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Document Title: Elevated-Temperature Corrosion of Iron-Base Alloys in Hanford Grande Ronde Groundwater		WBS No. or Work Package No. L234 CEI 002 <i>July 1983</i>																																																																													
Key Words: Iron-Base Alloys, Grande Ronde Groundwater		Prepared by (Name and Dept. No.) Author: R. E. Westerman (PNL) BWIP Contact R. L. Fisher <i>3/18/83</i> See reverse side for additional approvals																																																																													
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Abstract <p>The corrosion behavior of several iron-base materials was studied at 250°C and 150°C in synthetic Hanford Grande Ronde groundwater. The groundwater was pumped through autoclaves at ~35ml/hr from supply vessels continuously sparged with an Ar-20% O₂ mixture. Crushed basalt was placed in the autoclaves to simulate the repository environment.</p> <p>The uniform corrosion rates at both 150° and 250°C were relatively low at test times of three months and beyond; the uniform corrosion rates were <0.3 mil/yr at 250°C and <0.5 mil/yr at 150°C based on linear corrosion kinetics. Although non-uniform (pitting) attack was observed in most of the materials tested, no conclusions can be drawn with regard to non-uniform attack of canisters fabricated from these materials since conditions at the actual canister surface include contact with a basalt-bentonite packing material and a much lower groundwater oxygen concentration.</p> <p>This report contains data obtained by Pacific Northwest Laboratories between June 1981 and June 1983.</p>		<p style="text-align: center;">BASALT</p> <p style="text-align: center;">(Continued on reverse side)</p>																																																																													
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<p>GROUP PROJECT INTEGRATION</p> <p>NAME: <i>Basalt</i> DATES: <i>1/82</i></p> <p>REVIEWED FOR PATENTABILITY</p> <p>SENSITIVE MATERIAL</p> <p>B501070585 841116 PDR WMRES ECAEROS A-4165 PDR</p>		<p>Prepared By: Pacific Northwest Laboratory for RHO/BWIP</p> <p>Used By: Rockwell Hanford Operations/BWIP</p>																																																																													

Rockwell Hanford Operations

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<p>Approvals</p> <p><input type="checkbox"/> _____ Program Office</p> <p><input type="checkbox"/> _____ Research and Engineering</p> <p><input type="checkbox"/> _____ Plant Operations</p> <p><input type="checkbox"/> _____ Health, Safety and Environment</p> <p><input checked="" type="checkbox"/> M. E. Nicol <i>M. E. Nicol 5/9/84</i> Quality Assurance</p> <p><input type="checkbox"/> _____ Training <i>[Signature]</i></p> <p><input checked="" type="checkbox"/> R. W. Fitch, Mgr., Research & Licensing End Function <i>4/9/84</i></p> <p><input checked="" type="checkbox"/> M. J. Smith, Mgr., Engineered Barriers End Function</p> <p><input checked="" type="checkbox"/> <i>[Signature]</i> <i>4/17/84</i> Project Integration <i>9-4-84</i></p> <p><input checked="" type="checkbox"/> P. F. Salter, Mgr., Near-Field Geochemistry Gp.</p> <p><input checked="" type="checkbox"/> <i>[Signature]</i> <i>4/17/84</i> R. T. Wilde Mgr. Systems</p> <p><input type="checkbox"/> _____ <i>[Signature]</i> <i>3/25/84</i></p> <p><input checked="" type="checkbox"/> T. B. McCall, Mgr., Engineered Barriers Approval Authority Design Development & Analysis</p>		* Distribution	Name	Mail Address

SUMMARY OF REVISION

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Rev. Ltr./ Chg. No.	Description Of Changes	Approvals / Date
1.	The 12- and 17-month corrosion data listed in Table 4 of the subject document (Rev. 0-0) for 1-1/4% Cr, 1/2% Mo cast steel and 2-1/2% Cr, 1% Mo cast steel were accidentally interchanged.	<p>R. L. Zisch 4/26/84</p> <p>J. T. Murphy 4/27/84</p> <p>O. T. Murphy 5/4/84 BWIIP OK</p> <p>P. J. Roden 5/7/84</p> <p>P. J. Roden</p>
Release Stamp	Release Stamp	Release Stamp

Informal Report

ELEVATED-TEMPERATURE CORROSION OF
IRON-BASE ALLOYS IN HANFORD GRANDE RONDE GROUNDWATER

Submitted to

Basalt Waste Isolation Project
Rockwell Hanford Operations

by
R. E. Westerman
Battelle-Northwest

July, 1983

BACKGROUND

Battelle-Northwest is performing a study for the Basalt Waste Isolation Project that has as its objective the characterization and recommendation of metallic materials that have the ability to contribute to the long-term (to 1000 years) waste-containment function of high-level nuclear waste packages. During this period of time the canister's surface will be exposed to elevated-temperature groundwaters. The corrosive action of these groundwaters on the canister must be estimated by means of laboratory studies so that the waste package designer can provide an appropriate corrosion allowance. The maximum test temperature (250°C) used in the present study is higher than that expected over the major part of the container lifetime, and the potential corrosion-mitigating effect of a packing material has not been factored in to the present studies. Also, it is expected that the test conditions used in the present work would be more severe and produce higher corrosion rates than in a repository because lower flow rates and oxygen concentrations are expected under repository conditions. Thus, the results presented herein are considered conservative except for the fact that radiation effects are not included. The effects of irradiation have been discussed in recent reports.^(1,2)

The investigation of general (uniform) corrosion has been emphasized in the series of tests reported herein. Stress-corrosion-cracking, crevice corrosion, and weldment corrosion have not yet been specifically addressed.

EXPERIMENTAL

All of the corrosion studies were performed in refreshed (flowing) autoclave systems. The autoclaves were made either of Inconel 600 or Hastelloy C, both corrosion-resistant nickel-base alloys. Two test temperatures were used: 250° and 150°C. Synthetic Grande Ronde groundwater was pumped to the autoclaves at ~35 ml/hr from a storage reservoir continuously sparged with an Ar-20% O₂ mixture. The water, termed "oxic" because of its 6-8 ppm O₂ content, flowed upward through a bed of crushed basalt rock 3-5 cm (1-2 in.) deep prior to contacting the specimens. The effluent from the autoclaves was not recycled.

At the conclusion of the desired exposure selected specimens were removed from the autoclaves and stripped of their surface films by means of light abrasion and immersion in formaldehyde-inhibited HCl. Specimens were generally not reinserted after stripping, though it was occasionally done. Pre-stripped specimens did not appear to corrode at a rate appreciably different from fresh specimens that had no prior exposure. Pre-stripped specimen data are included in the tabulated data in this report, but they are not included in the graphical representations.

A more detailed description of the experimental procedure has been published elsewhere.⁽²⁾

MATERIALS

Early screening studies⁽³⁾ revealed the possibility of using relatively inexpensive iron-base alloys under the conditions expected in a repository

constructed in a basalt formation, and subsequent studies have tended to support the early findings.⁽²⁾ To date, a total of five iron-base materials have been tested: a ductile cast iron, a 2 1/2% Cr, 1% Mo cast steel; a 1 1/4% Cr, 1/2% Mo cast steel; a 1025 cast steel; and a 1020 wrought steel. The compositions of the materials are presented in Table 1.

TABLE 1. Composition of Iron-Base Alloys

Material	Element, wt%						
	C	Mn	Si	P	S	Mo	Cr
2 1/2% Cr, 1% Mo Cast Steel	0.116	0.57	0.57	0.020	0.004	1.02	2.46
1 1/4% Cr, 1/2% Mo Cast Steel	0.117	0.76	0.59	0.018	0.001	0.49	1.31
1025 Cast Steel (ASTM A-27, Grade 60-30)	0.240	0.60	0.49	0.012	0.018	-	-
Ductile Cast Iron (ASTM A536-77, Grade 60-40-18)	3.53	0.31	2.51	0.05	0.004	-	-
1020 Wrought Steel	0.19	0.36	-	0.029	0.043	-	-

Each of the first three materials listed were tested in the as-cast condition. The specimens of cast ferrous material were obtained from castings each weighing ~160 kg (350 lb) with a minimum dimension of 130 mm (5.0 in.). Specimens were cut so that an edge would lie on the surface of the casting. The 1020 wrought steel was supplied in the form of hot-rolled, de-scaled sheet nominally 1.5 mm (0.060 in.) thick.

The ductile cast iron is representative of a large group of cast irons with good impact and ductility properties relative to the still-cheaper gray irons. The low-alloy cast steels are more expensive than the cast irons but have superior mechanical properties. The low level of chromium/molybdenum in one of the cast steels represents an attempt to learn what beneficial effects, if any, such modest alloy additions might have on the corrosion resistance of cast ferrous materials.

Prior to corrosion testing, the cast steel specimens were ground with an aluminum oxide wheel to produce a surface finish of 32 to 63 μm rms. The same

wheel produced a surface finish of 8 μm rms on the ductile iron specimens. The wrought steel sheet was surface ground with a 50-grit disc prior to corrosion testing.

The approximate composition of the basalt rock⁽⁴⁾ and the Hanford Grande Ronde (basalt) groundwater⁽⁵⁾ used in the study are given in Tables 2 and 3.

TABLE 2. Composition of Umtanum Flow Basalt⁽⁴⁾

<u>Constituent</u>	<u>wt%</u>
SiO ₂	54.9
Al ₂ O ₃	14.3
FeO	13.2
CaO	7.2
MgO	3.4
Na ₂ O	2.6
TiO ₂	2.2
K ₂ O	1.6
P ₂ O ₅	0.4
MnO	0.2

TABLE 3. Composition of Hanford Grande Ronde (Basalt) Groundwater⁽⁵⁾

<u>Chemical Species</u>	<u>Concentration, mg/l</u>
Na ⁺	250
K ⁺	1.9
Ca ⁺²	1.3
Mg ⁺²	0.40
CO ⁻²	27
HCO ₃ ⁻	70
OH ⁻	1.4
H ₃ SiO ₄ ⁻	103
Cl ⁻	148
SO ₄ ⁻²	108
F ⁻	37

pH = 9.8-10

The basalt was obtained from a surface outcrop of the Umtanum Flow, Grande Ronde Formation, near the Hanford Site. The groundwater was made up to simulate the water contained in the aquifer lying just beneath the proposed Hanford Site repository horizon. The composition of the Grande Ronde groundwater used in the tests differs somewhat from the composition now considered to define the reference Grande Ronde groundwater.⁽⁶⁾ The slightly different chemistries are not expected to result in significantly different corrosion kinetics.

RESULTS AND DISCUSSION

The corrosion rates of the four candidate iron-base materials have been determined in oxic synthetic Grande Ronde groundwater at 250°C and 150°C. The rates obtained are plotted in Figures 1 and 2, respectively, based on a linear-kinetics interpretation of the data. The data of Figures 1 and 2 are presented in tabular form in Table 4. The bulk of the data is enclosed in cross-hatched scatter bands in these figures for ease of visualization of overall magnitudes and trends.

The corrosion rates observed at 250°C (Figure 1) showed a sharp decrease after a 1-month exposure. From 3 months to 12 months the corrosion rates were reasonably uniform among the materials tested, and the corrosion rates observed were low, i.e., $<8 \mu\text{m}/\text{yr}$ ($<0.3 \text{ mil}/\text{yr}$). Also, there was little sign of non-uniform attack, such as pitting corrosion. Between 12 and 17 months, however, pitting attack became evident, notably on specimens of the normally well-behaved 2 1/2% Cr, 1% Mo material. (These specimens lie well above the scatter band of Figure 1.) The pits were numerous on these specimens, averaging 1 to 2/cm², and were non-uniformly distributed over the specimen surfaces. They were typically 50-100 μm (2-4 mil) deep, and 500-1000 μm (20-40 mil) in diameter, though the size variation was great. A similar phenomenon was observed at 17 months exposure in the 150°C test (Figure 2), in that one cast ductile iron specimen and two 2 1/2% Cr, 1% Mo specimens showed significant evidence of nonuniform attack, manifesting itself primarily in formation of arrays of shallow pits. Prior to this 17-month-exposure observation, four other specimens had shown evidence of pitting attack at 150°C: three 1020 wrought steel specimens, after exposures of 2 to 5 months, and a 1 1/4% Cr, 1/2% Mo specimen after 12 months exposure. Obviously, susceptibility of the candidate alloys to pitting attack is of much greater concern at this time than the modest penetration rates associated with general corrosion. If this pitting attack concern can be put aside, the corrosion data presented in Figures 1 and 2 suggest that ferrous materials offer promise as waste package structural barrier elements in a repository constructed in basalt for time periods up to 1000 years. Of course, possible effects of radiation and the possible interaction of environmental and mechanical stresses, as in stress corrosion cracking, are not addressed in these general corrosion results.

The nature of the corrosion product films and deposition layers on the surfaces of selected corrosion specimens was determined by x-ray diffraction (XRD) techniques. After 5 months of exposure to the synthetic ground water at 250°C, a surface film on a specimen of ductile cast iron was found to consist of analcime, $\text{NaAlSi}_2\text{O}_6 \cdot n\text{H}_2\text{O}$, and nontronite, $(\text{Fe}, \text{Al}) \text{Si}_2\text{O}_5(\text{OH}) \cdot n\text{H}_2\text{O}$. The diffraction pattern was diffuse, indicative of a weakly organized crystal structure. No indication was found of hematite, Fe_2O_3 , or magnetite, Fe_3O_4 .

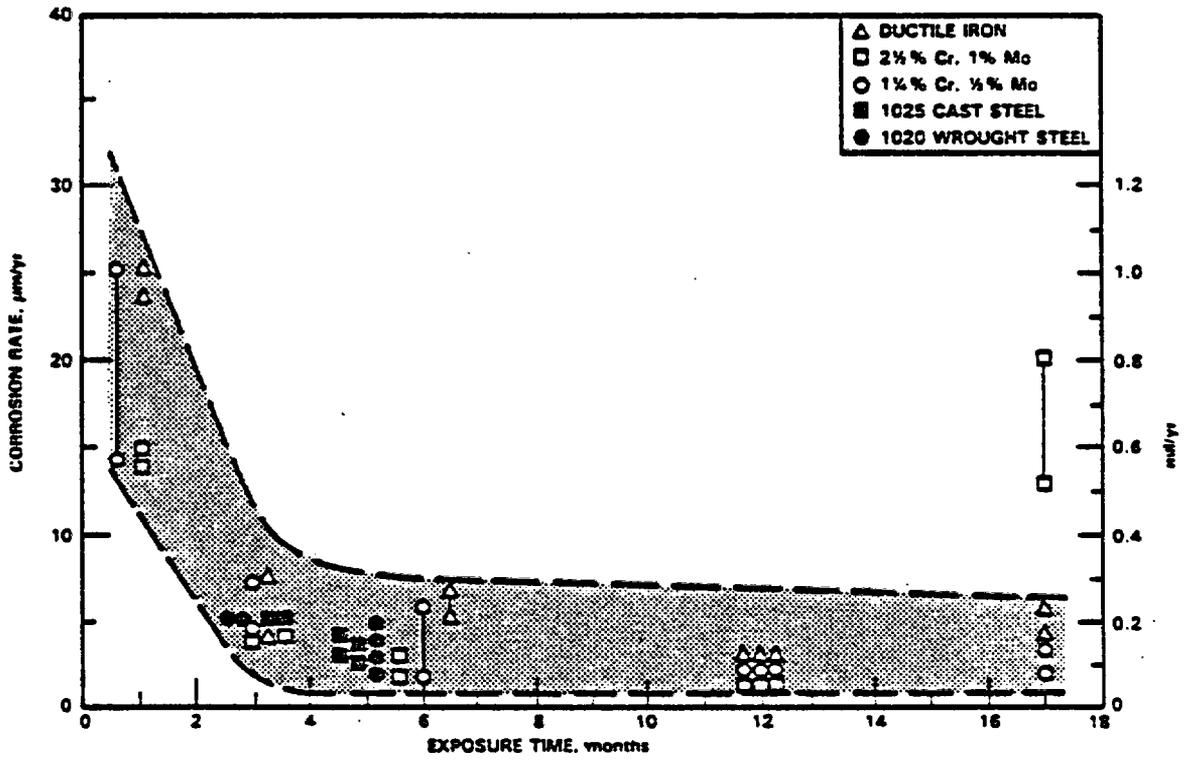


FIGURE 1. Corrosion of Iron-Base Alloys Exposed to Oxidic Synthetic Hanford Grande Ronde Basalt Groundwater at 250°C.

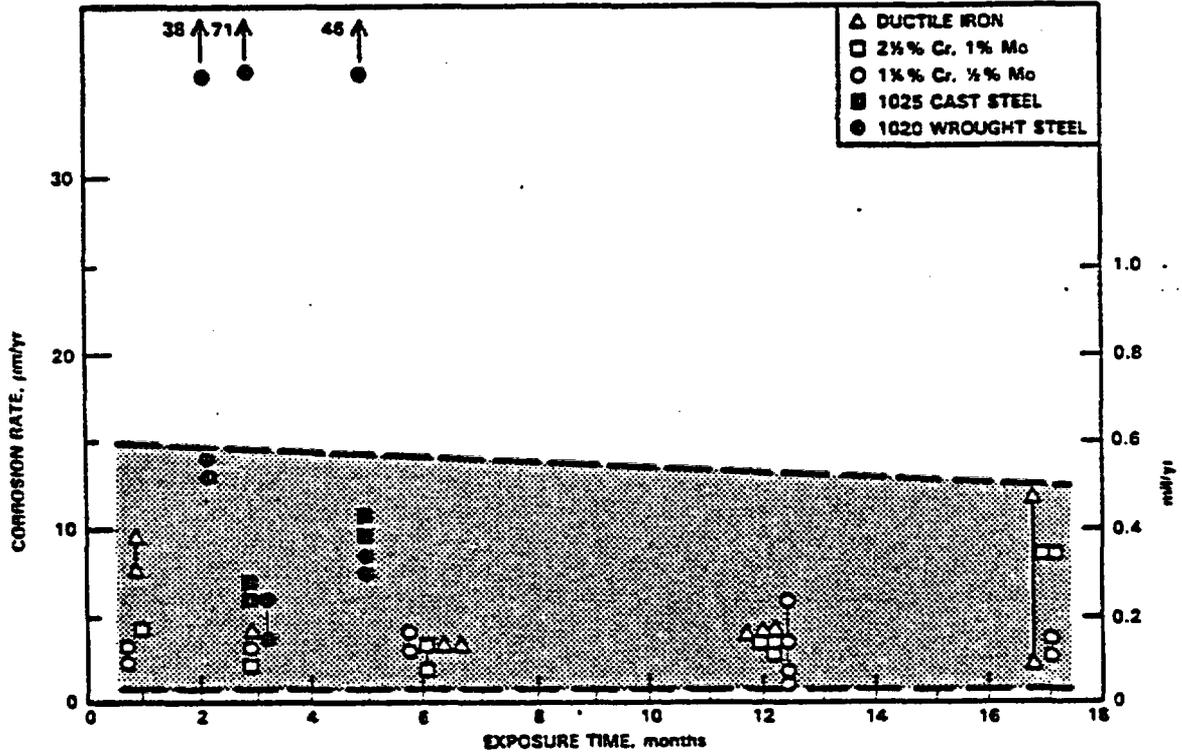


FIGURE 2. Corrosion of Iron-Base Alloys Exposed to Oxidic Synthetic Hanford Grande Ronde Basalt Groundwater at 150°C.

TABLE 4. Summary of Iron-Base Alloy Corrosion Data
Hanford Grande Ronde Basalt Groundwater

Material	Exposure Time, mo	Penetration, $\mu\text{m}/\text{yr}$ (mil/yr)	
		250°C	150°C
1 1/4% Cr, 1/2% Mo Cast Steel	1	14(0.54), 25(0.98)	2.0(0.080), 2.0(0.078)
	3	7.4(0.29) ^(a) , 4.3(0.17)	2.5(0.10), 3.6(0.14) ^(a)
	6	5.8(0.23), 1.8(0.067)	2.4(0.096), 3.1(0.12)
	12	1.3(0.050), 1.2(0.046), 1.2(0.047)	3.3(0.13), 2.8(0.11), 3.3(0.13)
	17	20(0.80) ^(b) , 13(0.52) ^(b)	8.6(0.34) ^(b) , 8.6(0.34) ^(b)
2 1/2% Cr, 1% Mo Cast Steel	1	14(0.56), 15(0.59)	3.3(0.13)
	3	3.6(0.14) ^(a) , 4.1(0.16)	1.4(0.056) ^(a) , 2.0(0.078)
	6	2.8(0.11), 1.5(0.059)	2.5(0.099), 1.2(0.048)
	12	1.3(0.050), 1.4(0.055), 1.6(0.064)	2.5(0.10), 1.4(0.055), 1.8(0.072), 5.6(0.22) ^(b)
	17	1.8(0.071), 2.8(0.11)	3.3(0.13), 2.4(0.095)
Cast Ductile Iron	1	25(1.0), 23(0.91)	8.9(0.35), 7.1(0.28)
	3	8.4(0.33) ^(a) , 7.9(0.31)	3.8(0.15), 43(1.7) ^(a,b)
	6	6.9(0.27), 4.8(0.19)	3.6(0.14), 3.6(0.14)
	12	2.5(0.099), 3.1(0.12), 2.5(0.097)	3.6(0.14), 3.8(0.15), 3.8(0.15)
	17	3.8(0.15), 6.1(0.24)	2.8(0.11), 12(0.46) ^(b)
1025 Cast Steel	3	5.1(0.20), 5.3(0.21)	6.9(0.27), 6.3(0.25)
	5	2.5(0.10), 3.0(0.12), 2.8(0.11) 3.6(0.14)	9.6(0.38), 11(0.44)
1020 Wrought Steel	2		14(0.55), 13(0.53), 38(1.5) ^(b)
	3	5.3(0.21), 5.6(0.22)	5.8(0.23), 3.6(0.14), 71(2.8) ^(b)
	5	3.0(0.12), 3.3(0.13), 3.6(0.14) 4.6(0.18)	8.1(0.32), 7.6(0.36), 46(1.8) ^(b)

(a) Specimen stripped once before final exposure.

(b) Specimen significantly pitted.

After 12 months exposure at 250°C specimens of 1 1/4% Cr, 1/2% Mo; 2 1/2% Cr, 1% Mo; and cast ductile iron were examined by XRD. The surface layers were found to consist primarily of analcime and Fe₃O₄ in all cases. After 12 months exposure at 150°C, on the other hand, only a very weak indication of Fe₃O₄ was found on similar specimens. The principal surface layer constituent on the 1 1/4% Cr, 1/2% Mo alloy and the cast ductile iron was found to be a zeolite of the phillipsite group, Na₃Al₃Si₅O₁₆·H₂O, with some nontronite. The 2 1/2% Cr, 1% Mo alloy showed some of the zeolite, but also a strong nontronite indication and some sign of Fe₃O₄. The films formed at 12 months exposure were not strongly adherent; and the protectiveness of the film did not increase with time, as the adherence to linear reaction kinetics is obvious. The high silica content of the ground water environment obviously favors the formation of surface layers consisting of silicate compounds rather than Fe₃O₄-Fe₂O₃.

The surface films formed on the specimens after 17 months exposure are currently undergoing analysis.

The observation of a tendency toward non-uniform attack with exposure time is cause for some concern, as the effect of the non-uniform attack on the metals' penetration rates is not known. Continued long-term testing under conditions more closely approximating those expected at the canister surface in a repository (e.g., with backfill present) will be necessary to assure that non-uniform attack will not result in unexpected penetration rates of candidate canister materials. This can only be accomplished by determining the rate of general corrosion and the rate of pit deepening once pits are nucleated. If non-uniform corrosion is found to be the predominant mode of corrosion of a given alloy, it does not necessarily signify the need to reject that particular material, as the pit depth may remain constant with time once a maximum depth is attained. These aspects of the corrosion problem remain to be defined in future tests, along with corrosion of weldments, crevice corrosion, and stress corrosion cracking.

CONCLUSIONS

- The general corrosion of candidate mild and low-alloy steels and a ductile cast iron in oxic Grande Ronde basalt groundwater at 250°C and 150°C is not overly severe. Barring concerns of non-uniform attack, the data obtained strongly suggest the suitability of these materials in waste package applications in a repository constructed in basalt.
- Non-uniform (pitting) attack has been found sporadically on specimens of mild and low-alloy steels in oxic Grande Ronde groundwater at 250°C and 150°C and time periods of one year or less. At 17 months exposure the occurrence of pitting has increased, with approximately half the specimens affected. The rates of the non-uniform attack are not readily predictable because it is not possible with the limited number of specimens available to determine the kinetics of pit growth. Conclusions cannot be drawn with regard to anticipated non-uniform (pitting) attack of canisters fabricated

from these iron-base alloys since conditions at the canister surface include contact with a basalt-bentonite packing material and a much lower groundwater oxygen concentration.

ACKNOWLEDGEMENTS

The experimental work described in this report was performed primarily by J. L. Nelson and D. H. Getchell, Corrosion Research and Engineering Section, BNW; and S. G. Pitman and H. E. Kissinger, Metallurgy Research Section, BNW.

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