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CHEMICAL COMPATIBILITY TESTING

FINAL REPORT

INCLUDING TEST PLANS

AND PROCEDURES

May 1994

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ABSTRACT

This report provides an independent assessment of information on mixed waste streams, chemical compatibility information on polymers, and standard test methods for polymer properties. It includes a technology review of mixed low-level waste (LLW) streams and material compatibilities, validation for the plan to test the compatibility of simulated mixed wastes with potential seal and liner materials, and the test plan itself. Potential packaging materials were reviewed and evaluated for compatibility with expected hazardous wastes. The chemical and physical property measurements required for testing container materials were determined. Test methodologies for evaluating compatibility were collected and reviewed for applicability. A test plan to meet US Department of Energy and Environmental Protection Agency requirements was developed. The expected wastes were compared with the chemical resistances of polymers, the top-ranking polymers were selected for testing, and the most applicable test methods for candidate seal and liner materials were determined. Five recommended solutions to simulate mixed LLW streams are described. The test plan includes descriptions of test materials, test procedures, data collection protocols, safety and environmental considerations, and quality assurance procedures. The recommended order of testing to be conducted is specified.

PREFACE

This report was prepared by the Environmental Technology & Education Center (ETEC), 3300 Mountain Road NE, Albuquerque, NM 87106-1920, under Contract AF-4541, for Transportation System Technology, Department 6642/MS 0717, Sandia National Laboratories, P. O. Box 5800, Albuquerque, NM 87185-0717.

This is a summary of work performed from April through November 1993. The Sandia project officer was Dr. Paul J. Nigrey. The ETEC principal investigator was Dr. Jon Nimitz. Dr. Ronald E. Allred and Mr. Brent W. Gordon of Adherent Technologies provided substantial technical assistance.

Sections 1 through 4 of this document constitute the final report for this project. Sections 5 through 8 and Appendices A through C constitute the test plans and procedures report.

Mention of any specific product in this report does not constitute endorsement by ETEC, Adherent Technologies, Sandia National Laboratories, or the U.S. Department of Energy.

ACRONYMS AND ABBREVIATIONS

Å	Angstrom
ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
atm	Atmospheres
BSI	British Standards Institution
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFC	Chlorofluorocarbon
cm	Centimeter
CM	Cross-linked chlorinated polyethylene
CO	Epichlorhydrin polymers
CPE	Chlorinated polyethylene
CR	Polychloroprene
CSPE	Chlorosulfonated polyethylene
DIN	Deutsches Institut für Normung
DMA	Dynamic mechanical analysis
DoD	U.S. Department of Defense
DOE	U.S. Department of Energy
DOT	U.S. Department of Transportation
ECO	Epichlorhydrin polymers
EIA	Ethylene interpolymer alloy
EPA	Environmental Protection Agency
EPDM	Ethylene propylene diene monomer
eV	Electron volt
FC	Perfluorocarbon
Freon	Dupont trade name for halocarbons
FTIR	Fourier transform infrared spectroscopy
g/L	Grams per liter
g/mL	Grams per milliliter
GTR	Gas transmission rate
GWP	Global warming potential
Gy	Gray

HDPE	High-density polyethylene
HDPE-A	High-density polyethylene/rubber alloy
IR	Infrared
IRRA	Infrared radiation absorption (same as FTIR)
ISO	International Organization for Standardization
J	Joule
Kg	Kilogram
LLDPE	Linear low-density polyethylene
LLW	Low-level waste
Mrad	Million rads, also known as megarads
M	Molar (moles/liter)
MILSPEC	Military specification
MILSTD	Military standard
MW	Molecular weight
NIST	National Institute for Standards and Technology (formerly NBS, National Bureau of Standards)
nm	Nanometer
NSF	National Sanitation Foundation
NTIS	National Technical Information Service
NVR	Nonvolatile residue
OA	Organic acid
ORNL	Oak Ridge National Laboratory
PCE	Perchloroethylene
PE-EP-A	Polyethylene ethylene/propylene alloy
PEL	Polyester elastomer
pH	The negative of the logarithm of the concentration of hydronium ions
ppm	Parts per million
PVC	Polyvinyl chloride
PVC-CPE	Polyvinyl chloride -- chlorinated polyethylene alloy
QA	Quality assurance
QC	Quality control
RCRA	Resource Conservation and Recovery Act
RDDT&E	Research, Development, Demonstration, Testing, and Evaluation

T-EPDM	Thermoplastic EPDM
TCA	1,1,1-trichloroethane
TCE	trichloroethylene
TN-PVC	Thermoplastic nitrile-polyvinyl chloride
USAF	United States Air Force
UV	Ultraviolet
VOC	Volatile organic compound
Z	Atomic number

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EXECUTIVE SUMMARY

The purpose of this effort is to provide chemical compatibility assurance for seals and liners for hazardous waste transportation containers. The major focus of this project was to develop a detailed experimental test plan to meet U.S. Department of Energy (DOE) and U.S. Environmental Protection Agency (EPA) requirements. This effort will assist in compliance with the environmental requirements of the Resource Conservation and Recovery Act (RCRA); the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA); the Atomic Energy Act; and state laws.

This report provides background information on mixed waste streams, chemical compatibility information on polymers, and standard test methods for polymer properties. It also provides an independent assessment of the applicability of this information for the development of a test plan for experimental testing of seal and liner materials for mixed waste sample containers and provides the test plan. The technology review consists of a review of DOE waste streams and known information on chemical compatibility of materials of interest. Validation for the test plan consists of assessments of established test procedures for polymers, selection of the most likely candidate materials for testing, and selection of the tests needed to validate their use in waste containers.

Strong alkali, organic solvents, and oxidants appear to constitute the main threats from the wastes of interest. Five solutions are selected to simulate the hazardous waste components of mixed wastes for testing materials compatibility of polymers:

- ❖ aqueous alkali containing dichromate ion
- ❖ a general mixed organic liquid consisting of equal parts by volume of 1,1,1-trichloroethane, hexane, toluene, methyl ethyl ketone, 1-butanol, acetonitrile, and dioxane
- ❖ three specific organic liquids consisting of (a) neat 1,1,1-trichloroethane to simulate chlorinated solvent waste, (b) a blend of equal parts by volume toluene, xylene, and dioxane to simulate scintillation cocktail wastes, and (c) a mixture of 90% by volume methyl isobutyl ketone (hexone) with 10% by volume kerosene.

If desired, other optional additives may be included (such as bismuth or praseodymium salts to simulate radionuclides), though these additives are judged extremely unlikely to have any effects on polymers.

A set of 10 polymers with anticipated high resistance to one or more of the anticipated types of waste environments is proposed for testing as potential seal or liner materials. These polymers are butadiene-acrylonitrile copolymer, cross-linked polyethylene, epichlorohydrin, ethylene-propylene rubber, fluorocarbon, glass-filled tetrafluoroethylene, high-density polyethylene, isobutylene-isoprene copolymer, polypropylene, and styrene-butadiene rubber.

The most applicable test methods are selected and specified. EPA Method 9090A is the only standard that attempts to evaluate the chemical resistance of liner materials comprehensively. It should therefore be used as the starting point for selection of appropriate tests. The major concerns for liner materials are dimensional stability, embrittlement, and permeability. Puncture resistance and swelling are secondary considerations because the liner will be supported by the canister. The same concerns apply to O-rings, however, swelling and compression set resistance must additionally be considered. If an O-ring swells or permanently deforms, its sealing properties can be lost, allowing the contents to leak. These concerns were used to select the most relevant test methods. The test plan is given for exposure of polymers to the simulated hazardous wastes (and radiation) and testing of mechanical properties.

Because of the low-level wastes involved, the distance from the sample container, the probable regular replacement of seals and liners, and the generally high resistance of polymers to radiation, it is not anticipated that the seals and liners considered in this study will be subjected to high enough radiation levels to cause any noticeable changes in properties. It has also been observed that radiation does not significantly affect chemical compatibility, so no synergistic destructive effects are expected. Thus, radiation testing is judged not essential for this test plan. For completeness, however, protocols for testing of effects of radiation exposure are included.

1.0 INTRODUCTION

1.1 Organization of Report and Test Plan

This report includes a technology review of mixed low-level waste (LLW) streams and material compatibilities, validation for the plan to test compatibility of simulated mixed wastes with potential seal and liner materials, and the test plan itself. The technology review consists of a review of U.S. Department of Energy (DOE) waste streams and known information on chemical compatibility of materials of interest. Validation for the test plan consists of assessments of established test procedures for polymers, selection of the most likely candidate materials for testing, and selection of the tests needed to validate their use in waste containers.

1.2 Purpose

The overall purpose of this effort is to provide significant chemical compatibility assurance for seals and liners for containers transporting hazardous wastes. The major focus of this project has been to develop a detailed experimental test plan to meet DOE and U.S. Environmental Protection Agency (EPA) requirements. This effort will assist in compliance with the environmental requirements of the Resource Conservation and Recovery Act (RCRA); the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA); the Atomic Energy Act; and state laws.

The specific objectives of this report include providing

- ❖ an independent technology review and assessment of the chemical and physical effects of hazardous and simulated mixed wastes on potential container seals and liners
- ❖ an independent assessment of appropriate test methodologies for chemical compatibility studies on potential container seals and liners
- ❖ a description of test plans and procedures for performing baseline experimental chemical compatibility evaluations with hazardous waste forms and candidate seals and liners.

This report assists in specifying container construction by assessing compatibility between wastes and potential packaging materials and by specifying tests for potential seal and liner materials with simulants for hazardous (but not mixed) wastes. The report focuses on the waste streams of interest and the top-ranking candidate seal and liner materials. The procedures specified in the test plan will guide laboratory testing to yield baseline data and data on simulated hazardous wastes, which will facilitate future testing of actual mixed wastes. The test plan allows flexibility in test parameters such as aging times, temperatures, and chemicals tested.

1.3 Background

During the past 45 years, substantial amounts of hazardous and mixed wastes have accumulated at several DOE facilities. At the end of 1991, the inventory of mixed LLWs at DOE sites totaled about 101,400 m³ (Ref. 1). During 1991, an additional 66,000 m³ of mixed LLW was generated throughout the DOE. Some of the major waste streams include aqueous nitrate/nitrite with low levels of inorganic, organic, and radioactive materials; chlorinated solvents with small amounts of radioactive materials; and hydrocarbons with small amounts of radioactive materials (Ref. 1). As described in the recent five-year Research, Development, Demonstration, Testing, and Evaluation (RDDT&E) Plan, the DOE strategy for managing mixed wastes includes minimizing generation, treating hazardous constituents, and permanent disposal (Ref. 2).

The Hanford, WA, site provides examples of some of the problems encountered with mixed wastes and strategies for their solutions. Hanford is illustrative because it has some of the largest quantities of mixed wastes in the DOE, and more thorough analyses of these wastes have been reported than for many other sites. The Hanford site, for example, has an estimated 6,900 metric tons of nuclear materials, including 4,100 metric tons of uranium and 15 metric tons of cesium-strontium capsules (Ref. 3). Often, nuclear materials have been mixed with other inorganic and organic chemicals; 770,000 metric tons of mixed waste is contained in storage tanks at Hanford. Scenarios similar to those found at Hanford occur elsewhere within the DOE, but generally on a smaller scale (Ref. 1).

The plan for safe disposal of these mixed wastes at Hanford is to retrieve all material from the tanks and separate it into high- and low-level components. The high-level wastes will be vitrified into glass logs and the LLWs will be made into grout (a cement-based matrix) and stored in underground vaults (Ref. 3). It is expected that the steady-state temperature in disposal vaults will be about 90°C (Ref. 4). Ground has been broken for the vitrification facility at Hanford, planned for startup in 1999.

To analyze the stored wastes, samples must be taken and transported for analysis. The containers for this transport must be highly resistant to leakage and must survive threats from impact, fire, and tampering. If the samples are expected to generate gases, the permeability of the containers to these gases must be understood and must not pose a threat. The purpose of this effort is to assist in selecting construction materials (seals and liners) for waste sample containers that will meet these requirements.

The envisioned sample container has a polymeric liner covering the entire inner surface and an elastomeric O-ring seal surrounding the lid-bottom mating surfaces. The exact geometry of the container and seal area remain to be determined. Seals and liners are not intended to be exposed directly to wastes except in the unlikely event of rupture of the inner container. However, to ensure safety in the case of rupture of the inner container, any seals or liners used in sample containers designed to transport mixed LLWs must have high chemical resistance to the wastes contained, be relatively impermeable, and maintain flexibility. Although the liner is not expected to be exposed to significant mechanical loads in the planned design, the seal must not permanently deform under compressive loads (*i.e.*, take a set).

Related issues such as ease and costs of changing seals and liners and their proper disposal are also relevant. The entire sample containers (including seals and liners) should be inexpensive. They may be designed to be either reusable or disposable. If the seals or liners take sets or become cracked or embrittled, they must be replaced. It is anticipated that under normal circumstances used seals and liners can be disposed of as normal solid waste. The only exception that would require seals or liners to be disposed of as hazardous or mixed wastes would be in the case of rupture of the inner container and exposure of the seal or liner materials to hazardous or mixed wastes.

1.4 Scope and Approach

The program plan for this effort consisted of three major tasks: (1) a technology review, (2) definition of evaluation parameters, and (3) development of the test plan. The purpose of Task 1 was to define the construction materials of interest, the actual waste components expected, and realistic simulants for these wastes. Known information on these topics was obtained, reviewed, and critically assessed. Sources of information included on-line databases, printed material, and interviews with experts in the field. Databases searched included those of Compendex, Chemical Abstracts, and the National Technical Information Service (NTIS). A list of search terms is given in Appendix A. Contacts at national laboratories and material manufacturers were also interviewed.

Potential packaging materials were reviewed and evaluated for compatibility with expected hazardous wastes. Emphasis was placed on potential waste-induced material property changes that could lead to failures. Possible undesirable property changes assessed include plasticization, stress cracking, permeability, and embrittlement. Issues regarding permeability include the possibility that, in the event of internal leakage, gases, liquids, or heavy metals would permeate the liner.

In Task 2, the information collected in Task 1 was reviewed to determine the chemical and physical property measurements required for testing container materials. Test methodologies for evaluating compatibility were collected and reviewed for applicability. Test methods were reviewed from the American Society for Testing and Materials (ASTM), the American National Standards Institute (ANSI), the National Sanitation Foundation (NSF), the British Standards Institution (BSI), the Deutsches Institut für Normung (DIN), the Environmental Protection Agency (EPA), and the International Organization for Standardization (ISO). It is expected that subtle changes in properties will provide initial indications of incompatibilities; therefore, techniques for evaluating small changes are discussed. Potential techniques include various spectroscopies (such as Fourier-transform infrared [FTIR], ultraviolet [UV] visible, fluorescence, and phosphorescence), thermal analysis, and dynamic mechanical analysis.

In Task 3, a test plan to meet DOE and EPA requirements was developed by comparing the expected wastes with the chemical resistances of polymers, selecting the top-ranking polymers for testing, and selecting the most applicable test methods for candidate seal and liner materials. The test plan includes descriptions of test materials, test procedures, data collection protocols, safety and environmental considerations, and quality assurance (QA) procedures. The recommended order of

testing to be carried out is specified.

It is anticipated that this testing will be conducted at QA Level 3. Some of the strategies for ensuring quality include statistical analysis and redundancy factors such as running multiple samples and testing identical samples using different laboratories and procedures.

2.0 GENERAL TECHNICAL CONSIDERATIONS

2.1 Liner Materials

The shipping container liner provides a barrier between the waste material(s) and the container structure. The liner itself is not expected to experience significant structural loads in service. Emphasis for liner material selection is on chemical compatibility. Liner material properties potentially most affected by chemical exposure are dimensional stability, permeability, and hardness. Stress cracking in the presence of some chemicals may also occur.

For the wide range of potential wastes to be shipped, only a few types of polymeric liner materials need to be considered. Liner materials for hazardous waste landfills have been tested for chemical compatibility with a variety of wastes and may be used as a guide for selecting shipping container liners (Refs. 4 through 9). EPA Standard 9090A (Ref. 7) separates liner materials into the following categories:

- ❖ **Thermoplastic materials** -- chlorinated polyethylene (CPE), chlorosulfonated polyethylene (CSPE), ethylene interpolymer alloy (EIA), polyvinyl chloride (PVC), polyvinyl chloride-chlorinated polyethylene alloy (PVC-CPE), thermoplastic nitrile-polyvinyl chloride (TN-PVC).
- ❖ **Vulcanized materials** -- butyl rubber, ethylene propylene diene monomer (EPDM), cross-linked chlorinated polyethylene (CM), epichlorohydrin polymers (CO and ECO), polychloroprene (CR).
- ❖ **Semicrystalline materials** -- high-density polyethylene (HDPE), high-density polyethylene/rubber alloy (HDPE-A), linear low-density polyethylene (LLDPE), polyethylene ethylene/propylene alloy (PE-EP-A), and thermoplastic ethylene propylene diene monomer (T-EPDM).

Additional materials of potential interest for shipping container liners include polypropylene and fluorocarbons such as polytetrafluoroethylene. Polypropylene has demonstrated good compatibility with high pH radioactive waste materials (Ref. 6). Fluorocarbons are well known for their chemical inertness, but were probably excluded from consideration as landfill liners because of cost considerations. Material costs are not an overriding concern for the small shipping containers under consideration here.

2.2 Seal Materials

Shipping container seals provide a barrier to the waste sample compartment when the lid is closed. In addition to chemical compatibility requirements, seal materials also have mechanical property requirements to function adequately. Mechanical properties that need to be evaluated in conjunction with chemical compatibility include compression set, hardness, and elongation, as well as dimensional stability and permeability.

Under strong oxidizing conditions, alkenes, disulfides, and amines are susceptible to oxidative cleavage. Because of the poor performance seen in the Battelle study (Ref. 4), polyester and polyurethane were eliminated from consideration immediately. Ester, amide, and urethane linkages are all subject to hydrolysis catalyzed by acid or base. To ensure low chemical reactivity and resistance to cleavage, it is our assessment that no carbon-to-carbon multiple bonds, disulfides, amines, amides, or esters should be present in seal and liner materials. Only the following (strong) single bonds are desirable: carbon-to-carbon, carbon-to-oxygen, carbon-to-halogen, silicon-to-fluorine, and silicon-to-oxygen.

Polymeric materials selected for initial screening as potential seals and liners are given in Table 1. These were selected for their mechanical properties and generally good resistance to chemicals, shown in Table 2. Within the various types of polymers given in Table 1, there are additional variables such as molecular weight and cross-link density. In addition, copolymers such as ethylene-perfluoroethylene also need to be considered. Cross-linked polyethylene may also provide a superior seal material. LLDPE includes polyethylenes with densities in the range of about 0.915 to 0.935 g/mL, while HDPE encompasses polyethylene with densities from about 0.935 to 0.970 g/mL (Ref. 9). Table 2 lists some of the primary classes of chemicals with which the polymers of interest are compatible.

2.3 Chemical Waste Streams

The anticipated waste samples may contain a wide range of inorganic, organic, and organometallic chemical species. For example, approximately 18 radioactive elements, 35 nonradioactive metals, and 70 organic chemicals have been identified to date in wastes from Hanford and Oak Ridge (Refs. 10 and 11). Even these characterizations are incomplete; it is expected that additional organic compounds will be identified, including some arising from reactions or radiolysis of waste components. Further complicating the waste handling and disposal problem is the fact that many different waste chemicals are often mixed together. Organic wastes are often commingled with heavy metals, transuranics, and fission products.

Table 1. Potential Seal and Liner Materials with Abbreviations and Selected Trade Names

Polymer	Abbr.	Trade Names
Butadiene-acrylonitrile copolymer	Nitrile	Breon, Buna-N, Butacril, Butakon, Butaprene FR-N, Chemigum, Elaprim, Europrene N, Hycar, Krynac, Marbon, Nilac, Nipol N, Nitrex, Ny Syn, Paracril, Perbunan N, SKN, Tylac
Chloroprene (polychloroprene)	--	Baypren, Butaclor, Butakon, Denkachloropren, Nairit, Neoprene, Switprene
Chlorosulfonated polyethylene	--	Hypalon
Epichlorohydrin rubber	ECH	Herclor, Hydryn
Ethylene propylene diene monomer	EPDM, EPM	Buna AP, Dutral Ter, Epcar, Epsyn, Intolan, Keltan, Mitsui EPT, Nordel, Royalene, Vistalon
Ethylene-propylene rubber	EPR	JSR, Dutral-CO, Epsyn, Epcar, Buna-AP, Intolan, Dypro, NPP
Fluorocarbon rubber	--	Fluorel, Kalrez, Kel-F, Poly-Fab, Tecnaflon, Viton
Fluorosilicone	--	Silastic L. S.
High-density polyethylene	HDPE	Paxon, Super Dylan, Hi-zex, Suntec, Vestolen, Wacker Polyethylen, Rigidex, Scolefin, Hiplex
Isobutylene-isoprene copolymer	Butyl	Bucar Butyl, Enjay-butyl, Exxon Butyl, Hycar-butyl, Petrotex-butyl, Polysar-butyl, Soca-butyl
Linear low-density polyethylene	LLDPE	Dowlex, Marlex TR 130, Ulzex, Innovex, Evaclear, Lotrex, Stamylex, Novapol, Sclair, Ladene
Phosphonitrilic fluoro elastomer	PNF	PNF-200
Polyacrylate	--	Cyanacryl, Hycar
Polypropylene	PP	Bapolene, Fortilene, Profax, Tenite, Noblen, Sunlet, Hostalen PP, Novolen, Vestolen P, Moplen, Lacqtene P, Propathene, Poprolin, Stamyln P, Eltrex P, Frizeta, Daplen
Polysulfide rubber	--	Thiokol
Polytetrafluoroethylene*	PTFE	Teflon, Thermocomp LF-1004, Tefzel HT-2004
Polyvinyl chloride	PVC	Dural, Geon, Kohinor, Vygen, Nipeon, Nipolit, Vinka, Vynchlone, Hostalit, Vestolit, Vinnol, Vinidur, Vinoflex, Trosiplast, Ravinil, Orgavyl, Corvic, Welvic, Benvic, Varlan, Rosevil, Ongrovil, Bovil, Hiplex, Juvinil, Zadrovil
Silicone rubber	--	Silopren, Wacker-Siliconkautschuk, GE Silicon Rubber, ICI Silicon Rubber, KE Rubber, Rhodia, Rhodorsil, Silastene, Silastic, Silastomer, SKT
Styrene-butadiene rubber	SBR	Ameripol, Austrapol, Buna-huls, Buna S, Cariglex S, Carom, Copo, Europrene, FRS, Gentro, GRS, Intol, ISR, Jetron, Krylene, Krynol, Krymix, Nipol, Petroflex, Philprene, Poly bd R 45, Polysar S, Sircis, SKS, Synapren, Ugipol

*May be glass-filled, which increases hardness but does not effect chemical resistance.

Table 2. General Chemical Resistance of Potential Seal and Liner Materials

Polymer Type	Resistance	Seals (S) or Liners (L)
Butadiene-acrylonitrile copolymer	Hydrocarbon oils, lubricants, water-based solutions	S
Chloroprene	Oxygen, freons, ammonia, acids and bases, silicate esters, alcohols and ketones	S
Chlorosulfonated polyethylene	Acids and bases, alcohols, freons, ketones, oxidants	S
Cross-linked polyethylene	Acids and bases, alcohols, ketones, oxidants, radiation	S, L
Epichlorohydrin	Oxygen, ozone, halocarbons, oils, amines	S
Ethylene-propylene rubber	Phosphate esters, water-based solutions, dilute acids and bases, ketones, alcohols, silicones, radiation	S
Fluorocarbon elastomers	Aliphatic and aromatic hydrocarbons, silicate esters, halocarbons, phosphate esters, bases, acids (except acetic)	L
Fluorosilicone	Amines, chlorinated biphenyls, hydrocarbon oils and fuels	S
Isobutylene-isoprene copolymer	Phosphate esters, ketones, silicones	S
Phosphonitrilic fluoro elastomer	Aromatic and aliphatic hydrocarbons, oxygen, ozone, esters	S
Polyacrylate	Hydrocarbon oils, oxidizing environments (ozone)	L
Polyethylene (all densities)	Acids and bases, alcohols, ketones, oxidants, radiation	L
Polypropylene	Acids and bases, aliphatic and aromatic hydrocarbons, water, radiation	L
Polysulfide	Acids and bases, most organics, water, oxygen	S
Polytetrafluoroethylene	Acids and bases, alcohols, ketones, hydrocarbons, oxidants	S, L
Polyvinyl chloride	Alcohols, acids and bases, oils, radiation	L
Silicone	Alcohols, bases, gases	S
Styrene-butadiene rubber	Acids and bases, alcohols and ketones, water, oxygen	S

Generation of gases by radiolysis must also be considered. Radiolysis of organics, nitrates, and nitrites is known to generate significant quantities of hydrogen (H₂) and nitrous oxide gas (N₂O) (Ref. 12). The mixture of these gases in air can be explosive. In designing seals and liners, the permeability to these gases and the ability to withstand pressure buildup must be considered. The rate of gas generation is highly temperature-dependent. Because the wastes will be removed from an elevated-temperature environment in the storage tanks (up to 140 F) and transported at ambient temperature, gas generation will be greatly reduced during transport.

Some insight on possible DOE mixed LLW streams can be obtained from examining commercial mixed LLW streams. Statistics on generation, storage, and treatment of some of the major commercial mixed LLW streams of concern in 1990 are given in Table 3 (Refs. 1 and 13).

Detailed preliminary analyses have been published on wastes at the Hanford and Oak Ridge sites (Ref. 11). The approximately 70 organic chemicals identified in these mixed wastes include a wide range of chemical classes: alcohols, aldehydes, alkanes, alkenes, aromatic hydrocarbons, chlorofluorocarbons (CFCs), chlorinated hydrocarbons, ketones, and nitriles. Major compounds identified include acetonitrile, acetone, 1-butanol, undecane, dodecane, methylundecane, tridecene, and tridecane. The concentration ranges for the relative amounts of chemicals listed in Tables 5 through 7 are defined in Table 4. Organic chemicals identified to date in DOE mixed LLW are listed in Table 5 with their chemical classes and relative amounts (Refs. 3, 10, and 11). Table 6 lists nonradioactive elements and cations identified in inorganic and organometallic compounds (Refs. 4 and 10). Table 7 lists the inorganic anions, and Table 8 lists radionuclides identified in DOE mixed LLWs (Refs. 1, 3, 4, 10, and 11).

Table 3. Characterization of Commercial Mixed Low-Level Wastes in the U.S. in 1990

Hazardous Stream	Waste Volume in m ³		
	Generated	Stored	Treated
liquid scintillation fluids	2837	363	3372
waste oil	149	178	139
chlorinated organics	71	27	23
chlorofluorocarbons	113	255	4
other organics	275	118	259
metals (lead, mercury, chromium, and cadmium)	123	1018	12
aqueous corrosives	80	12	3

Table 4
Definitions of Relative Amounts
in Tables 5 through 7

Category	g/L	ppb (micrograms/mL)
high	>100	>100,000
medium	>1 to 100	>1,000 to 100,000
low	0.01 to 1	10 to 1,000
trace	<0.01	<10

Table 5. Possible Organics in Waste Samples

Name	Formula	Class	Relative Amount
1,1,1-Trichloroethane	CCl_3CH_3	Chlorinated alkane	Unknown
1,1-Dichloroethylene	$\text{CCl}_2=\text{CH}_2$	Chlorinated alkene	Medium
1-Butanol	$\text{HO}(\text{CH}_2)_3\text{CH}_3$	Alcohol	High
1-Butene	$\text{CH}_2=\text{CHCH}_2\text{CH}_3$	Alkene	Medium
1-Pentene	$\text{CH}_2=\text{CHCH}_2\text{CH}_2\text{CH}_3$	Alkene	Medium
1-Propanol	$\text{HO}(\text{CH}_2)_2\text{CH}_3$	Alcohol	Medium
2,3-Dimethylpentane or 2-Pentanone ^a	$\text{CH}_3\text{CH}(\text{CH}_3)\text{CH}(\text{CH}_3)\text{CH}_2\text{CH}_3$ or $\text{CH}_3\text{CO}(\text{CH}_2)_2\text{CH}_3$	Hydrocarbon or ketone	Medium
2,6-Dimethylnonane	$(\text{CH}_3)_2(\text{CH}_2)_4\text{CH}(\text{CH}_3)(\text{CH}_2)_2\text{CH}_3$	Alkane	Medium
2-Butyl-1,1,3-trimethylcyclohexane	$\text{C}_{13}\text{H}_{26}$	Alkane	Medium
2-Heptanone	$\text{CH}_3\text{CO}(\text{CH}_2)_5\text{CH}_3$	Ketone	Medium
2-Hexanone	$\text{CH}_3\text{CO}(\text{CH}_2)_4\text{CH}_3$	Ketone	Medium
2-Methylbutane	$(\text{CH}_3)_2\text{CHCH}_2\text{CH}_3$	Alkane	Low
2-Methylheptane	$(\text{CH}_3)_2\text{CH}(\text{CH}_2)_4\text{CH}_3$	Alkane	Low
2-Methylpentane	$(\text{CH}_3)_2\text{CH}(\text{CH}_2)_2\text{CH}_3$	Alkane	Medium

**Table 5. Possible Organics in Waste Samples
(continued)**

Name	Formula	Class	Relative Amount
2-Methylpropane	$(\text{CH}_3)_3\text{CH}$	Alkane	Medium
2-Octanone	$\text{CH}_3\text{CO}(\text{CH}_2)_6\text{CH}_3$	Ketone	Medium
2-Propanol	$(\text{CH}_3)_2\text{COH}$	Alcohol	Medium
3-Heptanone	$\text{CH}_3\text{CH}_2\text{CO}(\text{CH}_2)_3\text{CH}_3$	Ketone	Medium
3-Methyl-1-butene	$\text{CH}_2=\text{CHCH}(\text{CH}_3)_2$	Alkene	Low
4-Methyl-2-pentanone	$\text{CH}_3\text{COCH}_2\text{CH}(\text{CH}_3)_2$	Ketone	Medium
6-Methyl-2-heptanone	$\text{CH}_3\text{CO}(\text{CH}_2)_3\text{CH}(\text{CH}_3)_2$	Ketone	Medium
Acetaldehyde	CH_3CHO	Aldehyde	Low
Acetone	CH_3COCH_3	Ketone	High
Acetonitrile	CH_3CN	Nitrile	High
Benzene	C_6H_6	Aromatic	Medium
Butanal	$\text{CH}_3(\text{CH}_2)_2\text{CHO}$	Aldehyde	Medium
Butane	$\text{CH}_3(\text{CH}_2)_2\text{CH}_3$	Alkane	Medium
Butanenitrile	$\text{CH}_3(\text{CH}_2)_2\text{CN}$	Nitrile	Medium
C5-Substituted cyclohexane ^a	$\text{C}_{11}\text{H}_{22}$	Alkane	Medium
Carbon disulfide	$\text{S}=\text{C}=\text{S}$	Sulfur-containing	Medium
Carbon tetrachloride	CCl_4	Chlorinated	Unknown
Carbonyl sulfide	$\text{O}=\text{C}=\text{S}$	Sulfur-containing	Medium
Chlorobenzene	$\text{C}_6\text{H}_5\text{Cl}$	Chlorinated, aromatic hydrocarbon	Unknown
Chloroform	CHCl_3	Chlorinated	Unknown
<i>cis</i> -2-Butene	$\text{CH}_3\text{CH}=\text{CHCH}_3$	Alkene	Medium
Citric acid	$\text{HOCH}(\text{CH}_2\text{COOH})_2$	Organic acid	Unknown
Cyclohexane	$\text{cyclo}-(\text{CH}_2)_6$	Aliphatic hydrocarbon	Unknown
Decane	$\text{CH}_3(\text{CH}_2)_8\text{CH}_3$	Aliphatic hydrocarbon	High
Decene ^a	$\text{C}_{10}\text{H}_{20}$	Alkene	Medium

Table 5. Possible Organics in Waste Samples(continued)

Name	Formula	Class	Relative Amount
Diethylenetriamine-pentaacetic acid (DTPA)	$\text{HO}_2\text{CCH}_2\text{N}(\text{CH}_2\text{CH}_2\text{N}(\text{CH}_2\text{CO}_2\text{H})_2)_2$	Chelant	Unknown
Dodecane	$\text{CH}_3(\text{CH}_2)_{10}\text{CH}_3$	Aliphatic hydrocarbon	High
Ethanol	$\text{CH}_3\text{CH}_2\text{OH}$	Alcohol	Medium
Ethyl acetate ^{b,c}	$\text{CH}_3\text{COOCH}_2\text{CH}_3$	ester	Unknown
Ethylbenzene	$\text{C}_6\text{H}_5\text{CH}_2\text{CH}_3$	Aromatic hydrocarbon	Unknown
Ethylenediamine tetraacetate	$((\text{HOOC})_2\text{NCH}_2)_2$	Chelant	Unknown
Formic acid	HCOOH	Organic acid	Unknown
Heptane	$\text{CH}_3(\text{CH}_2)_5\text{CH}_3$	Alkane	Medium
Heptanenitrile	$\text{CH}_3(\text{CH}_2)_5\text{CN}$	Nitrile	Low
Hexane	$\text{CH}_3(\text{CH}_2)_4\text{CH}_3$	Alkane	Medium
Hexanenitrile	$\text{CH}_3(\text{CH}_2)_4\text{CN}$	Nitrile	Medium
Methyl ethyl ketone (butanone)	$\text{CH}_3\text{COCH}_2\text{CH}_3$	Ketone	Unknown ^b
Methyl isobutyl ketone (hexone)	$\text{CH}_3\text{CO}(\text{CH}_2)_4\text{CH}_3$	Ketone	High
Methylene chloride	CH_2Cl_2	Chlorinated	Low
Methylundecane ^a	$\text{C}_{12}\text{H}_{26}$	Alkane	High
Nitrilotriacetic acid (NTA)	$\text{N}(\text{CH}_2\text{COOH})_3$	Chelant	Unknown
Nonane	$\text{CH}_3(\text{CH}_2)_7\text{CH}_3$	Alkane	Medium
Octane	$\text{CH}_3(\text{CH}_2)_6\text{CH}_3$	Alkane	Medium
Oxalic acid	HOOCCOOH	Organic acid	Unknown
Pentane	$\text{CH}_3(\text{CH}_2)_3\text{CH}_3$	Alkane	Medium
Pentanenitrile	$\text{CH}_3(\text{CH}_2)_3\text{CN}$	Nitrile	Medium
Perchloroethylene	$\text{CCl}_2=\text{CCl}_2$	Chlorinated	Unknown

Table 5. Possible Organics in Waste Samples (concluded)

Name	Formula	Class	Relative Amount
Propane	CH ₃ CH ₂ CH ₃	Alkane	Medium
Propanenitrile	CH ₃ CH ₂ CN	Nitrile	Medium
Propene	CH ₂ =CHCH ₃	Alkene	Medium
Propyne	HCCCH ₃	Alkyne	Low
Phthalic acid	ortho-C ₆ H ₄ (COOH) ₂	Organic acid	Unknown
Polychlorinated biphenyls (PCBs)	C ₁₂ H ₅ Cl ₅	Aromatic Hydrocarbon, Chlorinated	Unknown
Propanal	CH ₃ CH ₂ CHO	Aldehyde	Medium
Tetradecene	CH ₂ =CH(CH ₂) ₁₁ CH ₃	Alkene	Medium
Tetrahydrofuran or crotonaldehyde	cyclo-(CH ₂) ₄ O	Ether or Aldehyde	Medium
Toluene	C ₆ H ₅ CH ₃	Aromatic hydrocarbon	Medium
Trans-2-butene	CH ₃ CH=CHCH ₃	Alkene	Low
Tributyl phosphate	(<i>n</i> -BuO) ₃ PO	Phosphate ester	Unknown
Trichloroethylene	CHCl=CCl ₂	Chlorinated	Medium
Trichlorofluoromethane (CFC-11)	CFCl ₃	Chlorofluorocarbon	Medium
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113)	CCl ₂ FCClF ₂	Chlorinated	Low
Tridecadiene ^a	C ₁₃ H ₂₄	Alkene	Medium
Tridecane	CH ₃ (CH ₂) ₁₁ CH ₃	Alkane	Medium
Tridecene ^a	C ₁₃ H ₂₆	Alkene	High
Undecane	CH ₃ (CH ₂) ₉ CH ₃	Alkane	High
Undecene ^a	C ₁₁ H ₂₂	Alkene	Medium
Xylene	<i>o</i> , <i>m</i> , and <i>p</i> -C ₆ H ₄ (CH ₃) ₂	Aromatic Hydrocarbon	Unknown

a. Incomplete structural determination reported.

b. A common solvent speculated by the current authors to be present, but not yet reported.

c. If originally present, may have undergone hydrolysis to the corresponding carboxylic acid and alcohol under the highly alkaline storage conditions.

Table 6. Possible Nonradioactive Elements and Cations in Inorganic and Organometallic Compounds in Waste Samples

Name	Symbol	Possible Net Charge							Relative Amount
		0	1	2	3	4	5	6	
Aluminum	Al	❖			❖				Low
Ammonium	NH ₄		❖						Not reported
Antimony	Sb				❖		❖		Low
Arsenic	As				❖		❖		Low
Barium	Ba			❖					Low
Beryllium	Be			❖					Trace
Bismuth	Bi				❖		❖		Trace
Boron	B				❖				Low
Cadmium	Cd	❖		❖					Trace
Calcium	Ca			❖					Medium
Cerium	Ce				❖	❖			Trace
Chromium	Cr			❖	❖			❖	Low
Cobalt	Co	❖		❖	❖				Trace
Copper	Cu	❖	❖	❖					Low
Gallium	Ga				❖				Low
Hafnium	Hf					❖			Trace
Iron	Fe	❖		❖	❖				Low
Lanthanum	La				❖				Trace
Lead	Pb	❖	❖	❖					Low
Lithium	Li		❖						Low
Magnesium	Mg	❖		❖					Medium
Manganese	Mn			❖		❖			Trace
Mercury	Hg	❖	❖	❖					Trace
Molybdenum	Mo			❖	❖	❖	❖	❖	Trace
Nickel	Ni	❖	❖	❖					Low
Potassium	K		❖						Medium
Phosphorus	P				❖	❖	❖		Low
Selenium	Se					❖		❖	Low
Silicon	Si					❖			Low

Table 6. Possible Nonradioactive Elements and Cations in Inorganic and Organometallic Compounds in Waste Samples (concluded)

Name	Symbol	Possible Net Charge							Relative Amount
		0	1	2	3	4	5	6	
Silver	Ag	❖	❖						Trace
Sodium	Na		❖						High
Strontium	Sr			❖					Low
Sulfur	S	❖		❖		❖		❖	Low
Titanium	Ti	❖			❖	❖			Low
Tungsten	W	❖		❖	❖	❖	❖	❖	Low
Vanadium	V	❖		❖	❖	❖	❖		Trace
Zinc	Zn	❖		❖					Low
Zirconium	Zr					❖			Trace

Table 7. Inorganic Anions Expected in Waste Samples

Anion	Formula	Net Charge			Relative Amount
		-3	-2	-1	
Arsenate	AsO ₃		❖		Not reported
Bismuthate	BiO ₃			❖	Not reported
Borate	B ₄ O ₇		❖		Not reported
Bromide	Br			❖	Not reported
Carbonate	CO ₃		❖		Not reported
Chloride	Cl			❖	Medium
Dichromate	Cr ₂ O ₇		❖		Medium
Ferrocyanide	Fe(CN) ₆	❖			Medium
Fluoride	F			❖	Low
Hydroxide	OH			❖	High
Nitrate	NO ₃			❖	High
Nitrite	NO ₂			❖	High
Phosphate	PO ₄	❖			Medium
Selenate	SeO ₄		❖		Trace
Sulfate	SO ₄		❖		Medium

Table 8. Possible Radionuclides in Waste Samples

Name	Symbol	Isotope(s)	Type of Emitter			Possible Oxidation State							
			α	β	γ	0	1	2	3	4	5	6	7
Americium	Am	241	❖					❖ ^a	❖	❖	❖	❖	
Barium	Ba	137			❖			❖					
Carbon ^b	C	14		❖		❖	❖	❖	❖	❖			
Cesium ^c	Cs	134, 137		❖	❖		❖						
Cobalt	Co	60		❖	❖			❖	❖				
Curium	Cm	244	❖						❖	❖ ^a			
Europium	Eu	152, 154, 155		❖	❖			❖	❖				
Gold	Au	198				❖	❖		❖				
Hydrogen (tritium)	H or T	3		❖		❖	❖						
Niobium	Nb	95			❖				❖		❖		
Plutonium	Pu	238, 239, 240	❖						❖	❖	❖	❖	❖ ^a
Ruthenium ^d	Ru	106		❖				❖	❖	❖		❖	
Strontium ^c	Sr	90		❖				❖					
Technetium	Tc	99		❖									❖
Thorium	Th	232	❖						❖ ^a	❖			
Uranium	U	233, 235, 238	❖			❖			❖	❖	❖	❖	
Yttrium	Y	90		❖					❖				
Zirconium	Zr	95			❖					❖			

a. Less common oxidation state.
b. Negative oxidation states from -1 to -4 are also possible.
c. Present in high levels.
d. Oxidation state of +8 also possible.

Because of the wide variety of wastes expected, it is desirable that potential seal and liner materials be resistant to a broad range of chemicals. Resistance to chlorinated solvents, aliphatic and aromatic hydrocarbons, oxidants, base, and other inorganic ions (such as metals, nitrate, nitrite, sulfate, and phosphate) must all be considered.

Chlorinated solvents include 1,1,1-trichloroethane (TCA), trichloroethylene (TCE), chlorofluorocarbon-113 (CFC-113 or 1,1,2-trichloro-1,2,2-trifluoromethane), methylene chloride, chloroform, and carbon tetrachloride. Aliphatic hydrocarbons include hexane, heptane, octane, nonane, decane, undecane, dodecane, and petroleum distillates such as naphtha, kerosene, and mineral spirits. Aromatic hydrocarbons contain a benzene ring as part of their structure. Examples of this group include benzene, toluene, xylene, and chlorobenzene.

Common oxidants used by chemical laboratories and industry include dichromates and perchlorates, but these are not expected to be present in significant quantities in the mixed wastes because they were not widely used, were usually disposed of separately when used (because of their high oxidizing ability), and any trace quantities present in mixed wastes are likely to have reacted with traces of other chemicals, destroying the strong oxidizing agents.

In many cases, most chemical constituents are only present in trace amounts and are unlikely to have a significant effect upon liner and seal materials. Some waste streams consist of aqueous solutions containing the following ions: approximately 5.5 M sodium, 2.1 M nitrate, 0.7 M nitrite, 2.0 M hydroxide, and trace quantities of other cations, anions, organics, and radionuclides. These aqueous samples are strongly basic (with a pH near 14). Other waste streams consist of chlorinated or aromatic hydrocarbon solvents with traces of radionuclides. For example, 1,1,1-trichloroethane and CFC-113 have been used for uranium processing at the Rocky Flats Plant (Ref. 14). Scintillation test solutions from many sites reportedly contain aromatic hydrocarbons and dioxane.

2.4 Radiation

Several units of measure are used to describe the absorption of high-energy radiation by materials. The most common is the rad, which is being replaced by the new standard international (SI) unit of the Gray (Gy). Conversion among common units is shown in Equation (1).

$$1 \text{ Gy} = 100 \text{ rad} = 1 \text{ J/kg} = 10^4 \text{ erg/g} = 6.24 \times 10^{15} \text{ eV/g} \quad (1)$$

Plastics in general are relatively unaffected by radiation up to 10^4 Gy (10^6 rad) and are relatively easy to decontaminate (Refs. 15 through 17). Many plastics have been used on a large scale as components of nuclear reactors (Ref. 16). Concerns about radiation effects on plastics begin at exposure levels of about 10^5 Gy (10^7 rads) (Refs. 15 and 17). Near 10^5 Gy (10^7 rads) significant differences are observed among elastomers. The property most affected by gamma radiation is compression set. Set means a reduction in flexibility leading to the inability to return to the original shape. After exposure to 10^6 Gy (10^8 rads), all elastomers tested took over 85% set, a level expected to cause leakage. At levels higher than 10^5 Gy (10^7 rads), elastomeric seals should not be used.

For most plastics, high radiation doses induce cross-linking, making the plastics more brittle. At doses of several million rad (abbreviated megarad or Mrad), most polymers become harder to stretch, and the break elongation decreases dramatically (Ref. 18). For other plastics, radiation induces chain scission, which degrades the plastic and makes it weaker. Lists of polymers that undergo cross-linking and chain scission in the presence of radiation are found in References 16 and 17.

In general, polymers are sensitive only to total radiation dose and not to dose rate or the type of radiation. Average levels of about 10 to 30 Curies/m³ or approximately 0.01 to 0.03 Curies/liter are expected in LLW samples (Ref. 1). Even allowing for large (30- to 100-fold) variability in samples, a maximum of 1 Curie/liter should not be exceeded in the waste samples. Assuming a gamma ray energy equivalent to that from Cesium-137, a level of 1 Curie/liter corresponds to approximately 80 Gy/yr (8,000 rads/yr) being deposited in the liner and seal materials per liter of sample waste, which is a negligible amount. The calculation of 80 Gy/yr (8000 rads/yr) is made as follows. One megacurie of gamma radiation from Cobalt-60 produces a dose rate of 5 Gy/sec (500 rad/sec) in polymers. Because the gamma radiation is about half as energetic (per photon) from Cesium-137 as from Cobalt-60, Cesium-137 produces a dose rate of about 2.5 Gy/sec (250 rad/sec) (Ref. 4). Thus, one Curie of Cesium-137 per liter produces about 2.5×10^{-6} Gy/sec (2.5×10^{-4} rads/sec), which corresponds to about 80 Gy/yr (8,000 rads/yr).

The concern level of 10^7 rads is thus several orders of magnitude higher than any anticipated radiation exposures of the seals in this application. In order to approach an exposure of 10^7 rads, a seal or liner would have to be exposed continuously to a sample of far above average radioactivity for over 1,000 years. Because the expected radioactivity of the sample is one to two orders of magnitude lower than 1 Curie/liter and the expected total exposure time is on the order of months (three orders of magnitude shorter than 1,000 years), there is a margin of safety of four to five orders of magnitude before radiation exposure would be expected to affect the seals or liners.

Because of the LLWs, the distance from the sample container, the probable regular replacement of seals and liners, and the generally high resistance of polymers to radiation, it is not anticipated that the seals and liners considered in this study will be subjected to high enough radiation levels to cause any noticeable changes in properties. It has also been observed that radiation does not significantly affect chemical compatibility, so no synergistic destructive effects are expected (Ref. 6).

2.5 Chemical Compatibility

Polymer-environment interactions can be either reversible (absorption leading to plasticization and swelling) or irreversible (oxidation). These may also be referred to as physical (reversible) or chemical (irreversible) interactions, although the physical interactions have a significant chemical aspect in the breaking of secondary interchain bonds. In general, polymers are resistant to weak acids, weak bases, and salt solutions (Refs. 19 through 23). Strong acids can oxidize polymers leading to embrittlement. Such an effect is often seen in polyolefins. Fuels, fats, oils, and organic solvents cause swelling, softening, and eventually dissolution. These mechanisms are diffusion controlled. Most chemical degradation is system-specific for a particular polymer and fluid or gas. It is

unlikely that significant chemical compatibility information between polymers and the complex waste mixtures can be found in the literature; therefore, the materials selected will require compatibility testing with simulated wastes.

Few polymers or plastics are commercially available in pure form. Most contain antioxidants, plasticizers, heat stabilizers, processing aids, residual catalysts, and other additives and impurities. The chemical resistance of the material will depend upon the amount and type of these compounds present. The wide range of formulations presents the potential for lot-to-lot variations or supplier-to-supplier variations in chemical resistance for supposedly identical materials. If it is determined that such variability poses a significant chemical compatibility threat, then a plan to screen lots of material for compatibility will need to be implemented.

The most significant common threats to polymers (oxygen, ozone, and water) are probably not important to the selection of seal and liner materials with the exception of condensation polymers such as polyesters, polyamides, or urethane-based materials that are subject to hydrolysis. Absorption of strong alkali, organic solvents, and oxidants appear to constitute the main threats from the wastes of interest.

Solvent absorption leads to separation of thermoplastic chains without breaking primary bonds. Only secondary, interchain forces (Van der Waals or acid/base interactions) are broken. Secondary forces are less than one-tenth as strong as primary bonds. Since primary bonds are not involved, the interaction of solvents with thermoplastic polymers is similar to that of lower molecular weight organic compounds, and solubility parameters can be used as a guide to compatibility behavior.

Permeability is a key variable in the solvent degradation process. Polymers have an open structure or free volume content that allows the passage of small molecules. Reorganization of interchain bonds allows redistribution of free volume, which permits the subsequent diffusion of solvent molecules. Pressure gradients and solvation effects increase the rate of permeation. Larger molecules require larger molecular holes and have a slower diffusion rate. Diffusion in elastomers occurs much more easily when the elastomer is above its glass transition temperature, because of the larger free volume. Elastomeric seal materials at room temperature are above their glass transition temperatures, and diffusion may occur. Chemical reaction between the permeant and the polymer can significantly alter permeability. Permeability is a function of polymer composition, fluid or gas composition, temperature, and geometry. For a given polymer-permeant combination, Equation 2 gives the relationship between the amount of fluid penetrating and permeability coefficient (Ref. 19).

$$q = PtA(p_1 - p_2)/l \quad (2)$$

where

- q = quantity of fluid permeating (in g)
- P = permeability coefficient (in g/m • sec • Pa)
- t = time (in sec)
- A = exposed area (in m²)
- p₁, p₂ = partial pressures of fluid on two sides of material (in Pa)
- l = material thickness (in m).

Physical stress or chemical reaction can accelerate the permeation predicted from Equation 2. Permeation is generally limited to amorphous polymer regions, although, in some cases, permeants can solvate crystallites in semicrystalline polymers.

Solvent-induced changes can include environmental stress cracking, plasticization, solvent crazing, softening, swelling, and delamination. In most cases, the polymer must be engineered to resist the anticipated environment. Environmental stress cracking is limited to partially crystalline materials (liner materials) and is a surface phenomenon acting at stressed surfaces. The solvent acts to increase local stress. Solvent viscosity, surface tension, and wettability are important factors; solubility is not. Polymer yield stress is also important. Stresses may be either applied or residual from the fabrication process. Liner materials should be well annealed to reduce the probability of environmental stress cracking.

Solvent crazing is similar to environmental stress cracking but takes place in the bulk plastic. It can also occur in both amorphous and crystalline materials. Thorough annealing is also recommended to reduce susceptibility to solvent crazing.

Plasticization requires that the free energy of interaction between permeant and polymer be less than the polymer-polymer interaction energy. Plasticization effects can be estimated from solubility parameters.

Metal ions and most common inorganic ions generally do not significantly affect polymers. Potential threats include strong acid or base, oxidizing agents, oils, and solvents such as chlorinated, aliphatic, and aromatic hydrocarbons. Under strong oxidizing conditions, alkenes, disulfides, and amines are susceptible to oxidative cleavage. Because of the poor performance seen in the Battelle study (Ref. 4), polyester and polyurethane were eliminated from consideration immediately. Ester, amide, and urethane linkages are all subject to hydrolysis catalyzed by acid or base.

To ensure low chemical reactivity in general, it is our assessment that no carbon-to-carbon multiple bonds, disulfides, amines, amides, or esters should be present in seal and liner materials. Only the following (strong) single bonds are desirable: carbon-to-carbon, carbon-to-oxygen, carbon-to-halogen, silicon-to-fluorine, and silicon-to-oxygen. Radiation is less selective than chemical attack, and sufficiently energetic radiation can cleave all types of chemical bonds. It is also possible in principle that interactions of radiation with adsorbed compounds could initiate reactions that would degrade the polymer host. However, the quantity of radiation exposure in this application is extremely low and no noticeable effects are expected from degradation caused by radiation.

Reported information on the chemical compatibility of selected liner and seal materials is summarized in Table 9 (Refs. 9, 15, 16, and 24 through 31). For each polymer and each type of chemical, the compatibility is summarized as being excellent, good, or poor. The literature data are reported using several different ranking schemes. The classifications outstanding, excellent, and resistant have all been combined to the single rating excellent; the descriptors very good, good, and slight to moderate swelling have been combined in the category good; and the terms fair, poor, unsatisfactory, excessive swelling, and surface attack have been combined into the ranking poor. When different members of

a class (*e.g.*, different aqueous acids) gave widely varying compatibility for the same polymer or when widely conflicting reports have been published, the rating of "varies" was assigned. In the radiation resistance column, "poor" means noticeable changes in properties occur between 10^5 and 10^6 rads, "good" means noticeable changes in properties occur between 10^6 and 10^7 rads; "exc." (an abbreviation for excellent) means that noticeable changes in properties occur above 10^7 rads.

Table 9. Chemical Resistance of Potential Seal and Liner Materials

Polymer	ALCO- HOLS	ALIPHATIC HYDRO- CARBONS	AQUEOUS ACID	AQUEOUS BASE	AROMATIC HYDRO- CARBONS	CHLOR- INATED	ESTERS	KETONES	OILS	OXIDANTS	RADIA- TION	WATER
Butadiene-acrylonitrile copolymer	good	exc.	poor	poor	good	poor	poor	poor	good	poor	exc.	good
Chloroprene	exc.	good	varies	poor	poor	poor	poor	poor	good	varies	exc.	good
Chlorosulfonated polyethylene	exc.	good	exc.	exc.	poor	poor	poor	poor	good	exc.	exc.	good
Epichlorohydrin	poor	good	poor	poor	exc.	exc.	poor	poor	exc.	exc.	poor	good
Ethylene propylene diene monomer	exc.	poor	good	exc.	poor	poor	exc.	exc.	poor	good	good	exc.
Ethylene-propylene rubber	exc.	poor	good	exc.	poor	poor	exc.	exc.	poor	exc.	exc.	exc.
Fluorocarbon rubber	exc.	exc.	poor	poor	exc.	exc.	good	poor	exc.	exc.	good	good
Fluorosilicone	exc.	good	poor	good	good	poor	good	poor	good	exc.	good	exc.
Isobutylene-isoprene copolymer	good	poor	good	exc.	poor	poor	good	exc.	poor	good	good	poor
Phosphonitrilic fluoro elastomer	poor	exc.	poor	good	exc.	poor	poor	poor	exc.	poor*	good*	poor
Polyacrylate	good	good	poor	poor	poor	poor	poor	poor	exc.	exc.	good	poor
Polyethylene (cross-linked, linear low-density, and high-density)	good	poor	good	good	poor	poor	poor	good	poor	good	exc.	exc.
Polypropylene	good	good	exc.	exc.	good	poor	good	good	good	poor	exc.	exc.
Polysulfide	exc.	exc.	poor	varies	good	poor	good	good	good	exc.	poor*	exc.
Polytetrafluoroethylene	exc.	exc.	exc.	exc.	exc.	good	exc.	exc.	exc.	exc.	exc.	exc.
Polyvinyl chloride	exc.	poor	exc.	exc.	poor	poor	poor	poor	exc	good	exc.	exc.
Silicone	exc.	poor	varies	exc.	poor	poor	exc.	good	good	poor	exc.	poor
Styrene-butadiene rubber	exc.	poor	good	exc.	poor	poor	poor*	exc.	poor	poor	exc.	exc.

*Conservative (low) estimates by the authors based on chemical structures and compatibilities of similar polymers.

Table 10 lists the hardness, high- and low-temperature limits, and compression set resistance of the polymers under consideration (Refs. 15, 16, and 31). Hardness is the resistance of a material to indentation deformation. Because plastics vary over a wide range of hardness, a single type of hardness test is not applicable to all plastics. Durometer hardness testing is used for softer materials. Hardness is reported as a dimensionless number on either the A or D scale. The two hardness tests differ in the shape and dimensions of the indenter. Type A is softer than Type D.

Table 10. Hardness, High- and Low-Temperature Limits, and Compression Set Resistances of Polymers

Polymer	Hardness (A or D scale)	Cont. High-Temp Limit (°C)	Low-Temp. Limit (°C)	Compression Set Resistance
Butadiene-acrylonitrile copolymer	A 40-90	110 to 135	-20 to -55	very good
Chloroprene	A 40-80	90 to 150	-30 to -55	good
Chlorosulfonated polyethylene	A 50-90	120 to 125	-30 to -55	fair to poor
Cross-linked polyethylene	D 60-80 ^a	80	N/A	fair to good ^a
Epichlorohydrin	A 50-90	125 to 150	-40 to -55	fair to good
Ethylene propylene diene monomer	A 50-90	120 to 150	-50 to -55	very good
Ethylene-propylene rubber*	A 50-90 ^a	120 to 150 ^a	-50 to -55 ^a	very good ^a
Fluorocarbon rubber	A 70-90	190 to 225	-25 to -40	very good
Fluorosilicone	A 60-80	175	-65 to -75	very good
High-density polyethylene	D 60-70	50	N/A	fair
Isobutylene-isoprene copolymer	A 50-70	100 to 120	-30 to -55	fair to good
Linear low-density polyethylene	D 45-53	50	N/A	poor
Phosphonitrilic fluoro elastomer	A 50-90	175	-65	good
Polyacrylate	A 70-90	175	-20	fair
Polypropylene	D 70-80	120	N/A	fair ^a
Polysulfide rubber	A 50-80	100 to 120	-50 to -55	fair
Polytetrafluoroethylene	D 55-75	260	-40 to -50	fair to poor
Polyvinyl chloride	D 75-80	75	N/A	good
Silicone rubber	A 40-80	230 to 250	-75 to -115	excellent
Styrene-butadiene rubber	A 40-80	70 to 100	-30 to -55	good
* Estimates by the authors based on properties of similar polymers.				

3.0 STANDARD COMPATIBILITY TESTS

3.1 Background

The compatibility or effectiveness of a barrier material (seal or liner) with a waste environment depends on the composition and temperature of the waste stream, the duration of exposure, and the allowable leak rate. A material that is suitable for one type of waste environment may deteriorate when exposed to a different waste stream. If a material provides a hermetic seal for a year but rapidly degrades at the end of that period, it would be suitable only for short-term exposures. A seal that leaks at a rate of one gram per year would be adequate for some wastes, but would be intolerable for something as hazardous as plutonium-containing waste. A seal might work perfectly at room temperature or higher and fail catastrophically at low temperature. All factors must be considered when testing for compatibility between barrier materials and waste streams.

The safest way to evaluate the effectiveness of barrier materials is to expose them to standardized simulated waste streams and measure the changes in the physical properties of the materials. Any property that does not initially meet certain criteria would result in the elimination of that material. Where possible, a national standard should be used for the test procedure to ensure that the test is repeatable and that unforeseen errors will not occur.

Table 11 lists the national standards that apply to barrier material testing. The standards are graded as to their applicability for evaluating compatibility of seals and liners for mixed LLW containers. Section 3.3 provides a comparison of test procedures used in the standards. Section 3.4 is a description of equipment needed for the tests. Because universal test machines are an expensive and important part of the testing program, approximate costs are presented.

3.2 Test Methods

Table 11 describes the standard test methods and procedures for the evaluating rubbers, elastomers, and plastics. These tests are generally designed for use with flat pieces of the material and (with some exceptions) are not suitable for testing O-rings. O-ring testing is covered by ASTM Method D1414, which gives the necessary modifications to the other methods for their use with O-rings.

Table 11 is organized into seven columns. The first column lists the organization that developed the test method. The organizations are the American Society for Testing and Materials (ASTM), the American National Standards Institute (ANSI), the National Sanitation Foundation (NSF), the British Standards Institution (BSI), the Deutsches Institut für Normung (DIN), the United States Environmental Protection Agency (EPA), and the International Organization for

Table 11. Standard Test Methods for Rubbers, Elastomers, and Plastics

ORG.	METHOD	TITLE	MATERIAL	NOTES	SEALS	LINERS
ASTM	D297-81	Rubber Products□Chemical Analysis	Rubber	Rubber density and other properties.	2 [*]	2
ASTM	D395-89	Rubber Property□Compression Set	Rubber	Measures residual deformation after application of a deflection or specified force. Dept. of Defense (DoD) approved.	2 [*]	3
ASTM	D412-87	Rubber Properties in Tension	Rubber	Tensile stress, strength, ultimate elongation, and set. Replaces DoD Methods 4001, 4116, 4121, and 4411 of FTMS 601.	2 [*]	2 ^{**}
ASTM	D471-79(91)	Rubber Property□Effects of Liquids	Rubber	Changes in mass and volume after immersion in standard test oils and fuels. References ASTM Standards D412 and D751. Replaces DoD Methods 6001, 6111, 6121, 6211, 6231, and 6251 of FTMS 601.	2 [*]	3
ASTM	D543-87	Resistance of Plastics to Chemical Reagents	Plastics	Change in mass and volume after immersion in standard reagents. Does not address strength loss or other tests. DoD approved.	3	2
ASTM	D573-88	Rubber□Deterioration in an Air Oven	Rubber	Loss of properties caused by accelerated aging.	2 [*]	3
ASTM	D618-61(90)	Conditioning Plastics and Electrical Insulating Materials for Testing	Plastics	Conditioning procedure for other ASTM tests. DoD approved.	3	1
ASTM	D621-64(88)	Deformation of Plastics Under Load	Plastics	Deformation under compression of all plastics, both rigid and nonrigid. Replaces DoD Method 1101 of FTMS 406.	3	2
ASTM	D624-91	Tear Strength of Conventional Vulcanized Rubber and Thermoplastic Elastomers	Elastomers	Tear strength of rubber and thermoplastic elastomers using one of four techniques. DoD approved.	3	2 ^{**}
ASTM	D638-91	Tensile Properties of Plastics	Plastics	Tensile strength, tensile stress, elongation, and modulus of plastics between 0.04 and 0.55 inches thick. DoD approved.	3	1 ^{**}
ASTM	D751-89	Coated Fabrics	Rubber-Fabric	Fifteen different properties of rubber- coated fabrics. DoD approved.	3	2 ^{**}

**Table 11. Standard Test Methods for Rubbers, Elastomers, and Plastics
(continued)**

ORG.	METHOD	TITLE	MATERIAL	NOTES	SEALS	LINERS
ASTM	D790-91	Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials	Plastics	Flexural strength and modulus using three-point or four-point bending. Replaces DoD Method 1031 of FTMS 406.	3	1
ASTM	D792-91	Density and Specific Gravity (Relative Density) of Plastics by Displacement	Plastics	Density and specific gravity of all solid plastics. Replaces DoD Methods 5011 and 5012 of FTMS 406 and Methods 14011 and 14021 of FTMS 601.	2	1
ASTM	D814-86(91)	Rubber Property □ Vapor Transmission of Volatile Liquids	Rubber	Vapor transmission through rubber sheets 2.69 in. in diameter and less than 0.125 in. thick. The test liquid is not specified.	3	1
ASTM	D882-91	Tensile Properties of Thin Plastic Sheeting	Plastics	Tensile strength, tensile strength at break, percent elongation at break, yield strength, percent elongation at yield, elastic modulus, and tensile energy to break for thin (less than 0.04 in.) plastic sheeting. Replaces DoD Method 1013 of FTMS 406.	3	1 ^{**}
ASTM	D945-92	Rubber Properties in Compression or Shear (Mechanical Oscillograph)	Rubber	Measures resilience, static and dynamic modulus, kinetic energy, creep, and set using dynamic techniques on a specialized machine. DoD approved.	1	3
ASTM	D1004-90	Initial Tear Resistance of Plastic Film and Sheeting	Plastics	Tear resistance of flexible plastic film and sheeting at very low rates of loading (2 in./min.). Replaces DoD Method 1121 of FTMS 406.	3	2 ^{**}
ASTM	D1239-55(82)	Resistance of Plastic Films to Extraction by Chemicals	Plastics	Measures weight loss after immersion in standard reagent for a specified period.	3	1
ASTM	D1329	Evaluating Rubber Property □ Retraction at Lower Temperatures (TR Test)	Rubber	Measures temperature at which rubber retracts 10%.	2 [*]	3
ASTM	D1349-87	Rubber □ Standard Temperatures for Testing	Rubber	Standard conditions for testing rubbers. DoD approved.	2	2

**Table 11. Standard Test Methods for Rubbers, Elastomers, and Plastics
(continued)**

ORG.	METHOD	TITLE	MATERIAL	NOTES	SEALS	LINERS
ASTM	D1414-90	Rubber O-Rings	Rubber	Measures tensile strength, ultimate elongation, tensile stress, tension set, compression set, low-temperature retraction, relative density, immersion resistance, heat-aging, hardness, and corrosion-causing tendencies. Extends ASTM Methods D412, D395, D1329, D297, D471, D865, D573, D2240, and D1415. Part of FTMS 601.	1	3
ASTM	D1415	Rubber Property□International Hardness	Rubber	Similar to Durometer hardness, D2240, except suitable for small diameter O-rings.	2*	3
ASTM	D1434	Determining Gas Permeability Characteristics of Plastic Film and Sheeting	Plastics	Measures gas diffusion through a polymer. Similar to ISO 2556 and DIN 53380.	2	1
ASTM	D1693-70(88)	Environmental Stress-Cracking of Ethylene Plastics	Polyethylene	Bent specimens of the plastic are exposed to a surface active agent. DoD approved.	3	1
ASTM	D2240-91	Rubber Property□Durometer Hardness	Rubber	Hardness measurements made based on either initial indentation or indentation after a specified period of time.	2*	2**
ANSI/ NSF	54-1991	Flexible Membrane Liners	Liners	Minimum requirements for flexible membrane liners as measured by standard and modified ASTM procedures. An appendix of ASTM methods referenced is included.	3	1
DIN	53 521-1987	Determination of the behavior of rubber and elastomers when exposed to fluids and vapors	Rubbers and Elastomers	Measures changes in volume and mass. Tests for hardness, tensile strength, and elongation at break are included by reference. The list of test chemicals is similar to ISO/R 175. This standard is an extension of ISO 1817.	2	1
EPA	9090A-1990	Compatibility Test for Wastes and Membrane Liners	Liners	A wide variety of tests are used to measure the durability of membrane liners for hazardous waste. The tests are comprehensive.	2	1
* Used and extended in ASTM D1414.						
** Used in EPA 9090A.						

Standardization (ISO). In the "Method" column, the digits following the dash are the year of the latest revision, followed by the year of last reapproval in parentheses. The "Material" column shows the material covered by the standard. The "Notes" column gives a brief summary of the standard and lists any equivalent standards or replacements. The "Seals" and "Liners" columns rate the applicability of the standard to seals/seal materials and liners/liner materials, respectively. A rating of 1 means that the standard applies and is judged suitable for use in this program. A rating of 2 means the standard applies, but is not needed, not recommended, or is improved by some other standard. A rating of 3 means that the standard does not apply or is essentially identical to some other standard.

EPA Method 9090A is the only standard that attempts to evaluate the chemical resistance of liner materials comprehensively. It should therefore be used as the starting point for selection of appropriate tests. The major concerns for liner materials are permeability, dimensional stability and embrittlement. Puncture resistance and swelling are secondary considerations because the liner will be supported by the canister. In addition to the above concerns, for O-rings swelling and compression set resistance must also be considered. If an O-ring swells or permanently deforms, its sealing properties can be lost, allowing the contents to leak. These concerns were used to select the test methods with a ranking of 1. Note that some test methods are equivalent except for the material tested (*e.g.*, plastics *versus* rubber). An example of this is ASTM Methods D412 and D638. These methods were not ranked the same because it is anticipated that O-rings may contain rubber, whereas liners will not. This same reasoning applies to other tests that are similar.

The regulations of the U.S. Department of Transportation (DOT) and the Nuclear Regulatory Commission (NRC) regarding transport of hazardous and radioactive wastes were reviewed to determine which, if any, standard tests are specified for materials compatibility of polymers used in transport containers. In the DOT regulations (Ref. 32), no tests for polymers are mentioned. The regulations only specify performance and, for some metal containers, materials of construction. For example, DOT regulations state that "The package must be manufactured from suitable plastic material and be of adequate strength in relation to its capacity and intended use." Several ASTM test methods are cited for testing flammability of contents and metal strength and corrosion resistance, but no tests of polymers are cited. Similarly, the NRC regulations on the packaging and transportation of radioactive material (Ref. 33) makes no mention of tests for polymers. Only physical performance tests for the whole containers, including free drop, puncture, and compression, are specified. It can be deduced from these specifications that any seals and liners must survive the physical tests specified; however, these requirements do not directly indicate any specific materials compatibility tests for polymers. It may be deduced that polymers with higher temperature resistance may perform better in the fire-resistance tests. However, this performance is largely a function of container design and how hot the interior becomes during these tests; both unknown factors at this time.

3.3 Comparison of Standard Test Methods

The selected test methods in Table 11 describe one or more of the following parameters: mass and density, tensile properties, compression properties, leak rate, stress-cracking, or a combination of these. This section briefly summarizes these tests and describes how they apply to compatibility assessment. The test methods most applicable to evaluating materials for

shipping containers are listed in Section 4.

ASTM Methods D618 and D1349 specify how the material is to be conditioned before testing and the conditions during testing. Many of the methods have additional specifications on environmental conditions before and during testing.

Methods D297, D471, D543, D792, and D1239 measure either the mass or density of the material. These tests provide quick, simple, inexpensive selection of candidate materials. A material can fail by either a change in mass or a change in density.

A material that loses mass has either had bulk material removed or has had chemicals leached out of it. The loss of bulk material means that eventually enough material will be removed to form a hole, resulting in leakage. Chemical leaching causes a loss in physical properties. The components most subject to leaching are the plasticizers. Loss of plasticizers means that eventually the material will become brittle and subject to breakage.

A decrease in density of the material can indicate leaching or swelling. Swelling can cause an O-ring to leave its groove, resulting in a leak path. Swelling also can cause an increase in permeability. An increase in density is caused by absorption of the test liquid, indicating high permeability to the test liquid.

The tensile properties of the candidate material are usually not of primary importance. However, a change in these properties provides direct evidence of material degradation. ASTM Methods D412, D624, D638, D882, and D1004 all measure tensile properties. Tensile properties also form major parts of ASTM D751 and D1414, ANSI/NSF 54, DIN 53 521, EPA 9090A, and ISO 1817.

The three most important compression properties are set, deformation, and hardness. These are measured in ASTM Methods D395, D1415, and D2240. O-rings are most sensitive to these properties. An O-ring that takes a set (becomes permanently deformed) can only be used once. In some applications an O-ring that is too hard can deform the sealing surface, although that will not be a concern in this case because the polymers under consideration are all soft relative to the metal sealing surface. An O-ring that is too soft can leak if the pressure differential is too great. The permeation rate of liquids through polymers is described in Equation 2.

A barrier material may pass all of the tests and still be incompatible with the waste stream if its permeability is too high. Swelling is one indication of excess permeability. Another way to measure permeability is weight loss from an impermeable test bottle sealed with the test material. ASTM Method D814 uses this technique. A more sensitive way is to use a mass-spectrometer helium leak detector. Because of its small size and low molecular weight, helium gas will leak through a seal faster than any other substance. Leak rates of 4×10^{-11} cm³ per second at standard temperature and pressure are easily measurable; this is a leak rate of less than 200 nanograms per year. The drawback to helium leak detection is that the equipment is relatively costly. Other methods to measure vapor transmission (of water) include the roughly equivalent methods ASTM E96, ISO 1195, and DIN 53122.

Permeability of gases can be measured using the roughly equivalent methods found in ASTM D1434, ISO 2556, and DIN 53380. Gas transmission is defined as the cubic centimeters

of gas (under standard temperature and pressure) that diffuse through a sample with a surface area of one square meter in 24 hours under a pressure of one bar. The gas transmission rate (GTR), is given in units of $\text{cm}^3/24 \text{ hours} \cdot \text{m}^2 \cdot \text{bar}$ (Ref. 16). The GTR value can be converted to a vapor transmission rate (VTR) by converting the units to $\text{mg/s} \cdot \text{m}^2$. The permeability coefficient P_g is given in units of $10^{-3} \text{ m}^2 \text{ s}^{-1} \text{ Pa}^{-1}$ and can be calculated using Equation 3.

$$P_g = \text{GTR} \times \text{thickness in microns}/8640 \quad (3)$$

Gas permeability measurements may be taken on films 25 to 50 microns (1 to 2 mils) thick.

Stress cracking is mainly a problem for polyethylene liner material. Stress cracking occurs when the polymer chain is stretched. Eventually some part of the chain breaks. Certain chemicals, such as surfactants, promote this breakage. ASTM method D1693 qualitatively measures stress cracking by immersing bent samples in a surfactant and rating the amount of cracking that occurs.

Several of the test methods use a combination of tests to evaluate materials. The ASTM Method for O-rings, D1414, uses variations of all of the above tests and adds some tests not discussed above. ANSI/NSF 54, DIN 53 521, EPA 9090A, and ISO 1817 all use multiple tests.

No single test is sufficient to evaluate compatibility; a battery of tests is needed. Starting points for the selection of tests are the two comprehensive methods, EPA 9090A for liners and ASTM D1414 for O-rings.

EPA Method 9090A is the only standard that attempts to evaluate the chemical resistance of liner materials comprehensively. EPA Method 9090A provides a wide variety of tests for durability of elastomers. It includes tests for tear resistance, puncture resistance, tensile properties, hardness, elongation at break, modulus of elasticity, volatiles content, extractables content, ply adhesion, and hydrostatic resistance. It is useful as the starting point for selection of appropriate tests. ASTM D1414 includes a wide range of tests for O-rings.

3.4 New Test Methods

All of the tests discussed previously involve gross changes in properties. Subtle changes may be occurring in materials that would not be detected by these tests. For candidate materials that pass initial screening using the gross tests, it will be desirable to investigate the properties with more sensitive laboratory techniques, to find evidence of leaching, absorption, oxidation, and other chemical changes. Spectroscopic analysis, dynamic mechanical behavior, and thermal response are commonly used methods in the laboratory to detect small changes in polymers. For this test plan, the additional test methods that may be desirable include FTIR, UV-visible spectroscopy, fluorescence, phosphorescence, helium leak detection, dynamic mechanical analysis (DMA), and thermal analysis.

3.5 Test Equipment and Cost Considerations

Except for the tension and hardness tests, the test equipment required is of the type found in most laboratories. Many of the tests require a laboratory balance with an accuracy of 0.1 milligram, a dial or digital caliper, and a micrometer. Some of the tests require a constant

temperature bath. To measure hardness, a standard hardness tester is required. Universal testing machines can provide both tension and compression forces for a variety of tests. A fixture can be added to provide shear forces. These instruments do not provide torsion forces, but such forces are not required for the tests recommended in this report. The compression set test requires a clamp made of parallel plates with spacers. The tension tests require a universal testing machine and specialized grips.

Table 12 is a listing of selected universal testing machines. The listing is not meant to be comprehensive. The "Manufacturer" and "Model" columns are self-explanatory. The "Size" column lists the maximum load the machine frame can support in pounds force. Pounds force are the units of load applied to a sample. A machine capable of 2,000 pounds of force is judged to be the smallest size appropriate for this effort. A larger machine provides more rigidity, less error, and more versatility in testing (*e.g.*, the ability to handle larger or thicker specimens). A large machine can be used for very low load tests merely by changing the load cell; a small machine cannot be upgraded to do higher load testing. Increasing the size of the machine does not result in a proportionate increase in price: increasing the size by a factor of five only increases the price by half. The price is approximate and may vary by $\pm 10\%$ or more depending on options and accessories. The physical specifications for accuracy and resolution of all the machines are nearly identical, and the differences in accuracy and resolution are not judged to be significant. Therefore, no ranking of specifications is given. Evaluated instead are the literature received from the manufacturer, customer support, and testing software.

Table 12. Descriptions of Selected Universal Testing Machines

Manufacturer	Model	Size (lbf)	Approx. Price, \$	Lit.	Support	Software (Std/Opt)	Notes
Applied Test Systems	1401	1,000	25,000	B	B	E	Not computer-controlled. May not be large enough for all desired tests.
Instron Corporation	4466	2,250	35,000	B	A	E/A	Not computer-controlled. Smart control panel. Optional IEEE-488 interface.
Instron Corporation	4468	11,250	50,000	B	A	E/A	Not computer-controlled. Smart control panel. Optional IEEE-488 interface.
Instron Corporation	5566	2,250	50,000	A	A	B/A	Computer controlled. Optional software has most ASTM methods.
Interlaken Technology Corp.	3350-2.2	2,200	57,000	C	B	A	Servohydraulic System.
Interlaken Technology Corp.	3340-2.2-5-6	2,200	61,000	C	B	A	Servohydraulic System.
Interlaken Technology Corp.	3310-2.2-5-6	2,200	70,000	C	B	A	Servohydraulic System.
Measurements Technology, Inc.	MTI-5K	5,000	20,500	C	B	A	Software written in Pascal. Source code provided. All standard tests provided.
Measurements Technology, Inc.	MTI-30K	30,000	28,000	C	B	A	Software written in Pascal. Source code provided. All standard tests provided.
MTS Systems Corp.	810.19	3,300	60,000	A	C	B/A	Servohydraulic System.
SATEC	MATS II T5000	5,000	37,000	B	A	B/A	Closed loop digital control. Optional software has different versions for tension, flex, compression, and peel.
SATEC	APEX T5000	5,000	26,000	B	A	B/A	Computer monitored control. Optional software has different versions for tension, flex, compression, and peel.
SINTEC	2/S	2,250	40,000	B	C	B/A	Software may be used on other systems.

The rankings are somewhat subjective. In the "Lit." column, a ranking of A means that the manufacturer provided brochures on the machine, the software, and the accessories such as grips and extensometers, and that the brochures included specifications on the equipment. A ranking of B means that some information was not included. A rating of C means that the manufacturer sent only a flyer with no detailed information. In the support column, a rating of A means that a sales engineer visited and was knowledgeable. A rating of B means that the sales engineer phoned and worked to make sure that anything needed was provided. A rating of C means that a sales engineer did not call within 30 days after requesting literature. A software rating of A means that the testing software has support for ASTM testing methods, multiple information windows, and provides database management of test results. A rating of B means that one of these items was missing or deficient. A rating of E means that the system is not computer controlled and therefore does not need software. No software had a rating of C or D. The standard software rating is listed first, followed by a rating of optional software, if any.

Environmental chambers, also called temperature chambers, are used whenever tests must be conducted at a temperature other than ambient. Standard chambers provide for heating only; cooling is an option at extra cost. The maximum temperature is usually in the range of 250 to 350°C, higher maximums (up to 600°C) are available at additional cost. If testing is done at temperatures greater than 100°C, high-temperature grips and water-cooled pull rods are recommended. The minimum temperature is -70°C for liquid carbon dioxide cooling and -150°C for liquid nitrogen cooling. Chambers are usually designed to fit a specific series of test frames.

Cost depends on the size of the chamber, the usable temperature range, the mounting method, and the options selected. Base price for a chamber is about \$8,000. Cooling adds another \$1,500. Mounting the chamber directly to the test frame costs about \$500, while mounting it on wheels adds about \$3,500.

For constant temperature and constant relative humidity conditioning of test samples, an environmental chamber such as the Lindberg/Blue-M Model VP100AT1 may be used. It provides a dry bulb temperature range of room temperature to 77°C in a 1.6 ft³ chamber. The range of achievable relative humidity is 20 to 98%. The cost of this type of unit is about \$3,000.

4.0 GENERAL APPROACH TO COMPATIBILITY TESTING

4.1 Scope of Testing

The following properties must be determined to assess the applicability of potential seal and liner materials: hardness, mass, density, and dimensional changes from absorption or leaching, modulus of elasticity, permeability, tensile strength, and elongation. For seals, compression set must also be measured; for some liner materials, stress cracking must be tested.

It may be desirable to run a series of initial screening tests. This initial screening could consist of testing samples with representative simulated wastes under accelerated aging conditions (high temperatures and concentrations of solvents and corrosive wastes). The maximum temperature at which a polymer should be tested is above its transition temperature and below its melting temperature. If a polymer shows no changes in properties under these conditions, it is a prime candidate, and some intermediate tests may be unnecessary. If a polymer fails in initial tests, it may not warrant full investigation and may be eliminated from the test matrix.

A permeability or leak rate test should be conducted next. Any materials that pass this test are prime candidates. The remaining tests can then be conducted to fully evaluate material compatibility.

The tests listed in Table 11 were carefully reviewed to determine, for each property, which is the single most applicable test, and whether those tests are adequate for this test plan. The following conclusions were made:

- ❖ All tests shall be governed by the appropriate ASTM, EPA, and ANSI/NSF test procedures.
- ❖ ASTM D471-79 (reapproved 1991), "Standard Test Methods for Rubber Property -- Effect of Liquids," provides procedures for exposure of polymer test samples to chemicals of interest. This test method includes procedures for determining changes in mass and volume. It also references ASTM Method D2240 for determination of hardness and D412 for tensile strength and ultimate elongation.
- ❖ The primary test procedure governing all O-ring tests will be ASTM D1414. This test includes procedures to determine the following properties of interest: tensile strength, ultimate elongation, tensile stress, compression set, temperature retraction, relative density, and hardness. ANSI/NSF 54 will serve as a guide for allowable changes in material properties. If the ASTM procedures allow a choice in parameters, EPA 9090A and DIN will serve as guides. ASTM D618 will define environmental conditions and conditioning procedures unless otherwise specified in the specific ASTM procedure. Permeability will be tested using ASTM D1434.
- ❖ For liner materials, the primary procedure governing testing will be EPA 9090A. The following tests shall be used. ASTM D882 shall be used for tensile tests of liner

materials less than 0.04 inches thick; for liner thicknesses between 0.04 inches and 0.5 inches, ASTM D638 will be used. ASTM D792 shall be used for all density measurements. Permeability will be measured using ASTM D814. ASTM D1239 will be used to measure leaching. Stress-cracking will be evaluated using ASTM D1693. Permeability will be tested using ASTM D2684. The tests selected for use are listed in Table 13.

Table 13. Tests to be Conducted

Property or Procedure	Materials	Individual ASTM Test Method	Relationship to ASTM D1414	Relationship to EPA 9090A
chemical effects	seals, liners	D471	ref with changes	none
compression set	seals, liners	D395	ref with changes	none
flexural properties	liners	D790	none	none
hardness	seals, liners	D2240	ref	ref
permeability to liquids	liners*	D814	none	none**
permeability to gases	seals	D1434	none	none
specific gravity	seals, liners	D792	none	none
stress cracking	liners	D1693	none	none
tensile strength, modulus, and elongation	seals	D412	ref	ref
tensile strength, modulus, and elongation	liners > 1 mm thick	D638	independent	ref
tensile strength, modulus, and elongation	liners < 1 mm thick	D882	independent	ref
<p>* Although no specific test was found especially applicable for permeability of seals to liquids, a large sample of the seal material could be tested similarly to a liner, using a modification of ASTM D814 or ASTM D2684.</p> <p>** A procedure for testing permeability of aqueous solutions is given in EPA 9090A, but not a procedure for testing permeability of organics.</p>				

ASTM Methods D1414 and EPA 9090A are described herein as "comprehensive" tests because they specify the testing of many properties of plastic materials, both by incorporating test procedures and referencing other individual ASTM tests, with or without changes. In Table 13, the single most appropriate test method (excluding the comprehensive methods ASTM D1414 and EPA 9090A) for each property is listed under "Test Method."

Under the columns "Relationship to ASTM D1414" and "Relationship to EPA 9090A" are the relationships of the individual ASTM test methods to the comprehensive methods ASTM D1414 and EPA 9090A. If there is no overlap, the entry is "none." If the individual ASTM test is referenced by the comprehensive method, the entry is "ref;" this may be with or without changes. If the comprehensive method and the individual ASTM test provide procedures for the test, the entry is "independent." When the entry is independent, the governing comprehensive method will be applied, and portions of the individual ASTM test method will be used as needed.

If the primary methods ASTM D1414 and EPA 9090A are followed, including referenced ASTM tests, the only additional tests needed will be ASTM D814 for permeability and D1693 for stress cracking.

As a brief overview, the test plan for liners will require the following steps:

1. Choice of whether to use 10 polymers in a larger test plan or 6 polymers in a smaller test plan.
2. Collection of polymer samples and chemicals to make simulated waste solutions.
3. Preparation of simulated waste solutions (with or without optional additives).
4. Preparation of samples for testing of flexural properties (ASTM D790) and dimensional and mass changes (ASTM D471).
5. Testing of properties of unexposed materials.
6. Immersion of samples in simulant solutions.
7. Testing of properties of exposed materials.
8. [Optional] Irradiation of simulant-exposed samples, followed by testing of properties.
9. Elimination of polymers that exhibited unacceptable changes.
10. Carrying out Steps 4 through 9 for stress cracking (ASTM D1693) and permeability to liquids (ASTM D814), followed by the other tests for liners specified in Table 13.

For seals, the above steps are followed with the following changes in Steps 4 and 10:

4. Preparation of samples for testing of liquid absorption (ASTM D471) and compression set (ASTM D395).
10. Carrying out Steps 4 through 9 for tensile properties (ASTM D412) and permeability to liquids (ASTM D814), followed by the other tests for seals specified in Table 13.

4.2 Materials

The required mechanical properties of seal materials are related to the container design. Because the container designs may vary, it is necessary to consider chemical compatibilities for a variety of potential seal materials. The list of materials initially considered for seals is given in Table 1 of Section 2.2. Potential liner materials are discussed in Section 2.1.

As discussed in Section 2.3, a seal or liner for waste sample containers should have good to excellent resistance to one or more of the following chemical groups: strong aqueous base, aliphatic and aromatic hydrocarbons, halocarbons, and ketones. The candidates from Table 1 that were rejected for testing failed the initial screening for a variety of reasons. As shown in Table 9, chloroprene and polyacrylate have good resistance to aliphatic hydrocarbons and poor resistance to aromatic hydrocarbons, aqueous base, and halocarbons. They were eliminated because they did not have good to excellent resistance for any of the selected waste environments. Chlorosulfonated polyethylene has good to excellent resistance to aliphatic hydrocarbons and aqueous base, but poor resistance to aromatic hydrocarbons and halocarbons. The only waste stream it would be applicable to is aqueous base, and there are already four candidate materials for this waste stream with superior resistance to other chemicals. Both EPDM rubber and styrene-butadiene have residual unsaturated sites (carbon-to-carbon double bonds) that are subject to oxidation and other reactions. Fluorosilicone has good to excellent resistance to aliphatic and aromatic hydrocarbons and aqueous base, but poor resistance to halocarbons. Phosphonitrilic fluoroelastomer has good to excellent resistance to aliphatic and aromatic hydrocarbons and aqueous base, but poor resistance to halocarbons. Polyacrylate has

good resistance to aliphatic hydrocarbons, but poor resistance to aqueous base, haloarbons, and aromatic hydrocarbons. Polysulfide has good to excellent resistance to aliphatic and aromatic hydrocarbons and aqueous base, but poor resistance to halocarbons. Polysulfide was also eliminated from the test matrix because it contains a high percentage of sulfur, which is subject to oxidative breakdown and other chemical reactions. Silicone has excellent resistance to aqueous base, but poor resistance to aliphatic and aromatic hydrocarbons and halocarbons. Silicones in general have excellent thermal stability but high permeability. The high permeability is a consequence of the long silicon-to-oxygen bonds and the resulting higher free volume. This high permeability means silicones may not meet leak rate requirements for mixed waste transport.

The materials listed in Table 14 satisfy the requirements of resistance to aqueous base, aliphatic and aromatic hydrocarbons, halocarbons, and/or ketones and are relatively chemically inert in general. The materials listed in Table 14 include at least three materials with good to excellent resistance to each waste stream of interest. To be included in Table 14 as a material resistant to hydrocarbons, a material had to have good to excellent resistance to both aliphatic and aromatic hydrocarbons, since these are often found together (*e.g.*, in petroleum distillates such as kerosene).

Table 14. Summary of Chemical Resistance Properties of Top-Ranked Materials*

Polymer	Seal	Liner	Aqueous Base	Chlorinated	Hydrocarbons	Ketones	Radiation
Butadiene-acrylonitrile copolymer	❖				❖		❖
Cross-linked polyethylene	❖	❖	❖			❖	❖
Epichlorhydrin	❖			❖	❖		❖
Ethylene-propylene rubber	❖		❖			❖	❖
Fluoroelastomer		❖		❖	❖		❖
High-density polyethylene		❖	❖			❖	❖
Isobutylene-isoprene copolymer	❖		❖			❖	❖
Polypropylene		❖	❖		❖	❖	❖
Polytetrafluoroethylene	❖	❖	❖	❖	❖	❖	❖
Styrene-butadiene rubber	❖		❖			❖	❖
* "❖" indicates good to excellent resistance to the waste type.							

For waste samples containing large quantities of halocarbons without aqueous base or aliphatic or aromatic hydrocarbons, the elastomers with good to excellent resistance are

epichlorohydrin, fluorocarbon elastomers, and polytetrafluoroethylene (may be glass-filled). Both epichlorohydrin and fluorocarbon elastomers are strong candidates for waste streams not containing aqueous base.

For waste samples containing large quantities of hydrocarbons without aqueous base or halocarbons, the elastomers with good to excellent resistance are butadiene-acrylonitrile copolymer, epichlorohydrin, fluorocarbon elastomers, fluorosilicone, polytetrafluoroethylene, and polypropylene.

The most chemically resistant polymer is polytetrafluoroethylene. Seals and liners made of this material have outstanding resistance to a broad range of chemicals. Polytetrafluoroethylene may have poor resistance to high radiation levels, although radiation is not judged a significant threat in this application. Both tetrafluoroethylene and cross-linked polyethylene are fairly rigid materials and their usefulness will depend on the design of the sample container (the sealing surface, geometry, and forces of closure). They may also be subject to cold flow, and it is possible that they would have to be replaced after each use.

Ethylene propylene, isobutylene-isoprene, and styrene-butadiene have excellent resistance to aqueous base and to ketones, but should not be used with hydrocarbons or chlorinated solvents. Epichlorohydrin and fluorocarbon rubber have good to excellent resistance to hydrocarbons and chlorinated solvents, but should not be used with aqueous wastes. Polysulfide has good to excellent resistance to hydrocarbons, ketones, and aqueous base, but should not be used with chlorinated solvents. These observations are summarized in Table 14. Thus, the "short list" of polymeric materials that should be tested consists of the 10 listed in Table 14.

The approximate number of samples required for the test plan can be estimated. This analysis is only approximate because it does not account for samples tested more than once. In a very comprehensive test matrix, for each of the 10 candidate polymers, 5 samples will be prepared for each of the 11 tests in Table 13 conducted with each simulant solution. To estimate the total number of samples required, multiplying 10 polymers times 5 samples times 11 tests times 2 simulant solutions (plus nonexposed material, for a total of 3 exposure conditions) gives a total of 1,650 samples. However, other factors will affect the number of samples needed. By following the testing hierarchy described above, some candidate polymers may drop out early in the testing. If a variety of exposure times or radiation exposure is desired, more samples may be required. For some tests, samples can be tested and reimmersed.

A smaller test plan could eliminate 4 of the 10 candidate polymers. The polymer with the smallest range of chemical resistance (butadiene-acrylonitrile copolymer), the polymer with poor compression set resistance (polytetrafluoro-ethylene), and those with very low heat resistance (polyethylenes) could be ruled less attractive than other candidates. In general, only three samples are required per test. If 2 simulant solutions (plus unexposed samples) are used, the total number of samples required will be 6 polymers times 3 samples times 11 tests times 3 exposure conditions for a total of 594 samples.

4.3 Parameters

All testing will be based on the ASTM procedures referenced in Table 13. The

temperature range for testing will be -29°C (-20°F) to 38°C (100°F), the range of temperatures specified by the NRC (Ref. 33). Exposure level (time of immersion) is another important parameter. Samples may be immersed in simulated wastes for up to 3 months, and graphs of property values *versus* time will be prepared.

4.4 Choice of Simulant Solutions

The main threats to seals and liners from the anticipated wastes are judged to come from strong aqueous base, oxidants, chlorinated solvents, hydrocarbons, and ketones. The following species appear to pose virtually no threat to the polymers under consideration under the range of conditions anticipated: water, chelating agents, and ions without appreciable acidic, basic, or oxidizing properties such as nitrate, nitrite, sodium, potassium, heavy metals, and radionuclides. There is no evidence in the literature that any of the polymers under consideration are affected by these materials under the conditions (concentrations and temperatures) anticipated. In transport of mixed LLWs, therefore, all threats to seals and liners are expected to come from the hazardous, not the radioactive, wastes. Simulant solutions need not include simulants for materials that will not affect the polymers, although if desired such simulants could be added as options. Thus, for example, simulant solutions need not include simulants for sodium, nitrate, nitrite, and radionuclides.

Any seal or liner materials used in containers to transport mixed LLWs must be shown to be resistant to samples containing one or more of the following types of chemicals: strong aqueous base, oxidants, chlorinated solvents, hydrocarbons, and ketones. Because few polymers are resistant to all these chemicals, it is possible that different polymers will be chosen for different waste streams. Because of the wide variety of waste samples, it is desirable to use several simulant solutions.

If a single simulated waste could be used for much testing of potential seal and liner materials, the test matrix would be simplified. However, it is also likely that fewer candidate polymers would pass such a stringent test than tests with less complete wastes. This single simulated waste would need to contain a variety of compounds representing those groups most likely to pose threats to seal and liner materials. A single general simulated waste could contain strong aqueous base, oxidants, chlorinated solvents, hydrocarbons, and ketones. Simulated radionuclides could be added for completeness, although radionuclides and other heavy metals are not expected to pose significant threats. A single simulant solution could not, however, contain all conceivable threats because some cannot coexist. For example, a solution cannot be both basic and acidic. Thus, if it is anticipated that acidic waste streams may be encountered as well as the basic streams already identified, at least one basic and one acidic simulated waste solution should be used. Another incompatibility is that many relatively nonpolar organic compounds such as hydrocarbons and chlorinated solvents are not miscible with water. Problems inherent in two-phase systems include nonhomogeneity, the necessity of constant effective stirring, and difficulties in reproducing exposures and results. To avoid these problems, at least one organic and one aqueous solution should be used. Thus, to simulate all potential waste streams at least two simulated wastes are required:

- ❖ a basic aqueous solution

❖ an organic solution.

The basic aqueous solution (called in this report simulant solution A) should contain hydroxide and dichromate ions and may, if desired for completeness, include nitrate, nitrite, and sodium ions, although the latter three are judged no significant threat. The organic solution should contain chlorinated solvents, hydrocarbons, and ketones.

Within a chemical family (such as alcohols or hydrocarbons), the smaller molecular weight liquids generally pose the greatest threats to polymers because they can penetrate surfaces more easily than the higher molecular weight members. In general, liquids in bulk react more quickly than gases or solids because they come into more intimate contact with the other species present (in this case, polymers). Therefore, to make a representative, stringent, and relatively simple simulant solution of organic solvents, at least one liquid low-molecular-weight member of the groups of chlorinated solvents, hydrocarbons, and ketones should be included. For practical reasons of safety and ease of testing, when a choice of solvents with similar effects is available, the solvent with lower toxicity and/or lower volatility (rate of evaporation) is to be preferred, to minimize risks to experimenters and potential changes in concentrations of solutions.

Suitable chlorinated solvents include chloroform, carbon tetrachloride, and 1,1,1-trichloroethane, all of which have been reported in mixed LLWs. The 1,1,1-trichloroethane is much less toxic than chloroform or carbon tetrachloride. Thus 1,1,1-trichloroethane is preferred for testing of compatibility with chlorinated solvents. Preferred hydrocarbons include aliphatic (hexane or heptane) and aromatic (benzene and toluene). Hexane has a lower molecular weight than heptane and is similar in toxicity, so it is preferred. Toluene is much less toxic than benzene and has similar properties. Low-molecular weight ketones include acetone and methyl ethyl ketone. Methyl ethyl ketone is slightly less volatile and preferred for experimental convenience. Other organic solvents identified in high quantities in some mixed wastes and containing functional groups not yet included in the simulant solution are 1-butanol (an alcohol), acetonitrile (a nitrile), and dioxane (an ether). Thus, a single organic simulant solution (labeled simulant solution B) could be made consisting of 1,1,1-trichloroethane, hexane, toluene, methyl ethyl ketone, 1-butanol, acetonitrile, and dioxane. The recommended proportions for this organic test solution are equal parts by volume. The exact proportions of constituents in a test solution are not expected to affect compatibility results noticeably, as long as each is present in significant quantity. If a polymer is attacked by a certain chemical, the results will be similar whether that chemical is present in 5% or 90% concentration (Ref. 23). This effect occurs because if there is an affinity between the surface of the polymer and a particular chemical, the polymer will preferentially adsorb that chemical from the solution. Since the volume of the polymer is much less than the volume of the test solution (typically less than 5%) and polymers typically absorb less than 100% of their own weight even in extreme cases, a 5% concentration of a component should be adequate to saturate the polymer and show swelling if absorption or reaction occurs. The reaction may occur more slowly in a 5% solution than in a 90% solution, but in both cases the reaction should proceed to completion during the course of the test (a few days). Thus, if an incompatibility exists, it should be obvious in the tests whether the chemical incompatible with the polymer is present at a 5% or 90% concentration.

Significant quantities of methyl isobutyl ketone (hexone) mixed with kerosene and contaminated with radionuclides have been identified at Hanford (Ref. 3). This mixture is

already effectively simulated by the organic simulant solution B, which contains methyl ethyl ketone (a ketone similar to hexone and a stronger solvent than hexone) and hexane (a hydrocarbon simulant for kerosene and a stronger solvent than kerosene).

The advantage of simulant solution B is that it would provide relatively simple rapid, broad screening for compatibility with organic solvents. If a seal or liner is compatible with this mixture it would have good resistance to a wide range of organic chemicals. However, just because a polymer is not compatible with this solution does not mean it could not be used for more specific waste streams. For this reason it is also desirable to have more specific simulant solutions. A chlorinated solvent waste stream can be simulated with neat 1,1,1-trichloroethane (simulant C). Scintillation solvents can be simulated by a blend of toluene, xylene, and dioxane (equal parts by volume), labeled simulant D. The hexone/kerosene mixture identified at Hanford and consists of a mixture of 90% by volume methyl isobutyl ketone (hexone) with 10% by volume kerosene, labeled simulant E. Testing of polymers with the simulants C, D, and E would entail additional work but would also provide more detailed information on compatibility than testing with solution B alone. Instructions for preparing simulant solutions A through E are given in Appendix B.

Eight simulant solutions have been recommended for subsurface science research (studies of possible migration of mixed LLW underground) (Ref. 35). These solutions were considered and assessed for relevance but were judged not particularly relevant for this effort.

Recommended simulants for mixed waste therefore include the following:

- (1) an aqueous solution of 2.0 M hydroxide and 0.15 M dichromate
- (2) an organic solvent solution consisting of equal parts by volume 1,1,1-trichloroethane, hexane, toluene, methyl ethyl ketone, 1-butanol, acetonitrile, and dioxane.
- (3) neat 1,1,1-trichloroethane
- (4) equal parts by volume toluene, xylene, and dioxane
- (5) 90% by volume methyl isobutyl ketone and 10% kerosene

Although no evidence of acidic aqueous mixed LLWs from DOE was found, if it is desired to validate seals or liners for transport of acidic aqueous wastes, an optional sixth solution could be used. This solution would consist of strong acid (*e.g.*, 1 M nitric acid) in water.

Solution A could have the optional additives of sodium, nitrate, nitrite, and aluminum ions. Although none of these optional additives are expected to affect materials compatibility, they may be desired to make the simulant solution more realistic. Aluminum, for example, is the most abundant metal other than sodium found in the waste streams.

For initial testing of materials compatibility, simulants for radionuclides and other trace contaminants can be left out of the simulated wastes because they are expected to have undetectably small effects on the elastomers tested. However, the following analysis provides reasoning for selection of simulants for radionuclides if desired.

The main radioactive elements identified in expected DOE waste streams are cesium, strontium, uranium, plutonium, and americium. Radioactive cesium and strontium can be simulated using their nonradioactive isotopes. The ideal nonradioactive simulant for uranium, plutonium, and americium would have a similar range of oxidation states (3 to 6), coordination

numbers (6 to 8) and ionic radii (0.75 to 1.06). The oxidation states, coordination numbers, and ionic radii of common radionuclides and selected candidate simulants are shown in Table 15.

The only nonradioactive element closely matching the oxidation states, coordination numbers, and ionic radii of common radionuclides is praseodymium. A variety of praseodymium compounds are commercially available at prices from about \$10 to \$300 per gram. For example, ICN Biomedicals, Inc. sells praseodymium acetate (\$197/10g). Praseodymium compounds are expensive, and little is known about their toxicity and environmental effects.

For compatibility testing of materials, it is not deemed necessary to match the properties of actinides as closely as does praseodymium. Heavy metals have very little effect on polymers, and virtually all heavy metal ions are expected to have similar effects. Of heavy metals with atomic numbers above 70, only two possess all three of the following desirable properties: (1) not extremely toxic, (2) not posing significant environmental hazards, and (3) relatively low in cost. These two metals are tungsten (W) and bismuth (Bi). As examples of how these elements are used in households, the filaments in incandescent light bulbs are tungsten and the active ingredient in the common upset stomach remedy Pepto-Bismol[®] is bismuth subsalicylate. The ionic radius of Bi⁺³ is in the desired range, whereas that for W⁺⁴ is significantly smaller. Thus, from a standpoint of simulating radionuclides with the least health and environmental hazards and at the lowest cost, bismuth compounds are attractive. One example of a potentially useful

Table 15. Charges, Coordination Numbers, and Ionic Radii of Selected Radionuclides and Potential Simulants

Element	Charge	Coordinate Number	Ionic Radius (Å)
Am	+3	6	1.0
Am	+4	8	0.95
Bi	+3	5	0.99
Bi	+3	6	1.02
Bi	+3	8	1.11
Cs	+1	6	1.70
Cs	+1	9	1.78
Cs	+1	10	1.81
Cs	+1	12	1.88
Pr	+3	6	1.01
Pr	+3	8	1.14
Pr	+4	6	0.78
Pr	+4	8	0.99
Pu	+3	6	1.02
Pu	+4	6	0.80
Pu	+4	8	0.96
Sr	+2	6	1.16
Sr	+2	7	1.21
Sr	+2	8	1.25
Sr	+2	10	1.32
Sr	+2	12	1.44
U	+3	6	1.06
U	+4	7	0.98
U	+4	8	1.00
U	+4	9	1.05
U	+5	6	0.92
U	+5	7	0.96
U	+6	6	0.75
U	+6	7	0.88
W	+4	6	0.65
W	+6	4	0.41

bismuth compound for simulated wastes is bismuth nitrate pentahydrate, available from ICN for \$11/100g.

All simulant solutions have the option of adding small amounts of a bismuth salt as a simulant for uranium, plutonium, and americium. For the organic simulant solutions, a soluble bismuth salt is desirable. Addition of a chelating agent such as DTPA or NTA may assist solubility.

5.0 RADIATION TESTING

5.1 Introduction

As discussed in Section 2.4, it is highly unlikely that the radiation levels of the mixed LLWs being considered here will cause any detectable degradation of liner and seal materials. Previous reports indicate no synergistic effects of wastes and radiation on polymers (Ref. 6). However, in the presence of waste chemicals, synergistic degradation of liner and seal materials is remotely conceivable. Therefore, a limited amount of sample testing by irradiation after exposure to simulated wastes may be desired.

Several established procedures exist for radiation testing (Refs. 36 and 37). Detailed procedures for irradiation and testing of polymers are given in Reference 36. Reference 37 provides an additional useful guide for selecting irradiation conditions, instrumentation, and data analysis.

In general, the performance data on each material need to be obtained by standardized procedures designed to demonstrate the influence of radiation and waste compounds on significant properties for the application. Environmental conditions should be well controlled and documented during the measurement of radiation effects. For sample shipping container materials, the conditioned sample environment and, perhaps, compressive stress for seals are important conditions to maintain. Oxygen should also be present as it would be in the actual container to simulate radiation-induced oxidation reactions. At the low radiation doses expected for sample shipping containers, no effects of dose rate are expected to be observed and no associated controls will be required.

5.2 Sample Preparation

Samples for radiation testing should be representative of seal and liner materials in composition and thickness. Where possible, the samples should be premachined into the desired mechanical test specimens before exposure to wastes and radiation. If the test specimens are machined after conditioning and irradiation, this treatment should be noted in the testing report. Specimen dimensions should follow the appropriate ASTM or equivalent standards. Because the effects of radiation can depend upon specimen dimensions, the dimensions should be uniform for comparison of all material types.

For both seal and liner materials, specimen dimensions should be monitored for changes resulting from radiation exposure using ASTM D792 as a guide. Changes in tensile or flexural properties (modulus, yield strength, ultimate strength, and elongation) are of most interest for liner materials and will be tested using ASTM D638, D882, and D790 as guides. Hardness, compression set, and tensile properties should be tested for seal materials in accordance with ASTM D1414 and D945.

The effects of combined chemical compatibility and irradiation are likely to be time-dependent as a result of diffusion or chemical breakdown reactions (Refs. 38 and 39). For this reason, the specimens should be preconditioned in the waste solution as described in ASTM

D618 before being irradiated. Unconditioned control specimens should also be included in the radiation test matrix, as should nonirradiated conditioned specimens. After conditioning in the waste solution, care should be taken to maintain the conditioned environment before radiation exposure. Procedures for maintaining the specimen chemical environment will depend upon the volatility of the components of the waste solution.

5.3 Irradiation Exposure

For analogous experimental conditions (equal absorbed dose), changes in the properties of polymers are only slightly dependent on the type of radiation (Refs. 40 through 42). As such, the radiation source should be selected for ease of use where the absorbed dose can be determined easily and precisely. Gamma rays and fast electrons are, thus, the two radiation sources of choice. Many commercial companies have inexpensive ^{60}Co or ^{137}Ce gamma sources for rent that have well-characterized dose rates. Time to achieve the required absorbed dose is the main variable in such exposures, although care must be taken not to overload the irradiation cell with too many samples that could cause shadowing and nonuniform absorption. These types of sources do not have high enough dose rates to cause a significant rise in specimen temperature. Under high dose rate conditions, temperature must be monitored because many radiation-induced chemical reactions are temperature-dependent.

Maintaining the specimen in a conditioned environment during irradiation will require that the specimens be sealed in an appropriate container with the desired environment. This may be achieved by simply placing the conditioned specimens in a plastic bag if the bag is compatible with the waste composition. It is likely that different bag materials will be required for different wastes to ensure compatibility, as described in Section 4.2. The irradiated samples may be surrounded by the solution during irradiation provided that the solution thickness does not cause significant absorption of the incident radiation.

In general, organic materials can be handled with minimal safety equipment after irradiation. The standard safety goggles, lab coat, and gloves often provide adequate protection. Because residual reactive species are likely to be present in irradiated organic materials, it is desirable that they be tested as soon as possible after irradiation, certainly within one week. Care should be taken to monitor residual radiation from samples exposed to wastes containing heavy metals. These may require a considerable time to cool down in a radiation cell to drop to acceptable levels unless subsequent tests are conducted in a hot cell.

5.4 Data Entry

The test report will include a description of the material, type of radiation source, specimen conditioning and absorbed dose, and changes in material properties that occurred. Sample reports are given in Appendix A of IEC 544-2 (Ref. 37).

6.0 MATERIALS TESTS

6.1 Introduction

A hierarchy of testing will be established to maximize efficiency of the testing process. Only those materials passing the first set of tests go on to the second, and so on. The hierarchy of compatibility screening will be as follows. For rigid liner materials, first are flexural properties, dimensional changes, hardness, and mass changes. Second is stress cracking, third is permeability, and fourth is chemical changes (detectable by FTIR, *etc.*). For flexible liner materials, tensile characterization is used for qualifying materials. For seals, the first tests are absorption and compression set, second are tensile properties, third is permeability, and fourth is chemical change.

ASTM tests recommended in this section are from 2 to 13 pages and describe in detail procedures for preparation of samples and testing to determine properties. It is impossible to provide the details of these tests here without simply copying the ASTM tests verbatim. An experimenter planning to carry out the tests will need to refer to these methods for detailed procedures. However, for the convenience of the reader, brief summaries of these methods are provided here.

6.2 Testing Facilities and Equipment

A universal test machine with a capacity of at least 2,000 pounds will be used. Other equipment required will include micrometers for dimensional measurements, a controlled-temperature oven, an analytical balance, and common laboratory glassware. Containers for exposure of the samples to the simulated wastes will be needed; these could be glass containers with airtight lids. Mason jars (1/2 pint) will be needed for permeability testing using ASTM D814. A press with either a calibrated spring to provide constant tension or spacers to provide constant deflection is needed to determine compression set using ASTM D395.

6.3 Sample Handling

A set of at least five samples will be tested before exposure to the simulated waste solution to determine baseline properties. Unless otherwise specified, all samples are conditioned by storage for at least three hours at the test temperature (normally $23 \pm 2^\circ\text{C}$). If the samples are affected by humidity, they are conditioned by storage for at least 24 hours in an atmosphere of $50 \pm 5\%$ relative humidity. Samples removed from solutions are kept in inert airtight plastic bags and tested within three hours.

6.4 Visual Examination

The exposed samples will be examined visually for color changes and evidence of swelling, cracking, pitting, or other deterioration. Any changes will be recorded.

6.5 Liner Sample Measurements

All tests shall be governed by the appropriate ASTM test procedures. ANSI/NSF 54 will serve as a guide for allowable changes in material properties. If the ASTM procedures allow a choice in parameters, EPA 9090A and DIN 53 521 will serve as guides.

6.5.1 Liner Dimensional Measurements

ASTM D471 ("Rubber Property -- Effect of Liquids") will be used to determine dimensional changes for liner materials. This method involves cutting standard rectangular test specimens of 25 x 50 x 2.0 mm. The specimens are immersed in the test liquid in a glass test tube at the chosen temperature between -75 and 250°C for a time period of 22, 46, 70, 166, or 670 hours. The exposed specimens are brought to room temperature, dipped briefly in acetone, blotted dry with filter paper, and weighed. Dimensions are measured and the volume is measured by immersion in water.

6.5.2 Liner Specific Gravity

ASTM D792 ("Density and Specific Gravity [Relative Density] of Plastics by Displacement") shall be used for all density measurements. In cases where changes are observed, ASTM D1239 shall be used to measure leaching. In ASTM D792, samples may consist of sheets, rods, tubes, or molded items of volume not less than 1 cm³ and mass of 1 to 50 g. A sample is weighed, attached to a sinker if needed (if the density is less than that of water and the sample would float), and then suspended by a fine wire and immersed in air-free deionized water at 23°C. The immersed weight is determined. The immersed weight is less than the weight in air by an amount equal to the weight of the water displaced, or the volume of the sample times the density of water. The specific gravity is calculated using Equation 4.

$$\text{Sp. gr.} = a/(a + w - b) \quad (4)$$

where a is the apparent mass of the specimen in air without wire or sinker, b is the apparent mass of the specimen (and of the sinker if used) completely immersed and of the wire partially immersed, and w is the apparent mass of the totally immersed sinker (if used) and the partially immersed wire.

In ASTM D1239 ("Resistance of Plastic Films to Extraction by Chemicals"), 50-mm square test specimens are conditioned at 23°C and 50% relative humidity for at least 40 hours as described in ASTM D618. Each specimen is then immersed in a covered jar containing 400 mL of the test liquid. The specimens are maintained at either 23°C or 40°C for 4 or 24 hours. After this exposure, the samples are removed, then rinsed if appropriate (with water if the test liquid was an aqueous salt, soap, acid, or base solution or with ligroin if the test liquid was an oil), and wiped to dry them. The samples are weighed, and the weights are recorded. The weight loss is calculated as shown in Equation 5.

$$\text{Weight loss, \%} = [(W_1 - W_2)/W_1] \times 100 \quad (5)$$

where W₁ is the weight of the specimen after the conditioning period and W₂ is the weight of the

specimen after the test.

6.5.3 Liner Hardness

ASTM D2240 ("Rubber Property--Durometer Hardness") shall be used to determine liner hardness. In ASTM D2240, samples must be flat, at least 6 mm thick, and at least as large as a rectangle of 30 x 36 mm. (Note: this size was calculated by the author based on five readings per sample at least 6 mm apart and at least 12 mm from an edge). The durometer is calibrated by applying measured forces to the tip. The specimen is placed on a flat, hard surface and the durometer is held with the point of the indenter at least 12 mm from any edge. At least five measurements are made on each specimen, at least 6 mm apart. The arithmetic mean is the reported value.

6.5.4 Liner Flexural Strength and Modulus

ASTM D790 ("Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials") shall be used to determine the flexural strength and modulus of liner materials. In ASTM D790, samples are prepared by cutting bars of rectangular cross section from sheets, plates, or molded shapes, or by molding the bars directly. The dimensions of the samples depend on the thickness and rate of cross-head travel of the test machine; a table of recommended sample sizes is given in the procedure. If conditioning is required, specimens are conditioned at 23°C and 50% relative humidity for at least 40 hours as described in ASTM D618. The sample is placed on a support span in a universal test machine, with a loading nose pressing down in the center. As a force is applied to the loading nose, the deflection of the center of the sample is recorded. The test is run until the outer layer of the sample breaks. The maximum strain in the outer layer occurs at midspan and can be calculated by Equation 6.

$$r = 6Dd/L^2 \quad (6)$$

where r is the maximum strain in the outer layer (mm/mm), d is depth (mm), D is maximum deflection of the center of the beam (mm), and L is support span (mm).

The tangent modulus of elasticity is calculated using Equation 7.

$$E_B = L^3m/4bd^3 \quad (7)$$

where E_B is modulus of elasticity in bending (N/m²), L is the support span (m), m is the slope of the tangent to the initial straight-line portion of the load-deflection curve (N/m), b is the width of beam tested (m), and d is the depth of beam tested (m).

6.5.5 Liner Tensile Properties

ASTM D882 will be used for tensile tests of liner materials less than 1 mm (0.04 inches) thick. ASTM D638 will be used for liner thicknesses between 1 mm (0.04 inches) and 14 mm (0.55 inches). In ASTM D882, specimens are prepared consisting of strips of uniform width between 5 and 25 mm. The thickness is at least 50 mm longer than the grip separation used, and the width-to-thickness ratio of the sample is at least eight. If conditioning is required, specimens are conditioned at 23°C and 50% relative humidity for at least 40 hours as described

in ASTM D618. At least five specimens are tested from each sample. Each specimen is placed in the grips of the testing machine, and a record is made of load *versus* extension. The tensile strength is calculated by dividing the maximum load by the original minimum cross-sectional area of the specimen. The percent elongation at break is calculated by dividing the extension at the moment of rupture by the initial length of the specimen and multiplying by 100. Yield strength is calculated by dividing the load at the yield point by the original minimum cross-sectional area of the specimen. The elastic modulus is calculated by drawing a tangent to the initial linear portion of the load-extension curve, selecting any point on this tangent, and dividing the tensile stress by the corresponding strain. Tensile energy to break is calculated by integrating the energy per unit volume under the stress-strain curve. ASTM D638 is similar to ASTM D882 but the samples used are dumbbell-shaped, and the forces involved are greater because of the increased thickness of the samples.

6.5.6 Liner Permeability

Liner permeability shall be measured using ASTM D814. In ASTM D814, samples are 68-mm diameter circular disks cut from sheets up to 3.2 mm thick. These are placed into the lids of 1/2-pint Mason jars (vapor transmission jars), supported with a wire mesh disk if necessary, and the lids are screwed onto the jars each of which contains 200 mL of the test liquid. The jars are inverted and held in a rack at 23°C. They are weighed after periods of 5 days and 8 days. The difference is taken as the vapor transmitted in 3 days. The vapor transmission rate (VTR) is calculated using Equation 8.

$$\text{VTR (mg/s - m}^2\text{)} = 4.05 \times \text{average mass of liquid lost per 24 hours (g)} \quad (8)$$

6.5.7 Liner Stress Cracking

Stress-cracking shall be measured using ASTM D1693 ("Environmental Stress-Cracking of Ethylene Plastics"). In ASTM D1693, bent specimens of plastic (38 x 13 mm) with controlled imperfections are exposed to the action of a solution. The percentage of samples that crack in a given time is observed. If conditioning is required, specimens are conditioned at 23°C and 50% relative humidity for at least 40 hours as described in ASTM D618. Specimens are cut in a blanking die and nicked in a jig. They are placed in a holder that maintains them in a bent position and immersed in glass tubes containing the solution. The tubes are maintained in a constant-temperature bath at 50°C or 100°C. The samples are inspected at intervals of 48 hours for failure. The time to 50% failure is reported, along with the standard deviation in failure time.

6.5.8 Liner Chemical Changes

Chemical changes will be detected by spectroscopic analysis, dynamic mechanical behavior, and thermal response as needed. Test methods that may be used include FTIR, UV-visible spectroscopy, fluorescence, phosphorescence, helium leak detection, DMA, and thermal analysis.

6.6 Seal Sample Measurements

ASTM D1414 ("Standard Test Methods for Rubber O-Rings") shall govern all tests of

O-rings. This procedure includes tests for tensile strength, ultimate elongation, tensile stress, compression set, relative density, dimensional and volume changes on immersion, and hardness, as well as several other properties of less interest in this test plan.

6.6.1 Seal Dimensional Measurements

ASTM D1414 and the dimensional test referenced therein (ASTM D471, "Rubber Property -- Effect of Liquids") shall be used to determine dimensional changes in seals. The thickness or cross section is measured by either (a) a hand micrometer, (b) a dial indicator, or (c) an electronic micrometer. Four readings are taken at points equally distributed around the circumference in both the radial and axial directions; an average measurement is used for calculation. To measure the diameter of the O-ring, a stepped cone or optical comparator is used. Dimensional measurements are taken before and after exposure to the test solution.

6.6.2 Seal Specific Gravity

ASTM D792 shall be used to determine changes in specific gravity. This method is described in Section 6.5.2 of this test plan. For small O-rings (less than 1 g), extreme care is required to obtain accurate results with this test. The suspending wire should be the smallest diameter corrosion-resistant wire that will support the specimen weight. If judged necessary, the surface tension on the wire and air bubbles on the sample surface are reduced by adding a few drops of wetting agent.

6.6.3 Seal Tensile Properties

Tensile strength and elongation shall be determined using ASTM D412 ("Rubber Properties in Tension"). Test samples may be in the shape of dumbbells, rings, or straight pieces of uniform cross section. Tests are carried out at 23°C. Samples are conditioned for at least 3 hours at 23°C before testing and, if the material is affected by moisture, the samples are conditioned at 50% relative humidity for at least 24 hours before testing. Two benchmarks (reference points for measurement of elongation) are placed on the sample using ink or powder. Tests are performed on a calibrated universal testing machine using a rate of grip separation of 500 mm/min. The force at the elongation specified and at the time of rupture are determined and recorded. The tensile stress at specified elongation is calculated according to Equation 9.

$$\text{Tensile stress} = F/A \quad (9)$$

where F is the observed force at the specified elongation and A is the cross-sectional area of the unstretched specimen. The tensile strength is calculated using Equation 9 where F is the force needed to break the specimen. Ultimate elongation (E , in percent) is calculated according to Equation 10.

$$E = 100(L - L_0)/L_0 \quad (10)$$

where L is the length between benchmarks of the specimen at rupture and L_0 is the unstretched length between benchmarks. The data obtained in ASTM D412 also allow calculation of the modulus of elasticity, defined as the stress (elongation) divided by the strain (force applied).

Another test, ASTM D638 (discussed elsewhere) also provides information on modulus of elasticity.

6.6.4 Seal Hardness

Seal hardness shall be determined using ASTM D2240. This method is described in Section 6.5.3 of this test plan.

6.6.5 Seal Permeability

The permeability of seals to liquids shall be determined by using a large sample of the seal material (shaped into a sheet) and testing it similarly to a lining material using ASTM D814 ("Vapor Transmission of Volatile Liquids"). This method is described in Section 6.5.6.

The permeability of seals to gases shall be determined using ASTM D1434. Samples must be in sheet form, free of imperfections, and cut to fit into a gas transmission cell. They are conditioned in a desiccator at 23°C. For the test, a sample is mounted in a gas transmission cell so as to form a sealed semibarrier between two chambers. One chamber contains the test gas at a high pressure and the other, at a lower pressure, receives the permeating gas. The pressure in the receiving cell is monitored as a function of time using a capillary tube filled with mercury. The plot gives a straight line. The volume-flow rate, V_r , in microliters per second is calculated from the slope of the pressure-time line as shown in Equation 11.

$$V_r = \text{slope} \times a_c \quad (11)$$

where slope is the rate of pressure rise in mm/sec and a_c is the cross-sectional area of the capillary in mm². The GTR is calculated according to Equation 12.

$$\text{GTR} = 10^{-6} \times p_0 \times V_r / ART \quad (12)$$

where p_0 is the ambient pressure in Pa, A is the transmitting area of the specimen in mm², R is the universal gas constant, and T is the ambient temperature in K.

6.6.6 Seal Compression Set

The compression set of seals will be determined using ASTM D1414 and ASTM D395 ("Rubber Property -- Compression Set") referenced therein. Specimens are cylindrical disks 12.5 mm thick and 29 mm in diameter. The test specimen is compressed for a certain time at a certain temperature either by a specified force or to a specified deflection. This is achieved by placing the sample in a press with either a calibrated spring to provide constant force or spacers to provide constant deflection. The compression set is calculated as a percentage of the original deflection. For example, if constant deflection is used, compression set is calculated according to Equation 13.

$$C_B = [(t_0 - t_f) / (t_0 - t_n)] \times 100 \quad (13)$$

where C_B is compression set, t_0 is the original thickness of the specimen, t_f is the final thickness

of the specimen, and t_n is the thickness of the spacer bar used.

6.6.7 Seal Chemical Changes

Chemical changes will be detected by spectroscopic analysis, dynamic mechanical behavior, and thermal response as needed. Test methods that may be used include FTIR, UV-visible spectroscopy, fluorescence, phosphorescence, helium leak detection, DMA, and thermal analysis.

Radiation causes cross-linking of a small percentage of polymer strands and primarily affects mechanical, not chemical, properties. Even at high radiation doses, detection of chemical changes is difficult. If a new type of bond (not present in the original polymer) is formed on irradiation, at high radiation exposures it might be detectable by FTIR spectroscopy. At the doses expected in this study no detectable chemical changes caused by radiation are expected.

7.0 DATA REDUCTION AND ANALYSIS

Methods of data reduction, analysis, and reporting are discussed in each applicable standard; these guidelines will be followed. In addition, the following guidelines will apply. In cases of disagreement between the standard and these guidelines, whichever requirement is more stringent will apply.

All data except that on special test data sheets will be recorded in ink in permanent hardbound notebooks. Data sheets will be kept in a three-ring binder, and all entries will be in ink. Except when in use, the binder will be kept in a locked drawer in a location unlikely to be subjected to water or fire damage. No measurement data is to be entered until the date, investigator's name, and unique sample ID are entered. At the end of each day, data sheets containing new data will be photocopied, and the copies will be removed to a safe place at another site for storage. Observations are to be recorded immediately as they are made. If an error is made, it is to be crossed through with a single line.

At least five measurements will be made for each datum. This provides additional confidence in the values compared to the three measurements sometimes specified and decreases the confidence interval. It also means that if one test result is determined to be in error and is discarded, at least four values will remain.

For each procedure carried out, a standard statistical analysis of the results will be performed (Ref. 43). This analysis includes calculation of the mean value, standard deviation, and 95% confidence interval. In the case of outlying values, the Q test will be applied to determine whether the value should be included or excluded.

8.0 QUALITY ASSURANCE

It is anticipated that this effort will be conducted at QA Level 3. Some of the strategies for ensuring quality will include redundancy factors such as running multiple samples and testing identical samples using different laboratories and procedures. The number of samples run for each test will be at least the minimum specified in the ASTM test method. Statistical analysis will be performed to determine the mean, standard deviation, and confidence interval for each data set. All data sheets will be cross-referenced to the preceding sheets for the same samples.

If more than one lot of a polymer is used in the test plan, testing and analysis will be carried out to determine lot-to-lot variation.

8.1 Records and Reports

Records that furnish documentary evidence of quality will be specified, prepared, and maintained. Records will be legible, identifiable, and retrievable. Records will be protected against damage, deterioration, or loss. Duplicate copies will be made and stored at separate, remote, safe locations. Requirements and responsibilities for record transmittal, distribution, retention, maintenance, and disposition will be published and documented.

8.2 Measuring and Testing Equipment

A calibration check will be conducted before and after each set of sample measurements and documented with the data taken.

8.3 Procedures

In general, the standard procedures in each ASTM test method will be followed. If modifications are needed, these will be carefully documented.

8.4 Training

The technician conducting the testing should have at least a bachelor's degree in a physical science or equivalent plus experience in materials testing. Specific training on the test equipment can be provided by the manufacturer(s).

8.5 Verification

Samples will be sent to other qualified laboratories for testing and verification of results. The test results from different testing laboratories will be compared as described in ASTM D412. This process involves testing identical samples of several materials at a number of testing laboratories, with tests repeated on different days. Standard deviations within laboratories and between laboratories obtained will indicate the precision, reproducibility, and bias of the test method.

8.6 Inventory Control

Each sample will be permanently marked with a unique identifier when it is cut. Marking pens may be used or, if the markings are removed by the test solution, the samples will be notched in a non-tested area. Marking pens have the advantage that they do not affect the material properties; however, marking pen ink may be removed by certain solvents (especially alcohols, ketones, hydrocarbons, and chlorinated solvents). Notching is more permanent but must be done carefully in an area of the sample that will not affect later testing. Samples will be kept only in specified areas, and periodic inventories will be taken to ensure that all samples are present.

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APPENDICES

APPENDIX A

SEARCH TERMS USED WITH ON-LINE DATABASES

Note: In the search terms, a pound sign (#) means a single additional character may or may not be present. This truncation is used to include plurals. A question mark indicates that any character string may follow. The designation (w) means the two words so connected must be adjacent, in the order given. The designation (xn) where x is a small number means that the two words connected by this designation must appear within x words of each other.

acid#	mixed (3n) waste
actinide#	oil#
alcohol#	oxida?
aliphatic	Pantex
aromatic	phosphonitrilic
base#	polyacrylate
Buna-N	polyethylene
butadiene	polypropylene
chlorinated	polysulfide
chloroprene	polytetrafluoroethylene
compatib?	polyvinyl (w) chloride
Department (2n) Energy	radioactive
DOE	resistan?
epichlorohydrin	review
fluorocarbon	Rocky (w) Flats
fluorosilicone	Savannah
glass (w) filled	silicone
Hanford	solvent#
hydrocarbon#	storage
inventor?	stream
isobutylene	styrene
ketone#	transuranic
low-level	Y-12
metal#	

APPENDIX B

COMPOSITIONS OF SIMULATED WASTES

The aqueous hazardous waste stream can be simulated using either a very simple mixture or a slightly more complex mixture with optional additives. Because hydroxide ion and the strong oxidant Cr^{6+} are judged to pose the most severe threats to polymers, these must be included. No evidence was found that nitrate, nitrite, or sodium affect any of the polymers considered. However, these ions may be added if desired to more closely simulate realistic waste streams. Aluminum, the most prevalent metal ion other than sodium, is also not expected to affect polymers, but may optionally be added in concentrations approximating those in the waste stream if desired. An optional bismuth (or other simulant metal) salt can be added to simulate the radionuclides present in mixed LLW. It should be noted that, if optional components are added, their counterions may affect concentrations in the mixture. For example, if bismuth or aluminum is added as the nitrate, the concentration of nitrate ion is increased, and this should be accounted for (perhaps by decreasing the sodium nitrate added). The constituents of the recommended aqueous simulant solution, called Solution A (with optional additives), are listed in Table B-1.

Table B-1. Components of Aqueous Simulant Solution

Species	Required or Optional*	Molarity Desired	Add As	Molarity to Add	Molecular Weight of Compound	Weight to Add per Liter (g)
OH^-	Required	2.05	NaOH	2.05	40.0	82.0
Cr^{6+}	Required	0.015	$\text{Na}_2\text{Cr}_2\text{O}_7$	0.0075	262.0	1.97
NO_3^-	Optional	2.10	NaNO_3	2.10	85.0	178.5
NO_2^-	Optional	0.73	NaNO_2	0.73	69.0	50.4
Na^+	Optional	5.48	Na_2CO_3	0.59**	106.0	62.5
Bi^{3+}	Optional	0.10	$\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$	0.10	485.0	48.5
Al^{3+}	Optional	0.66	$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	0.66	375.0	247.5
<p>* If adding optional components, molarities and weights added may need to be changed from those shown in the table to achieve the desired concentrations of all species.</p> <p>** Assuming simultaneous addition of NaNO_3 and NaNO_2. No extra Na_2CO_3 may be required if enough NaNO_3, NaNO_2, $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, and/or $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ are added to provide the desired sodium ion concentration.</p>						

To prepare Solution A, the required and desired optional components are weighed into a glass or stainless steel container equipped with adequate stirring (magnetic or mechanical). Water is added until the desired total volume is reached, and the solution is stirred to effect dissolution.

Solution B simulates mixed solvent waste and consists of equal parts by volume of 1,1,1-trichloroethane, hexane, toluene, methyl ethyl ketone, 1-butanol, acetonitrile, and dioxane. Solution B can be used for rapid screening for general compatibility with organic solvents. Solutions C, D, and E simulate more specific waste streams. Solution C simulates chlorinated solvent waste streams and consists of neat 1,1,1-trichloroethane. Solution D simulates scintillation solvents and consists of a blend of toluene, xylene, and dioxane (equal parts by volume). Solution E simulates the hexone/kerosene mixture identified at Hanford and consists of a mixture of 90% by volume methyl isobutyl ketone (hexone) with 10% by volume kerosene.

APPENDIX C
TEST DATA SHEETS

Compression Set Data Input
Materials Type:

	A	B	C	D	E	F
1	Duration: 0 Days	Exposure Temperatur		Waste Type:		
2	Specimen No.	1	2	3	AVE.	STD. DEV.
3						
4	Original Thickness (mm)					
5	Final Thickness (mm)					
6	Difference in Thickness(=B5-B4	=B5-B4	=B5-B4	=AVERAGE(B6:D6)	=STDEV(B6:D6)
7	Compression Set	=B6/B4	=C6/C4	=D6/D4	=AVERAGE(B7:D7)	=STDEV(B7:D7)
8						
9	Duration:	Exposure Temperatur		Waste Type:		
10	Specimen No.	1	2	3	AVE.	STD. DEV.
11						
12	Original Thickness (mm)					
13	Final Thickness (mm)					
14	Difference in Thickness(=B13-B12	=B13-B12	=B13-B12	=AVERAGE(B14:D14)	=STDEV(B14:D14)
15	Compression Set	=B14/B12	=C14/C12	=D14/D12	=AVERAGE(B15:D15)	=STDEV(B15:D15)
16						
17	Duration:	Exposure Temperatur		Waste Type:		
18	Specimen No.	1	2	3	AVE.	STD. DEV.
19						
20	Original Thickness (mm)					
21	Final Thickness (mm)					
22	Difference in Thickness(=B21-B20	=C21-C20	=D21-D20	=AVERAGE(B22:D22)	=STDEV(B22:D22)
23	Compression Set	=B22/B20	=C22/C20	=D22/D20	=AVERAGE(B23:D23)	=STDEV(B23:D23)
24						
25	Duration:	Exposure Temperatur		Waste Type:		
26	Specimen No.	1	2	3	AVE.	STD. DEV.
27						
28	Original Thickness (mm)					
29	Final Thickness (mm)					
30	Difference in Thickness(=B29-B28	=C29-C28	=D29-D28	=AVERAGE(B30:D30)	=STDEV(B30:D30)
31	Compression Set	=B30/B28	=C30/C28	=D30/D28	=AVERAGE(B31:D31)	=STDEV(B31:D31)
32						
33	Duration:	Exposure Temperatur		Waste Type:		
34	Specimen No.	1	2	3	AVE.	STD. DEV.
35						
36	Original Thickness (mm)					
37	Final Thickness (mm)					
38	Difference in Thickness(=B37-B36	=C37-C36	=D37-D36	=AVERAGE(B38:D38)	=STDEV(B38:D38)
39	Compression Set	=B38/B36	=C38/C36	=D38/D36	=AVERAGE(B39:D39)	=STDEV(B39:D39)

Compression Set Data Input
Materials Type:

	A	B	C	D	E	F
1	Duration: 0 Days	Exposure Temp:		Waste Type:		
2	Specimen No.	1	2	3	AVE.	STD. DEV.
3						
4	Original Thickness (mm)					
5	Final Thickness (mm)					
6	Difference in Thickness(mm)					
7	Compression Set					
8						
9	Duration:	Exposure Temp:		Waste Type:		
10	Specimen No.	1	2	3	AVE.	STD. DEV.
11						
12	Original Thickness (mm)					
13	Final Thickness (mm)					
14	Difference in Thickness(mm)					
15	Compression Set					
16						
17	Duration:	Exposure Temp:		Waste Type:		
18	Specimen No.	1	2	3	AVE.	STD. DEV.
19						
20	Original Thickness (mm)					
21	Final Thickness (mm)					
22	Difference in Thickness(mm)					
23	Compression Set					
24						
25	Duration:	Exposure Temp:		Waste Type:		
26	Specimen No.	1	2	3	AVE.	STD. DEV.
27						
28	Original Thickness (mm)					
29	Final Thickness (mm)					
30	Difference in Thickness(mm)					
31	Compression Set					
32						
33	Duration:	Exposure Temp:		Waste Type:		
34	Specimen No.	1	2	3	AVE.	STD. DEV.
35						
36	Original Thickness (mm)					
37	Final Thickness (mm)					
38	Difference in Thickness(mm)					
39	Compression Set					

Dimensional Testing Data Input
Materials Type:

	A	B	C	D	E	F
1	Duration: 0 Days	Exposure Temperatu		Waste Type:		
2	Specimen No.	1	2	3	AVE.	STD. DEV.
3	Length, side a-b (mm)					
4	Length, side c-d (mm)					
5	Length, through center (m)					
6	Average Length (mm)	=AVERAGE(B3:B5)	=AVERAGE(C3:C	=AVERAGE(D3:D	=AVERAGE(B6:D6)	=STDEV(B6:D6)
7						
8	Width, side a-d (mm)					
9	Width, side b-c (mm)					
10	Width, through center (m)					
11	Average Width (mm)	=AVERAGE(B8:B10)	=AVERAGE(C8:C	=AVERAGE(D8:D	=AVERAGE(B11:D1	=STDEV(B11:D11)
12						
13	Thickness, corner a (mm)					
14	Thickness, corner b (mm)					
15	Thickness, corner c (mm)					
16	Thickness, corner d (mm)					
17	Thickness, center e (mm)					
18	Average Thickness (mm)	=AVERAGE(B15:B1	=AVERAGE(C13:	=AVERAGE(D13:	=AVERAGE(B18:D1	=STDEV(B18:D18)
19	Volume (mm ³)	=(B6*B11)*B18	=(C6*C11)*C18	=(D6*D11)*D18	=AVERAGE(B19:D1	=STDEV(B19:D19)
20						
21	Specimen Weight (g)				=AVERAGE(B21:D2	=STDEV(B21:D21)
22						
23	Duration:	Exposure Temperatu		Waste Type:		
24	Specimen No.	1	2	3	AVE.	STD. DEV.
25	Length, side a-b (mm)					
26	Length, side c-d (mm)					
27	Length, through center (m)					
28	Average Length (mm)	=AVERAGE(B25:B2	=AVERAGE(C25:	=AVERAGE(D25:	=AVERAGE(B28:D2	=STDEV(B28:D28)
29						
30	Width, side a-d (mm)					
31	Width, side b-c (mm)					
32	Width, through center (m)					
33	Average Width (mm)	=AVERAGE(B30:B3	=AVERAGE(C30:	=AVERAGE(D30:	=AVERAGE(B33:D3	=STDEV(B33:D33)
34						
35	Thickness, corner a (mm)					
36	Thickness, corner b (mm)					
37	Thickness, corner c (mm)					
38	Thickness, corner d (mm)					
39	Thickness, center e (mm)					
40	Average Thickness (mm)	=AVERAGE(B35:B3	=AVERAGE(C35:	=AVERAGE(D35:	=AVERAGE(B40:D4	=STDEV(B40:D40)
41	Volume (mm ³)	=(B28*B33)*B40	=(C28*C33)*C40	=(D28*D33)*D40	=AVERAGE(B41:D4	=STDEV(B41:D41)
42						
43	Specimen Weight (g)				=AVERAGE(B43:D4	=STDEV(B43:D43)
44						
45	% Change in Length	=(B28-B6)/B6	=(C28-C6)/C6	=(D28-D6)/D6	=AVERAGE(B45:D4	=STDEV(B45:D45)
46	% Change in Width	=(B33-B11)/B11	=(C33-C11)/C11	=(D33-D11)/D11	=AVERAGE(B46:D4	=STDEV(B46:D46)
47	% Change in Thickness	=(B40-B18)/B18	=(C40-C18)/C18	=(D40-D18)/D18	=AVERAGE(B47:D4	=STDEV(B47:D47)
48	% Volume Change	=(B41-B19)/B19	=(C41-C19)/C19	=(D41-D19)/D19	=AVERAGE(B48:D4	=STDEV(B48:D48)
49	% Weight Change	=(B43-B21)/B21	=(C43-C21)/C21	=(D43-D21)/D21	=AVERAGE(B49:D4	=STDEV(B49:D49)

Dimensional Testing Data Input
Materials Type:

	A	B	C	D	E	F
1	Duration: 0 Days	Exposure Temperature:		Waste Type:		
2	Specimen No.	1	2	3	AVE.	STD. DEV.
3	Length, side a-b (mm)					
4	Length, side c-d (mm)					
5	Length, through center (mm)					
6	Average Length (mm)					
7						
8	Width, side a-d (mm)					
9	Width, side b-c (mm)					
10	Width, through center (mm)					
11	Average Width (mm)					
12						
13	Thickness, corner a (mm)					
14	Thickness, corner b (mm)					
15	Thickness, corner c (mm)					
16	Thickness, corner d (mm)					
17	Thickness, center e (mm)					
18	Average Thickness (mm)					
19	Volume (mm ³)					
20						
21	Specimen Weight (g)					
22						
23	Duration:	Exposure Temperature:		Waste Type:		
24	Specimen No.	1	2	3	AVE.	STD. DEV.
25	Length, side a-b (mm)					
26	Length, side c-d (mm)					
27	Length, through center (mm)					
28	Average Length (mm)					
29						
30	Width, side a-d (mm)					
31	Width, side b-c (mm)					
32	Width, through center (mm)					
33	Average Width (mm)					
34						
35	Thickness, corner a (mm)					
36	Thickness, corner b (mm)					
37	Thickness, corner c (mm)					
38	Thickness, corner d (mm)					
39	Thickness, center e (mm)					
40	Average Thickness (mm)					
41	Volume (mm ³)					
42						
43	Specimen Weight (g)					
44						
45	% Change in Length					
46	% Change in Width					
47	% Change in Thickness					
48	% Volume Change					
49	% Weight Change					

Hardness Testing Data Input
Materials Type:

	A	B	C	D	E	F	G
1	Duration: 0 Days	Ambient Temperature					
2	Specimen No.	1	2	3	4	AVG.	STD. DEV.
3	Durometer Point Reading						
4	Durometer Point Reading						
5	Durometer Point Reading						
6	Durometer Point Reading						
7	Durometer Point Reading						
8	Average	=AVERAGE(B3:B7)	=AVERAGE(C3:C7)	=AVERAGE(D3:D7)	=AVERAGE(E3:E7)	=AVERAGE(B8:E8)	=STDEV(B8:E8)
9							
10	Duration:	Exposure Temperature		Waste Type:			
11	Specimen No.	1	2	3	4	AVG.	STD. DEV.
12	Durometer Point Reading						
13	Durometer Point Reading						
14	Durometer Point Reading						
15	Durometer Point Reading						
16	Durometer Point Reading						
17	Average	=AVERAGE(B12:B16)	=AVERAGE(C12:C16)	=AVERAGE(D12:D16)	=AVERAGE(E12:E16)	=AVERAGE(B17:E17)	=STDEV(B17:E17)
18	% Hardness Change	(B17-B8)/B8	(C17-C8)/C8	(D17-D8)/D8	(E17-E8)/E8	=AVERAGE(B18:E18)	=STDEV(B18:E18)
19							
20	Duration:	Exposure Temperature		Waste Type:			
21	Specimen No.	1	2	3	4	AVG.	STD. DEV.
22	Durometer Point Reading						
23	Durometer Point Reading						
24	Durometer Point Reading						
25	Durometer Point Reading						
26	Durometer Point Reading						
27	Average	=AVERAGE(B22:B26)	=AVERAGE(C22:C26)	=AVERAGE(D22:D26)	=AVERAGE(E22:E26)	=AVERAGE(B27:E27)	=STDEV(B27:E27)
28	% Hardness Change	(B27-B8)/B8	(C27-C8)/C8	(D27-D8)/D8	(E27-E8)/E8	=AVERAGE(B28:E28)	=STDEV(B28:E28)
29							
30	Duration:	Exposure Temperature		Waste Type:			
31	Specimen No.	1	2	3	4	AVG.	STD. DEV.
32	Durometer Point Reading						
33	Durometer Point Reading						
34	Durometer Point Reading						
35	Durometer Point Reading						
36	Durometer Point Reading						
37	Average	=AVERAGE(B32:B36)	=AVERAGE(C32:C36)	=AVERAGE(D32:D36)	=AVERAGE(E32:E36)	=AVERAGE(B37:E37)	=STDEV(B37:E37)
38	% Hardness Change	(B37-B8)/B8	(C37-C8)/C8	(D37-D8)/D8	(E37-E8)/E8	=AVERAGE(B38:E38)	=STDEV(B38:E38)
39							
40	Duration:	Exposure Temperature		Waste Type:			
41	Specimen No.	1	2	3	4	AVG.	STD. DEV.
42	Durometer Point Reading						
43	Durometer Point Reading						
44	Durometer Point Reading						
45	Durometer Point Reading						
46	Durometer Point Reading						
47	Average	=AVERAGE(B42:B46)	=AVERAGE(C42:C46)	=AVERAGE(D42:D46)	=AVERAGE(E42:E46)	=AVERAGE(B47:E47)	=STDEV(B47:E47)
48	% Hardness Change	(B47-B8)/B8	(C47-C8)/C8	(D47-D8)/D8	(E47-E8)/E8	=AVERAGE(B48:E48)	=STDEV(B48:E48)

Hardness Testing Data Input
Materials Type:

	A	B	C	D	E	F	G
1	Duration: 0 Days	Ambient Temperature					
2	Specimen No.	1	2	3	4	AVG.	STD. DEV.
3	Durometer Point Reading						
4	Durometer Point Reading						
5	Durometer Point Reading						
6	Durometer Point Reading						
7	Durometer Point Reading						
8	Average						
9							
10	Duration:	Exposure Temperature		Waste Type:			
11	Specimen No.	1	2	3	4	AVG.	STD. DEV.
12	Durometer Point Reading						
13	Durometer Point Reading						
14	Durometer Point Reading						
15	Durometer Point Reading						
16	Durometer Point Reading						
17	Average						
18	% Hardness Change						
19							
20	Duration:	Exposure Temperature		Waste Type:			
21	Specimen No.	1	2	3	4	AVG.	STD. DEV.
22	Durometer Point Reading						
23	Durometer Point Reading						
24	Durometer Point Reading						
25	Durometer Point Reading						
26	Durometer Point Reading						
27	Average						
28	% Hardness Change						
29							
30	Duration:	Exposure Temperature		Waste Type:			
31	Specimen No.	1	2	3	4	AVG.	STD. DEV.
32	Durometer Point Reading						
33	Durometer Point Reading						
34	Durometer Point Reading						
35	Durometer Point Reading						
36	Durometer Point Reading						
37	Average						
38	% Hardness Change						
39							
40	Duration:	Exposure Temperature		Waste Type:			
41	Specimen No.	1	2	3	4	AVG.	STD. DEV.
42	Durometer Point Reading						
43	Durometer Point Reading						
44	Durometer Point Reading						
45	Durometer Point Reading						
46	Durometer Point Reading						
47	Average						
48	% Hardness Change						

Permeability Testing Data Input
Material Type:

	A	B	C	D	E	F
1	Exposure Duration: 0 days	Exposure Temp:		Waste Type:		
2	Specimen	1	2	3	AVE.	STD. DEV.
3	Weight of Jar/Lid/Specimen (gm)					
4	Weight of Jar/Lid/Specimen/Waste (gm)					
5	Weight Difference (gm)	=B4-B3	=C4-C3	=D4-D3		
6	Time (hours)					
7	Radius of Specimen (mm)					
8	Radius of Specimen (meter)	=B7/1000	=C7/1000	=D7/1000		
9	Area of Specimen (m ²)	=3.14*(B8 ²)	=3.14*(C8 ²)	=3.14*(D8 ²)		
10	Permeability (g/hr/m ²)	=B5/(B6*B9)	=C5/(C6*C9)	=D5/(D6*D9)	=AVERAGE(B10:D10)	=STDEV(B10:D10)
11						
12	Exposure Duration: 7 days	Exposure Temp:		Waste Type:		
13	Specimen	1	2	3	AVE.	STD. DEV.
14	Weight of Jar after Time Duration (gm)					
15	Weight Difference (gm)	=B4-B14	=C4-C14	=D4-D14		
16	Time (hours)					
17	Radius of specimen (mm)					
18	Radius of Specimen (meter)	=B17/1000	=C17/1000	=D17/1000		
19	Area of Specimen (m ²)	=3.14*(B18 ²)	=3.14*(C18 ²)	=3.14*(D18 ²)		
20	Permeability (g/hr/m ²)	=B15/(B16*B19)	=C15/(C16*C19)	=D15/(D16*D19)	=AVERAGE(B20:D20)	=STDEV(B20:D20)
21	% Permeability Change	=((B20-B10)/B10)*10	=((C20-C10)/C10)*10	=((D20-D10)/D10)*10	=AVERAGE(B21:D21)	=STDEV(B21:D21)
22						
23	Exposure Duration: 14 days	Exposure Temp:		Waste Type:		
24	Specimen	1	2	3	AVE.	STD. DEV.
25	Weight of Jar after Time Duration (gm)					
26	Weight Difference (gm)	=B4-B25	=C4-C25	=D4-D25		
27	Time (hours)					
28	Radius of specimen (mm)					
29	Radius of Specimen (meter)	=B28/1000	=C28/1000	=D28/1000		
30	Area of Specimen (m ²)	=3.14*(B29 ²)	=3.14*(C29 ²)	=3.14*(D29 ²)		
31	Permeability (g/hr/m ²)	=B26/(B27*B30)	=C26/(C27*C30)	=D26/(D27*D30)	=AVERAGE(B31:D31)	=STDEV(B31:D31)
32	% Permeability Change	=((B31-B21)/B21)*10	=((C31-C21)/C21)*10	=((D31-D21)/D21)*10	=AVERAGE(B32:D32)	=STDEV(B32:D32)
33						
34	Exposure Duration: 28 days	Exposure Temp:		Waste Type:		
35	Specimen	1	2	3	AVE.	STD. DEV.
36	Weight of Jar after Time Duration (gm)					
37	Weight Difference (gm)	=B4-B36	=C4-C36	=D4-D36		
38	Time (hours)					
39	Radius of specimen (mm)					
40	Radius of Specimen (meter)	=B39/1000	=C39/1000	=D39/1000		
41	Area of Specimen (m ²)	=3.14*(B40 ²)	=3.14*(C40 ²)	=3.14*(D40 ²)		
42	Permeability (g/hr/m ²)	=B37/(B38*B41)	=C37/(C38*C41)	=D37/(D38*D41)	=AVERAGE(B42:D42)	=STDEV(B42:D42)
43	% Permeability Change	=((B42-B32)/B32)*10	=((C42-C32)/C32)*10	=((D42-D32)/D32)*10	=AVERAGE(B43:D43)	=STDEV(B43:D43)
44						
45	Exposure Duration: 180 days	Exposure Temp:		Waste Type:		
46	Specimen	1	2	3	AVE.	STD. DEV.
47	Weight of Jar after Time Duration (gm)					
48	Weight Difference (gm)	=B4-B47	=C4-C47	=D4-D47		
49	Time (hours)					
50	Radius of specimen (mm)					
51	Radius of Specimen (meter)	=B50/1000	=C50/1000	=D50/1000		
52	Area of Specimen (m ²)	=3.14*(B51 ²)	=3.14*(C51 ²)	=3.14*(D51 ²)		
53	Permeability (g/hr/m ²)	=B48/(B49*B52)	=C48/(C49*C52)	=D48/(D49*D52)	=AVERAGE(B53:D53)	=STDEV(B53:D53)
54	% Permeability Change	=((B53-B42)/B42)*10	=((C53-C42)/C42)*10	=((D53-D42)/D42)*10	=AVERAGE(B54:D54)	=STDEV(B54:D54)

Permeability Testing Data Input
Material Type:

	A	B	C	D	E	F
1	Exposure Duration: 0 days	Exposure Temp:		Waste Type:		
2	Specimen	1	2	3	AVE.	STD. DEV.
3	Weight of Jar/Lid/Specimen (gm)					
4	Weight of Jar/Lid/Specimen/Waste (g)					
5	Weight Difference (gm)					
6	Time (hours)					
7	Radius of Specimen (mm)					
8	Radius of Specimen (meter)					
9	Area of Specimen (m ²)					
10	Permeability (g/hr/m ²)					
11						
12	Exposure Duration: 7 days	Exposure Temp:		Waste Type:		
13	Specimen	1	2	3	AVE.	STD. DEV.
14	Weight of Jar after Time Duration (g)					
15	Weight Difference (gm)					
16	Time (hours)					
17	Radius of specimen (mm)					
18	Radius of Specimen (meter)					
19	Area of Specimen (m ²)					
20	Permeability (g/hr/m ²)					
21	% Permeability Change					
22						
23	Exposure Duration: 14 days	Exposure Temp:		Waste Type:		
24	Specimen	1	2	3	AVE.	STD. DEV.
25	Weight of Jar after Time Duration (g)					
26	Weight Difference (gm)					
27	Time (hours)					
28	Radius of specimen (mm)					
29	Radius of Specimen (meter)					
30	Area of Specimen (m ²)					
31	Permeability (g/hr/m ²)					
32	% Permeability Change					
33						
34	Exposure Duration: 28 days	Exposure Temp:		Waste Type:		
35	Specimen	1	2	3	AVE.	STD. DEV.
36	Weight of Jar after Time Duration (g)					
37	Weight Difference (gm)					
38	Time (hours)					
39	Radius of specimen (mm)					
40	Radius of Specimen (meter)					
41	Area of Specimen (m ²)					
42	Permeability (g/hr/m ²)					
43	% Permeability Change					
44						
45	Exposure Duration: 180 days	Exposure Temp:		Waste Type:		
46	Specimen	1	2	3	AVE.	STD. DEV.
47	Weight of Jar after Time Duration (g)					
48	Weight Difference (gm)					
49	Time (hours)					
50	Radius of specimen (mm)					
51	Radius of Specimen (meter)					
52	Area of Specimen (m ²)					
53	Permeability (g/hr/m ²)					
54	% Permeability Change					

**Specific Gravity Testing Data Input
Materials Type:**

	A	B	C	D	E	F	G
1	DATA SUMMARY						
2	Days	0	7	14	28	180	
3	Specific Gravity 23/23	=F13	=F20	=F28	=F36	=F44	
4	% Specific Gravity Change	0	=F21	=F29	=F37	=F45	
5	Standard Deviation	=G13	=G21	=G29	=G37	=G45	
6							
7							
8	Exposure Duration: 0 days	Exposure Temperature:		Waste Type:			
9	Specimen	1	2	3	4	AVE.	STD. DEV.
10	Mass in air (mg)			*			
11	Apparent mass of specimen (mg)						
12	Apparent mass of wire/sinker (mg)						
13	Specific Gravity 23/23 C	= (B10)/(B10+B12-B11)	= (C10)/(C10+C12-C11)	= (D10)/(D10+D12-D11)	= (E10)/(E10+E12-E11)	= AVERAGE(B13:D13)	= STDEV(B13:E13)
14							
15	Exposure Duration: 7 days	Exposure Temperature:		Waste Type:			
16	Specimen	1	2	3	4	AVE.	STD. DEV.
17	Mass in air (mg)						
18	Apparent mass of specimen (mg)						
19	Apparent mass of wire/sinker (mg)						
20	Specific Gravity 23/23 C	= (B17)/(B17+B19-B18)	= (C17)/(C17+C19-C18)	= (D17)/(D17+D19-D18)	= (E17)/(E17+E19-E18)	= AVERAGE(B20:D20)	= STDEV(B20:E20)
21	% Specific Gravity Change	= (B20-B13)/B13	= (C20-C13)/C13	= (D20-D13)/D13	= (E20-E13)/E13	= AVERAGE(B21:D21)	= STDEV(B21:E21)
22							
23	Exposure Duration: 14 days	Exposure Temperature:		Waste Type:			
24	Specimen	1	2	3	4	AVE.	STD. DEV.
25	Mass in air (mg)						
26	Apparent mass of specimen (mg)						
27	Apparent mass of wire/sinker (mg)						
28	Specific Gravity 23/23 C	= (B25)/(B25+B27-B26)	= (C25)/(C25+C27-C26)	= (D25)/(D25+D27-D26)	= (E25)/(E25+E27-E26)	= AVERAGE(B28:D28)	= STDEV(B28:E28)
29	% Specific Gravity Change	= (B28-B13)/B13	= (C28-C13)/C13	= (D28-D13)/D13	= (E28-E13)/E13	= AVERAGE(B29:D29)	= STDEV(B29:E29)
30							
31	Exposure Duration: 28 days	Exposure Temperature:		Waste Type:			
32	Specimen	1	2	3	4	AVE.	STD. DEV.
33	Mass in air (mg)						
34	Apparent mass of specimen (mg)						
35	Apparent mass of wire/sinker (mg)						
36	Specific Gravity 23/23 C	= (B33)/(B33+B35-B34)	= (C33)/(C33+C35-C34)	= (D33)/(D33+D35-D34)	= (E33)/(E33+E35-E34)	= AVERAGE(B36:D36)	= STDEV(B36:E36)
37	% Specific Gravity Change	= (B36-B13)/B13	= (C36-C13)/C13	= (D36-D13)/D13	= (E36-E13)/E13	= AVERAGE(B37:D37)	= STDEV(B37:E37)
38							
39	Exposure Duration: 180 days	Exposure Temperature:		Waste Type:			
40	Specimen	1	2	3	4	AVE.	STD. DEV.
41	Mass in air (mg)						
42	Apparent mass of specimen (mg)						
43	Apparent mass of wire/sinker (mg)						
44	Specific Gravity 23/23 C	= (B41)/(B41+B43-B42)	= (C41)/(C41+C43-C42)	= (D41)/(D41+D43-D42)	= (E41)/(E41+E43-E42)	= AVERAGE(B44:D44)	= STDEV(B44:E44)
45	% Specific Gravity Change	= (B44-B13)/B13	= (C44-C13)/C13	= (D44-D13)/D13	= (E44-E13)/E13	= AVERAGE(B45:D45)	= STDEV(B45:E45)

Specific Gravity Testing Data Input
Materials Type:

	A	B	C	D	E	F	G
1	DATA SUMMARY						
2	Days	0	7	14	28	180	
3	Specific Gravity 23/23						
4	% Specific Gravity Change						
5	Standard Deviation						
6							
7							
8	Exposure Duration: 0 days						
9	Specimen	1	2	3	4	AVE.	STD. DEV.
10	Mass in air (mg)						
11	Apparent mass of specimen (mg)						
12	Apparent mass of wire/sinker (mg)						
13	Specific Gravity 23/23 C						
14							
15	Exposure Duration: 7 days						
16	Specimen	1	2	3	4	AVE.	STD. DEV.
17	Mass in air (mg)						
18	Apparent mass of specimen (mg)						
19	Apparent mass of wire/sinker (mg)						
20	Specific Gravity 23/23 C						
21	% Specific Gravity Change						
22							
23	Exposure Duration: 14 days						
24	Specimen	1	2	3	4	AVE.	STD. DEV.
25	Mass in air (mg)						
26	Apparent mass of specimen (mg)						
27	Apparent mass of wire/sinker (mg)						
28	Specific Gravity 23/23 C						
29	% Specific Gravity Change						
30							
31	Exposure Duration: 28 days						
32	Specimen	1	2	3	4	AVE.	STD. DEV.
33	Mass in air (mg)						
34	Apparent mass of specimen (mg)						
35	Apparent mass of wire/sinker (mg)						
36	Specific Gravity 23/23 C						
37	% Specific Gravity Change						
38							
39	Exposure Duration: 180 days						
40	Specimen	1	2	3	4	AVE.	STD. DEV.
41	Mass in air (mg)						
42	Apparent mass of specimen (mg)						
43	Apparent mass of wire/sinker (mg)						
44	Specific Gravity 23/23 C						
45	% Specific Gravity Change						

**Stress Cracking Data Input
Materials Type:**

	A	B	C	D	E	F	G	H	I	J	K	L
1	DATA SUMMARY											
2	Days	0	7	14	28	180						
3	% Failure	0	=L17	=L24	=L31	=L38						
4												
5	Exposure Duration: 0 Days	Exposure Temp:		Waste Type								
6	Specimen No.	1	2	3	4	5	6	7	8	9	10	Total
7												
8	Pass											
9	Fail											
10	% Failure											
11												
12	Exposure Duration: Days	Exposure Temp:		Waste Type								
13	Specimen No.	1	2	3	4	5	6	7	8	9	10	Total
14												
15	Pass											
16	Fail											
17	% Failure											
18												
19	Exposure Duration: Days	Exposure Temp:		Waste Type								
20	Specimen No.	1	2	3	4	5	6	7	8	9	10	Total
21												
22	Pass											
23	Fail											
24	% Failure											
25												
26	Exposure Duration: Days	Exposure Temp:		Waste Type								
27	Specimen No.	1	2	3	4	5	6	7	8	9	10	Total
28												
29	Pass											
30	Fail											
31	% Failure											
32												
33	Exposure Duration: Days	Exposure Temp:		Waste Type								
34	Specimen No.	1	2	3	4	5	6	7	8	9	10	Total
35												
36	Pass											
37	Fail											
38	% Failure											

Tensile Testing Data Input
Material Type:

	A	B	C	D	E	F	G	H
1	Duration: 0 Days	Exposure Temp.						
2	Specimen No.	1	2	3	4	5	AVE.	STD. DEV.
3								
4	Neck Thick (in.)							
5	Neck Thick (in.)							
6	Neck Thick (in.)							
7	Average Thickness (in.)	= AVERAGE(B4:B6)	= AVERAGE(C4:C6)	= AVERAGE(D4:D6)	= AVERAGE(E4:E6)	= AVERAGE(F4:F6)	= AVERAGE(B7:F7)	= STDEV(B7:F7)
8								
9	Neck Width (in.)							
10	Neck Width (in.)							
11	Neck Width (in.)							
12	Average Width (in.)	= AVERAGE(B9:B11)	= AVERAGE(C9:C11)	= AVERAGE(D9:D11)	= AVERAGE(E9:E11)	= AVERAGE(F9:F11)	= AVERAGE(B12:F12)	= STDEV(B12:F12)
13								
14	Ave. Cross-Section Area (in. ²)	= B7*B12	= C7*C12	= D7*D12	= E7*E12	= F7*F12	= AVERAGE(B14:F14)	= STDEV(B14:F14)
15	Maximum Load (lbf)						= AVERAGE(B15:F15)	= STDEV(B15:F15)
16	Tensile Strength at Yield or Break (lbf/in. ²)	= B15/B14	= C15/C14	= D15/D14	= E15/E14	= F15/F14	= AVERAGE(B16:F16)	= STDEV(B16:F16)
17								
18	Length between benchmarks (in.)							
19	Elongation at Yield (in)							
20	% Elongation at Yield	= (B19/B18)*100	= (C19/C18)*100	= (D19/D18)*100	= (E19/E18)*100	= (F19/F18)*100	= AVERAGE(B20:F20)	= STDEV(B20:F20)
21	Elongation at Break (in.)							
22	% Elongation at Break	= (B21/B18)*100	= (C21/C18)*100	= (D21/D18)*100	= (E21/E18)*100	= (F21/F18)*100	= AVERAGE(B22:F22)	= STDEV(B22:F22)
23								
24	Stress (linear)							
25	Strain (linear)							
26	Modulus of Elasticity (lbf/in. ²)	= (B24/B25)/B14	= (C24/C25)/C14	= (D24/D25)/D14	= (E24/E25)/E14	= (F24/F25)/F14	= AVERAGE(B26:F26)	= STDEV(B26:F26)
27								
28	Duration:	Exposure Temp.						
29	Specimen No.	1	2	3	4	5	AVE.	STD. DEV.
30								
31	Neck Thick (in.)							
32	Neck Thick (in.)							
33	Neck Thick (in.)							
34	Average Thickness (in.)	= AVERAGE(B31:B33)	= AVERAGE(C31:C33)	= AVERAGE(D31:D33)	= AVERAGE(E31:E33)	= AVERAGE(F31:F33)	= AVERAGE(B34:F34)	= STDEV(B34:F34)
35								
36	Neck Width (in.)							
37	Neck Width (in.)							
38	Neck Width (in.)							
39	Average Width (in.)	= AVERAGE(B36:B38)	= AVERAGE(C36:C38)	= AVERAGE(D36:D38)	= AVERAGE(E36:E38)	= AVERAGE(F36:F38)	= AVERAGE(B39:F39)	= STDEV(B39:F39)
40								
41	Ave. Cross-Section Area (in. ²)	= B34*B39	= C34*C39	= D34*D39	= E34*E39	= F34*F39	= AVERAGE(B41:F41)	= STDEV(B41:F41)
42	Maximum Load (lbf)						= AVERAGE(B42:F42)	= STDEV(B42:F42)
43	Tensile Strength at Yield or Break (lbf/in. ²)	= B42/B41	= C42/C41	= D42/D41	= E42/E41	= F42/F41	= AVERAGE(B43:F43)	= STDEV(B43:F43)
44								
45	Length between benchmarks (in.)							
46	Elongation at Yield (in)							
47	% Elongation at Yield	= (B46/B45)*100	= (C46/C45)*100	= (D46/D45)*100	= (E46/E45)*100	= (F46/F45)*100	= AVERAGE(B47:F47)	= STDEV(B47:F47)
48	Elongation at Break (in.)							
49	% Elongation at Break	= (C46/C49)*100	= (D46/D49)*100	= (E46/E49)*100	= (F46/F49)*100	= (G46/G49)*100	= AVERAGE(B49:F49)	= STDEV(B49:F49)
50								
51	Stress (linear)							
52	Strain (linear)							
53	Modulus of Elasticity (lbf/in. ²)	= (B51/B52)/B41	= (C51/C52)/C41	= (D51/D52)/D41	= (E51/E52)/E41	= (F51/F52)/F41	= AVERAGE(B53:F53)	= STDEV(B53:F53)

Tensile Testing Data Input
Material Type:

	A	B	C	D	E	F	G	H
1	Duration: 0 Days	Exposure Temp.						
2	Specimen No.	1	2	3	4	5	AVE.	STD. DEV.
3								
4	Neck Thick (in.)							
5	Neck Thick (in.)							
6	Neck Thick (in.)							
7	Avg Thickness(in.)							
8								
9	Neck Width (in.)							
10	Neck Width (in)							
11	Neck Width (in)							
12	Average Width(in.)							
13								
14	Ave. Cross-Section Area (in.^2)							
15	Maximum Load (lbf)							
16	Tensile Strength at Yield or Break (lbf/in^2)							
17								
18	Length between benchmarks (in.)							
19	Elongation at Yield (in)							
20	% Elongation at Yield							
21	Elongation at Break (in.)							
22	% Elongation at Break							
23								
24	Stress (linear)							
25	Strain (linear)							
26	Modulus of Elasticity (lbf/in.^2)							
27								
28	Duration:	Exposure Temp.						
29	Specimen No.	1	2	3	4	5	AVE.	STD. DEV.
30								
31	Neck Thick (in.)							
32	Neck Thick (in.)							
33	Neck Thick (in.)							
34	Average Thickness(in.)							
35								
36	Neck Width (in.)							
37	Neck Width (in)							
38	Neck Width (in)							
39	Average Width(in.)							
40								
41	Ave. Cross-Section Area (in.^2)							
42	Maximum Load (lbf)							
43	Tensile Str at Yield or Break (lbf/in^2)							
44								
45	Length between benchmarks (in.)							
46	Elongation at Yield (in)							
47	% Elongation at Yield							
48	Elongation at Break (in.)							
49	% Elongation at Break							
50								
51	Stress (linear)							
52	Strain (linear)							
53	Modulus of Elasticity (lbf/in.^2)							

APPENDIX D

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