-tolerant, desiccation-tolerant, and radiation-tolerant bacteria in the simulated dilute water at 60°C (Vessel 29). Simulated dilute water has a pH near 10 and is ten times more concentrated than the well J-13 water. Analyses of the simulated groundwater in Vessel 25 (simulated acidified water at 60°C) also showed the presence of heat-tolerant, desiccation-tolerant, and acidtolerant bacteria. Simulated acidified water is pH 3 and is approximately 1,000 times more concentrated than J-13 water. Heat and desiccation tolerant microorganisms were also detected in the analysis of rock from the repository site. That is, there are a few types of microorganisms that were found both in the LTCTF vessels and at the repository site. However, most of the organisms that survived in the LTCTF were the ones best adapted to the severe conditions in the vessels, and could indicate the types of microorganisms that will colonize the repository if sufficient water is available and extreme conditions are maintained. These tests in the LTCTF are continuing. The 5-year immersion tests from LTCTF showed, in general, low corrosion rates both for the welded and nonwelded Alloy 22 coupons (10 nm/yr and lower).

In addition, electrochemical corrosion rate results have been obtained on welded Alloy 22 coupons exposed to 100× J-13 water plus 0.1% glucose at ambient temperature. The testing was carried out in both Yucca Mountain microorganism-inoculated and sterile environments using the techniques of ASTM G 59-97, Standard Test Method for Conducting Potentiodynamic Polarization Resistance Measurements, as performed on base metal. However, the testing conditions for the welded coupons are believed to be more aggressive than the conditions used for the nonwelded coupons shown in Figure I-3. For the nonwelded coupon testing, the tests were run in a batch mode with no active aeration or deaeration. In contrast, for the welded coupon testing, there is a continuous flow of the electrolyte, and, in addition, the test cell is actively aerated. While the nutrient level in the aqueous solution used for the nonwelded coupon batch testing is initially higher than that in the aqueous solutions used in the welded coupon continuous flow testing, the welded coupons are exposed to greater total nutrient because of the long duration of the testing. Recent initial results show (Figure I-2) that the corrosion rates obtained for welded coupons in nonsterile environments were slightly higher than those in sterile environments and consistent with the enhancement factor obtained from the nonwelded coupon data shown in Table I-1. Data in Figure I-2 also show that the corrosion rates for welded coupons are essentially the same as those for nonwelded coupons in Table I-1. The data in Figure I-2 are for three testing cells in sterile conditions and three testing cells in nonsterile conditions. Some of the data points in sterile conditions appear to be high (e.g., for times between 70 and 90 days); however, this was the result of contamination of the sterile control which required autoclaving of the cell (resterilization). After a few days of testing the results for the sterile cells return to the lower baseline.

Based on the consistency in the corrosion rates measured in the welded and base metal samples (previous section) no apparent dealloying is expected in the welded samples. However, detailed

surface analyses are planned for the welded coupons now being tested in sterile and inoculated environments.

# I.4.4 Analysis of Test Media from Corrosion Tests

After 5 months of incubation, at the termination of linear polarization studies on base metal, aqueous media from both bacterially inoculated and sterile corrosion vessels were sampled and subjected to inductively coupled plasma atomic emission spectrometry analysis to determine the concentrations of solubilized Alloy 22 elements (Table I-2). For comparison with the data on Alloy 22, Table I-2 also presents concentration of dissolved metals data for other materials. Fresh medium, which had never been exposed to corrosion vessels, was likewise assessed for comparison. Results showed chromium concentrations (approximately 1 ppm) in test vessels that had been inoculated and incubated with Yucca Mountain bacteria, but no chromium was detected in sterile vessels incubated for the same period or in unexposed media. Additionally, nickel (0.1 ppm) was found solubilized in vessels incubated with Yucca Mountain bacteria, but none was detected in media from sterile control experiments or in fresh medium. Iron was detected in all media tested. The presence of solubilized chromium and nickel in vessels incubated under biotic conditions suggested that the applied Yucca Mountain bacteria might be causing surface dissolution from Alloy 22. In order to further assess the degradation process, elemental analyses of the surface of Alloy 22 specimens were performed (see below).

Conditions/Material	Chromium (ppm)	Nickel (ppm)	Molybdenum (ppm)
Sterile Stainless Steel Type 304	ND	ND	ND
Bacteria + Stainless Steel Type 304	1.03	0.04	ND
Sterile I625	ND	ND	ND
Bacteria + I625	1.07	0.12	ND
Sterile Alloy 22	ND	ND	ND
Bacteria + Alloy 22	1.05	0.1	ND

Table I-2.Concentrations of Dissolved Metals from Alloy 22 and Other Materials in Medium after<br/>5-Month Linear Polarization Testing

Source: Lian et al. 1999.

NOTE: ND = not detectable.

Other investigators (Dunn et al. 2001) have found apparent noncongruent dissolution of potentiostatically polarized Alloy 22 in 95°C, 0.028 mol/L NaCl solution without inoculated bacteria based solely on solution analysis, with posttest solution analyses indicating chromium enrichment and essentially no presence of nickel. However, after further analyses of deposits on some of the posttest corrosion test cell surfaces (a platinum electrode surface and the specimen surface), significant differences were found in the Ni:Cr ratios on each surface, and both surface ratios were different from the ratio in the solution. These differences made it difficult to confirm congruent dissolution without performing a complete mass balance for the entire system.

In a subsequent study, Dunn and Brossia (2002) modified the electrochemical test cell to maximize the Alloy 22 surface area relative to the solution volume. Following the test, they chemically dissolved all plated-out or precipitated Alloy 22 corrosion products before

performing chemical analysis using inductively coupled plasma mass spectroscopy to determine the total inventory of soluble corrosion products. The tests were performed in 60°C KCl with 1,000 ppm chloride, and the concentration of soluble species was monitored over time (at approximately 100-hour intervals) for about 950 hours. They found that the soluble chromium content remained relatively constant over time (the first analysis was at 96 hours exposure), whereas the nickel content and, to a lesser extent, the molybdenum content increased in an approximately linear manner. Thus, the Ni:Cr ratio increased to an equilibrium state with time, indicating an approach to congruent dissolution rather than dealloying.

# I.4.5 Surface Analysis of Alloy 22 Corrosion Test Samples

Surface Analysis of Sample from 5-Month Linear Polarization Study-Scanning electron microscopy images at 1,000× to 10,000× magnification showed particulates on the surfaces of Alloy 22 coupons incubated in linear polarization studies; these were very numerous on the bacterially inoculated coupons (data not shown). The corrosion cell design that was used for these experiments had the Alloy 22 sample, the working electrode, emplaced at the base of the cell, forming the floor of the apparatus (Figure I-3). Therefore, if homogeneous general corrosion was occurring and some of the elements had reprecipitated, then it was possible that at least some of this material might be deposited on the Alloy 22 sample (Figure I-4a). Energy dispersive spectral analysis was used to determine the elemental composition of detected precipitates on coupon surfaces. Energy dispersive spectral analyses in spot mode showed that one particulate on a nonsterile coupon was chromium rich, although most were silica enriched. The observed silica precipitated from the Yucca Mountain groundwater fraction of the growth media, while the chromium was most probably a result of the reduction of solubilized chromium(VI) to chromium(III) with subsequent precipitation of insoluble chromium complexes, which occurs readily in solutions containing organic matter (Baetjer et al. 1974). Energy dispersive spectral analysis dot maps of six separate areas each on inoculated and unexposed coupons, however, did not show any significant differences in elemental compositions between any of the inspected regions. This indicates that no reprecipitated alloying elements with significantly different composition compared to the base metal and present in sufficient quantity were discernable on biotically incubated coupon surfaces. The analysis is inconclusive to discern between the two scenarios in Figures I-4a and I-4b because neither the alloy element precipitates nor a dealloyed layer could be discerned from the signature of the base metal

**Depth Profile of Alloy 22 Samples from Linear Polarization Study**–The depth of penetration of the electron beam used for energy dispersive spectral analysis on the surface of the studied linear polarization coupons may be as great as 1  $\mu$ m. It was, therefore, possible that impinging electrons could penetrate as far as unaffected base metal, accounting for the lack of elemental difference that was evident using energy dispersive spectral analysis dot mapping between differently treated samples (see above). Therefore, less penetrating, higher-resolution sputter x-ray photoelectron spectroscopy analysis was used to evaluate the surface layers of coupons to determine elemental compositions on an inoculated coupon and one incubated under sterile conditions. Sputtering into the surface of sterile, inoculated, and unexposed coupons at a rate of 2 nm/min coupled with x-ray photoelectron spectroscopy analysis of sputtered material at predetermined intervals showed marked differences between sterile, inoculated, and unexposed coupons.



(c) Resolution of Sputtering X-Ray Photoelectron Spectroscopy Analysis



NOTE: (a) Extant layered material if generalized stoichiometric dissolution of Alloy 22 is occurring; note that even if only select alloying elements are found solubilized, others could exist as precipitates on the surface of the coupon; (b) extant layered material if selective dissolution of Alloy 22 is occurring. The aim of sputtering x-ray photoelectron spectroscopy analysis is to distinguish between the scenario depicted in (a) versus (b); (c) the roughness of the Alloy 22 coupons (600 grit) and associated layers of material. x-ray photoelectron spectroscopy analysis (1 mm) covers a greater area than the scale of roughness of the Alloy 22 coupons. Therefore, for any given depth of analysis, more than a single layer of material can be sampled, producing a loss of resolution in analytical results between the layers.

Figure I-4. Configuration of Potential Layers of Materials on Biotically Incubated Alloy 22 Coupons

The inoculated coupon had a thick layer of carbonaceous material that asymptotically fell to low levels after sputtering approximately 400 nm into the surface. The sterile control coupon, in contrast, had a thin layer of surface carbon that decreased to less than 10% of the total carbon present within 30 nm of the surface (Figure I-5). Therefore, at any given depth of analysis, differently treated coupons could not be compared; clearly, the inoculated surface of Alloy 22 after 5 months of incubation had an extensive biofilm coating that was not present on the sterile control. Furthermore, because the surfaces of these coupons were polished to 600 grit they were relatively rough, while the area of analysis was comparatively large, on the order of a square millimeter. Therefore, at any given depth of analysis, more than a single layer of material could be analyzed (Figure I-4c), producing poor definition of any defined layers that may have been present. Specifically, because of the roughness of the coupon combined with the relatively large area of x-ray photoelectron spectroscopy analysis, definitive thin layers may not have been discerned (Figure I-4c).

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Source: DTN: LL040311312251.082.

The depth profile of the specimen in the sterile media shows essentially the alloying elements of Alloy 22 under a thin surface layer containing carbon species. The carbon species layer was about 40 nm. The source of the carbon may be from organics in the test media that were deposited during the testing and also from adsorption of airborne carbon species after the testing.

The depth profile of the specimen in the nonsterile biotic media shows there was a thick carboncontaining surface layer, the biofilm. Within the biofilm, the alloying elements of Alloy 22, most notably nickel and chromium, are present. As with the sterile sample, the alloying elements

Figure I-5. Atomic Percent of Major Elements as a Function of Depth in Inoculated and Sterile Nonwelded Alloy 22 Coupons

dominate the composition when the carbon signal drops below 10%. The significant feature in the depth profile is that the alloying elements of Alloy 22 are contained in the biofilm, indicating the degradation of the alloy under the biofilm.

When x-ray photoelectron spectroscopy analysis of several different surface areas (without sputtering, purely surficial analysis) of sterile- and nonsterile-incubated coupons was performed, no nickel or chromium was detected on the Alloy 22 surface incubated with Yucca Mountain bacteria, while both nickel and chromium were evident on some examined areas of the sterile-incubated coupon (Table I-3). Coincident with the presence of nickel and chromium on sterile surfaces, oxygen content was also greater (Table I-3); therefore, the chromium and nickel that were detected were probably areas of passive film metal oxides exposed between areas of media precipitates on the surface of this sterile-incubated coupon. Supporting this supposition is the finding that oxygen levels on nonsterile surfaces are significantly less than those on sterile surfaces, and these lower values are similar to those observed using the x-ray photoelectron spectroscopy depth analysis of the biofilm. Given that no Alloy 22 components were evident on the surface of the biofilm present on the nonsterile coupon does not exclude the possibility that there may be metal precipitates present in other locations or within the biofilm, but no metal precipitates were detectable in this particular analysis.

	Nonsterile Surface (atomic % <sup>a</sup> )				Sterile Surface (atomic % <sup>a</sup> )			
Element	Spot 1 <sup>b</sup>	Spot 2 <sup>b</sup>	Spot 3 <sup>b</sup>	Spot 4 <sup>c</sup>	Spot 1 <sup>b</sup>	Spot 2 <sup>b</sup>	Spot 3 <sup>b</sup>	Spot 4 <sup>c</sup>
Nickel	ND	ND	ND	ND	ND	ND	0.1	0.2
Chromium	ND	ND	ND	ND	ND	0.3	1.2	0.9
Carbon	94.8	95.5	95.8	95.6	81.7	83.6	72.2	78.7
Oxygen	3.8	3.3	3.0	3.2	13.7	12.2	19.6	14.8
Silicon	1.4	1.2	1.2	0.8	3.9	3.9	3.4	3.7

Table I-3. X-Ray Photoelectron Spectroscopy Elemental Analysis of Surface Sites on Alloy 22

Source: DTN: LL040303712251.079.

NOTE: <sup>a</sup> ±0.5 atomic %. <sup>b</sup> 1 mm diameter. <sup>c</sup> 30 mm<sup>2</sup> area ND = not detectable.

Silicon was detected on both the nonsterile and the sterile Alloy 22 surface; silica precipitates had to originate from the Yucca Mountain groundwater solvent, which was the only source of silicon present. Significantly more silica was evident on the sterile surface, suggesting that the biofilm present on the biotic coupon may have been masking siliceous precipitates, just as it may have been occluding detection of metal precipitates. Therefore, while the detection of precipitates using surficial x-ray photoelectron spectroscopy analysis could be an indicator of general dissolution; so far, this approach has not been promising due to the possible occlusion of putative precipitates by biofilm material.

# I.4.6 Continuation of Testing to Verify Enhancement Factor

In response to the third item (continue testing, including simulated saturated repository environment to confirm enhancement factor) of KTI agreement CLST 1.02, testing under representative repository conditions was continued. The testing system used simulates a

saturated repository environment that includes aseptically collected and crushed Yucca Mountain rock continually fed (2 mL/hr) with Yucca Mountain groundwater at 10-fold concentration and supplemented with 0.1% glucose (Figure I-6). These "microcosm" experiments have been incubated for extended periods at temperatures up to 30°C. Small (1 cm<sup>2</sup>) coupons of Alloy 22 were incubated in these systems and periodically removed, cleaned, and examined. The crushed Yucca Mountain rock contained in these systems was left with the full complement of Yucca Mountain microorganisms and compared to results of sterile control microcosms in which the rock had been presterilized by radiation.

Figure I-1 shows scanning electron microscopy images of the surface of the unexposed specimen and of the surfaces exposed to sterile and microbial environments. After nearly five years of exposure, the specimen exposed to nonsterile microbial environment shows some evidence of surface modification with features resembling pinholes or pores, and some specimen polishing ridges are still discernible. While no quantitative estimates of corrosion rates are possible from the scanning electron microscopy examination of these specimens, the limited amount of surface attack observed is qualitatively consistent with the microbially influenced corrosion effects discussed in Section I.4.2.



NOTE: Microcosm reactors were each composed of a 2-L reservoir containing sterile media that was fed through a 0.2-μm in-line filter by means of a peristaltic pump into a 500-mL modified spin flask test cell that contained crushed Yucca Mountain rock (either sterile or nonsterile) and coupon material. Bacteria were maintained within the spin flask test cell by means of second 0.2-μm in-line filter. The outflow rate was regulated by a second peristaltic pump. Sterility was maintained throughout the system except within the rock-containing vessel.

Figure I-6. Configuration of Microcosms Test Apparatus

# I.5 REFERENCES

## I.5.1 Documents Cited

Baetjer, A.M.; Birmingham, D.J.; Enterine, P.E.; Mertz, W.; and Pierce, J. O. 1974. "Chapter 1 - Properties of Chromium." *Chromium*. National Academy of Sciences. 1–6. Washington, D.C.: National Academy of Sciences. TIC: 255533.

Dunn, D.S. and Brossia, C.S. 2002. "Assessment of Passive and Localized Corrosion Processes for Alloy 22 as a High-Level Nuclear Waste Container Material." *Corrosion/2002, 57th Annual Conference & Exposition, April 7-11, 2002, Denver, Colorado*. Paper No. 02548. Houston, Texas: NACE International. TIC: 254579.

Dunn, D.S.; Brossia, C.S.; and Pensado, O. 2001. "Long-Term Dissolution Behavior of Alloy 22: Experiments and Modeling." *Corrosion/2001, 56th Annual Conference & Exposition, March 11-16, 2001, Houston, Texas, USA*. Paper No. 01125. Houston, Texas: NACE International. TIC: 208528.

Horn, J.; Carrillo, C.; and Dias, V. 2003. "Comparison of the Microbial Community Composition of Yucca Mountain and Laboratory Test Nuclear Repository Environments." *Corrosion/2003, 58th Annual Conference & Exposition, March 16–20, 2003, San Diego, California.* Paper No. 03556. Houston, Texas: NACE International. TIC: 255527.

Kerrisk, J.F. 1987. *Groundwater Chemistry at Yucca Mountain, Nevada, and Vicinity.* LA-10929-MS. Los Alamos, New Mexico: Los Alamos National Laboratory. ACC: NNA.19870507.0017.

Lian, T.; Martin, S.; Jones, D.; Rivera, A.; and Horn, J. 1999. "Corrosion of Candidate Container Materials by Yucca Mountain Bacteria." *Corrosion 99, 54th Annual Conference and Expo, San Antonio, Texas, April 25-30, 1999.* Paper No. 476. Houston, Texas: NACE International. TIC: 245833.

Reamer, C.W. and Gil, A.V. 2001. Summary Highlights of NRC/DOE Technical Exchange and Management Meeting of Range on Thermal Operating Temperatures, September 18-19, 2001. Washington, D.C.: U.S. Nuclear Regulatory Commission. ACC: MOL.20020107.0162.

Schlueter, J. 2000. "U.S. Nuclear Regulatory Commission/U.S. Department of Energy Technical Exchange and Management Meeting on Container Life and Source Term (September 12-13, 2000)." Letter from J. Schlueter (NRC) to S. Brocoum (DOE/YMSCO), October 4, 2000, with enclosure. ACC: MOL.20010731.0161.

## I.5.2 Standards

ASTM G 59-97. 1998. *Standard Test Method for Conducting Potentiodynamic Polarization Resistance Measurements*. West Conshohocken, Pennsylvania: American Society for Testing and Materials. TIC: 249897.

## I.5.3 Data, Listed by Tracking Number

LL040303612251.078. Microbiology Influenced Corrosion (MIC) Effects on Microcosm-Incubated Alloy 22. Submittal date: 03/29/04.

LL040303712251.079. Sputtering and Surfacial X-Ray Photoelectron Spectroscopic Analysis of Sterile and Yucca Mountain Bacteria-Inoculated Alloy-22. Submittal date: 02/25/04.

LL040311312251.082. X-Ray Photoelectron Spectroscopic (XPS) Surface Analysis of Alloy 22 Sterile and Non-Sterile Coupons. Submittal date: 03/30/04.

LL040402912251.085. Microbiologically Induced Corrosion (MIC) Linear Polarization Corrosion Rate of Alloy 22 Weldments for Sterile and Non-Sterile Coupons. Submittal date: 04/20/04.

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