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# Test Plan for the Irradiation of Nonmetallic Materials

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L.H. Brush<sup>1</sup>, C. Ottinger<sup>1</sup>, F. Gelbard<sup>1</sup>, M. Dahl<sup>2</sup>, C.C. Joslyn<sup>3</sup>, and T.J. Venetz<sup>3</sup>

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# ABSTRACT

A comprehensive test program to evaluate nonmetallic materials use in the Hanford Tank Farms is described in detail. This test program determines the effects of simultaneous multiple stressors at reasonable conditions on in-service configuration components by engineering performance testing.

Key Words: Hose-in-Hose Transfer Lines, HIHTL, Test Plan, Nonmetallic Materials, Irradiation, EPDM, HIHTL Life Extension, Teflon O-rings

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# ACRONYMS AND ABBREVIATIONS

β	Ionizing radiation consisting of beta particles (electrons) emitted from the nucleus of a decaying radionuclide
γ	ionizing radiation consisting of gamma rays (high-energy, short-
	wavelength photons) emitted from the nucleus of a decaying radionuclide
Al(OH) <sub>3</sub>	Aluminum hydroxide
ASME	American Society of Mechanical Engineers
ASTM	ASTM International
Ba, <sup>13/m</sup> Ba	Barium, a radioactive isotope of barium with an atomic mass of 137 amu
Cl	Chloride ion
Co, <sup>60</sup> Co	Cobalt, a radioactive isotope of Co with an atomic mass of 60 amu
CO <sub>3</sub>	Inorganic carbon
Cs, <sup>137</sup> Cs	Cesium, a radioactive isotope of cesium with an atomic mass of 137 amu
CSR	Compression stress-relaxation
DLO	Diffusion-limited oxidation
DNFSB	Defense Nuclear Facilities Safety Board
DOE	U.S. Department of Energy
DST	Double-shell tank
EPDM	Ethylene propylene diene monomer, a synthetic rubber
ES&H	environmental, safety and health
F	Fluoride ion
GIF	Gamma Irradiation Facility (SNL's)
$\mathrm{H},\mathrm{H}_{2},\mathrm{H}^{+}$	Hydrogen, diatomic hydrogen, hydrogen ion
Hg	Mercury
HIHTL	Hose-in-hose transfer line
HLW	High-level waste
ID	Inner diameter
K, <sup>40</sup> K	Potassium, a radioactive isotope of potassium with an atomic mass of
	40 amu
KNO <sub>3</sub>	Potassium nitrate
Kr, <sup>85</sup> Kr	Krypton, a radioactive isotope of krypton with an atomic mass of 85 amu
Ν	North
Na <sub>2</sub> CO <sub>3</sub>	Sodium Carbonate
$Na_2SO_4$	Sodium sulfate
Na <sub>3</sub> PO <sub>4</sub>	Sodium phosphate
NaCl	Sodium chloride
NaNO <sub>2</sub>	Sodium nitrite
NaNO <sub>3</sub>	Sodium nitrate
NaOH	Sodium hydroxide
$O, O_2$	Oxygen, diatomic oxygen
OD	Outer diameter
ORP	Office of River Protection
OUO	Official Use Only
Р	Pressure
PEEK	Polyether ether ketone
pН	the negative, common logarithm of the activity of $H^+$

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QA	quality assurance
R&A	Review and approval
River Bend	River Bend Transfer Systems, LLC
SNL	Sandia National Laboratories
Sr, <sup>90</sup> Sr	Strontium, a radioactive isotope of strontium with an atomic mass of
	90 amu
SST	Single-shell tank
Т	Temperature
TWINS	Tank Waste Information Network System
VTR	Vapor Transport Rate
WRPS	Washington River Protection Solutions, LLC
Y, <sup>90</sup> Y	Yttrium, a radioactive isotope of yttrium with an atomic mass of 90 amu
Units	
%	percent
/s	per second
0	degree(s)
°C	degrees Centigrade
°F	degrees Fahrenheit
amu	atomic mass units
bar, mbar	bar(s), millibar(s), both units of pressure
cm	centimeter
eV, MeV	electron volts, megaelectron volts, both units of energy
ft	foot or feet
g	gram
Gy	Grays
hr	Hour
in	inch(
kL	kiloliter
krad	kilorad, a unit of dose of ionizing radiation
М	molar
mbar	millibar, a unit of pressure
mCi	millicurie
MeV	megaelectron volt
ml, mL	milliliter
mm	millimeter
MPa	megapascal, Pressure Unit
nm	nanometer
psi	pounds per square inch
psig	pounds per square inch (gauge pressure)
R	roentgen(s), a unit of exposure to ionizing radiation
rad, krad	rad(s), kilorad(s), both units of dose of ionizing radiation
STP	Standard temperature and pressure
uCi	microcurie
wt, wt%	weight, weight %
Yr	year(s)

# TEST PLAN FOR THE IRRADIATION OF NONMETALLIC MATERIALS

# 1. OVERVIEW

This Test Plan proposes a study of the possible synergistic effects of simultaneous exposure of nonmetallic materials to radiation and other stressors, and the effects of exposure to these multiple stressors on their performance and properties. The nonmetallic materials are used by Washington River Protection Solutions, LLC, (WRPS) in the Hanford tank farm s in the waste transfer system. These materials include the primary (inner) hoses in hose-in-hose transfer lines (HIHTLs), Teflon®<sup>1</sup> gaskets, ethylene propylene diene monomer (EPDM, a synthetic rubber) O-rings, and (perhaps) other nonmetallic materials. The stressors include  $\beta$  and  $\gamma$  radiation; elevated temperatures; a nonradioactive, caustic (high-pH) supernatant HLW simulant of the tank farms; and elevated pressures.

The study described in this Test Plan will include at least three features that will be unique, or at least atypical of, previous studies of radiation effects. First, it will emphasize the irradiation of nonmetallic materials in their in-service configurations, not just the irradiation of coupons. Second, this study will simulate in-service exposure of these nonmetallic materials to simultaneous multiple stressors (i.e., radiation, elevated temperatures, nonradioactive caustic solutions, and elevated pressures). Third, it will emphasize postexposure mechanical performance testing to quantify the synergistic effects of simultaneous exposure to multiple stressors.

This Test Plan considers two options: a multi-phase baseline test program to perform comprehensive testing on hoses, gaskets and o-rings and an alternative offering reduced cost and schedule that performs comprehensive testing on hoses with a reduced scope, proofing type test on gaskets and o-rings. The baseline program will comprise four phases, testing HIHTLs, Teflon gaskets and EPDM O-rings concurrently. Phase 1 will compare the effects of simultaneous β and  $\gamma$  irradiation of nonmetallic materials to those from  $\gamma$  only. Phase 1 of this study must be carried out at ambient laboratory temperatures and pressures and in the absence of caustic solutions, because the sealed  $\beta$  sources to be used for this phase can only be used under ambient conditions to avoid rupture. Phase 2 will determine the effects of simultaneous exposure of nonmetallic materials to  $\gamma$  irradiation and elevated temperatures (up to 180 °F for hoses and up to 200 °F for other components). Phase 3 will investigate the effects of simultaneous exposure of these materials to  $\gamma$  irradiation, elevated temperatures, and nonradioactive caustic solutions. Finally, Phase 4 will quantify the effects of simultaneous exposure of nonmetallic materials to  $\gamma$ irradiation; elevated temperatures; a chemical simulant; and elevated pressures (400 or 425 psig). The reduced cost option would eliminate Phase 1 beta/gamma comparison tests, perform Phase 2, 3, and 4 testing on hoses and only phase 3 testing on Teflon gaskets and EPDM O-rings.

<sup>&</sup>lt;sup>1</sup> Teflon and Tefzel are registered trademarks of E. I. Du Pont de Nemours and Company, Wilmington, Delaware.

Most of Phase 1 and all of Phases 2, 3, and 4 will be carried out in Sandia National Laboratories' (SNL's) Gamma Irradiation Facility (GIF) pool. These tests will use gamma irradiation requiring the shielding provided by the GIF pool. Part of Phase 1 ( $\beta$  irradiation only or unirradiated controls) will be conducted in the GIF but outside the pool (probably in the high bay). The  $\beta$  exposure rate will be 100 R/hr, the  $\gamma$  exposure rates will be 100 or 1000 R/hr for Phase 1, and 1000 R/hr for Phases 2, 3, and 4. Sealed line sources containing <sup>85</sup>Kr will be used for  $\beta$  irradiation during Phase 1. Sealed source pins containing <sup>60</sup>Co will be used for  $\gamma$  irradiation in Phases 1, 2, 3 and 4.

Annular stainless steel vessels will be used to contain the specimens of nonmetallic materials for these tests. Each vessel will have an outer diameter of approximately 2.5 ft, a length of about 4 or 5 ft, and a central cavity with a diameter of about 1 ft that is open to the GIF pool. The  $\beta$  irradiations for Phase 1 will be carried out by inserting the sealed <sup>85</sup>Kr line sources inside the primary hoses and other materials, and placing these materials inside the enclosed, annular portions of these containers that surround the central cavities. The  $\gamma$  irradiations for Phases 1, 2, 3 and 4 will be conducted by placing sealed <sup>60</sup>Co source pins in the central cavities. A total of 18 containers will be required for the entire baseline program; a total of 12 would be needed for the reduced cost option.

Preliminary calculations were carried out to determine the required activity of each sealed <sup>60</sup>Co source pin and the total number of source pins needed to maintain a  $\gamma$  exposure rate of 1000 R/hr for the nonmetallic materials proposed for this study. However, additional calculations will be necessary for the final test design and prior to the start of irradiation tests. These follow-on calculations will provide more robust definitions of the final test configurations, the number of <sup>60</sup>Co sources required, and the cost of these sources.

A test control and monitoring system will be developed and deployed for all 18 or 12 of the vessels, respectively, in the proposed baseline test program or reduced cost option. One or more of the following test variables will be controlled in all four phases of the baseline program or the reduced cost option: (1) the  $\beta$  and/or  $\gamma$  exposure rates, (2) temperatures; (3) pressures, (4) the flow rate(s) of compressed air through the vessels to maintain ambient concentrations of O<sub>2</sub> in the vessels. The values of the following test variables will be monitored and recorded for all of the vessels: (1) total  $\beta$  and/or  $\gamma$  exposure, (2) temperature, (3) pressure, (4) the flow rate(s) of compressed air, and (5) the concentrations of O<sub>2</sub>. All of these control and monitoring instruments will be calibrated according to the quality assurance (QA) requirements that are being developed for this study.

The schedule for the baseline test program, as shown in Figure 1, includes a year for preparations (October 2013 through September 2014). These preparations include final test-configuration calculations, design and fabrication of test components, procurement of test specimens and sealed sources, safety approvals, and verification testing. Phases 1 and 2 would be placed in the GIF pool from October through December 2014 and from January through March 2015, respectively. They would be removed from the pool from October through December 2016 and from January through March 2017, respectively. Phases 3 and 4 would be placed in the GIF pool from October through December 2016 and from January through March 2017, respectively. They would be removed from October through December 2018 and from January through March 2017, respectively.

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through March 2019, respectively. The proposed baseline schedule also includes posttest analysis and data reports for each of these phases. A final report on the entire program would be completed by September 2019. This schedule is based on three crucial assumptions: First, it is assumed that the funding for the first year of the baseline test program (preparations) will arrive at SNL at the beginning of October 2013. Earlier or later arrival of funding would result in commensurate changes for Phases 1, 2, 3, and 4. Second, it is assumed that all 14 vessels required for Phases 1 and 2, and all 12 required for Phases 3 and 4 can be placed in the GIF pool simultaneously. Third, it is assumed that all 14 vessels required for Phases 3 and 4 can be placed in the pool from October 2014 through March 2015; and that all 12 required for Phases 3 and 4 can be placed in the pool while those used for Phase 1 and 2 are being removed.



## Figure 1. Project Schedule for Baseline Test Program.

The reduced cost option will eliminate the Phase 1 beta/gamma comparison tests. The issue of relative damage from beta versus gamma radiation will be settled through alternate means. Hoses will still be tested at the varying levels of the 4 primary stressors as described in Phases 2, 3, and 4, allowing a better service life prediction model to be developed. PUREX connector gaskets and O-rings will only be tested as described in Phase 3, with chemical stimulant, varying temperature and varying radiation exposure up to two years total. This will demonstrate gasket acceptability in a "proofing" sense and give insight into the effect of increasing radiation levels to the gasket material in the confined geometry of the PUREX connector.

The primary advantages for the reduced scope program are significant cost reductions and reduction of the schedule. Cost saving come from elimination of the high cost beta sources, reduction in the number of test vessels, test hoses, gaskets, and coupons, less post exposure testing and analysis, and a reduced number of test, instrument, and control loops. Most significantly, this option also reduces the size requirements such that all test vessels required can fit into the pool at the Gamma Irradiation Facility at the same time, allowing for Phase 2, 3 and 4 to occur simultaneously. The preparation time is similar, but this option would cut two years off the total schedule.

This document does not include an estimate of the cost for execution of this test plan.. Instead, the cost estimate is found in a separate document that includes the cost of the baseline test program and the reduced cost option, RPP-PLAN-54568, *Test Plan for the Irradiation of Nonmetallic Materials: Cost Estimate Summary*. The cost estimate is marked as Official Use Only (OUO) because it contains Sandia Proprietary Information.

Larry: Ken suggests a one-liner in the Overview: RPP-PLAN-50529, Rev. 0. Washington River Protection Solutions (U.S. Department of Energy Contract DE-AC27-08RV14800

# 2. INTRODUCTION

# 2.1 Objectives

This Test Plan proposes a study of the possible synergistic effects of simultaneous exposure of nonmetallic materials to radiation and other stressors, and the effects of exposure to these multiple stressors on their performance and properties. The nonmetallic materials are used by WRPS in the Hanford tank farms waste transfer system. They include the primary (inner) hose of the HIHTLs (hereafter referred to as "hoses"), Teflon gaskets, and ethylene propylene diene monomer (EPDM) O-rings used in the tank farms. The stressors include radiation (both  $\beta$  and  $\gamma$ ); elevated temperatures; a nonradioactive, caustic, supernatant HLW simulant of the tank farms; and elevated pressures.

This Test Plan originates from a Defense Nuclear Facilities Safety Board (DNFSB) recommendation, contained in an April 26<sup>th</sup>, 2011 letter to the U.S. Department of Energy (DOE), to conduct post-mortem examination of HIHTL hoses and Teflon gaskets to improve the existing technical basis. The DOE-Office of River Protection (ORP) responded in a letter dated July 25<sup>th</sup>, 2011, recognizing the value in enhancing the technical basis of HIHTL and other nonmetallic materials while stating that worker exposure concerns outweighed the potential benefits of post-mortem examinations. In letter 11-NSD-069, DOE-ORP directed WRPS to provide a cost and schedule for development of this Test Plan and to identify the Test Plan in the Documented Safety Analysis as a planned improvement.

The study described in this Test Plan will include at least three features that will be unique, or at least atypical of, previous studies of radiation effects. First, it will emphasize the irradiation of nonmetallic materials in their in-service configurations (see Subsection 2.2), not just the irradiation of coupons. Second, this study will simulate in-service exposure of these nonmetallic materials to simultaneous multiple stressors (i.e., various combinations of  $\beta$  and  $\gamma$  irradiation; elevated temperatures; the presence of a nonradioactive, caustic, a chemical simulant; and elevated pressures). (Subsections 2.3 and 2.4 describe these stressors in more detail.) Third, it will emphasize postexposure mechanical performance testing (e.g., burst tests and leak tests), independent of mechanical properties testing, to quantify the synergistic effects of simultaneous exposure to multiple stressors. Implementation of performance-based testing will allow WRPS to use the results directly in the safety basis required to complete its tank farms mission. (Subsection 2.5 discusses the difference between mechanical performance and properties testing; Subsection 2.6 explains the unique or atypical features of this test program in more detail.)

This Test Plan considers two options: the baseline test program and alternative reduced cost option. The baseline program will be comprised of four phases, testing hoses, Teflon gaskets and EPDM O-rings concurrently. Phase 1 will compare the effects of simultaneous  $\beta$  and  $\gamma$  irradiation of hoses and Teflon gaskets to those from  $\gamma$  only. Phase 2 will determine the effects of simultaneous exposure of hoses, Teflon gaskets, and EPDM O-rings to  $\gamma$  irradiation and elevated temperatures. Phase 3 will investigate the effects of simultaneous exposure of the same materials used in Phase 2 to  $\gamma$  irradiation, elevated temperatures, and a chemical simulant. Finally, Phase 4 will quantify the effects of simultaneous exposure of the same materials used in Phases 2 and 3 to  $\gamma$  irradiation, elevated temperatures, the chemical simulant, and elevated

pressures. This phased approach was established during a meeting of WRPS, SNL, and other personnel held July 31-August 1, 2012, in Richland, Washington. This baseline program will allow for testing of the three highest-priority materials using the space available in SNL's irradiation facility to the maximum extent possible. Other nonmetallic materials are not included in the baseline test program because of the space and time required to complete this option with the three highest-priority materials. However, the baseline could be expanded, at the discretion of WRPS, by adding more materials or different combinations of stressors (see below).

Figure 2 illustrates the logic of the proposed four-phase baseline test program. Table 1 identifies the test variables and their values for each of the four phases of this baseline program. This four-phase sequence will provide WRPS with the most important information needed to address the questions posed by the DNFSB as soon as possible. Furthermore, it reflects the anticipated time required to obtain environmental, safety and health (ES&H) approvals for each phase of the baseline program, listed from the shortest to the longest expected approval process.

The radiation to which nonmetallic materials are exposed in the Hanford tank farms include both  $\beta$  from <sup>90</sup>Sr and <sup>90</sup>Y, which are associated mainly with the solids in the tanks; and  $\gamma$  from <sup>137</sup>Cs and <sup>137m</sup>Ba, associated mainly with the supernatant solutions (TFC-ENG-STD-34, *Standard for the Selection of Non-metallic Materials in Contact with Tank Waste*, Attachment E).

Sealed  $\beta$  sources will be used to avoid contamination of the irradiated samples of nonmetallic materials (see Section 3.0). The disadvantages of worker exposure resulting from testing contaminated samples would outweigh the benefits of the data obtained. Furthermore, the use of sealed sources will greatly simplify the postexposure mechanical testing that will be used to quantify the effects of  $\beta$ ,  $\gamma$ , and simultaneous  $\beta$  and  $\gamma$  irradiation on these samples. However, the sealed  $\beta$  sources identified for this Test Plan can only be used at ambient laboratory pressures and temperatures, and only in the absence of the chemical simulant to avoid breaching the thin walls of these sealed  $\beta$  sources (see Subsection 3.3).

The effects of  $\beta$  and  $\gamma$  irradiation must be determined under ambient laboratory conditions without the simulated supernatant solution by comparing the effects of simultaneous  $\beta$  and  $\gamma$ irradiation to those from  $\gamma$  only. This comparison will provide a " $\beta$  enhancement factor" or " $\beta$ factor" to reflect how much additional damage, if any, results from simultaneous irradiation with  $\beta$  and  $\gamma$  relative to that from  $\gamma$  only. (Subsection 3.1 explains the establishment of the  $\beta$  factor in detail). This factor will be used to correct the results obtained from simultaneous exposure to  $\gamma$ irradiation and one or more additional stressors (elevated temperatures, simulated supernatant solutions, and elevated pressures). It is practical to combine  $\gamma$  irradiation and these other stressors because the  $\gamma$  radiation from sealed sources outside the stainless steel vessels will penetrate the vessels (and in some of the tests) the chemical simulant without contaminating the nonmetallic test materials.

The baseline program outlined in Figure 2 and Table 1 could be expanded in two ways, at additional cost and impact to the schedule, by adding: (1) more materials to the program; and (2) different combinations of stressors. Adding more materials could be used to expand the  $\beta$ - $\gamma$  comparison or the assessment of the combined effects of  $\gamma$  irradiation and elevated temperatures (Phase 2) while awaiting ES&H approval for Phase 3. Adding different combinations of stressors could be used to complete a fractional factorial matrix to identify which binary or

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Figure 2. Logic for the Proposed Baseline Test Program Proposed for the Irradiation of Nonmetallic Materials.

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Test		Materials			
Phase	Description	Hoses	Teflon	EPDM O-Rings	Remarks
1	β- $γ$ comparison at ambient high-bay or GIF-pool P and T, no chemical simulant. β = 100 R/hr. γ = 100 and 1000 R/hr.	30-in long primary (2-in ID) hoses in their in-service configuration & coupons of composite hose materials.	Gaskets confined in connectors in their in-service configurations, unconfined gaskets, & coupons.	Will not be included in $\beta$ - $\gamma$ comparison (will not be exposed to $\beta$ radiation in their in-service configurations).	It is anticipated that the $\beta$ - $\gamma$ comparison will require the least amount of time for ES&H R&A, so this series of tests will probably start first.
2	Effects of $\gamma$ & elevated T without chemical simulant, ambient GIF-pool P. $\gamma = 1000$ R/hr. Remove samples at $10^5$ , $10^6$ , $10^7$ , & $> 10^7$ R (exact durations TBD).	Same sample configurations as above. T = 70, 130, & 180 °F (ambient, operating & design, respectively).	Same sample configurations as above. T = 70, 130, & 200 °F (ambient, operating & design, respectively).	O-rings confined in connectors in their in-service configurations, & (perhaps) unconfined O- rings & coupons. $T = 70, 130, \& 200 \ ^{\circ}F$ (ambient, operating & design, respectively).	It is anticipated that the effects of $\gamma$ & elevated T will require more time for ES&H R&A than that for the $\beta$ - $\gamma$ comparison, so this series of tests will probably start second.
3	Effects of $\gamma$ , elevated T, & chemical simulant at ambient GIF- pool P. $\gamma = 1000$ R/hr. Remove samples at $10^5$ , $10^6$ , $10^7$ , & $> 10^7$ R (exact durations TBD).	Same sample configurations as above, same Ts as above.	Same sample configurations as above, same Ts as above.	Same sample configurations as above. Same Ts as above.	It is anticipated that the effects of $\gamma$ , elevated T & chemical simulant will require more time for ES&H R&A than that for the effects of $\gamma$ & elevated T, so this series of tests will probably start third.
4	Effects of γ, elevated P & T, &	Same sample configurations	Same sample configurations	Same sample configurations as above,	It is anticipated that the effects of $\gamma$ , elevated P & T, &

#### Table 1. Identification of the Baseline Experimental Variables and Values for the Irradiation of Nonmetallic Materials.

chemical simulant.	as above, $P = 425$	as above, $P = 400$	P = 400  psig.	chemical simulant will require
$\gamma = 1000$ R/hr.	psig, same Ts as	psig, same Ts as	Same Ts as above.	more time for ES&H R&A
Remove samples at	above.	above.		than that for the effects of $\gamma$ ,
$10^5, 10^6, 10^7, \& >$				elevated T & chemical
$10^7 \mathrm{R}$ (exact				simulant, so this series of tests
durations TBD).				will probably start last.

Notes:

$\beta$ = beta radiation from sealed <sup>85</sup> Kr	ID = inner diameter	R&A = review and approval
sources	$\gamma = \gamma$ radiation from sealed <sup>60</sup> Co or	T = temperature
EPDM = ethylene propylene diene	<sup>137</sup> Cs sources	TDD to be determined
monomer	P = pressure	IBD = to be determined
ES&H = environmental, safety and		
health	psig = pounds per square in (gauge)	
°F = degrees Fahrenheit		
GIF = SNL's Gamma Irradiation		

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ternary combinations of stressors produce the greatest synergistic effects (if any). The baseline program outlined in Figure 2 and Table 1 could also be modified to reduce the work scope, shorten the schedule, or alter the order in which the testing is carried out. All modifications to the baseline program are at the discretion of WRPS.

# 2.2 Materials

Washington River Protection Solutions has identified the inner hoses from the HIHTLs, Teflon gaskets, and EPDM O-rings as the three highest-priority nonmetallic materials for this study. The HIHTL assembly is comprised of two hoses, the primary (inner) hose and the secondary (outer) hose. Both of the hoses are constructed similarly, however, only the primary hose will be the focus of this work. Phase 1, the  $\beta$ - $\gamma$  comparison, will include hoses and Teflon gaskets, but not EPDM O-rings, because the O-rings are exposed to  $\gamma$  irradiation, but not  $\beta$ , in their in-service configurations in the tank farms. Phases 2, 3, and 4 will include all three of these materials.

The samples of hoses will comprise 30-in long primary (2-in nominal inner diameter, or ID) hoses in their in-service configuration, but with blank end fittings; and flat coupons of the composite hose materials provided by River Bend Transfer Systems, LLC, the manufacturer of the HIHTLs. The hose materials are nominally 3/8-in thick and consist of: (1) an inner layer of EPDM rubber; (2) a layer of woven polyester; (3) a central layer of EPDM embedded with two helical steel wraps; (4) another layer of woven polyester; and (5) an outer layer of EPDM. The helical coils embedded in the central layer of EPDM consist of high-tensile-strength, 1/16-in diameter carbon steel, each with 10 ft of steel per ft of hose. The burst pressure reported for new primary hoses is typically 2800–3000 psig.



Figure 3. Sample Primary HIHTL Hose with 6" Ruler for Scale.

River Bend personnel refer to the 30-in long primary hoses in their in-service configuration as "coupons." In this Test Plan, these samples are referred to as "30-in long primary hoses," "30-in long hoses," or as previously stated "hoses." "Coupons" refers to flat pieces of the five-layer composite material (EPDM-woven polyester-EPDM embedded with two helical steel wraps-woven polyester-EPDM) described above, which will be included along with the hoses in Phases 1 through 4 of this study. (See Table 1.) This usage is consistent with that used in the materials-science literature.



Figure 4. EPDM O-Ring on HIHTL End Fitting.

The samples of the Teflon gaskets will consist of gaskets confined in connectors in their inservice configurations, unconfined gaskets, and coupons of the same Teflon used for the confined and unconfined gaskets. The EPDM samples will include O-rings confined in connectors in their in-service configurations, and (perhaps) unconfined O-rings & coupons.





## 2.3 $\beta$ and $\gamma$ Irradiation Rates

The radioactive source term to which the nonmetallic materials described in this Test Plan are exposed in the Hanford tank farms include mainly: (1)  $\beta$  from 90Sr and 90Y, which are associated mainly with the solids in the tanks; and (2)  $\gamma$  from 137Cs and 137mBa, associated mainly with the supernatant solutions.

The relative proportions of  $\beta$  and  $\gamma$  radiation to which the nonmetallic materials are exposed depend on factors such as the concentrations of 90Sr and 90Y associated with the solids at the bottoms of the tanks, the concentrations of these solids suspended in the solutions pumped

through the primary hoses, the concentrations of 137Cs and 137mBa in the supernatant solutions, and the duration of pumping.

WRPS has specified that the primary  $\gamma$  irradiation rate used for this study will be 1000 R/hr2. 100 R/hr will also be used during phase 1 to determine the effects of dose rate. The 1000 R/hr irradiation rate bounds the rates provided in the histogram of estimated double-shell tank (DST) supernatant (137Cs and 137mBa)  $\gamma$  irradiation rates in TFC-ENG-STD-34, Figure E-1 (included herein as Figure 6). Therefore, the irradiation rate specified by WRPS is more representative than Nigrey's (2000) dose rate of 95 krad/hr. Use of this more representative irradiation rate will require longer irradiation times to achieve the exposures requested by WRPS, and thus largely eliminate the issue of limited time for O2 diffusion into the test specimens (Burnay and Hitchon, 1985; Gillen et al., 1989; Wise et al., 1997; Gillen et al., 2003), which could bias the results in favor of reduced damage.



Figure 6. Histogram of DST Supernatant Estimated Dose Rates.

Hanford tank farms nonmetallic materials are evaluated to a nominal 250 Rad/hr based on the above histogram detailing supernatant exposure. For the purposes of this testing, a more detailed source term was developed in Section 2.7.5. Sealed  $\beta$  sources capable of producing an exposure rate of 100 R/hr were identified as the only  $\beta$  sources suitable for the  $\beta$ - $\gamma$  comparison (see Subsection 3.4). This rate is close to that established as a reasonably conservative, bounding  $\beta$  irradiation rate.<sup>3</sup> Gamma irradiation rates of 100 R/hr are proposed for the  $\beta$ - $\gamma$ 

 $<sup>^2</sup>$  The specified dose rate herein uses the units of Roentgen for the purposes of source sizing and selection, as it is independent of the test material being exposed. The radiation unit of concern for nonmetallic material degradation is the Rad, Calculations herein assume materials exhibit absorption behavior similar to tissue, wherein 1 R approximately equals 1 Rad. As explained later, the test configuration will use shielding to 'tune' the radiation field produced from the sources to the desired exposure rate. This shielding approach should allow enough flexibility to account for inaccuracy, as determined in detailed radiation calculations during design, in the absorption behavior assumption.

<sup>&</sup>lt;sup>3</sup> Here, bounding is used to describe a reasonable upper limit. The exact cause of greater damage at lower dose rates is not completely resolved within literature. It is assumed that eliminating diffusion-limited oxidation effects minimizes concern around evidence of lower dose rates causing greater levels of damage on accumulated basis.

comparison (see Subsection 3.1). The WRPS-specified  $\gamma$ -irradiation rate of 1000 R/hr will be used for Phases 2 through 4 of this study.

# 2.4 Values Specified for Other Stressors

This subsection describes the temperatures, the composition of the nonradioactive, caustic solution that will simulate the supernatant HLW solutions, and the pressures that will be used in the baseline test program. Table 1 summarizes the temperatures, whether a nonradioactive, caustic, supernatant HLW simulant will be present or absent, and the pressures for each of the four phases of this study.

Phase 1 of this study (see Table 1) must be carried out at ambient laboratory temperature and pressure, and in the absence of caustic solutions simulating the supernatant tank solutions (see Section 3.0). This is because the sealed  $\beta$  sources for this study (Subsection 3.3) can only be used under ambient conditions to avoid rupture of the thin walls of these sealed  $\beta$  sources (see Subsection 4.3) and contamination of the irradiated samples of nonmetallic materials. This will facilitate the postexposure mechanical testing (Section 5.0).

Phase 2 will be conducted at temperatures of 70, 130, and 180 °F for the hoses and coupons that simulate the hoses. However, the confined and unconfined Teflon gaskets and Teflon coupons; and the confined EPDM O-rings and (perhaps) the unconfined O-rings and EPDM coupons will be tested at 70, 130, and 200 °F. Phase 2 will be performed in the absence of the chemical simulant and at ambient pressure.

Phase 3 will be run at temperatures of 70, 130, and 180 °F for the hoses and coupons; and 70, 130, and 200 °F for the Teflon gaskets and coupons; and the EPDM O-rings and (perhaps) coupons. Phase 3 will include the chemical simulant, but will be performed at ambient pressure. Subsection 2.7.4.1 provides the composition of the simulant.

Finally, Phase 4 will be carried out at the same temperatures used for Phases 2 and 3 with the chemical simulant. However, a pressure of 425 psig will be used for the hoses and coupons; and 400 psig will be used for the Teflon gaskets and coupons; and the EPDM O-rings and (perhaps) coupons.

WRPS specified all of these material-specific temperatures and pressures for the test program. These values are generally defined as ambient, normal operating and design conditions.

It must be emphasized that the simulated supernatant solution described in Subsection 2.7.4.1 will not contain any of the radionuclides present in HLW; rather, this chemical simulant will be nonradioactive, except for very low concentrations of naturally occurring radioisotopes in the reagent-grade chemicals used to synthesize the simulant (e.g., naturally occurring  $^{40}$ K in the reagent-grade K-bearing compounds).

## 2.5 Postexposure Testing

Postexposure mechanical testing will provide the results used to compare the effects of  $\beta$  and  $\gamma$  irradiation of nonmetallic materials, and to quantify the possible synergistic effects of simultaneous exposure of these materials to the four stressors described above.

Postexposure testing will include quantification of both mechanical performance and mechanical properties. For the purposes of this Test Plan, postexposure mechanical performance testing refers to measurements that are directly applicable to the primary hoses, Teflon gaskets, and EPDM O-rings as used by WRPS in the tank farms (i.e., in their in-service configurations). Examples include measurements of the pressure required to burst the hoses, or the torque required to prevent leakage of Teflon gaskets and leak rate of EPDM O-rings in their confined configurations. Postexposure mechanical properties testing, on the other hand, refers to standardized procedures such as the ASTM International (ASTM) test for compression set or elongation at rupture. ASTM procedures will be used for coupons of the composite hose materials, Teflon, or EPDM. Section 5.0 of this Test Plan describes these procedures in detail.

## 2.6 Relation of this Test Plan to Other Studies

The study described in this Test Plan will include at least three features that will be unique, or at least atypical of, the approaches used in other studies of radiation effects. First, this study will emphasize the irradiation of nonmetallic materials in their in-service configurations, not just the irradiation of coupons (see Subsection 2.2). This feature will facilitate the direct application of the results of postexposure mechanical testing to the performance of these materials in the Hanford tank farms. Second, this study will simulate in-service exposure of these nonmetallic materials to simultaneous multiple stressors (i.e., various combinations of  $\beta$  and  $\gamma$  irradiation; elevated temperatures; the presence of a nonradioactive, synthetic solution representative of the supernatant solutions in the single-shell tanks (SSTs) and DSTs; and elevated pressures). Third, this study will emphasize postexposure mechanical performance testing, not just mechanical properties testing, to quantify the synergistic effects of simultaneous exposure to multiple stressors. Subsection 2.5 explains the difference between mechanical performance and mechanical properties testing.

## 2.7 Technical Justifications

This Test Plan is designed to address technical questions about the use of nonmetallic materials with Hanford tank farms using representative conditions to simulate exposure. Justifications of the conditions selected are presented herein.

## 2.7.1 Materials

The materials identified in this Test Plan were selected based on their application within the Hanford waste transfer system and for the added value in addressing regarding the longevity. This value is manifested in an improved safety basis for operating safety-significant equipment, beneficial cost and schedule gains due to potentially increased service lives, and reduced risk to the cost and schedule baseline by developing a service-life basis which may be used for improved run/repair/replace decisions. Collectively, materials evaluated for inclusion in this Test Plan are listed in TFC-ENG-STD-34. Of this list of materials, testing for three of these materials is initially included in this Test Plan. After review of the extent of testing required to address technical issues and the durations required, the focus of the Test Plan was narrowed to HIHTLs, Teflon gaskets, and EPDM O-rings. The methodology developed herein can be extended to the remaining materials with appropriate postexposure tests selected, during or after the approximate six-year schedule as found prudent.

Discussed in Section 2.1, the primary focus of the Test Plan are the primary hoses of the HIHTL assemblies. HIHTLs are currently used within waste retrieval systems due to the extensive cost savings provided over the use of pipe-in-pipe systems. HIHTLs provide flexibility to support multiple tank retrievals without requiring extensive excavation and removal that a piped system requires and minimizes the space required to support retrieval transfers compared to piped systems. The EPDM hoses are also used in flexible jumpers in the DST transfer system.

HIHTLs are a composite rubber hose, consisting of an inner and outer layer of EPDM, two intermediate layers of polyester fibers surrounding central dual helical steel wire reinforcement with EPDM filler. Swaged stainless steel threaded connectors are fitted on the ends of the hose. In general, the EPDM layers protect the hose from environmental exposure, the polyester fibers provide axial strength, and the steel wire reinforcement provides radial strength. The swaged fittings have performed exceptionally and have not been a known point of failure in any previous testing or operating experience.

Teflon gaskets used in PUREX connectors are the secondary focus of the Test Plan, primarily due to the discrepancy with the exemplary service record at Hanford compared to the poor radiation tolerance of Teflon<sup>4</sup>. PUREX connectors have a unique confining geometry that may limit factors contributing to the damaging mechanism. PUREX connectors are used to make remote connections to minimize worker exposure in waste transfer pits and boxes. The Teflon gasket is the sealing surface between the connection points.

EPDM O-rings are a common sealing material within pressure boundary components, found as backup rings in valve seats and in the HIHTL end fittings. Their inclusion in this set of testing is relatively simple and does not require a significant amount of space. Additionally, the inclusion of an EPDM-only component may provide general qualitative evidence of the performance of the EPDM component in HIHTLs.

Of the remaining TFC-ENG-STD-34 materials, two major component types can be identified. Tefzel®1, Kynar®<sup>5</sup>, and Ultra-High Molecular Weight Polyethylene (UHMWPE) are employed as valve seat materials. Two specific flat gasket materials, Garlock®<sup>6</sup> Blue-Gard®6 3000 and 3700, are identified. The valve seat materials would require similar space and confined geometry considerations as the Teflon gaskets, yet these materials do not have the outstanding conflict between literature radiation data and operating experience of Teflon use at Hanford. The flat gasket materials would require greater space than the EPDM O-rings, are less prevalent and do not provide additional insight into HIHTL durability as the O-rings potentially do. Other materials listed in TFC-ENG-STD-34 find more specialized applications (i.e., are much less prevalent in use) requiring either explicit test specificities that reduce the potential value if other applications are later employed, or generalizations that reduce the direct applicability. Either option is less ideal than prioritizing testing the materials identified above. The methodology described in this Test Plan can be implemented into testing for these materials with relatively

<sup>&</sup>lt;sup>4</sup> Teflon radiation tolerance is frequently cited at approximately  $10^4$  Rad. Usage of Teflon at Hanford suggests this critical dose has been exceeded routinely with no noted failures.

<sup>&</sup>lt;sup>5</sup> Kynar is a registered trademark of Arkema, Inc., King of Prussia, Pennsylvania.

<sup>&</sup>lt;sup>6</sup> Garlock and Blue-Gard are registered trademarks of Garlock Sealing Technologies LLC, Palmyra New York.

little additional effort. It was determined to be prudent to focus on the specified materials, while ensuring the applicability of the remaining materials.

### 2.7.1.1 In-Service Configurations

Testing components in their in-service configurations is an essential component of this Test Plan. Testing 'coupons,' or small samples, of material are useful for making comparative, mechanistic, or modeling determinations. The goal of this Test Plan is to develop a technical basis for an expected service life of nonmetallic components. In order for this to be achieved, the testing must be as representative of the in-service exposure as possible. The efforts to achieve representative conditions with regard to configuration are discussed herein.

Significant physical geometry will be preserved to the maximum extent possible. This includes metallic components as part of the assembly containing metallic materials, the directional source of radiation, chemical attack, and thermal exposure. Using Teflon PUREX gaskets as an example, the retaining ring geometry will be entirely preserved, while the sealing surfaces and immediate surrounding surface of the connector block and of the nozzle will be preserved. An alternative clamping/compression mechanism may be selected and the remainder of the assembly form may be adjusted to minimize space and maximize penetration from an external radiation source. The HIHTL primary hoses will have their configuration conserved in their entirety. EPDM O-rings may be contained in the hose assembly on the end fittings.

All components will be internally exposed to a simulated supernatant liquid solution to represent the chemical environment during waste transfers. Components will be connected or compressed in the same manner as in field deployment or in a representative manner resulting in similar physical force applied.

## 2.7.2 Temperature

Temperature is well known as a critical parameter in the degradation of polymers. Temperature not only drives the rate of degrading chemical reactions, but also can result in property changes of the polymer material affecting transport of reactants and breakdown or leeching of additives. Additionally, mechanical properties are often temperature sensitive; therefore, any synergistic effects based on mechanical stresses may also have a temperature contribution. The effects of temperature on polymer degradation have been the explicit focus for a number of accelerated aging studies to examine time-temperature superposition, solely to account for and predict the significant effect temperature has on degradation.

Test temperatures are specified to be at realistic conditions. In doing so, the need for extrapolation from elevated test temperatures to field exposure conditions is eliminated. Interpolation within the test temperature range is less likely to produce erroneous results that result from changes in the degradation regime, and is more likely to exhibit known and predictable polymer degradation behavior as evidenced by the applicability of time-temperature superposition in some materials. Temperatures selected in Table 2 will nominally represent ambient or low-end operating conditions, intermediate or heat-traced conditions, and design temperatures. There is some evidence that increased levels of degradation may occur at intermediate temperatures, as evidenced by SAND94-1104C, *Explanation of Enhanced Mechanical Degradation Rate for Radiation-Aged Polyolefins as the Aging Temperature is* 

*Decreased.* This phenomenon is known as the inverse temperature effect. This has been demonstrated for polyolefins in the above paper, however it supports maintaining an intermediate temperature in case this phenomena exists for other materials as well.

_	Ambient	Intermediate	Design
HIHTL	70ºE	1209	180°F
Other	/0 Г	150 Г	200°F

 Table 2. Specified Test Temperatures Representing Nominal Conditions.

## 2.7.3 Pressure

Mechanical pressure will exist in two forms during testing: internal fluid pressure and applied compression force to sealing components. Applied compression forces will be imposed by using representative in-service configurations. Internal fluid pressure will be applied at design conditions, 425 psig for HIHTL and 400 psig for other materials. There will be some cyclic pressurization due to replenishment of the chemical simulant. Generally, internal fluid pressure is expected to have only a minor contribution to material degradation, based on previous work detailed in RPP-6711, Appendix L. It should be noted that the testing reported in Appendix L of RPP-6711 was a static pressurization and did not include any pressure cycling. However, compressive and stress relaxation properties may be significant for sealing components.

Cyclic mechanical stress effects are a relatively well-understood phenomenon. Polymers, specifically elastomers, generally exhibit elastic behavior, recovering completely from applied stress. Over time as the polymer degrades, it may become fatigued or embrittled and begin to take on a compression set for repeated or consistently applied stress. It is expected that a component degraded from other exposure will take on mechanical changes such as compression set earlier than a non-exposed component subjected to similar mechanical stress. Additionally, if flaws develop in a component, such as a small crack, it would be expected that applied pressure would result in a concentration of stress at the crack, potentially resulting in a worsening of the flaw. However, literature searches have yielded little information on enhancing synergistic degrading effects in nonmetallic materials. While other conditions may have been necessary for development of the flaw, the worsening of the flaw may be primarily dependent on mechanical pressure. Although mechanical stress may or may not possess synergistic qualities with respect to the other stressors, its effects may contribute to material degradation and will be included to properly represent conditions during waste transfers and retrieval.

## 2.7.4 Chemistry

Chemical exposure will be provided using a nonradioactive, caustic, supernatant HLW simulant of the Hanford tank farms. The chemical simulant will approximate the exposure conditions created during waste retrieval. This simulant will expose components to its constituents, radiolysis-generated reactive products, and leach mobile products out of the polymer. In order to maintain a consistent chemical environment, the simulant will be replaced approximately every two months. Expected potentially reactive species include, but are not limited to nitrates, nitrites, oxygen, hydroxide, peroxides, free radicals, and various intermediate products. Of these, oxygen is the most attributed and studied component to polymer degradation.

Organic chemicals are also well known to be degrading to polymeric materials. However, the resistance to degradation from organic chemicals is highly specific to the organic chemical and polymer exposed. Selection of a representative organic component has been determined to be unfeasible since it is believed that no organic simulant could be both inclusive of a significant number of possible organic chemicals of concern and remain representative of the majority of waste. It is recommended that organic chemicals of concern be evaluated for compatibility on a case-by-case basis.

Stress corrosion cracking has been observed in a few polymers in case studies, predominantly acetals, in acidic environments (Wright 2006). Stress corrosion cracking is caused by hydrolysis of susceptible functional groups, including ester, amide, imide, and carbonate groups. Other chemically aggressive species such as condensed halogens or their acids have been identified as responsible in case studies, notably PVDF. Such conditions are not expected on wetted surfaces in the Hanford waste transfer system. Polyethylene terephthalate, commonly called polyester, exists as the fiber reinforcement within the HIHTL hose. Polyester is known to be subject to stress corrosion cracking with exposure to NaOH, however the polyester within the hose assembly is not in direct contact with the process fluid, separated by a layer of EPDM. Generally, materials in contact with process fluid are suitably resistant, as evaluated in TFC-ENG-STD-34. However, evolution of polymer degradation products may concentrate within a constrained geometry to create a microenvironment conducive to crevice or stress corrosion cracking of either the polymer material or the surrounding steel components. Such a mechanism has not been observed at Hanford, however examinations of equipment removed from service are generally not conducted due to the extent of contamination.

#### 2.7.4.1 Chemical Simulant

Testing will utilize a reasonably bounding chemical source term developed in TFC-ENG-STD-34, shown in Table 3. This source term was developed using the Tank Waste Information Network System (TWINS) database for inclusion of 95% by mass of DST supernatant analytes, with inclusion of Cl<sup>-</sup>, F<sup>-</sup>, and PO<sub>4</sub><sup>3-</sup> because of material or process sensitivities, resulting in a cumulative wt% of 97%. Total inorganic carbon is assumed to be Na<sub>2</sub>CO<sub>3</sub>.

There is no existing procedure for synthesizing a simulant of this composition; thus, one will need to be developed. The approach taken for development of an AN-107 simulant in WTP-RPT-115, *Gas Generation Testing and Support for the Hanford Waste Treatment and Immobilization Plant*, may be useful, wherein two solutions were prepared such that ideal mixing would result in the target composition after addition of nonreactive salts. Precipitates were allowed to form and drop out of solution.

Compound	Wt%	Molarity
KNO <sub>3</sub>	1.31	0.17
NaNO <sub>3</sub>	11.41	1.7
NaNO <sub>2</sub>	7.38	1.4
Na <sub>2</sub> CO <sub>3</sub>	3.99	0.5
NaOH	4.61	1.5
Al(OH) <sub>3</sub>	1.93	0.55
Na <sub>2</sub> SO <sub>4</sub>	0.64	0.06
NaCl	0.47	0.11
Na <sub>3</sub> PO <sub>4</sub>	0.49	0.04
NaF	0.10	0.03
Balance of $Na^+$	1.19	

No solid particulate matter will be included in the simulant, as erosion is not being considered within this test plan. The existing erosion basis will continue to apply to nonmetallic material usage. Specifically for HIHTL hoses, in RPP-6711, EPDM rubber is cited for its resistance to abrasive erosion due to its ability to deform elastically under impact and its common applications in tires and pumps. Furthermore, a meaningful test program for erosion requires extensive test controls over a wide variety of parameters, resulting in the extensive complication of delivering uniform erosive conditions to a hundred or more samples within an underwater radiation environment. Including erosive material would also have significant design challenges and complicate the safety review and approval process.

#### 2.7.4.2 Oxygen Conditions

Oxygen plays a vital role in degradation mechanisms of nonmetallic materials. The sources and transport of oxygen needs to be understood to ensure it is accounted for properly during testing for unconstrained geometries such as HIHTL hoses. For constrained geometries, diffusion limited oxidation (DLO) effects are suspected in service due to the lack of replenishment of oxygen. During waste transfers, HIHTLs are expected to be full of the pumped fluid due to minimum flow requirements practiced to prevent settling of solids during transfer. The oxygen content of waste is explored further in this section. While installed, the hose-in-hose geometry connects the annulus space between the primary and secondary hose to be exposed to the air space in the tanks. It is expected that this allows for a source of oxygen replenishment for the exterior of the primary hose, and assumes that the surface hose is exposed to a chemically ambient air.

Oxygen generation and depletion in Hanford wastes has been a topic of study within the past two decades as part of the safety-related effort in determining hydrogen gas evolution rates to protect against flammable gas and buoyant gas displacement events. Radiolysis caused by water being present in a radiation field results in the generation of hydrogen and oxygen. While hydrogen was the primary focus of these studies, oxygen plays a role in flammable gas mixtures and it has

been found that the presence of an oxygen atmosphere increases the hydrogen generation rate of Hanford wastes, especially those containing organic material. This work is summarized in WTP-RPT-115.

WTP-RPT-115 concluded that oxygen is depleted by actual tank wastes and in simulants for both high organic content and no organic content. The results from WTP-RPT-115 are shown in Table 4 with the simulant composition shown in Table 4, compared to the specified TFC-ENG-STD-34 simulant. The depletion of oxygen matches process knowledge that quiescent tanks contain oxygen-depleted supernatant.

	Cover Gas, 20% O <sub>2</sub> in Neon		
Tank Type	O <sub>2</sub> depletion, mol/kg/day	Standard dev	TOC (minus oxalate), wt%
AN-102	-1.83E-02	1.19E-02	1.99
AN-106	-4.23E-03	1.41E-03	0.02
AN-107	-2.90E-02	1.82E-02	2.86
AW-101	-3.13E-03	1.26E-03	0.14
U-106	-2.30E-02	1.58E-02	2.70
AN-107 simulant	-1.18E-02	4.44E-03	1.37
AN-107 simulant, no-organics	-1.94E-03	3.25E-04	0

Table 4. WTP-RPT-115 Oxygen Depletion Rates for Actual and Simulated Wastes.

Mechanisms presented in WTP-RPT-115 are generally hydrogen focused. Mechanisms focusing on oxygen depletion all used degradation of organics to explain oxygen consumption, with no inorganic mechanisms postured. Appendix C of WTP-RPT-115 discusses possible mechanisms that would be responsible for increasing hydrogen generations rates due to oxygen; however, they are deemed insignificant to organic degradations. This makes it difficult to understand mechanistically what is responsible for the observed oxygen depletion organic-less AN-107 simulant, AN-106 waste and perhaps AW-101 waste, as shown in Table 4. However, the oxygen depletion of the AN-107 simulant without organics provides confidence that the TFC-ENG-STD-34 specified simulant will also be oxygen depleting under irradiation.

Thus, for primary hoses, the external surface is exposed to atmospheric oxygen, while the internal surface is not exposed during waste transfers. The test configuration for test Phases 3 and 4 including chemical exposure via simulant will model this environment well. However, it must be determined that the oxygen exposure and dose rate specified within Subsection 2.7.5, 1,000 R/hr gamma, will not create diffusion-limited oxidation (DLO) effects. A methodology for determining when DLO effects occur, developed by AV Cunliffe and A Davis and presented by R Clough and K Gillen in *Irradiation Effects on Polymers*, will be applied to determine if DLO effects will be present for HIHTL hose irradiation at 1000 Rad/hr. Cunliffe and Davis' criteria is presented below, where values greater than eight indicate DLO effects are present.<sup>7</sup>

<sup>&</sup>lt;sup>7</sup> The following analysis and corresponding values used are found in *Irradiation Effects on Polymers*.

$$\frac{L^2 \Phi}{P_{ox} p} \ge 8$$

Where: L =sample thickness (cm)

 $\Phi$  = oxygen consumption rate (ml STP/ml/s)

 $P_{ox} = oxygen permeation rate (ml STP/cm/cm Hg/s)$ 

p = partial pressure of atmospheric oxygen (cm Hg)

 $\Phi$  can be determined by multiplying a G(-O<sub>2</sub>) value (ml STP/ml/Gy) by the dose rate (units of Gy/s, by dimensional analysis). A quantitative test of the Cunliffe-Davis theory is discussed wherein P<sub>ox</sub> was found to be 7.3 × 10<sup>-9</sup> ml STP/cm/cm Hg/s at 70 °C and G(-O<sub>2</sub>) was found to be 5.8 × 10<sup>-10</sup> mol O<sub>2</sub>/ml/Gy for a commercial EPDM rubber. Application of the ideal gas law at STP converts G(-O<sub>2</sub>) to  $1.3 \times 10^{-5}$  ml STP/ml/Gy. While not explicitly stated, it is assumed this criterion assumes atmospheric oxygen diffusion in two dimensions on a sample of thickness L. In our application, oxygen diffusion is available in only one direction; therefore, L will be twice the thickness of HIHTL hose (3/8 inch), plus an additional 1/16 inch to account for EPDM tape used to apply heat trace, equaling 7/8 inch or 2.2 cm. 1000 Rad/hr corresponds to 2.78 × 10<sup>-3</sup> Gy/s. Partial pressure of oxygen at SNL is approximately 13.2 cm Hg.

$$\frac{(2.2cm)^2 * \left(1.3 * 10^{-5} \frac{ml STP}{ml * Gy}\right) * \left(2.78 * 10^{-3} \frac{Gy}{s}\right)}{\left(7.3 * 10^{-9} \frac{ml STP}{cm * cm Hg * s}\right) * 13.2cm Hg} = 1.82 < 8$$

The Cunliffe-Davis criterion indicates that there will not be DLO effects for irradiation of HIHTL filled with chemical simulant at 1000 Rad/hr.

#### 2.7.5 Radiation

Degradation caused by irradiation of nonmetallic materials is a growing concern in both the commercial and defense nuclear industries. Commercially, nuclear power plants seeking to extend their license beyond their original design life are evaluating degradation of insulating cables and other materials. These plants are evaluating the remaining service life as well as the remaining ability to withstand loss of coolant accidents after years of exposure. The defense sector work includes this Test Plan, as well as degradation of sealing materials in packaging and shipping containers.

This Test Plan, to our knowledge, is unique in delivering low dose rate exposure over a significant period of time with simultaneous exposure to a chemical solution, elevated temperatures and pressures. This approach will allow for the study of synergistic effects that may accelerate or mitigate known degradation mechanisms. These mechanisms will be covered briefly herein; extensive detail of these mechanisms is available in literature.

Radiation-induced damage of polymeric materials can be generalized as a change in the polymeric structure, usually predominated by the long carbon chain backbone. This chain can undergo scission and cross-linking; changes which are generally understood and modeled as a chemical system by the generation, propagation, and termination of radicals. Free radicals are highly reactive, especially in the presence of oxygen, as reflected by its prevalence in reactive pathway studies and kinetic models. Changes in material properties can be generally attributed to either chain scission or cross-linking, such as increased hardness, strength, brittleness and reduced flexibility and elasticity for cross-linking polymers. However, changes in material properties, desirable or not, are the consequence of a dynamic environment wherein the competition between chain scission and cross-linking can change. The non-linear behavior of material properties as a function of dose rate is discussed in Wilski (1986), while Nigrey (2000) demonstrates the non-linear behavior of material properties as a function of absorbed dose. In short, the half value dose may suggest a final cross-linked or scissioned state, but the journey to that state may have local maxima in either direction for both mechanism and properties.

Possible synergistic effects of radiation-induced damage are based on interactions with the oxygen-radical pathways. Reaction rates of these degradation pathways may be temperature sensitive, whether it be from increased diffusion and transport of oxygen, Arrhenius behavior of initiating, intermediate, and termination reactions, promotion of competing intermediate reactions, or increased radical mobility resulting in changes in observed oxidation reaction rates. The presence of a chemical solution may make available a number of additional reactive and oxidative chemicals, including nitrates and nitrites, or radiolysis products such as peroxides. Mechanical stress, in the form of internal pressure, or compression in the case of sealing materials, may change mass transfer via altered micropore structure, physical alterations of the polymer chain configuration from stretching, straining, or otherwise, resulting in possible changes in energy absorption properties, relative physical relocation of existing radicals, and changes in the apparent activation energy of scission or cross-linking reactions.

The objective of this Test Plan is to expose materials to a dose as representative as possible while achieving results in a reasonable time frame, yet improving on past irradiation experiments. To this end, exposure rates of 1000 R/hr of gamma radiation and 100 R/hr of both gamma and beta radiation have been specified. This improves on historical irradiation-only experiments at dose rates on the order of  $10^{5}$ - $10^{7}$  Rad/hr.

#### 2.7.5.1 Radiation Type

This Test Plan must align the types of radiation from Hanford wastes with suitable test sources capable of surviving the test environment. Both topics are addressed here.

Hanford wastes are a product of plutonium production from the 1940s up to 1985, and the separations processes continued up to 1988. Alpha, beta, and gamma emitters are all present in Hanford wastes in varying quantities. However, since reactor production ceased in 1985, the majority of remaining Hanford waste radioactivity are attributed to the approximately 30 year half-life fission products of <sup>137</sup>Cs and <sup>90</sup>Sr, and their respective daughter products <sup>137m</sup>Ba and <sup>90</sup>Y. Various alpha emitters are present; however, their exclusion in the Test Plan is a consequence of the progeny and scope of the Test Plan. Post-mortem (in this case, taken out of service, not failed) examination of components were considered prior to direction to develop this Test Plan, but not deemed worth the cost of worker exposure in handling the contaminated materials

(7/25/2011 U.S. Department of Energy Response to the Defense Nuclear Facilities Safety Board). Alpha sources are too fragile to withstand the environments to be produced during testing and working directly with an alpha-emitting solution resulting in contaminated equipment undermines the inherent direction of the Test Plan scope.

Testing will be conducted using sealed gamma and beta sources. Feasible gamma sources include <sup>137</sup>Cs and <sup>60</sup>Co in a pencil configuration. <sup>60</sup>Co pencil sources may be more readily available and offer other advantages over using a <sup>137</sup>Cs source which provides the best matching radiological energy spectrum. A beta source configuration has been found capable of producing the required dose rate in the desired configuration; a <sup>85</sup>Kr line source has been identified. High beta dose rates are typically generated in beams, in relatively fragile packing to allow transmission, or with small windows that do not provide the desired coverage. Further details on source selection are contained in Subsection 4.3.

An important element of this Test Plan is focused on potentially different effects from gamma versus beta irradiation. The existing background of most knowledge about the radiation resistance of nonmetallic materials is based on accelerated testing using high rates of gamma irradiation only. A series of reports from a joint U.S. and French research program examined the comparative effects for gamma and beta irradiation concluded that the effects can be correlated on the basis of absorbed dose (SAND86-0366<sup>8</sup>). However, these tests generally used thin specimens. It is not clear, due to the different energy deposition properties of gamma and beta radiation, if they will have the same effect on a bulk component, such as HIHTL hoses.

Beta irradiation, compared to gamma, has a strong interaction with matter owing to its charge. Electrons essentially continuously interact, and deposit energy, to the surrounding matter. These interactions occasionally cause secondary electrons to be emitted from the path of the primary electron resulting in a branched pattern of energy deposition in a short range in dense matter. Beta radiation from a <sup>90</sup>Sr source will deposit approximately 90% of its energy in 4 mm of water.

Gamma irradiation is modeled as a particle that does not continuously interact with the matter around it. Instead, it is modeled as having a particular chance to 'collide' with matter it passes through. Several feet of water is often used as shielding in pool irradiator facilities. For the photon energies of concern, Compton scattering is the dominant phenomenon upon interaction in matter. In Compton scattering, a photon interacts with an outer electron, ejecting it and producing a photon of lesser energy in a random direction, hence scattering. This effect leads to the phenomenon that the absorbed dose of a material subject to gamma irradiation is higher slightly under the surface of the material than at the surface. The decay of gamma irradiation dose deposition generally follows the Beer-Lambert law, with mass attenuation coefficients unique to individual materials. 1 cm of water will absorb less than 10% of the dose rate from the <sup>137m</sup>Ba photon emission.

<sup>&</sup>lt;sup>8</sup> Buckalew, W.H., et al., U.S./French Joint Research Program Regarding the Behavior of Polymer Base Materials Subjected to Beta Radiation Volume 2: Phase-2a Screening Tests.

### 2.7.5.2 Irradiation Dose Rate

Dose rate selection must consider multiple factors: expected radionuclide content of Hanford waste streams, test durations to reach critical doses, facility capabilities, and DLO effects. In order to make this determination, irradiation of HIHTL hoses will be used as a basis given their importance in the Test Plan and on their lack of confined geometry. DLO effects are discussed in section 2.8.4.2. Additionally, the capabilities of SNL's GIF and associated facilities far exceed the requirements of this Test Plan.

Hanford waste is stored in 177 underground storage tanks containing 56 million gallons of liquid and solid waste. The waste was produced from multiple reactors and several processing and postprocessing plants and methods, leading to a considerable variety in radionuclide content. Waste transfer operations may involve pumping supernatant liquid only or solids suspended in water or supernatant liquid. Due to the fact that <sup>137</sup>Cs and <sup>90</sup>Sr are highly partitioned to the liquid phase and solid phase, respectively, the slurry stream of waste solids suspended in supernatant liquid produces the largest amount of energy deposited in an HIHTL and most conservative to use as a basis. Based on volumetric material balances, averaged solids loading during retrieval are typically 5% solids or less; with one retrieval approaching 10% solids (RPP-RPT-50852, *Operating History Statistics of POR104 Portable Valve Box*). Accounting for the possibility of more efficient retrieval technologies, 15% solids will be used as a conservative basis.

Radionuclide loadings are tracked at Hanford as part of the TWINS. The information is segregated into many parameters, including tank, waste layer designation, volume, and constituent concentration. The relevant parameters listed below with respective units/descriptions.

- Waste Phase
  - o Sludge
  - o Saltcake
  - o Supernatant
- Analyte
  - o <sup>137</sup>Cs
  - o <sup>90</sup>Sr
  - Inventory
    - o Ci
  - Volume
    - o kL
- Component Density
  - o g/mL
- Adjusted Concentration
  - o uCi/mL (for liquids)
    - $\circ$  uCi/g (for solids)

The waste phase designation for sludge and saltcake layers carries several different variations (e.g., 'Sludge [Liquid & Solid]' and 'Sludge Solid'); however, no phase shares a descriptor as both saltcake and sludge. Any variation of sludge was designated as sludge, and so forth. All

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SSTs and DSTs were included. Data was retrieved on 6/4/2012 for analytes <sup>90</sup>Sr and <sup>137</sup>Cs with a decay date of 1/1/2008. The data were processed using simple arithmetic as follows:

- 1. Solids concentrations were adjusted to volumetric basis by multiplication with the component density.
- 2. Radionuclide inventory and waste volume values were summed for total values.
- 3. Segregated data by waste phase and analyte.
- 4. Sorted data from smallest to largest concentration.
- 5. Calculated waste volume and radionuclide inventory as a percentage of total volume or inventory for each waste layer.
- 6. Calculated percent cumulative waste volume and radionuclide inventory in ascending order with ascending concentration.

Matching process knowledge, it was found that sludge solids contribute to dose more than saltcake; therefore, sludge waste is used as the basis for solid radionuclide contribution. Plotting cumulative volume and radionuclide inventory versus analyte concentration for <sup>137</sup>Cs in supernatant and <sup>90</sup>Sr in sludge results in Figures 7 and 8.



Figure 7. <sup>137</sup>Cs Supernatant Concentrations Versus Cumulative Waste Volume and Radionuclide Inventory.

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Figure 8. <sup>90</sup>Sr Sludge Waste Layer Concentrations Versus Cumulative Waste Volume and Radionuclide Inventory.

Figure 7 displays the range of <sup>137</sup>Cs concentrations existing in Hanford tank farms supernatant waste. From this graph, a reasonably bounding <sup>137</sup>Cs concentration can be identified to provide context for selection of a testing dose rate. Over 90% of supernatant waste by volume has a <sup>137</sup>Cs concentration of about 450  $\mu$ Ci/mL or less. An approximate 25% increase to 580  $\mu$ Ci/mL bounds two additional supernatant concentrations and an additional 5-6% of total <sup>137</sup>Cs inventory. A single supernatant composition is not bounded by 580  $\mu$ Ci/mL, which at 1470  $\mu$ Ci/mL represents a 250% increase over the selected basis of 580  $\mu$ Ci/mL. This value is bounding for 95.8% of all supernatant waste, excluding only Tank 241-AZ-101 supernatant.

Figure 8 displays the range of <sup>90</sup>Sr concentrations existing in Hanford tank farms sludge waste layers. From this graph, a reasonably bounding <sup>90</sup>Sr concentration can be identified to provide context for selection of a testing dose rate. Over 90% of supernatant waste by volume has a <sup>90</sup>Sr concentration of 1700 –Ci/mL or less. Increasing the bounding value beyond this point, returns little additional bounded volume for a large increase in concentration. The next plateau in the graph occurs at 6400  $\mu$ Ci/mL, representing an increase of over 350% to bound an additional 2.6% of sludge waste. At a total volume of 42,000 kL, or 1.11 million gallons, the 8.3% not bounded by the 1700 –Ci/mL value represents ~92,000 gallons of sludge waste or less than 1% of the total of 56 million gallons of Hanford tank farms waste. Furthermore, sludge conservatively bounds the <sup>90</sup>Sr content of saltcake layers.

Collectively, use of 580  $\mu$ Ci/mL <sup>137</sup>Cs supernatant and 1700  $\mu$ Ci/mL <sup>90</sup>Sr sludge as a basis for a slurry transfer bounds all possible transfers, excluding AZ-101 supernatant and 92,000 gallons of
sludge. These source concentrations can be used to calculate a dose rate produced from a reasonably bounding 15% by volume sludge slurry transfer. By assuming the <sup>137</sup>Cs contribution from the sludge is insignificant relative to the supernatant and the <sup>90</sup>Sr contribution from the supernatant is insignificant relative to the sludge, a slurry source term is calculated by simple multiplication by 85% and 15%, respectively, resulting in 493  $\mu$ Ci/mL <sup>137</sup>Cs and 255  $\mu$ Ci/mL <sup>90</sup>Sr.

Both <sup>137m</sup>Ba and <sup>90</sup>Y exhibit secular equilibrium with their parent radionuclides. Approximately 95% of <sup>137</sup>Cs decays result in <sup>137m</sup>Ba, with the remaining 5% going directly to the stable <sup>137</sup>Ba. <sup>90</sup>Sr decays directly to <sup>90</sup>Y. The resulting concentrations of <sup>137m</sup>Ba and <sup>90</sup>Y are 468.4  $\mu$ Ci/mL and 255  $\mu$ Ci/mL, respectively. A summary of the radionuclide concentrations, and their respective emission energies are shown in Table 5.

To apply this source term to determine a dose rate, a rule of thumb from *The Health Physics and Radiological Health Handbook* will be applied. This rule of thumb is listed in the same form for both gamma and beta sources; however, their applicability varies due to the nature of the radiation type. The rule of thumb is to determine dose rate for tissue immersed in an infinite solution of uniform radionuclide concentration. The rule of thumb can be modified by a factor of 0.5 to calculate the dose rate at the surface of an infinite solution. Because no input parameters are based on the object being irradiated, including density and thickness, and the infinite field assumption, the rule of thumb calculates a maximum possible dose rate for a given concentration. Given the penetration depth of the beta energies of concern, the diameter and thickness of the HIHTL hose, the infinite solution assumption is valid. For gamma irradiation, the penetration depth is much greater than the thickness and diameter of HIHTL hose, meaning that the rule of thumb is very conservative. Furthermore, it is assumed for this calculation that the f-factor for dose deposition in tissue compared to nonmetallic materials is 1.

Dose Rate 
$$(\frac{Rad}{hr}) = \sum_{i} 0.5 * 2.12 * \frac{E_i * C_i}{\rho}$$

Where:  $E_i = Emission energy for radionuclide i (MeV)$ 

 $C_i$  = Concentration of radionuclide i (uCi/mL)

 $\rho$  = Density of solution (g/mL)

Application of the rule of thumb results in the values listed in Table 5. A density of 1.25 g/mL will be conservatively used for a 20% by volume sludge slurry solution, as a lower density value results in a higher dose rate.

	Emission			Dose Rate to	Total Dose
Radionuclide	Туре	Emission Energy	Concentration	Tissue	Rate to Tissue
<sup>137</sup> Cs	Beta	0.174 MeV	493 uCi/mL	74.1 Rad/hr	Gamma
<sup>137m</sup> Ba	Gamma	0.662 MeV	468.4 uCi/mL	267.7 Rad/hr	267.7 Rad/hr
<sup>90</sup> Sr	Beta	0.196 MeV	255 uCi/mL	43.2 Rad/hr	Beta
<sup>90</sup> Y	Beta	0.934 MeV	255 uCi/mL	205.7 Rad/hr	322.9 Rad/hr

 Table 5. Application of Rad. Health Handbook Rule of Thumb

For gamma irradiation of tissue, the conversion from Roentgen to Rad is approximately a factor of 1. It must be reemphasized that these values are determined by applying a rule of thumb rather than a rigorous calculation, with simplifying assumptions to determine a slurry stream composition, with bounding source term values and a conservative solids loading. However, this estimation provides much needed context for a reasonably bounding dose rate for test plan implementation. This calculation is not intended to be directly comparable to existing dose rate estimation practices employed for Hanford tank farms.

Radiation resistance of materials is often presented in literature in the form of critical dose, which is typically defined as the amount of dosage required to cause a 50% reduction in an specific material property, usually tensile strength. Most materials have a cited critical dose on the order of  $5 \times 10^6$  Rad to  $5 \times 10^7$ , with notable exceptions of Teflon at  $10^4$  and polyether ether ketone (PEEK) at 10<sup>8</sup> or higher. EPDM and polyethylene, components of HIHTL hose, are commonly cited at around  $1-2 \times 10^7$  Rad. Usually these critical doses are determined at dose rates on the order of 10,000-100,000 Rad/hr (100-1000 Gy/hr), resulting in irradiation times of 3 days to 12 weeks. Extremely thin samples are required to prevent DLO effects at these rates. However, as previously discussed, this Test Plan is designed to improve upon these bases by using a more realistic dose rate on actual components to simulate actual exposure conditions. If the combined dose rate of 590.6 Rad/hr developed above was used to obtain  $2 \times 10^7$  Rad, the total irradiation time is approximately 4 years. For comparison, dose rates calculated using the same rule of thumb in RPP-6711, Appendix T, for retrievals of C-103, C-106, C-108, C-109 had a maximum of 34 Rad/hr, while retrieval of S-112 calculated 365 Rad/hr. These two dose rates result in considerably longer exposure times, which are impractical. Due to space restrictions, cost concerns, and the desire for data after a reasonable time, a slightly higher dose rate is selected. However, this dose rate must not be so high as to introduce DLO effects in the unconstrained HIHTL hose. It is suspected that DLO effects are observed in field configurations due to limited access to oxygen replenishment from the constrained geometry. The exposure for the majority of testing is specified to be 1000 R/hr gamma. Assuming the energy absorption of EPDM is roughly similar to tissue, the specified dose rate is roughly 1000 Rad/hr. This rate of exposure results in an integrated dose of  $8.8 \times 10^6$  Rad after 1 year,  $1.31 \times 10^7$  Rad after 1.5 years, and  $1.75 \times 10^7$  Rad after 2 years.

To investigate the effects of dose rate and type, Phase 1 of testing includes testing gamma at 100 R/hr and 100 Rad/hr beta, the limited of feasible beta sources.

#### 2.7.5.3 Exposure

Recreating reasonable testing radiation conditions is largely dependent on geometry. Since using a radioactive simulant solution is not possible for this Test Plan, no configuration will be able to perfectly mimic radiation source geometry, which affects dose uniformity and dose deposition profiles.

Hanford waste transfers can be assumed to produce a uniform concentration of radionuclides. Any significant non-uniformity would be present due to a stratification of suspended solids, which would result in a significant disparity of <sup>90</sup>Sr concentration. Of tank waste layers that have been analyzed for <sup>90</sup>Sr particle size distributions, observed particle sizes in sludge have been less than 200 nm (PNNL-20646, *Hanford Waste Physical and Rheological Properties: Data and Gaps*), matching process knowledge that strontium's insolubility results in early precipitation of fine particles. Particles of this size will be easily suspended during transfers. Incorporation of these fine particles into aggregates could result in association with larger particle sizes; however, such aggregation would also provide a degree of shielding. Regardless, limitations in source selection have bounded the maximum beta dose rate available. The mineral nastrophite (NaSrPO<sub>4</sub>·9H<sub>2</sub>O) has been observed in saltcake layers as particles as large as 200 µm; however, saltcake wastes have significantly lower <sup>90</sup>Sr concentrations.

Line sources will be used to approximate a pipe or hose filled with an approximately uniform radionuclide concentration. This configuration provides the same 360° coverage and uniform dosage if placed in the center of the pipe or hose, for an isolated system. For beta irradiation, this configuration will be used, due to the penetration characteristics of beta exposure. Achieving a uniform field with beta sources using an array to irradiate multiple samples would be bulky and the dose deposition profile would vary drastically from the field condition it is intended to represent, with approximately zero dose on the inner surface of the component if an external array were used. In regards to beta irradiation, each hose can be treated as an isolated system with an internal source.

In contrast with beta irradiation, the penetration of gamma irradiation requires neighboring sources to be considered and permits having an external source produce an approximately uniform dose to all sides of a component. In order to maximize the radiation field generated from a gamma source, an annular vessel with the sources in the center will be deployed. This approach will minimize the number of sources required. If significant dosage differences are expected for the component facing towards the sources versus away from the sources, then a method of periodic rotations could be deployed to ensure an even cumulative dose. Using external gamma sources does result in an inverted dose deposition profile; however, to a much lesser degree than compared to beta irradiation.

# 3. PHASE 1: COMPARISON OF B AND $\Gamma$ IRRADIATION OF NONMETALLIC MATERIALS

## 3.1 Objectives and Strategy

The nonmetallic materials used in the Hanford tank farms are exposed to both  $\beta$  and  $\gamma$  radiation. The sources of  $\beta$  radiation are mainly <sup>90</sup>Sr and <sup>90</sup>Y, which are associated with the solids in the tanks. The sources of  $\gamma$  are mainly <sup>137</sup>Cs and <sup>137m</sup>Ba and are associated with the supernatant solutions.

It is not practical to use a  $\beta$  source dispersed in an aqueous or gaseous medium in direct contact with the insides of the hoses, Teflon gaskets, or EPDM O-rings for the  $\beta$ - $\gamma$  comparison. Rather, it is highly desirable that sealed  $\beta$  sources be used to avoid contamination of the irradiated nonmetallic materials. This will greatly simplify the postexposure mechanical testing to be used to quantify the effects of  $\beta$ ,  $\gamma$ , and simultaneous  $\beta$  and  $\gamma$  irradiation on these materials. However, the sealed  $\beta$  sources identified for this Test Plan can only be used at ambient laboratory temperatures and pressures, and only in the absence of caustic solutions simulating the supernatant tank solutions.

Therefore, the possible synergistic effects of simultaneous  $\beta$  and  $\gamma$  irradiation must be determined under ambient laboratory conditions without the simulated supernatant solution by comparing the effects of simultaneous  $\beta$  and  $\gamma$  irradiation to those from  $\gamma$  only (see below). This comparison will provide a " $\beta$  enhancement factor" or " $\beta$  factor" to reflect how much additional damage, if any, results from simultaneous irradiation with  $\beta$  and  $\gamma$  relative to that from  $\gamma$  only. This factor will be used to correct the results obtained from simultaneous exposure to  $\gamma$  irradiation and one or more additional stressors (elevated temperatures, simulated supernatant solutions, and elevated pressures). It is practical to combine  $\gamma$  irradiation and these stressors because the  $\gamma$  radiation from sealed <sup>60</sup>Co sources outside the stainless steel vessels will penetrate the vessels without contaminating the samples inside the vessels.

Figure 9 provides the baseline test matrix proposed for the  $\beta$ - $\gamma$  comparison. Comparisons of the results measured by postexposure mechanical testing after Paths 2 and 3 of Phase 1 will provide a " $\beta$  enhancement factor" or " $\beta$  factor" to quantify how much additional damage (if any) results from irradiation with both  $\beta$  and  $\gamma$  (Path 3) relative to that from  $\gamma$  only (Path 2). This factor will be used to correct the results obtained from Phases 2, 3, and 4 of this study, which will use  $\gamma$  only, to simulate the additional effects of  $\beta$ . The proposed use of the  $\beta$  factor is illustrated in Figure 2, the waterfall diagram (see Section 2.0).

Use of this  $\beta$  enhancement factor to correct the results from Phases 2, 3, and 4 will necessitate the assumption that this factor is identical for the conditions used for Phase 1 (the  $\beta$ - $\gamma$ comparison) and those used for Phases 2, 3, and 4 (effects of simultaneous exposure to multiple stressors). These other stressors include: (1) elevated temperatures (Phase 2); (2) elevated temperatures and the presence of a caustic solution to simulate the supernatant liquid in the tanks (Phase 3); and (3) elevated temperatures, the caustic solution, and elevated pressures (Phase 4). Therefore, use of the  $\beta$  factor to correct the results from Phases 2, 3, and 4 will require the assumption that this factor is independent of temperature, the caustic chemical conditions in the tanks, and pressure, up to temperatures of 200  $^{\circ}$ F and pressures up to 425 psig.

The proposed  $\beta$  irradiation rate of 100 R/hr is the maximum exposure rate achievable with commercially available line sources (see Subsection 3.3). However, this exposure rate is close to the reasonably conservative, bounding  $\beta$  irradiation rate provided by WRPS (see Subsection 2.3). WRPS has specified that the  $\gamma$ -irradiation rate for Phases 2, 3, and 4 of this study (see Section 4.0) will be 1000 R/hr (Subsection 2.3). Therefore, Path 1 of Phase 1 will be carried out at a  $\beta$  irradiation rate of 100 R/hr, Path 2.1 at a  $\gamma$  irradiation rate of 1000 R/hr, and Path 3.1 at a  $\beta$  irradiation rate of 100 R/hr and a  $\gamma$  irradiation rate of 1000 R/hr.

Paths 2.1 and 3.1 are the most important elements in the proposed baseline matrix (Figure 9), because comparison of the results of these tests will provide an estimate of the additional effects that would be observed if  $\beta$  irradiation were added to the results of Phases 2, 3, and 4. However, comparison of the sum of the effects of Paths 1 and Path 2.1 to those from Path 3.1 will reveal if simultaneous  $\beta$  and  $\gamma$  irradiation results in synergistic effects that cannot be predicted by adding the effects observed separately in Paths 1 and 2.1.

Furthermore, it will also be useful to conduct Path 2.2 at a  $\gamma$ -irradiation rate of 100 R/hr, and Path 3.2 at a  $\beta$  irradiation rate of 100 R/hr and a  $\gamma$  irradiation rate of 100 R/hr. Performing Paths 2.2 and 3.2 would reveal whether the  $\beta$  factor depends on the  $\gamma$  irradiation rate. This is potentially important because, although a  $\gamma$ -irradiation rate of 1000 R/hr is a reasonably conservative, bounding rate for the nonmetallic materials used in the tank farms, all of the observed  $\gamma$  irradiation rates are actually less than 1000 R/hr (see TFC-ENG-STD-34, Figure E-1). In fact, the mean of the estimated DST supernatant (<sup>137</sup>Cs and <sup>137m</sup>Ba)  $\gamma$  exposure rates is 161 rad/hr; the median is 133 rad/hr; and the modal interval of the histogram is < 100 rad/hr (TFC-ENG-STD-34, Figure E-1). Therefore, it would be prudent to ascertain if the  $\beta$  factor depends on the  $\gamma$  irradiation rate, as well as determining the effects of varying  $\gamma$  dose rate.

Finally, Path 4 is proposed as a control. Path 4 will quantify the effects (if any) of shipment of the samples of hoses and Teflon to SNL and storage of these materials under ambient conditions there. It is unlikely that these operations will affect the performance of these materials. However, inclusion of control samples would be relatively easy (no additional safety approvals would be required) and hence relatively inexpensive.



Figure 9. Baseline Test Matrix Proposed for the  $\beta$ - $\gamma$  Comparison.

#### 3.2 Facility, Test Vessels and Samples, and Test Conditions and Durations

Paths 1 and 4 of the  $\beta$ - $\gamma$  comparison, which will include a  $\beta$ -irradiation rate of 100 R/hr or no irradiation, respectively, will be carried out in a dry, shielded area of SNL's GIF outside the dry irradiation cells (probably in the high bay). The test vessels to be used for Path 4 (see below) will not — of course — require shielding, but will be co-located with those used for Path 1 to ensure that the ambient laboratory conditions for the controls are as similar as possible to those for Path 1. It would not be practical to place any control samples in the GIF pool because of the space limitations anticipated for this part of the facility. Paths 2.1, 2.2, 3.1, and 3.2 must be conducted in the GIF pool, because they will include <sup>60</sup>Co  $\gamma$  sources capable of irradiating the nonmetallic test materials at 100 or 1000 R/hr.

A total of 12 annular stainless steel vessels will be used to contain the specimens of nonmetallic materials for Phase 1. Each vessel will have an outer diameter (OD) of approximately 2.5 ft, a length of about 4 or 5 ft, and a central cavity with a diameter of about 1 ft that is open to the GIF pool. The  $\beta$ irradiations for Phase 1 will be carried out by inserting the sealed <sup>85</sup>Kr sources inside the primary hoses and other materials, and placing these materials inside the enclosed, annular portions of these containers that surround the central cavities. The  $\gamma$  irradiations for Phase 1 (and for Phases 2, 3 and 4) will be conducted by placing sealed <sup>60</sup>Co sources in the central cavities. The objective of using these vessels during Phase 1 will be to contain any radioactive gas ( $^{85}$ Kr) that might leak from the sealed  $\beta$  sources (see Subsection 3.3) during the tests. Although it is unlikely that the sealed  $\beta$  sources would leak, the use of leak-proof vessels will facilitate SNL's ES&H R&A process for Phase 1. The 12 vessels required for Phase 1 need only be leak-proof at the ambient temperatures and pressures of the GIF high bay (4 vessels) or GIF pool (8 vessels). Therefore, these vessels might not have to be certified for use at elevated temperatures and pressures, and for exposure to the simulated supernatant solutions, because Phase 1 will be carried out at ambient temperatures and pressures in the high bay of the GIF or in the GIF pool. However, SNL's ES&H R&A process will probably be easier to fulfill, and might even result in the requirement that the 8 vessels to be placed in the GIF pool, or even all 12 vessels to be used for Phase 1, must be identical to those used for Phases 2 through 4 (i.e., certified for use at elevated temperatures and pressures, and for exposure to the simulated supernatant solutions). Furthermore, six of the vessels used for Phase 1 could be reused for Phase 3 if these vessels were certified for elevated temperatures, for exposure to the chemical simulant, and for elevated pressures. This would obviate the need to purchase six vessels for these phases in addition to six of the leak-proof vessels for Phase 1. Therefore, it is assumed that the vessels to be used for Phase 1 will be identical to those used for Phases 2, 3, and 4.

Two vessels will be used for each path or subpath in Figure 9. One of these vessels will contain 12 primary hoses and 12 coupons of the composite hose materials. The other will hold 12 confined Teflon gaskets, 12 unconfined Teflon gaskets, and 12 Teflon coupons The six vessels to be used for hoses will have dimensions that are identical to the six to be used for the Teflon gaskets. Figures 10, 11, and 12 illustrate these vessels.

Four of these vessels will be placed in the GIF high bay (two for Path 1 and two for Path 4). The other eight will be placed in the GIF pool.



4 in diameter, 30 in long hose test specimens, with β sources in center of hose 100 R/hr gamma in central annulus



4 in diameter, 30 in long hose test specimens, with β sources in center of hose 1000 R/hr gamma in central annulus

4 in diameter, 30 in long hose test specimens 100 R/hr gamma in central annulus



Side view

(Not all hoses shown for ease of viewing.)



4 in diameter, 30 in long hose test specimens 1000 R/hr gamma in central annulus

#### Figure 10. Schematic Diagrams of the Vessels to Be Used for the Hose Materials. Legend in Figure 12.

All 12 of these vessels to be used for Phase 1 will be allowed to equilibrate with the ambient temperatures and pressures in the GIF high bay or pool. The temperatures and pressures expected in the high bay are typical of those described as "ambient laboratory conditions," except that ambient laboratory pressures at the elevation of the GIF (a little over 5,000-ft above mean seal level) is a little over 80% of the average pressure at sea level (1013 mbar). The temperatures and pressures expected in the pool, however, will be somewhat greater than ambient laboratory conditions, because: (1)  $\gamma$  irradiation of the vessels might increase the temperature of the water in the pool somewhat, and (2) the vessels used for Paths 2.1, 2.2, 3.1, and 3.2 of Phase 1 (and for Phases 2, 3, and 4) will be placed at the bottom of the 18-ft deep pool to achieve the maximum shielding available from the water in the pool.

For Paths 1, 2.1, and 3.1, in which the  $\beta$  and/or  $\gamma$  exposure rates will both be 100 R/hr, the samples will be irradiated for periods of 6, 12, 18, and 24 months. This will correspond to total exposures of  $4.38 \times 10^5$ ,  $8.77 \times 10^5$ ,  $1.31 \times 10^6$ , and  $1.75 \times 10^6$  R, respectively. Three of the 12 primary hoses and 3 of the 12 coupons of the composite hose materials; and 3 of the 12 confined Teflon gaskets, 3 of the 12 unconfined Teflon gaskets, and 3 of the 12 Teflon coupons will be removed after each of these irradiation periods or total exposures. This will provide triplicate samples of each of these materials for postexposure mechanical testing after each irradiation period or exposure.



~6 in diameter Teflon gasket test specimen confinement fitting, with β sources in center 100 R/hr gamma in central annulus



~6 in diameter Teflon gasket test specimen confinement fitting, with β sources in center 1000 R/hr gamma in central annulus



-6 in diameter Teflon gasket test specimen confinement fitting, 100 R/hr gamma in central annulus



(Not all gaskets shown for ease of viewing.)

~6 in diameter Teflon gasket test specimen confinement fitting, 1000 R/hr gamma in central annulus

#### Figure 11. Schematic Diagrams of the Vessels to Be Used for the Teflon Gaskets. Legend in Figure 12.

For Paths 2.2 and 3.2, in which the  $\beta$  exposure rate will be 100 R/hr and the  $\gamma$  exposure rate will be 1000 R/hr, the samples will be irradiated to total  $\gamma$  exposures of approximately 10<sup>5</sup>, 10<sup>6</sup>, and 10<sup>7</sup> R. According to TFC-ENG-STD-34, Appendix B, Table B.1, these exposures correspond approximately to the "normal, reasonably bounding conditions for [the] selection of nonmetallic materials, [in particular, the] total absorbed radiation dose source term:

- For nonmetallic materials subject to continuous immersion in DST supernatant,  $1.1 \times 10^7$  Rad
- For nonmetallic materials in equipment such as valves and piping, subject to infrequent exposure to DST supernatant waste,  $1.3 \times 10^6$  Rad
- For nonmetallic materials in readily accessible, self-shielded equipment arrangements, such as above-ground pits,  $1.3 \times 10^5$  Rad"

An additional total  $\gamma$  exposure of > 10<sup>7</sup> R will also be included as an overtest condition. The results from this total  $\gamma$  exposure of > 10<sup>7</sup> R will provide information as to whether these nonmetallic materials can successfully sustain total exposures higher than the maximum expected for nonmetallic materials subject to continuous immersion in the DST supernatant solutions.

As in the case of Paths 1, 2.1, and 3.1, 3 of the 12 samples of each type of nonmetallic material will be removed after total  $\gamma$  exposures of approximately  $10^5$ ,  $10^6$ ,  $10^7$ , and  $> 10^7$  R, which will provide triplicate samples of each of these materials for postexposure testing.

$\bigcirc \bigcirc$	Teflon gasket and hose test specimens, remove at $4.38 \times 10^5 \text{ R}$ total exposure	(6 months)
$\bigcirc \bigcirc$	Teflon gasket and hose test specimens, remove at 8.77 x $10^5$ R total exposure	(12 months)
$\bigcirc \bigcirc$	Teflon gasket and hose test specimens, remove at $1.31 \times 10^{6}$ R total exposure	(18 months)
$\bigcirc \bigcirc$	Teflon gasket and hose test specimens, remove at $1.75 \ge 10^6$ R total exposure	(24 months)
	1000 R/hr	
$\bigcirc$	Teflon gasket and hose test specimens, remove at $4.38 \times 10^5 R$ total exposure	(6 months)
	Teflon gasket and hose test specimens, remove at $8.77 \times 10^5 R$ total exposure	(12 months)
	Teflon gasket and hose test specimens, remove at $1.31 \times 10^6$ R total exposure	(18 months)
$\bigcirc \bigcirc$	Teflon gasket and hose test specimens, remove at $1.75 \times 10^6$ R total exposure	(24 months)

100 R/hr

#### Figure 12. Legend for Figures 10 and 11.

#### 3.3 $\beta$ and $\gamma$ Sources

Sealed  $\beta$  line sources containing <sup>85</sup>Kr will be used for Phase 1 (the  $\beta$ - $\gamma$  comparison). Each source will consist of an approximately 10-in long,0.25-in. OD, 0.006-in. thick, aluminum-1100 or 3003 tube with 7.5 mCi/in of gaseous <sup>85</sup>Kr in stable gaseous Kr at a total pressure slightly below atmospheric. The half-life of <sup>85</sup>Kr is 10.73 yr. It emits a  $\beta$  particle with an end-point energy of 0.897 MeV and a 0.514 MeV  $\gamma$  ray, and decays to stable <sup>85</sup>Rb.

These sealed <sup>85</sup>Kr line sources will be placed inside the 30-in long, 2-in nominal ID primary hoses and the confined and unconfined Teflon gaskets. Spacers (washers) will be used to align the line sources close to the centerlines of the hoses. These sources will produce exposure rates of about 100 R/hr at a distance of 1 in from these sources (i.e., on the insides of the hoses), based on information provided by the vendor. This is about 50% of the estimate of the reasonably conservative, bounding  $\beta$  exposure rate provided by WRPS (see Subsection 2.3). The flat hose and Teflon coupons will be bent around other sealed <sup>85</sup>Kr line sources. The sealed <sup>85</sup>Kr line sources could be irradiated with 100 or 1000 R/hr of  $\gamma$  irradiation for Path 3 (see Figure 9), because they do not contain Mylar®<sup>9</sup> windows.

Other possible  $\beta$  sources were considered before selecting the sealed <sup>85</sup>Kr line sources described above. Sealed <sup>90</sup>Sr  $\beta$  point sources are available, for example. These point sources consist of right-circularcylindrical pellets with a diameter of about 0.25 in and a height of about 0.3 in, with a 0.210-in diameter ceramic disc containing the <sup>90</sup>Sr. The sources are covered with 0.002-in thick pieces of stainless steel. These sources could also be irradiated with 1000 R/hr of  $\gamma$  radiation, because they do not contain Mylar windows. Furthermore, these sources would produce a maximum  $\beta$  dose of about 1,900 R/hr at a distance of 1 in from the source, based on information from the vendor. This would be more than

<sup>&</sup>lt;sup>9</sup> Mylar is a registered trademark of DuPont Teijin Films, Wilmington, Delaware.

adequate to achieve the reasonably conservative, bounding exposure rate provided by WRPS. (See Subsection 2.3.) However, such a point source or sources could not be used to uniformly irradiate the insides of the hoses. This is because the radiation emitted by the source would be confined to a narrow beam or conical region, and it would not be practical to irradiate hoses or Teflon gaskets in their in-service configurations.

Electron-beam (e-beam) irradiation of the materials specified for this study was also considered. Buckalew et al. (1984) and Buckalew (1989) used a Pelletron-Raster system, an electrostatic particle accelerator similar to a Van de Graaff generator with scanning capability, to compare the effects of  $\beta$ and  $\gamma$  irradiation of coupons of nonmetallic materials at Sandia. Buckalew's Pelletron-Raster system could not be located, nor could anyone currently employed at Sandia who is familiar with this equipment or its use. A vendor was identified, but it was decided not to propose reestablishing this capability at Sandia because: (1) it would not be practical to irradiate hoses or Teflon gaskets in their inservice configurations (Buckalew carried out his studies with coupons only), and (2) commercially available Pelletron-Raster systems cost in excess of \$500,000.

Sealed <sup>60</sup>Co source pins will be used for the  $\gamma$  irradiations in Phase 1 (and for those in Phases 2, 3, and 4). Each of these 18-in long source pins will contain a total activity of 1000 Ci of <sup>60</sup>Co. This is the lowest activity of <sup>60</sup>Co that the vendor can provide. The preliminary test configuration calculations described in Subsection 4.4 suggest that a total activity of 100 Ci of <sup>60</sup>Co will be required in each source pin to produce an exposure rate of 1000 rad/hr on the inner sides of the nonmetallic materials in the enclosed regions surrounding the inner cavities of the test vessels. Therefore, shielding sleeves containing different thicknesses of various metals will be used to reduce the effective loading of the <sup>60</sup>Co source pins from 1000 to 100 Ci. These sleeves will be replaced periodically with less dense or thinner sleeves to maintain an essentially constant exposure rate of 1000 R/hr on the inner sides of the test which SNL's Non-Reactor Nuclear Facilities Dept. 1387 personnel typically purchase  $\gamma$  sources for use in the GIF. All of the <sup>60</sup>Co source pins to be used in the irradiations of nonmetallic materials for this study will have to be purchased because (1) Dept. 1387 personnel are currently using all of their <sup>60</sup>Co source pins, and (2) they typically use <sup>60</sup>Co source pins with a total activity of 10,000 Ci.

## 3.4 Test Configuration

Subsection 4.4 describes the test configuration for Phase 1 (the  $\beta$ - $\gamma$  comparison) along with those for Phases 2, 3, and 4 (effects of simultaneous exposure to multiple stressors). The configuration for Phase 1 is discussed along with those for Phases 2, 3, and 4, because eight of the vessels used for Phase 1 would be placed in the GIF pool essentially simultaneously with the six for Phase 2 in the baseline test program. The reduced cost alternative does not include Phase 1 testing. All phases will utilize similar temperature and air flow configurations.

## 3.5 Test Control and Monitoring

Subsection 4.5 describes the test control and monitoring system that will be designed and deployed for all 30 test configurations of 18 test vessels in the proposed baseline test program. This description includes the control and monitoring to be carried out for Phase 1 (the  $\beta$ - $\gamma$  comparison).

## 4. PHASES 2, 3, AND 4: QUANTIFICATION OF SYNERGISTIC EFFECTS OF SIMULTANEOUS EXPOSURE OF NONMETALLIC MATERIALS TO MULTIPLE STRESSORS

#### 4.1 Objectives and Strategy

The nonmetallic materials used in the Hanford tank farms (see Subsection 2.2) are simultaneously exposed to four stressors: (1) radiation ( $\beta$  and  $\gamma$ ), (2) elevated temperatures, (3) a nonradioactive, caustic (high pH), supernatant HLW simulant, and (4) elevated pressures.

It is not possible to use the sealed  $\beta$  line sources (see Subsection 3.3) in experiments at elevated pressures and temperatures, or in the presence of simulated supernatant solutions. Therefore, the effects of simultaneous exposure of nonmetallic materials to  $\gamma$  irradiation, elevated temperatures, simulated supernatant solutions, and elevated pressures will be quantified. Then, the  $\beta$  enhancement factor or  $\beta$  factor obtained from Phase 1 of this study (the  $\beta$ - $\gamma$  comparison) will be used to include the additional effects (if any) of  $\beta$  irradiation. Subsection 3.1 describes the establishment of the  $\beta$  factor in detail.

The proposed baseline test program (see Figure 2 and Table 1) is repeated here for ease of reference. The baseline program would include four phases. Phase 1 (see Section 3.0) would compare the effects of simultaneous  $\beta$  and  $\gamma$  irradiation of hoses and Teflon gaskets to those from  $\gamma$  only. Phase 2 would determine the effects of simultaneous exposure of hoses, Teflon gaskets, and EPDM O-rings to  $\gamma$  irradiation and elevated temperatures. Phase 3 would investigate the effects of simultaneous exposure of the same materials used in Phase 2 to  $\gamma$  irradiation, elevated temperatures, and a nonradioactive, simulated supernatant solution. Finally, Phase 4 would quantify the effects of simultaneous exposure of the same materials used in Phases 2 and 3 to  $\gamma$  irradiation, elevated temperatures, the chemical simulant, and elevated pressures.

A reduced cost alternative test program is also considered: it would eliminate Phase 1 tests, carry out the remaining three phases with the hoses only, and carry out only Phase 3 tests with the Teflon gaskets and EPDM O-rings. Section 7.1 of this Test Plan describes this reduced cost alternative in detail.

## 4.2 Facility, Test Vessels and Samples, and Test Conditions and Durations

Phases 2, 3, and 4 will be carried out in the GIF pool. The vessels for Phases 2, 3, and 4 will be certified for use at elevated temperatures and pressures, and for exposure to the simulated supernatant solutions. All of the vessels will be annular with an OD of approximately 2.5 ft, a length of about 4 or 5 ft, and a central cavity with a diameter of about 1 ft that is open to the GIF pool. Figures 13, 14, and 15 (see next three pages) show these vessels.

Six vessels will be used for each of Phases 2, 3, and 4. Three of these vessels will contain 12 primary hoses and 12 coupons of the composite hose materials. The other three will hold 12 confined Teflon gaskets, 12 unconfined Teflon gaskets, and 12 Teflon coupons; and 12 confined EPDM O-rings and 12 unconfined EPDM O-rings and 12 EPDM coupons.

For Phase 2, the three vessels with the hoses will be maintained at a temperature of 70, 130, or 180 °F and ambient (GIF-pool) pressures without the simulated supernatant solution. The three vessels with the Teflon gaskets and EPDM O-rings will be maintained at 70, 130, or 200 °F and ambient pressure without the chemical simulant. (As explained in Subsection 3.2, these pressures will be somewhat higher than atmospheric at the elevation of the GIF, because the vessels will be placed at the bottom of the 18-ft deep pool.)

For Phase 3, the three vessels with the hoses will be maintained at a temperature of 70, 130, or 180  $^{\circ}$ F and ambient (GIF pool) pressures, and will be filled or nearly filled with the simulated supernatant solution. The three vessels with the Teflon gaskets and EPDM O-rings will be maintained at a 70, 130, or 200  $^{\circ}$ F and ambient pressure with the caustic chemical simulant.

In the case of Phase 4, the vessels with the hoses will be maintained at a temperature of 70, 130, or 180 °F and a pressure of 425 psig with the simulated supernatant solution. The vessels with the Teflon gaskets and EPDM O-rings will be maintained at a 70, 130, or 200 °F and a pressure of 400 psig with the chemical simulant.

For Phases 2 through 4, the  $\gamma$  exposure rate will be 1000 R/hr (see Subsection 2.3). The samples will be irradiated to total  $\gamma$  exposures of approximately 10<sup>5</sup>, 10<sup>6</sup>, 10<sup>7</sup>, and > 10<sup>7</sup> R. Three of the 12 primary hoses and 3 of the 12 coupons of the composite hose materials will be removed after each of these total exposures. Similarly, 3 of the 12 confined Teflon gaskets, 3 of the 12 unconfined Teflon gaskets, and 3 of the 12 Teflon coupons; and 3 of the 12 confined EPDM O-rings and (if present) 3 of the 12 unconfined EPDM O-rings and 3 of the 12 EPDM coupons will be removed after each of these total exposures. This will provide triplicate samples of each of these materials for postexposure mechanical testing after each total exposure.



4 in diameter, 30 in long hose test specimens 1000 R/hr gamma in central annulus



Side view

(Not all hoses shown for ease of viewing.)

Figure 13. Schematic Diagrams of the Vessels to Be Used for the Hose Materials. Legend in Figure 15.



 $\sim$  6 in diameter gasket test specimen confinement fitting, and  $\sim$  4 in diameter O-rings and fitting test specimens, 1000 R/hr in central annulus



(Not all gaskets & O-ring stacks shown for ease of viewing.)

## Figure 14. Schematic Diagrams of the Vessels Be Used for the Teflon Gaskets and EPDM O-Rings. Legend in Figure 15.

#### 1000 R/hr

Teflon gasket, O-ring, and hose test specimens, remove at 10<sup>5</sup> R total exposure
 Teflon gasket, O-ring, and hose test specimens, remove at 10<sup>6</sup> R total exposure
 Teflon gasket, O-ring, and hose test specimens, remove at 10<sup>7</sup> R total exposure
 Teflon gasket, O-ring, and hose test specimens, remove at >10<sup>7</sup> R total exposure

Figure 15. Legend for Figures 13, 14, 18, 19, 20, and 21.



Figure 16. Schematic Diagram of Two Staggered <sup>60</sup>Co Source Pins.

## 4.3 $\gamma$ Sources

Subsection 3.3 describes the sealed <sup>60</sup>Co source pins will be used for the  $\gamma$  irradiations in Phase 1 (and for those in Phases 2, 3, and 4). Each of these 18-in long source pins will contain a total activity of 1000 Ci of <sup>60</sup>Co, which is the lowest activity of <sup>60</sup>Co that the vendor can provide. A total activity of 100 Ci of <sup>60</sup>Co will be required in each source pin to produce an exposure rate of 1000 rad/hr on the inner sides of the nonmetallic materials in the enclosed regions surrounding the inner cavities of the test vessels (see Subsection 4.4). Therefore, shielding sleeves will be used to reduce the effective loading of the <sup>60</sup>Co source pins from 1000 to 100 Ci. These sleeves will be removed or replaced periodically (see below) to maintain an exposure rate of ~1000 R/hr on the test materials as the <sup>60</sup>Co decays.

## 4.4 Test Configuration

This subsection describes the test configuration for Phase 1 (the  $\beta$ - $\gamma$  comparison) along with those for Phases 2, 3, and 4 (effects of simultaneous exposure to multiple stressors) of the baseline test program. The configurations for all four phases are discussed herein because eight of the vessels used for Phase 1 will be placed in the GIF pool essentially simultaneously with the six for Phase 2 in the baseline test program. The reduced cost alternative will allow placement of Phases 2, 3, and 4 simultaneously.

Preliminary test configuration calculations were carried out with the TIGER codes (Franke et al., 2009) to establish the required activity of each sealed <sup>60</sup>Co source pin and the total number of source pins needed to maintain a y exposure rate of 1000 R/hr for the nonmetallic materials proposed for this study. Figure 16 illustrates the test configuration used for the preliminary calculations. A staggered configuration will be used for ease of source handling. Furthermore, staggering the <sup>60</sup>Co source pins allows the active portions of each pin to be aligned with the inactive portion of the other pins, thereby avoiding  $\gamma$ -free regions in the cavities. Figure 16 shows two staggered <sup>60</sup>Co source pins in the cavity of an annular vessel. The results of these preliminary calculations are that: (1) a total activity of 100 Ci of <sup>60</sup>Co will be required in each source pin to produce an exposure rate of 1000 rad/hr on the inner sides of the hoses, and (2) three 18-in long <sup>60</sup>Co source pins would be preferable to two in order to prevent decreases in the exposure rates at the ends of the hoses. The lowest-activity <sup>60</sup>Co source pins that the vendor can provide have a total activity of 1000 Ci. Therefore, shielding sleeves containing different thicknesses of various metals will be used to reduce the effective loading of the <sup>60</sup>Co source pins from 1000 to 100 Ci. These sleeves will be removed or replaced periodically with less dense or thinner sleeves to maintain an exposure rate as close as possible to 1000 R/hr on the nonmetallic materials as the <sup>60</sup>Co decays. Staggering the source pins will allow independent removal or replacement of the shielding sleeves (i.e., removal or replacement of the shielding sleeves from one of the source pins without having to remove one or both of the other source pins to gain access).

Additional test configuration calculations with the TIGER codes will be necessary prior to the start of any irradiation testing. These follow-on calculations will produce final test configurations with more definitive requirements for the numbers and placement of the sealed <sup>60</sup>Co source pins, definitions of the requirements for the densities and thicknesses of the shielding sleeves, how closely the vessels can be spaced in the GIF pool, etc. These follow-on calculations could in turn result in adjustments of the cost of these sources. These calculations could be performed during the preparations for the baseline program from October 2013 through September 2014.

Pending the results of the follow-on test configuration calculations described above, a conceptual test configuration for the baseline program is proposed based on the results of the preliminary calculations. Figure 17 illustrates the GIF pool, with all dimensions given.

The aqua-colored areas of the pool (Figure 17) are the portions that could potentially be used for the 26 of the 30 vessels that will be exposed to  $\gamma$  irradiation in the baseline test program. These vessels will include those in Paths 2.1, 2.2, 3.1, and 3.2 of Phase 1; and all of the vessels in Phases 2, 3, and 4 of the baseline program. The gray area of the pool (Figure 17) is the approximately  $4 \times 7$ -ft cask set-down area. This area cannot be used for testing.

Figure 18 provides our proposed, conceptual configuration for the deployment of Phases 1 and 2 in the GIF pool. The eight-vessel array proposed for Phase 1 appears to fit in the approximately  $7 \times 13$ -ft testing area to the right (south) of the cask set-down area, pending the results of the test configuration calculations described above. Figure 19 shows our conceptual configuration for the deployment of Phases 3 and 4 in the pool. The six vessels that will be used for Phases 2 and 4 are situated along the bottom and side of the sideways U-shaped area that begins at the upper-left part of the cask set-down area in Figures 18 and 19. Figure 20 and 21 show enlargements of the eight-vessel array proposed for Path 1, and the six-vessel array for Path 3.

## 4.5 Test Control and Monitoring

A test control and monitoring system will be developed and deployed for all 18 or 12 of the vessels, respectively, in the proposed baseline test program or the reduced cost option. Table 6 (which appears after Figures 17, 18, and 19) shows the test control and monitoring to be applied to each of the four phases of this study. The test control and monitoring system will be designed to be as similar as possible for all vessels to be deployed in Phases 1, 2, 3, and 4.

One or more of the following test variables will be controlled in all four phases of the baseline program or the reduced cost option: (1) the  $\beta$  and/or  $\gamma$  exposure rates, (2) temperature; (3) pressure, (4) the flow rate(s) of gas (compressed air) through the vessels and thus the concentrations of O<sub>2</sub> in the vessel atmospheres.

The values of the following test variables will be monitored and recorded for all of these vessels: (1) total  $\beta$  and/or  $\gamma$  exposure (by radiation dosimetry in the vessels), (2) temperatures (with thermocouples in the vessels), (3) pressures (with pressure transducers in the vessels), (4) the flow rate(s) of compressed air in the effluent from the vessels, and (5) the concentrations of O<sub>2</sub> in the effluent.

Thermocouples, pressure transducers, and  $\beta$  and  $\gamma$  radiation dosimeters suitable for the exposure rates defined for this Test Plan will be placed inside each of the vessels. Although the thermocouples and pressure transducers will be sheathed with stainless steel or other suitable alloys, they will probably have to be replaced, perhaps each time that samples of nonmetallic materials are removed for postexposure mechanical properties testing.

Continuous flow of compressed air through each of the vessels will be necessary to ensure that  $O_2$  is present throughout these tests. Maintaining oxic conditions in the vessels will replicate in-service conditions, but might not address diffusion-limited oxidation (Burnay and Hitchon, 1985; Gillen and Clough, 1989; Wise et al., 1997; Gillen et al., 2003), which could bias the results in favor of reduced damage. Oxic conditions could also be representative of those in the primary hoses used in the Hanford tank farms. This is because air accumulates in these hoses after each use, and is probably present for at least some of time that supernatant solutions and suspended solids are pumped through the hoses. Therefore, oxic conditions are certainly a relevant end-member condition when the insides of these hoses are exposed to  $\beta$  from <sup>90</sup>Sr and <sup>90</sup>Y associated with the suspended solids, and  $\gamma$  from <sup>137</sup>Cs and <sup>137m</sup>Ba associated with the supernatant solutions. Furthermore, oxic conditions may persist during much of the time that solutions and solids are pumped through the hoses. Thus, if anoxic conditions develop and persist in the tests proposed in this Test Plan, the results could be criticized because the test conditions were nonconservative.

Compressed air is inexpensive relative to other inert carrier gases that could be used to maintain atmospheric  $O_2$  concentration. However, compressed air might be inappropriate, or might have to be pretreated to remove  $CO_2$ , during Phases 3 and 4. This is because the  $CO_2$  in the compressed air would react with the caustic simulated supernatant solutions to be used in Phase 3. The composition of the simulant should provide sufficient buffering given the regular replenishment of the simulant.



Figure 17. Schematic Diagram of the GIF Pool. See text for explanation of colors.



Figure 18. Schematic Conceptual Configuration for the Deployment of Phases 1 and 2 in the GIF Pool. Legend in Figure 15.



Figure 19. Schematic Conceptual Configuration for the Deployment of Phases 3 and 4 in the GIF Pool. Legend in Figure 15.



Figure 20. Schematic of the Eight-Vessel Area Proposed for Phase 1. Legend in Figure 15.

1000 R/hr		1000 R/hr
gamma	1000 R/hr	gamma
70 °F	gamma	200 °F
chemical	130 °F	chemical
simulant	chemical	simulant
gaskets &	simulant	gaskets &
O-rings	hoses	O-rings
Phase 3	Phase 3	Phase 3



1000 IV/III	1000 10/11	1000 IQ/III	
gamma	gamma	gamma	
70 °F	130 °F	180 °F	
chemical	chemical	chemical	
simulant	simulant	simulant	1 ft
hoses	gaskets &	hoses	
Phase 3	O-rings	Phase 3	
	Phase 3		

Figure 21. Schematic of the Six-Vessel Area Proposed for Phases 2, 3, and 4. Legend in Figure 15.

Test	Radiation Temperature		Pressure	Compressed Air	Remarks	
Phase				Flow Rate &		
				O <sub>2</sub> Concentration		
1	Control: $\beta = 0 \text{ or } 100 \text{ R/hr};$ $\gamma = 0, 100, 1000 \text{ R/hr}$ Monitor w dosimetry	Equilibrate w ambient conditions in GIF high bay or pool Monitor w thermocouples in vessels	Equilibrate w ambient conditions in GIF high bay or pool Monitor w P transducers in vessels	Control flow rate so that $O_2$ concentration in effluent is at or close to atmospheric Monitor effluent w flow meters & $O_2$ sensors	Ambient T & P in the GIF pool will both be somewhat higher than ambient T & P in the GIF high bay.	
2	Control: $\gamma = 1000$ R/hr Monitor w dosimetry	Control: $T = 70, 130, \& 180 \degree F$ (hoses & hose coupons) $T = 70, 130, \& 200 \degree F$ (Teflon gaskets & EPDM O-rings Monitor w thermocouples in vessels	Equilibrate w ambient conditions in GIF pool Monitor w P transducers in vessels	Control flow rate so that $O_2$ concentration in effluent is at or close to atmospheric Monitor effluent w flow meters & $O_2$ sensors	Ambient P in the GIF pool will be somewhat higher than that typical of ambient laboratory conditions.	
3	Control: $\gamma = 1000$ R/hr Monitor w dosimetry	Control: $T = 70, 130, \& 180 \degree F$ (hoses & hose coupons) $T = 70, 130, \& 200 \degree F$ (Teflon gaskets & EPDM O-rings Monitor w thermocouples in vessels	Equilibrate w ambient conditions in GIF pool Monitor w P transducers in vessels	Control flow rate so that $O_2$ concentration in effluent is at or close to atmospheric Monitor effluent w flow meters & $O_2$ sensors	<ul><li>Ambient P in the GIF pool will be somewhat higher than that typical of ambient laboratory conditions.</li><li>Chemical simulant will be replenished on a regular basis.</li></ul>	
4	Control: γ = 1000R/hr Monitor w dosimetry	Control: $T = 70, 130, \& 180 \degree F$ (hoses & hose coupons) $T = 70, 130, \& 200 \degree F$ (Teflon gaskets & EPDM O-rings Monitor w thermocouples in vessels	Control: P = 425 psig (hoses & hose coupons) P = 400 psig (Teflon gaskets & EPDM O-rings Monitor w P transducers in vessels	Control flow rate so that $O_2$ concentration in effluent is at or close to atmospheric Monitor effluent w flow meters & $O_2$ sensors	Chemical simulant will be replenished on a regular basis.	

Table 6. Test Control and Monitoring for the Irradiation of Nonmetallic Materials	. (2 sheets)

#### Table 6. Test Control and Monitoring for the Irradiation of Nonmetallic Materials. (2 sheets)

Notes:

 $\beta$  = beta radiation from sealed <sup>85</sup>Kr sources

EPDM = ethylene propylene diene monomer

F = Fahrenheit

 $\gamma = \gamma$  radiation from sealed <sup>60</sup>Co or <sup>137</sup>Cs sources

Phase 1:  $\beta$ - $\gamma$  comparison at ambient high-bay or GIF-pool T & P, no chemical simulant.  $\beta = 100$  R/hr;  $\gamma = 100$  or 1000/R,

Phase 2: Effects of  $\gamma$  & elevated T without chemical simulant at ambient GIF-pool P.  $\gamma = 1000$  R/hr. Remove samples at  $10^5$ ,  $10^6$ ,  $10^7$ ,  $\& > 10^7$  R (exact durations TBD)

Phase 3: Effects of  $\gamma$ , elevated T, & chemical simulant at ambient GIF-pool P.  $\gamma = 1000 \text{ R/hr}$ . Remove samples at  $10^5$ ,  $10^6$ ,  $10^7$ , & >  $10^7 \text{ R}$  (exact durations TBD)

Phase 4: Effects of  $\gamma$ , elevated P & T, & chemical simulant.  $\gamma = 1000 \text{ R/hr}$ . Remove samples at  $10^5$ ,  $10^6$ ,  $10^7$ ,  $\& > 10^7 \text{ R}$  (exact durations TBD)

psig = pounds per square in (gauge)

R&A = review and approval

P = pressure

T = temperature

w = with

Compressors are less reliable than compressed air tanks, so tanks of compressed air will be used instead of a compressor to maintain a slow flow rate and  $O_2$  concentrations at or close to atmospheric in the vessels. The initial pressure of air tanks (~10,000-20,000 psig) is more than sufficient to maintain flow through the vessels, and will also be used, along with a back-pressure regulator, to pressurize the vessels for Phase 4. Flow rates will be set as low as reasonably achievable consistent with maintaining  $O_2$  concentrations at or close to atmospheric to minimize the number of the compressed air tanks required for these tests.

Flow meters and  $O_2$  sensors are planned for each of the vessels to be placed in the GIF high bay or in the GIF pool. A maximum of 18 flow meters and  $O_2$  sensors (plus a few extra in case of malfunctions) in the baseline test program, or 12 flow meters and  $O_2$  sensors (plus a few extra) for the reduced cost option would be required. (These are the maximum number of vessels to be placed into test at one time; see Subsection 7.1.) The other option would be to use a switching system that would allow one or a few pairs of flow meters and  $O_2$  sensors to monitor the effluent from all of the vessels. However, the use of a dedicated flow meter and  $O_2$  sensor for each vessel would enable continuous and more reliable monitoring of these parameters.

The flow meters and  $O_2$  sensors will be placed on the exhaust outlet outside of the GIF pool, to prevent exposure to radiation. Oxygen sensors that use a laser diode oxygen analyzer, such as those manufactured by Oxigraf, are robust and would thus be suitable for long-term, continuous monitoring of effluent  $O_2$  concentrations, because they do not use any consumable components that would require replacement or replenishment during the tests.

All of these control and monitoring instruments will be calibrated according to the QA requirements that are being developed for this study (see Section 6.0).

The temperatures and pressures in the vessels, the flow rate(s) of effluent compressed air, and the concentration of  $O_2$  in the effluent will be monitored essentially continuously (e.g., every 10 minutes or so) by a data acquisition system assembled from commercially available components. The data will be continuously recorded and saved, and archived on duplicate backup systems. The objective is to make these data retrievable online to authorized SNL personnel in Technical Areas 1 and 5, and to WRPS personnel at the Hanford Site in Washington, both via password-controlled access. Furthermore, password-restricted control of the test conditions to the SNL technologist(s) in overall charge of the tests will also be established. However, any and all remote access to the data acquisition system and remote control of the test system must be compatible with the ES&H and security requirements established by SNL for the GIF.

## 5. POSTEXPOSURE MECHANICAL TESTING

Postexposure testing will be based primarily on performance testing of the specific components (e.g., burst testing of hose specimens and leak testing of PUREX connector gaskets. This will provide a direct indication of component damage and impairment of function from the test stressors. In addition to the performance testing, material coupons will be subject to selected materials properties testing. Although more abstract, the changes in material properties will allow comparison to other historical materials testing and relative comparisons within this Test Plan of the effects of various stress factors.

Specific detailed postexposure performance testing procedures will be developed and will follow ASTM practice to the extent possible, given the specialized performance testing application. The coupon material testing will follow ASTM procedures where applicable. Testing will occur onsite at Sandia facilities shortly after removal from the irradiation cell as reasonably possible to minimize changes in the postexposure time period.

## 5.1 Hose Burst Testing

A specific hose testing procedure will be developed and approved before testing. The hose burst testing procedure will follow the Riverbend test procedure and guidelines in ASTM D380, *Standard Test Methods for Rubber Hose*, as applicable.

The size of the 30-in samples used for the burst test will comply with ASTM D380, Section 5.1.3, "Straight Burst—For hose nominal 76 mm (3 in.) and smaller, 450 mm (18 in.)"; using a specimen 3 ft in length, or twice the minimum. The use of straight versus bent burst configurations is selected to be comparable to existing hose qualification. Burst tests will be performed with water at ambient temperature.

The burst test procedure will identify set-up; indicate how the system was displaced of air and identify how pressure was introduced; and reference applicable photos, drawings, and documents. The hose end-fittings will be blanked and equipped with test ports on each end to allow for pressure line measurement and purging of trapped air. A calibrated gauge will be plumbed in the pressure line to monitor pressure throughout the test. Water will be introduced to the system to displace all air. The pressure will be incrementally raised until failure. The ultimate pressure and type of failure will be recorded (e.g., hose split, termination failure, rupture, ply separation, etc.). Any visible deformation or leakage is typically classified as failure as good practice. Figure 22 shows similar burst tests on hose coupons.

A minimum specified burst pressure of 1700 psig, or 4 times design would be considered successful, but coupons will be taken to failure to understand allowable margin in the exposed coupons.



Hydro-testing of HIHTL coupons after crush test



Typical hose failure near swaged end fitting

#### Figure 22. Previous Burst Testing of HIHTL Hoses.

## 5.2 PUREX Connector Gasket Testing

A specialized leak testing procedure will be developed that tests the ability of the connector gaskets to form an adequate seal. Each gasket will be exposed and tested while in the retainer, to ensure representative conditions and prevent damage solely from removal/reinsertion into the retaining ring. A fixture similar to the one shown in Figure 23 is anticipated, which was used for hydro-testing a specialized connector component. A fixture consisting of a connector nozzle and blanked connector head will be constructed. The fixture will be equipped with test ports for application of the leak test fluid, pressure measurement, and a flow meter or other means to measure leak rate. Field criterion for suitable PUREX connector connection is no visible leakage, which will likely be used as a test criterion.

After exposure, the gasket/retainer assembly will be inserted into the test blank connector. The connector will be placed onto the test nozzle and the connector torqued to normal design torque (*For a 2 inch connector, torque to minimum 70 ft-lbs, maximum 150 ft-lbs; torque may be increased to 300 ft-lbs to provide sealing in the event of connector leaks*, Note 6 from connector design document H-2-32420, Sheet 3, *Assembly, Horizontal and Vertical 2" Connector*). The test fixture will be pressurized to the design pressure (400 psig), and the connection inspected for leaks. If leaks are seen, the leak rate will be measured. The torque will then be gradually increased until the leak rate stops. If the leak cannot be stopped, the connector can be loosened and retightened to attempt to get a better seal. Leak rates at test pressures in excess of the design pressure (1.5x or 2x) may be desired to better understand in-service design margins. Any gaskets failure will be recorded and photographed.



Hydro-test setup for PUREX connector



Connector test assembly (nozzle bottom is fitted with test port)

#### Figure 23. Previous Test Setup for Hydro-test of PUREX Connectors

During development of the connector gasket test procedure, consideration will be given to ASTM F2378, *Standard Test Method for Sealability of Sheet, Composite, and Solid Form-in-Place Gasket Material.* It can be developed as a separate test or applied to connector leak test discussed above. This ASTM test method utilizes a test specimen compressed in increasing stages between the surfaces of two flat steel platens. After the specified press load is applied, fluid introduced into the center of the annular gasket compressed between platens, and a pressure of 4 MPa (580 psi) is applied. The fluid leak rate is measured. The test fluid pressure is relieved and the press load is increased to the next level. The fluid pressure is reapplied and the leak rate measured again. The cycle is repeated five times until a final press load of 32 MPa (4640 psi) is achieved. The press loads, internal test pressures, pressurizing fluids, and number of cycles can be varied as appropriate. Figure 24 shows a schematic of a test fixture used in this test.

Another similar standard gasket test procedure is ASTM F37, *Standard Test Methods for Sealability of Gasket Materials*. This test method provides a method of evaluating the sealing properties of sheet and solid form-in-place gasket materials at room temperature, and is suitable for evaluating the sealing characteristics of a gasket material under different compressive flange loads. A test specimen is compressed between the surfaces of two smooth steel flange faces. After the specified flange load is applied, the test fluid is introduced into the center of the annular gasket compressed between the flanges, the specified test pressure is applied, and the leakage rate is measured.



FIG. 1 Test Assembly for Determining Sealability of Gasket Materials

## Figure 24. Test Assembly for Determining Sealability of Gasket Materials Taken from ASTM F2378.

Other gasket tests considered were ASTM F36, *Standard Test Method for Compressibility and Recovery of Gasket Materials*. This test method covers determination of the short-time compressibility and recovery at room temperature of gasket materials. It is not intended as a test for compressibility under prolonged stress application, or for recovery following such prolonged stress application, the inverse of which is generally referred to as "compression set." Also, it is not intended for tests at other than room temperature. A resiliency characteristic (the amount recovered expressed as a percentage of the compressed thickness), may also be calculated from the test data where desired.

## 5.3 Coupon Testing

Similar postexposure testing<sup>10</sup> has been performed on coupon samples in the past and provides guidelines for tests in this Test Plan. After irradiation and exposure to chemical waste stimulant, coupons were tested for a variety of materials property changes. The comprehensive testing consisted of measuring the specific gravity, dimensions, mass, hardness, compression set, Vapor Transport Rate (VTR) and tensile properties (tensile strength, ultimate elongation yield, and tensile stress). Most of these properties were evaluated using standardized test methods developed by the ASTM. For specific gravity measurements, ASTM D792<sup>11</sup> was used. In

<sup>&</sup>lt;sup>10</sup> SAND2000-0466, Response of Ethylene Propylene Diene Monomer Rubber (EPDM) to Simulant Hanford Tanks Waste

<sup>&</sup>lt;sup>11</sup> ASTM D792, Standard Test Method for Density and Specific Gravity (Relative Density) of Plastics by Displacement

measuring dimensions and mass, ASTM D543<sup>12</sup> was used. For hardness measurements, ASTM D2240<sup>13</sup> was used. In evaluating compression set, ASTM D395<sup>14</sup> – Method B was used. For VTR measurements, ASTM D814<sup>15</sup> was used. Finally, for evaluating tensile properties, ASTM D412<sup>16</sup> – Method A was used.

Other Sandia studies on nonmetallic materials, including EPDM, have used compression stressrelaxation (CSR) measurements to support aging prediction models (SAND99-0623C, *Better Methods for Predicting Lifetimes of Seal Materials* [Celina, M., 1999], and SAND99-0553J, *Methods of Predicting More Confident Lifetimes of Seals in Air Environments* [Celina, M., 1999]). A standard test method is mentioned and Shawbury-Wallace Compression Stress Relaxometer was used. The ASTM provides a relevant standard, ASTM D6147, *Standard Test Method for Vulcanized Rubber and Thermoplastic Elastomer – Determination of Force Decay* (*Stress Relaxation*) in Compression.

A smaller set of materials properties tests is anticipated for this Test Plan. For all coupons, changes in mass and hardness will be measured (ASTM D2240). For the hose-in-hose material coupons, additionally tensile strength and ultimate elongation will be tested (ASTM D412). For Teflon material coupons, Compressibility and Recovery (ASTM F36, *Test Method for Compressibility and Recovery of Gasket Materials*) and Creep Relaxation (ASTM F38, *Test Methods for Creep Relaxation of a Gasket Material*) will be measured. For O-rings, compression set (ASTM D395. Sample preparation will utilize standardized test methods to cut, condition, and test the materials. The geometry of the material samples is specified by the test method.

Other studies on nonmetallic materials, including EPDM, have used CSR measurements to support aging prediction models considering only thermal degradation and oxidation at elevated temperature (SAND97-2181C, *Evidence that Arrhenius High-Temperature Aging Behavior for an EPDM O-Ring does not extrapolate to Lower Temperatures* [Gillen, K.T., et al., 1997], SAND99-0553J, KCP-613-5806, *Accelerated Aging of EPDM and Butyl Elastomers*). A standard test method is mentioned and Shawbury-Wallace Compression Stress Relaxometer was used. The ASTM provides a relevant standard, ASTM D6147. CSR measurement will be considered on the materials coupons as well.

A summary of the materials tests that may be performed on the coupons is given in Table 7.

Consideration will be given to additional laboratory evaluation of the material coupons and failed specimens from performance testing. Analysis will be selected if it provides insight into the damage mechanism or the depth of polymer degradation. The number of samples for which these analyses will be performed will also be limited by the funds available, with the highest priority given to performance testing (i.e., burst testing of hose specimens and leak testing of PUREX connector gaskets) as discussed above. The methods may include Fourier Transform Infrared Spectroscopy, Thermogravimetric Analysis, Differential Scanning Calorimetry, photo-

<sup>&</sup>lt;sup>12</sup> ASTM D543, Standard Practices for Evaluating the Resistance of Plastics to Chemical Reagents

<sup>&</sup>lt;sup>13</sup> ASTM D2200, Standard Test Method for Rubber Property – Durometer Hardness

<sup>&</sup>lt;sup>14</sup> ASTM D395, Standard Test Method for Rubber Property – Compression Set

<sup>&</sup>lt;sup>15</sup> ASTM D814, Standard Test Method for Rubber Property – Vapor Transmission of Volatile Liquids in Tension

<sup>&</sup>lt;sup>16</sup> ASTM D412, Standard Test Method for Rubber Properties in Tension

microscopy, Scanning Electron Microscopy, and other examinations of the polymer microstructure.

Hose	Teflon	O-Rings	
Mass Change	Mass Change	Mass Change	
Hardness	Hardness	Hardness	
Tensile Strength	Compressibility and Recovery	Compression Set	
Ultimate Elongation	Creep Relaxation		
Compression stress-relaxation	Compression stress-relaxation	Compression stress-	
		relaxation	
ASTM D412, D395, D2240,	ASTM F36, F38, D395, D2240,	ASTM D395, D2240,	
D6147	D6147	D6147	

Table 7. Summary of Materials Property Test Proposed for Coupons

## 6. QUALITY ASSURANCE

WRPS has specified that the QA Program be based on the requirements of the American Society of Mechanical Engineers (ASME) NQA-1, *Quality Assurance Requirements for Nuclear Facility Applications*.

A project specific quality assurance program place will be developed during the preparation timeframe. Since this involves testing on safety significant components, all data will be collected under an approved NQA-1 program. Testing will be conducted to written, approved procedures and follow ASTM practices where applicable. Sandia National Labs will be evaluated by Hanford site QA personnel. Pending results of the evaluation, SNL will be added to the Mission Support Alliance-Acquisition Verification Services Evaluated Supplier List.

## 7. FACILITIES, EQUIPMENT, SCHEDULES, AND COSTS

This section describes the facilities, equipment, and schedules (Subsection 7.1) that were identified and estimated for the proposed baseline test program and reduced cost option. This section also presents a list of other alternatives, along with their advantages and disadvantages (Subsection 7.1).

## 7.1 Facilities, Equipment, and Schedules

Table 8 provides the facilities, equipment, and schedule for the proposed baseline test program. It is important to note that the schedule in Table 8 is based on three crucial assumptions: First, it is assumed that the funding for the first year of the baseline test program (preparations) will arrive at SNL at the beginning of October 2013. Earlier or later arrival of funding would result in commensurate changes in the schedule for Phases 1, 2, 3, and 4. Second, it is assumed that all 14 vessels required for Phases 1 and 2, and all 12 to be used for Phases 3 and 4 can be placed in the GIF pool simultaneously (see Subsection 4.4). Third, it is assumed that all 14 annular stainless steel vessels required for Phases 1 and 2 can be placed in the pool from October 2014 through March 2015; and all 12 required for Phases 3 and 4 can be placed in the pool while those used for Phase 1 and 2 are being removed. As explained in Subsection 4.4, additional test configuration calculations with the TIGER codes (Franke et al., 2009) will be necessary prior to the start of the preparations for the baseline program in October 2013, or during this preparations for the baseline program.

Table 8 includes all of the annular stainless steel vessels required for all four phases of the baseline program. It is assumed that the vessels to be used for Phase 1 will be identical to those used for Phases 2, 3, and 4 (see Subsection 3.2).

Note that, for the baseline program, there will be 18 annular stainless steel vessels in test essentially simultaneously (Phase 1: 4 in the GIF high bay and 8 in the GIF pool, Phase 2: 6 in the pool). After the completion of Phases 1 and 2, 12 vessels would be in test simultaneously (Phase 3: 6 in the pool, Phase 4: 6 in the pool). These requirements will increase the number of vessels that must be purchased for the baseline program, and increase the initial space requirements for the GIF pool relative to those for the reduced cost option (see Table 8).

To save space, Table 8 does not include the control and monitoring equipment or the postexposure mechanical testing equipment required for Phases 1, 2, 3, and 4 of the baseline program. However, the costs for the control and monitoring equipment, and the postexposure mechanical testing equipment are included in the cost estimate for the baseline program. Additional test configuration calculations with the TIGER codes (Franke et al., 2009) will be necessary prior to the start of the preparations for the baseline program in October 2013 or during this preparation period to confirm the number of the sealed  $\beta$  and  $\gamma$  sources required for the baseline program (see Subsection 4.4).

Table 9 provides another breakout of the baseline test program which provides, by phase, the number of test vessels and their allocation to test articles, instrument, monitoring and control, data needs, and post test requirements. Table 10 provides this information for the reduced cost option to allow comparison of resource needs. The reduced cost option is described in greater detail below.

## Table 8. Facilities, Equipment, and Schedule for the Irradiation of Nonmetallic Materials: Proposed Baseline Test Program (2 sheets)

Preparations	Set up and carry out code calculations (exposure rate-absorbed- dose, shielding,				
1	test configurations, etc.); design, purchase, & annular stainless steel test vessels;				
	design, purchase, assemble, & test control and monitoring equipment and				
	postexposure mechanical testing equipment; SNL ES&H R&A SNL leak test;				
	and final WRPS R&A of preparations and procedures for the baseline program,				
	materials, QA, schedule, etc.				
Schedule	Start preparations: Oct. 2013; complete preparations: Sep. 2014				
Phase 1	$\beta$ - $\gamma$ comparison at ambient lab P and T, no chemical simulant				
Facility	<b>Paths 1</b> (β only) & 4 (control): 4 annular stainless steel vessels (see below) in a				
	dry, shielded area of the GIF outside the dry irradiation cells (probably the high				
	bay)				
	Paths 2.1, 2.2, 3.1, & 3.2: 8 vessels (see below) in the GIF pool				
Equipment	12 P- & T-rated annular stainless steel vessels				
	Each vessel would be 1.5 or 2 ft OD $\times$ 3 or 4 ft high				
	Each vessel would contain 12 30-in primary hoses & 12 hose coupons; or 12				
	confined Teflon gaskets, 12 unconfined Teflon gaskets, & 12 Teflon coupons				
	Total quantities of <sup>85</sup> Kr $\beta$ sources & <sup>60</sup> Co $\gamma$ sources TBD				
Schedule	Place vessels in the GIF pool or the high bay: Oct.–Dec. 2014				
	Remove vessels from the GIF pool or the high bay: OctDec. 2016				
	Complete posttest analyses & Phase 1 data report: JanMar. 2017				
Phase 2	Effects of $\gamma$ (1000 Rad/hr) & elevated T (70, 130, & 180 or 200 °F), ambient lab				
	P, no chemical simulant. Remove samples at $10^5$ , $10^6$ , and $10^7$ Rad (exact				
	durations TBD)				
Facility	All 6 annular stainless steel vessels (see below) in the GIF pool				
Equipment	6 P- & T-rated annular stainless steel vessels ( $P_{max} = 425 \text{ psig}; T_{max} = 200 ^\circ\text{F}$ )				
	Each vessel would be 1.5 or 2 ft OD $\times$ 3 ft or 4 ft high				
	Each vessel would contain 12 30-in primary hoses & 12 hose coupons; or 12				
	confined Teflon gaskets, 12 unconfined Teflon gaskets, & 12 Teflon coupons; and				
	12 confined EPDM O-rings & (perhaps) 12 unconfined EPDM O-rings				
	& 12 EPDM coupons				
	Total quantity of ${}^{60}$ Co $\gamma$ sources TBD				
Schedule	Place vessels in the GIF pool: JanMar. 2015				
	Remove vessels from the GIF pool: JanMar. 2017				
	Complete posttest analyses & Phase 2 data report: AprJun. 2017				
Phase 3	Effects of $\gamma$ (1000 Rad/hr), elevated T (70, 130, & 180 or 200 °F), & chemical				
	simulant (TFC-ENG-STD-34) at ambient lab P. Remove samples at $10^5$ , $10^6$ , and				
	$10^{7}$ Rad (exact durations TBD)				

Facility	All 6 annular stainless steel vessels (see below) in the GIF pool					
Equipment	6 P- & T-rated annular stainless steel vessels ( $P_{max} = 425 \text{ psig}; T_{max} = 200 ^\circ\text{F}$ )					
	Each vessel would be 1.5 or 2 ft OD $\times$ 3 ft or 4 ft high					
	Each vessel would contain 12 30-in primary hoses & 12 hose coupons; or 12					
	confined Teflon gaskets, 12 unconfined Teflon gaskets, & 12 Teflon coupons; and					
	12 confined EPDM O-rings & (perhaps) 12 unconfined EPDM O-rings					
	& 12 EPDM coupons					
	Total quantity of ${}^{60}$ Co $\gamma$ sources TBD					
Schedule	Place vessels in the GIF pool: Oct.–Dec. 2016					
	Remove vessels from the GIF pool: Oct.–Dec. 2018					
	Complete posttest analyses & Phase 3 data report: JanMar. 2019					
Phase 4	Effects of γ (1000 Rad/hr), elevated T (70, 130, & 180 or 200 °F) & P (400 or 425					
	psig) & chemical simulant (TFC-ENG-STD-34). Remove samples at $10^5$ , $10^6$ , and					
	10 <sup>7</sup> Rad (exact durations TBD)					
Facility	All 6 annular stainless steel vessels (see below) in the GIF pool					
Equipment	6 P- & T-rated annular stainless steel vessels ( $P_{max} = 425 \text{ psig}; T_{max} = 200 ^\circ\text{F}$ )					
	Each vessel would be 1.5 or 2 ft OD $\times$ 3 ft or 4 ft high					
	Each vessel would contain 12 30-in primary hoses & 12 hose coupons; or 12					
	confined Teflon gaskets, 12 unconfined Teflon gaskets, & 12 Teflon coupons; and					
	12 confined EPDM O-rings & (perhaps) 12 unconfined EPDM O-rings					
	& 12 EPDM coupons					
	Total quantity of ${}^{60}$ Co $\gamma$ sources TBD					
Schedule	Place vessels in GIF pool: Jan.–Mar. 2017					
	Remove vessels from GIF pool: JanMar. 2019					
	Complete posttest analyses & Phase 4 data report: AprJun. 2019					
	Complete final report: JulSep. 2019					

**Note:** This table does not include the control and monitoring equipment or the postexposure mechanical testing equipment for Phases 1, 2, 3, and 4, to save space. Furthermore, this table does not include the sealed  $\beta$  and  $\gamma$  sources, because this information is not available yet (see text).

Baseline Test Program						
	Phase	1	Phase 2	Phase 3	Phase 4	
Total Test Vessels (in- pool and out- of-pool)	10		6	6	6	
Vessel allocation and test article loading			1		1	
Out-of-Pool Vessel - Gasket/O-rings	1		0	0	0	
Out-of-Pool Vessel - Hoses	1		0	0	0	
In-Pool Vessels - Hose	4		3	3	3	
In-Pool Vessels – Gaskets & O-rings	4		3	3	3	
Hoses per vessel	12		12	12	12	
Gaskets & O-rings per vessel	24		48	48	48	
Instrumentation, Monitoring and Control			I		I	
Thermocouples - 2 per In-pool hose vessel , 8 per in-						
pool gasket vessel, 1 per out-of-pool vessel		130 96		96	96	
Air flow rate 1 per vessel		10 6		6	6	
Air oxygen concentration 1 per vessel			6	6	6	
Compressed air pressure supply/control	0		0	0	6	
Leak detector, 2 per in-pool vessel			12	12	12	
Pressure transducer, 2 per vessel with pressurized						
items			0	0	12	
Heater controllers, 1 per vessel	0		6	6	6	
Blank data channels	10		10	10	10	
Data Needs			1		•	
Total computer data channels		176		136	154	
Simultaneous readings		312		290		
Post Tests						
Hoses to burst test	60 36		5	36	36	
Gaskets & O-rings to leak test	120	14	4	144	144	
Total Burst and Leak tests	180 180 180		180			

Table 9. Vessel, Instrument, Data and Post Test Summary for the Baseline Test Program
Table 10 Vessel	Instrument	Data and Post	Test Summary	v for the F	Reduced Cost C	ntion
	,	Data and 1 03t	rest ourinnar			puon

Reduced Cost Option			
Hoses: Phases 2, 3, and 4; Gaskets & O-rings: Phase 3 only			
	Phase 2	Phase 3	Phase 4
Total Test Vessels (in- pool and out- of-pool)	3	6	3
Vessel allocation and test article loading	1	1	
Out-of-Pool Vessel - Gasket/O-rings	0	0	0
Out-of-Pool Vessel - Hoses	0	0	0
In-Pool Vessels - Hose	3	3	3
In-Pool Vessels – Gaskets & O-rings	0	3	0
Hoses per vessel	12	12	12
Gaskets & O-rings per vessel	0	48	0
Instrumentation, monitoring and control			
Thermocouples - 2 per In-pool hose vessel , 8 per in-pool gasket vessel, 1 per out-of-pool vessel	72	96	72
Air flow rate 1 per vessel	3	6	3
Air oxygen concentration 1 per vessel	3	6	3
Compressed air pressure supply/control	0	0	3
Leak detector, 2 per in-pool vessel	6	12	6
Pressure transducer, 2 per vessel with pressurized items	0	0	6
Heater controllers, 1 per vessel	3	6	3
Blank data channels	10	10	10
Data Needs		•	
Total computer data channels	97	136	106
Simultaneous readings	339	1	
Post Tests			
Hoses to burst test	36	36	36
Gaskets & O-rings to leak test	0	144	0
Total Burst and Leak tests	36	180	36

The reduced cost option will eliminate the Phase 1 beta/gamma comparison tests. The issue of relative damage from beta versus gamma radiation will be settled through alternate means. Novel to this test plan, other irradiation studies do not include beta irradiation. The issue may be addressed by a thorough literature study, expert analysis/opinion, or by limited materials coupon study. This can be determined at later date, and if a beta/gamma adjustment is required, it can be applied to the results of the Phase 2, 3, and 4 studies, which can only be performed with gamma sources. Hoses will still be tested at the varying levels of the 4 primary stressors as described in Phases 2, 3, and 4, allowing for meaningful data collection and a better service life prediction model to be developed.

In the reduced cost option, PUREX connector gaskets and O-rings will only be tested as described in Phase 3, with chemical stimulant, varying temperature and varying radiation exposure up to two years total. This will demonstrate gasket acceptability in a "proofing" sense and give insight into the effect of increasing radiation levels to the gasket material in the confined geometry of the PUREX connector.

The primary advantage to the reduced cost option is significant reduction of the cost and reduction of the schedule. The primary savings in the reduced scope program are

- Eliminates the high cost and handling of beta sources
- Reduces the number of test vessels from 18 to 12
- Reduces the number of test hoses, gaskets, and coupons by almost one half
- Reduces the amount of post exposure testing and analysis required
- Reduces the number of test, instrument, and control loops required

Most significantly, the reduced cost option reduces the size requirements such that all test vessels can fit into the pool at the Gamma Irradiation Facility at the same time, allowing for Phase 2, 3 and 4 to occur simultaneously. This will cut two years off the total schedule and allow for significant labor savings. Data collection will be completed on the hoses much sooner that the baseline.

#### 7.2 Costs

The cost estimate for the work described in this study is included in RPP-PLAN-54568, *Test Plan for the Irradiation of Nonmetallic Materials: Cost Estimate Summary*. This document is marked as Official Use Only because in contains Sandia Proprietary Information.

Table 11 identifies five other possible alternatives, along with some advantages and disadvantages of these options. The effects of these alternatives on the number and/or size of the vessels, the number of vessels that could be placed in the GIF pool simultaneously and to what extent this would shorten the overall schedule, etc., have not been evaluated. However, WRPS may specify within the statement of work to conduct this testing to include these alternatives, however the budget estimates provided do not apply to these alternatives and would need to be addressed for an accurate cost.

Potential Alternative	Advantage(s)	Disadvantage(s)
Use 10-in instead of 30-in	• Would decrease the number	Would decrease
samples of primary hoses, and	and/or size and hence the cost of	defensibility of the results
similarly smaller samples of	vessels required	
confined Teflon gaskets and	• Would increase the number of	
confined EPDM O-rings	vessels that could be placed in	
	the GIF pool simultaneously	
	and shorten the schedule	
Use coupons only instead of	• Would decrease the number	Would decrease
along with materials in their	and/or size and hence the cost of	defensibility of the results
in-service configurations	vessels required beyond that	beyond that sustained with
	achievable with 10-in hoses, etc.	10-in hoses, etc.
	• Would increase the number of	
	vessels that could be placed in	
	the GIF pool simultaneously	
	and shorten the schedule beyond	
	that achievable with 10-in	
	hoses, etc.	
Reduce the number of	• Would decrease the number	Would decrease
samples exposed to	and/or size and hence the cost of	defensibility of the results
identical conditions for	vessels required	
identical durations from	• Would increase the number of	
three to two or even one.	vessels that could be placed in	
	the GIF pool simultaneously	
	and shorten the schedule	*** 111
Carry out $\gamma$ irradiations for	• Would decrease the number	Would limit the reliability
Phases 2, 3, and 4 at just	and/or size and hence the cost of	of interpolating or
two temperatures instead of	vessels required	extrapolating performance
three	• Would increase the number of	data to other temperatures
	the CIE need simultaneously	
	and shorten the schedule	
Limit total exposures to	• Would decrease the total time	Would not obtain results
$\leq 0.8 \times 10^7 \text{ D}$	• would decrease the total lille required for each phase of	for total does of $1.1 \times 10^7$
< 0.0 × 10 K	the test program and hence	$\mathbf{P}$ (the expected fatel doce
	shorten the schedule	for EDDM) and for
	shorten ne senedule	overtest dose
		overtest dose

# Table 11. Additional Alternatives That Might be Considered to Reduce the Required Timeor the Cost of Irradiation of Nonmetallic Materials. (2 sheets)

Potential Alternative	Advantage(s)	Disadvantage(s)
Use single upper-level	• May be able to use single larger	Size limitations may not
temperature and pressure	vessel to minimize procurement	allow full realization of
instead of individual design	cost of vessels and sources.	potential cost savings.
temperatures and pressures		GIF safety and handling
		concerns. Reduced
		defensibility by requiring
		extrapolation at bounding
		conditions.

## 8. **REFERENCES**

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## APPENDIX A. GANTT CHART FOR THE BASELINE TEST PROGRAM

March 22, 2013

D	Task Name	Start	Finish	Predecessors		2014		2015		2016		2017		2018		2
	75 m (2)				H2	H1	H2	H1	H2	H1	H2	H1	H2	H1	H2	
1	Phase 1											-				
2	Phase 1 Preparations															
3	Design, order, acquire vessels and modify PUREX connector/test fixture	Tue 10/1/13	Fri 5/30/14		C											
4	Acquire hoses	Mon 11/4/13	Fri 4/4/14													
5	Design, order, install and test instrumentation (wiring, tubing	Tue 10/1/13	Fri 5/30/14		C											
6	Send WRPS design	Mon 2/3/14	Mon 2/3/14			I										
7	Water tank testing	Thu 6/5/14	Fri 8/29/14	5,3,4		1										
8	Radiation calculations	Wed 10/2/13	Mon 12/16/13	1952		1										
9	Acquire beta sources	Tue 12/17/13	Thu 6/19/14	8	i	<b>1</b>										
10	Acquire gamma source	Tue 12/17/13	Thu 6/19/14	8	i											
11	GIF ES&H	Tue 10/1/13	Mon 9/1/14		C											
12	Complete Readiness Review	Tue 9/2/14	Fri 9/5/14	7,8,9,10,11			Ĩ									
13	Phase 1 Preparations Completed	Thu 9/18/14	Thu 9/18/14	11,12			Ť									
14	Phase 1 Experiment					-										
15	Install in GIF	Tue 9/30/14	Fri 10/31/14	11,12,13			The second									
16	Beta tests	Mon 11/3/14	Mon 11/28/16	15,8			Ľ				5					
17	Beta & Gamma tests	Mon 11/3/14	Mon 11/28/16	15,8			Ľ				3					
18	Gamma tests	Mon 11/3/14	Tue 11/29/16	15,8			E									
19	Send WRPS interim results	Thu 5/15/14	Wed 11/30/16			E					1					
20	Posttest analysis	Fri 2/27/15	Tue 2/28/17									3				
21	Phase 1 Documentation															
22	Verify QA documentat	Tue 10/1/13	Fri 3/3/17		E											
23	Submit draft report to WRPS	Fri 3/3/17	Fri 3/3/17	20								Ĩ				

D	Task Name	Start	Finish	Predecessors		2014		2015		2016		2017		2018		2019	
-					H2	H1	H2	H1	H2	H1	H2	H1	H2	H1	H2	H1	H2
24	Complete report to WRPS	Thu 3/30/17	Thu 3/30/17	23								I					
25	Phase 1 Completed		Fri 3/31/17									3—					r I
26	Phase 2						-										
27	Phase 2 Preparations						-										
28	Acquire vessels & hose	Tue 10/1/13	Mon 2/3/14														
29	Design, order, and install instrumentation, wiring, and tubing (dosimeters, thermocouples, pressure, flow, oxygen, puddle,	Tue 10/1/13	Fri 5/30/14			3											
30	Water tank testing	Mon 6/2/14	Mon 9/1/14	29,28		E											
31	Complete GIF ES&H	Tue 9/2/14	Tue 9/2/14	30,8			Ŧ										
32	Complete Readiness Review	Thu 9/18/14	Thu 9/18/14	31			Ť										
33	Phase 2 Preparations Completed		Wed 10/1/14	32,30,31			Ť										
34	Phase 2 Experiment																
35	Install in GIF	Thu 10/2/14	Thu 1/15/15	33			Ľ	<b>.</b>									
36	Gamma tests	Mon 2/2/15	Fri 2/24/17	35				Ľ				3					
37	Send WRPS interim results	Fri 7/10/15	Tue 2/28/17								-						
38	Posttest analysis	Tue 3/3/15	Fri 4/14/17					E				<b>1</b> 1					
39	Documentation											-					
40	Verify QA documentation	Fri 1/2/15	Thu 3/2/17					C									
41	Submit draft report to WRPS	Mon 4/17/17	Mon 4/17/17	35,36,38								Ŧ					
42	Complete report to WRPS	Wed 5/3/17	Wed 5/3/17	41								Ţ					
43	Phase 2 Completed	Mon 6/5/17	Mon 6/5/17	42								Ť					

D	Task Name	Start	Finish	Predecessors	112	2014	112	2015	112	2016	112	2017	112	2018	20	019	
44	Phase 3		_		HZ	H1	HZ	H1	HZ	HI	HZ	HI	HZ	H1	HZ   I	HI	HZ
45	Phase 3 Preparations																
46	Develop chemical reci	Mon 3/2/15	Wed 9/23/15		ΙΓ			C	Ъ							-	
47	Purchase chemicals	Thu 9/24/15	Mon	46					-								
	and prepare solutions	1110 572 17 15	12/14/15	10													
48	prepare vessels, hoses, chemicals	Mon 12/21/15	Mon 4/18/16	47					ì								
49	Install instrumentation, wiring, and tubing (dosimeters, thermocouples, pressure, flow, oxygen, puddle, temperature	Tue 4/19/16	Mon 6/20/16	48						Ľ							
50	Complete water tank testing with chemical stimulant	Tue 7/5/16	Tue 7/5/16	48,49						1							
51	Radiation calculations with chemical	Fri 10/2/15	Sun 12/20/15														
52	Complete GIF ES&H	Wed 7/6/16	Wed 7/6/16	50,51						É	7						
53	Complete Readiness Review	Thu 7/7/16	Thu 7/7/16	52						4	7/7						
54	Phase 3 Preparations Completed		Thu 9/1/16	53							ž						
55	Experiment									-	_						
56	Install in GIF	Mon 10/3/16	Fri 10/28/16	50,52							The second						
57	Gamma tests with solution renewal every 2 months	Tue 11/1/16	Fri 2/1/19	56							Ľ						
58	Send WRPS interim results	Tue 5/10/16	Mon 2/11/19							C					1		
59	Posttest analysis	Wed 2/1/17	Tue 2/12/19												1		
60	Documentation								1								

D	Task Name	Start	Finish	Predecessors		2014	( server '	2015		2016		2017		2018		2019	101201
<b>C1</b>	N/- 15 OA	Th. 10/1/15	E: 0/1E/10		H2	H1	H2	H1	H2	H1	H2	H1	H2	H1	H2	H1	H2
01	documentation	Thu 10/1/15	Ffi 2/15/19													-	
62	Submit draft report to WRPS	Wed 2/13/19	Wed 2/13/19	59												Ĩ	
63	Complete report to WRPS	Thu 3/28/19	Thu 3/28/19	62												Ŧ	
64	Phase 3 Completed	Fri 3/29/19	Fri 3/29/19	63												<b>X</b> 3,	29
65	Phase 4																
66	Phase 4 Preparations																
67	Install instrumentation, wiring, and tubing (dosimeters, thermocouples, pressure, flow, oxygen, puddle, temperature controller, back-pressure, computer	Wed 6/1/16	Mon 8/1/16														
68	Complete water tank testing	Tue 8/2/16	Tue 8/2/16	67							8/:	2					
69	Complete GIF ES&H	Wed 9/28/16	Thu 9/29/16								T.	٦					
70	Complete Readiness Review	Mon 10/3/16	Mon 10/3/16	68,69								10/3					
71	Phase 4 Preparations Completed	Mon 10/3/16	Thu 11/10/16	70													
72	Experiment																
73	Install in GIF	Tue 1/3/17	Tue 1/31/17	69,70								<b>D</b>					
74	Gamma tests with solution renewal every 2 months	Wed 2/1/17	Wed 5/1/19	73								Ľ				3	
75	Send WRPS interim results	Thu 8/10/17	Wed 5/8/19										۲			3	
76	Posttest analysis	Fri 2/1/19	Mon 6/3/19														
77	Documentation																
78	Verify QA documentat	Mon 1/2/17	Wed 5/1/19									<b>C</b>				3	

ID	Task Name	Start	Finish	Predecessors		201	.4		2015		2016		2017		2018		2019		
					H2	2 H:	1	H2	H1	H2									
79	Submit draft report to WRPS	Wed 6/5/19	Wed 6/5/19	76													I		
80	Complete report to WRPS	Fri 6/28/19	Fri 6/28/19	79													Ť		
81	Phase 4 Completed	Fri 6/28/19	Fri 6/28/19														•	6/2	8
82	Draft Cumulative Final Report to WRPS	Thu 8/1/19	Thu 8/1/19	81,64,43,25													1		
83	Revise Cumulative Final Report to WRPS	Mon 9/2/19	Mon 9/2/19	82														Ť	
84	Cumulative Final Report Issued	Thu 9/26/19	Thu 9/26/19	83														*	9/
85																			
86																			
87																			
				P	age 5														_



# APPENDIX B. LIFETIME PREDICTION MODEL

#### **B-1. LIFETIME PREDICTION MODEL**

It will be necessary to develop a comprehensive lifetime prediction model based on accelerated aging methodology so test data results can be used to determine service life for the polymeric materials used in waste retrieval systems. The new model will evaluate the effect of all four stressors evaluated in the test plan (temperature, pressure, chemistry, and radiation). Only ethylene propylene diene monomer (EPDM) hose-in-hose transfer line (HIHTL) assemblies (and similarly constructed EPDM hose jumper assemblies) are required to be tracked and assessed for future use. As a result, for the purposes of this appendix, it is assumed that only the EPDM HIHTL assemblies will be subjected to a lifetime prediction model.

Currently, the HIHTL assemblies are limited to three years of service life unless a formal life extension analysis is performed per the recommendations in RPP-6711, *Evaluation of Hose-in-Hose Transfer Line Service Life*, Appendix L. The accelerated aging and life predication techniques that were developed in RPP-6711 are based on concepts employed by the Arrhenius, Eyring, William Landel Ferry, and Palmero and DeBlieu methodologies. Additionally, the well-established Palmgren-Miner concept (Miner's Rule) was applied to account for the cumulative damage of the hoses due to the cyclic nature of their use for retrieval efforts. The life prediction efforts resulted in a service life equation that uses temperature and pressure to determine the total remaining life in the selected hoses.

The service life equation was based on sound methodology, but a number of deficiencies in the approach were noted during testing, as described below:

- 1. Testing was performed using only water as the internal media,
- 2. Radiation dose was not applied during testing, and
- 3. The test temperatures did not reflect temperatures expected during field operations.

Namely, the synergistic effects of chemicals, temperature, radiation, and pressure on the hose's useful life were not studied. It is anticipated that the testing described in this test plan will account for the synergistic effects of the listed stressors.

Historically, the majority of accelerated aging studies have utilized the Arrhenius methodology to determine the expected life of a component (SAND2000-3037C). The Arrhenius methodology is a chemical rate theory that assumes that linear behavior can use temperature to predict useful lifetime of a component. Additionally, as described in further work by Gillen (Gillen, 2001, Gillen, 1985, and Burnay, 1991), additional stressors may be taken into account by using the Time-temperature superposition (described below), or similar equation modification methods. Burnay identified an advantage of time-temperature superposition in that it does not require detailed knowledge of the degradation reaction mechanisms, but also identified limitations in that a single mechanism must be dominant. Burney concluded his superposition was of most use where extrapolations were made to lower temperatures and dose rates than those tested experimentally. Further analysis will be required to determine an appropriate comprehensive aging model that incorporates all four stressors.

### **B-2. TIME-TEMPERATURE SUPERPOSITION**

Time-temperature superposition was developed by Gillen and Clough (Gillen 2001) as a method for consolidating data on a measured variable that is a function of time and another parameter such as temperature. For example, the variable percent of elongation-at-break (PEAB), for a polymer is dependent on time and temperature. To obtain these data, polymer samples are aged at a fixed temperature in a vessel. At predetermined time intervals, some samples are withdrawn and the PEAB is measured for each sample. From these measurements, a PEAB curve is generated as a function of exposure time. The experiment is repeated again at a few more temperatures, from which additional PEAV curves are generated as a function of exposure time. To consolidate these curves, Gillen proposed that for each curve there is one "shift" parameter for each temperature that multiplies the exposure time. This shift parameter is obtained by first selecting a base curve, typically the curve with the longest exposure time required for a given PEAB. Then for each remaining curve, a shift parameter is determined that minimizes the difference between the base curve and other curves taken different temperatures than the base curve. A fit of all the shift parameters is determined as a function of temperature. For the PEAB, Gillen found that the logarithm of the shift parameter is linear with the inverse of absolute temperature. This is a classical Arrhenius fit, and the slope of the curve provides the activation energy. Departures from linearity of the shift parameter indicate that the activation energy is temperature-dependent. As an alternative to assuming that the shift parameter follows an Arrhenius fit, the shift parameter may be added to the exposure time instead of multiplying by the shift parameter (Gillen and Celina, 2001). In this case a linear fit of the shift parameter with temperature may provide a better basis for extrapolation.

For example, consider hypothetical measurements of the burst pressure as a function of exposure time at 1000 R/hr for three temperatures, 50 °C, 65 °C, and 90 °C. The hypothetical data are given in Figure 25.

To extrapolate to other temperatures, we find that if the exposure time for the data at 65 °C and 90 °C are multiplied by 2.5 and 10.0, respectively, the data collapses to one curve as given in Figure 26. (These data were artificially created, and thus there is no scatter for the collapsed curve of burst pressure. For realistic applications some scatter in the collapsed curve should be expected.)



Figure B-1. Hypothetical Burst Pressure Measurements at Three Temperatures.



Figure B-2. Hypothetical Burst Pressure as a Function of Shifted Exposure Time, Where the Shift Factor A is Determined so that the Data Collapses to a Single Curve.

A plot of the time shift parameter is given in Figure 27. The curve is extrapolated to an inverse temperature of 0.0034, corresponding to a temperature of 21 °C. The value of the shift parameter at this temperature is 0.167. Thus the extrapolated burst pressure curve may now be calculated with the shift parameter, and is given below in Figure 28. Essentially, for each point on the base curve, the time value is multiplied by 1/0.167 for the same value as the burst pressure. In this example, from Figure 28, after 6000 days (16.4 years), of exposure at 21 °C, the burst pressure would be 550 psig.



Figure B-3. Time Shift Parameter as a Function of Inverse Temperature Extrapolated Above and Below the Three Values of the Time Shift Parameter Obtained from the Burst Pressure given in Figure 25.



Figure B-4. Hypothetical Burst Pressure Data Shown with Open Symbols, and the Extrapolated Burst Pressure Shown with Filled Circles. The Extrapolated is Based on an Arrhenius Fit to the Time Shift Parameter Given in Figure 26.

### B-3. MINER'S RULE

To account for cumulative HIHTL damage, the Miner's Rule must also be applied, and the data fitted appropriately. Although used in a different context in RPP-6711, Miner's Rule describes the simple concept that degradation is cumulative, and that failure is a result of the accumulation of damage with time. Damage is simply a function of an applied stressor (or multiple stressors) and the number of cycles that the stressors are applied. The application of this method relies on the experimental completion of aging the HIHTL to a selected failure criterion such as PEAB. Discussion on the proper selection of failure criterion is further addressed in literature.

The final HIHTL life predictions will be based on the extrapolated results of the combined Arrhenius and Miner's Rule equations. However, to ensure accurate results, two main factors must be considered during testing to avoid non-Arrhenius behavior of the data: 1) Diffusion limited oxidation effects (discussed in Section 2.7.4.2), and 2) Ensuring that the results are not extrapolated far from the test data. As further described in Gillen, 2001, more confidence can be giving to results if test data mimics the intended results as accurately as possible.

### **B-4. TESTING CONDITIONS**

This test plan specifies four different stressors that will need to be incorporated into comprehensive models. Previous work has only looked at time, temperature, and radiation, where radiation testing was done at high dose rates requiring a large amount of extrapolation to predict performance. In this testing three out of four stressors (i.e., temperature, pressure, and chemistry) are in the anticipated operation range. The fourth stressor, radiation dose rate, will be elevated over most field conditions to facilitate realistic testing times. The reasonably bounding dose rates selected for this test plan are an order of magnitude (or greater) improvement over historical irradiation aging test conditions (e.g.SAND2000-0466).

Due to the selection of three stressors, temperature, pressure, and chemistry, within operating conditions, the modeling will primarily account for the extrapolation of radiation. The model will also account for the remaining stressors, but should not require significant extrapolation. Therefore, the service life determined from the data gathered from this testing will be less dependent on modeling and more dependent on actual performance as test specimens are aged under realistic test conditions. The resulting resolution of may permit justification for extended service life while still maintaining an adequate margin of safety.

#### **B-5. CONCLUSIONS**

Although an appropriate aging model cannot be selected before the data is collected, based on past experience, the Arrhenius methodology will most likely be used to predict HIHTL life. Due to multiple additional stressors associated with this testing, an appropriately determined comprehensive model will be required. Additionally, Miner's Rule should be applied to account for cumulative damage of the HIHTLs. By selecting stressors closely mimicking field conditions, much errant and non-Arrhenius behaviors can be greatly reduced.

#### **B-6. REFERENCES**

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