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# IMMOBILIZATION OF SODIUM NITRATE WASTE WITH POLYMERS

## TOPICAL REPORT

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## Abstract

This report describes work performed at Brookhaven National Laboratory (BNL) on the development of solidification systems for sodium nitrate waste. Sodium nitrate waste was solidified in several types of polymers: polyethylene, polyester-styrene (PES), and water-extendible polyester-styrene (WEP). Evaluations were made of the properties of waste forms containing various amounts of sodium nitrate by leaching (ANS 16.1), immersion in water, measuring compressive strengths and by the EPA Extraction Procedure.

The process, developed for solidification of the salt waste in polyethylene, utilizes a commercially available single-screw extruder which continuously discharges a prescribed polyethylene-waste mixture from the hoppers to the output die where it is extruded into a container while still in the molten form. The molten mixture ( $\sim 110$ - $120^{\circ}\text{C}$ ) conforms to the shape of the container and solidifies upon cooling. Proportional feeders maintain quality control and homogeneity of the waste form over a wide range of waste-binder ratios. Present studies utilize dry wastes although wet solid wastes also can be processed using vented extruders of the type used for the solidification of bitumen. The simplicity of the system ensures reproducible, homogenous waste forms.

Results of the leaching test are presented as cumulative fraction leached (CFL), incremental leaching rate, and average leaching indices (LI). For waste forms containing 30 to 70 wt% sodium nitrate, the CFL ranged from  $9.0 \times 10^{-3}$  to  $7.3 \times 10^{-1}$  and the LI from 11 to 7.8. After ninety days immersion in water, the compressive strengths ranged from 720 psi to 2550 psi. The nitrate releases from these samples using the EPA Extraction Procedure were below 500 ppm.

The two basic types of PES used were ordinary PES resins, which can be used to solidify dry wastes, and water extendible polyester (WEP), capable of immobilizing either dry or wet wastes. A promoter-catalyst system facilitated curing at room temperature.

The nitrate releases from PES waste forms were similar to those from polyethylene waste forms at the same waste loadings. The compressive yield strengths, measured after ninety-day immersion in water, ranged between 2070 and 7710 psi. In the case of WEP waste forms, only 30 wt% loaded samples passed the immersion test.

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## 1. INTRODUCTION

Sodium nitrate waste represents one of the high volume waste streams common to many Department of Energy (DOE) facilities which are involved in the processing and reprocessing of nuclear fuel.<sup>1</sup>

Methods for the treatment or disposal of this type of waste include thermal destruction of nitrate salts, chemical reduction of nitrate compounds, biodenitrification, and physical disposal. Disposal of the salt waste at DOE burial sites requires prior solidification.

Currently, the most common methods of solidification of sodium nitrate wastes for transportation/disposal involve the use of portland cements, with and without additives. Cement is an open-cell structure because of the porosity that develops while it cures. As such, it is permeable to water and susceptible to radionuclide and nitrate salt loss by leaching. Cementitious grouts of portland cement containing high concentrations of fly-ash and clays are also being considered. Although the use of such admixtures tends to reduce the leachability of some of the waste components, the amount of waste which can be incorporated (volumetric efficiency) also is lowered.

The more stringent standards for drinking water established in recent years limit the amount of nitrates that can be released into the ground. Therefore, several solidification agents are being considered which have the potential for greater durability, lower leachabilities, and higher volumetric efficiencies than the cementitious materials now in use. Studies are presently being conducted at Oak Ridge National Laboratory (ORNL) on the immobilization of nitrate salts with bitumen.

This report describes the work performed at Brookhaven National Laboratory (BNL) on the development of polyethylene and polyester-styrene processes for the immobilization of dried and wet nitrate wastes.

## 2. NITRATE SALT WASTE

Nitrate salt waste is produced in fuel cycle operations which use nitric acid. These operations include fuel fabrication, spent fuel reprocessing, purification and enrichment of uranium, regeneration of ion exchange resins, and decontamination. In fuel reprocessing, the first raffinate is considered as high-level waste (HLW). However, when HLW waste is treated to reduce its volume, large volumes of low-level nitrate salt waste are generated. Presently, more than 250 million kilograms of nitrate salt waste<sup>2</sup> are stored, primarily in DOE facilities. Although some of the sites are modifying their operations to reduce the rate at which nitrate waste is generated, large volumes are anticipated with the prolongation of the nuclear industry.

The waste streams can vary greatly in radiological, chemical, and physical characteristics, depending on the processing technologies and the subsequent treatment of the "as-generated" waste. This is exemplified in Table 1 which lists the major chemical components of Rocky Flats Plant (RFP) and Savannah River Plant (SRP) nitrate salt waste. Although the most abundant component of the waste streams is  $\text{NaNO}_3$ ,  $\text{KNO}_3$  is present in the RFP waste while  $\text{NaNO}_2$ ,  $\text{Na}_2\text{CrO}_4$ , and  $\text{NaB}(\text{C}_6\text{H}_5)_4$  are present in the SRP waste. Salt wastes can also contain phosphates, sulfates, ammonia, fluorides, and various organic compounds.

Some of the nitrate salt waste streams are classified as "mixed waste" because of the presence of toxic materials such as chromium, mercury, and certain organic compounds, even if they are present in relatively low concentrations.

Table 1

Chemical Compositions of Sodium Nitrate Waste at the Savannah River Plant  
(SRL) and Rocky Flats Plant (RFP)

<u>ROCKY FLATS PLANT</u> (Spray-Dried Nitrate Waste)		<u>SAVANNAH RIVER PLANT</u> (Decontaminated, Aged Salt Solution)	
<u>COMPOUND</u>	<u>(WT %)</u>	<u>COMPOUND</u>	<u>SOLUTION (WT%)</u>
NaNO <sub>3</sub>	53.9	H <sub>2</sub> O	68.0
KNO <sub>3</sub>	33.9	NaNO <sub>3</sub>	15.6
NaCl	3.2	NaNO <sub>2</sub>	3.9
KCl	2.2	NaOH	4.2
Na <sub>2</sub> SO <sub>4</sub>	2.8	NaCO <sub>3</sub>	1.7
K <sub>2</sub> SO <sub>4</sub>	1.8	NaAlO <sub>2</sub>	3.6
Na <sub>3</sub> PO <sub>4</sub>	0.9	Na <sub>2</sub> SO <sub>4</sub>	1.9
K <sub>3</sub> PO <sub>4</sub>	0.6	NaF	0.06
NaF	0.4	NaCl	0.12
KF	0.3	Na <sub>2</sub> SiO <sub>3</sub>	0.04
		Na <sub>2</sub> CrO <sub>4</sub>	0.05
		Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>	0.31
		Na <sub>3</sub> PO <sub>4</sub>	0.13
		NaB(C <sub>6</sub> H <sub>5</sub> ) <sub>4</sub>	0.06
		Other Salts	0.20

Mixed wastes are subject to regulations under the EPA Resource Conservation and Recovery Act (RCRA) until immobilized to prevent the release of the toxic materials above regulatory limits.

Since NaNO is the major component of nitrate waste streams, it was used to simulate the salt waste both in the process development and evaluation of the waste form. Simulated SRP and RFP wastes were used for studies of

binder-waste compatibility. However, some compatibility experiments also were performed with actual SRP waste. Because polyethylene and ordinary polyester-styrene (PES) are not compatible with aqueous wastes, dried wastes were used with these binder materials. However, salt solutions and slurries were used in studies with water-extendible polyester-styrene (WEP).

### 3. SOLIDIFICATION AGENTS

#### 3.1 Polyethylene

Polyethylene is a thermoplastic organic polymer of crystalline-amorphous structure formed through the polymerization of ethylene gas. Thermoplastic polymers consist of branched or linear polymer chains that normally are not crosslinked. However, these chains can be cross-linked by the addition of cross-linking agents or exposure to radiation, both of which transform the polymer from a thermoplastic to a thermoset material. At elevated temperatures thermoplastic polymers change from a hard material to a rubbery flowable liquid. On cooling, the polymers revert to their original form. Examples of other thermoplastic polymers are polystyrene and polymethyl methacrylate (Lucite).

The use of polyethylene to encapsulate low-level waste (LLW) was originally suggested by researchers at Oak Ridge National Laboratory (ORNL)<sup>3</sup>. Polyethylene has been used for solidification of low- and intermediate-level wastes in The Netherlands (unpublished) and in Argentina<sup>4,5</sup>. Currently researchers in Japan<sup>6</sup> are investigating various methods of encapsulating in polyethylene waste generated at nuclear power plants. Our studies on the development of polyethylene as an improved solidification agent for sodium nitrate waste are an extension of previous work performed at BNL on the immobilization of PWR and BWR evaporator concentrates and ion exchange resins in polyethylene, using an extrusion process<sup>7,8,9</sup>. The development of the polyethylene process was based on such considerations as compatibility with waste, efficiency of solidification, material properties, availability of materials, economic feasibility and ease of processibility. Since the solidification

process does not depend upon complex chemical reactions as in the case of hydraulic cements and thermosetting polymers, the processing is simplified, and solidification is assured upon cooling of the melt.

3.1.1 Properties of Polyethylene. Virtually all properties of polymers are affected by the incorporation of wastes or other materials into the matrix. The degree to which this occurs depends on the type and amount of waste incorporated, chemical interactions between the components of the waste and the monomer or polymer system, and the processing parameters used to produce the waste forms. Therefore, an independent approach to each waste-binder system is required to understand the behaviour and application of the selected material.

Polyethylene is resistant to most acids, bases, and organics normally encountered in waste streams. The superior mechanical properties of polyethylene (i.e., compressive strength) allow higher waste loadings than normally can be incorporated into other materials such as cement or bitumen, without compromising the integrity of the waste form.

Tables 2-4 list some of the more pertinent properties of polyethylene<sup>10, 11</sup>. Table 2 gives a comparison of the various properties of polyethylenes at different density ranges. The properties of low-density polyethylene are compared to those of other types of polymers in Tables 3 and 4.

Some of the more important factors which affect the properties of polyethylene are density, molecular weight, molecular weight distribution, melt index, and crosslinking.



Table 2

## Properties of Polyethylenes

Property	Low Density	Medium Density	High Density
Density, g/cc	0.910 - 0.925	0.926 - 0.940	0.941 - 0.965
Tensile Strength, MPa (psi)	4.14 - 15.86 600 - 2300	8.27 - 24.13 1200 - 3500	21.37 - 37.92 3100 - 5500
Compressive Strength, MPa (psi)	---	---	18.61 - 24.82 2700 - 3600
Water Absorbance (24 hr, 1/8" thick, %)	<0.01	<0.01	<0.01
Flammability (Burn Rate, in/min)	1.04	1.00 - 1.04	1.00 - 10.4
Average Extent of Burning, in.	0.8	0.6	---
Average Time of Burning, sec.	<5 - 25	10 - 60	---
Effect of Weak Acid	Resistant	Very Resistant	Very Resistant
Effect of Strong Acid	Attacked by Oxidizing Acids	Attacked Slowly by Oxidizing Acids	Attacked Slowly by Oxidizing Acids
Effect of Weak Alkalies	Resistant	Very Resistant	Very Resistant
Effect of Strong Alkalies	Resistant	Very Resistant	Very Resistant
Effect of Organic Solvents	Resistant Below 60°C Except to Chlorinated Solvents	Resistant Below 60°C Except to Chlorinated Solvents	Resistant Below 80°C

Table 3

## Physical, Mechanical and Thermal Properties of Polymers

<u>Properties</u>	<u>ASTM Test Method</u>	<u>Epoxy (thermoset)</u>	<u>Polyester- Styrene (thermoset)</u>	<u>Polystyrene (thermoplastic)</u>	<u>Low-Density Polyethylene (thermoplastic)</u>
<u>PHYSICAL</u>					
Specific Gravity	D792	1.11-1.40	1.1-1.5	1.04-1.09	0.917-0.925
Water Absorption 24 hr., 1/8-in. thick, %	D572	0.08-0.15	0.15-0.6	0.03-0.1	<0.1
<u>MECHANICAL</u>					
Tensile Strength, psi	D638	4,000-13,000	6,000-13,000	5,000-12,000	600-2,300
Elongation, %	D638	3.0-6.0	-	1.0-2.5	90-800
Compres. Strength, psi	D695	15,000-25,000	13,000-30,000	11,500-16,000	No Break Point
Hardness, Rockwell	D785	M80-110	M70-115	M65-80	M124-128
<u>THERMAL</u>					
Coef. of Linear Thermal Expansion, $10^{-5}$ in/in/°C	D696	4.5-6.5	5.5-10.0	6.0-8.0	2.5-6.0
Thermal Conductivity $10^{-4}$ cal-cm/sec-cm <sup>2</sup> -°C	C177	4.0-5.0	4.0	2.4-3.3	3.0-6.0

Table 4

## Resistance Characteristics of Polymers

<u>Properties</u>	<u>ASTM Test Method</u>	<u>Epoxy (thermoset)</u>	<u>Polyester- Styrene (thermoset)</u>	<u>Polystyrene (thermoplastic)</u>	<u>Low-Density Polyethylene (thermoplastic)</u>
Burning Rate, in./min.	D635	Slow	Slow to Self Extinguishing	<1.5 (Slow)	1.04
Effect of Weak Acids	D543	None	None	None	Resistant
Effect of Strong Acids	D543	Attacked by Some	Attacked	Attacked Slowly by Oxidizing Acids	Attacked by Oxidizing Acids
Effect of Weak Alkalies	D543	None	Slight	None	Resistant
Effect of Strong Alkalies	D543	Slight	Attacked	None	Resistant
Effect of Organic Solvents	D543	Generally Resistant	Slight to Considerable	Soluble in Aromatics	Resistant Below 60°C except to chlorinated solvents

Density - The degree of crystallinity of polyethylene determines its density which in turn affects a range of material properties. A variety of polyethylenes is commercially available, ranging from soft waxes to very tough plastics. The American Society of Testing and Materials (ASTM) grades polyethylene into three types: low, medium, and high, with respective density ranges of 0.910 to 0.925, 0.926 to 0.940, and 0.941 to 0.965g/cc.

The higher density polyethylenes (HDPE) have relatively few side branches allowing the chains to pack together more closely in an orderly fashion, giving it a hard crystalline character. Since the chains are closely packed, higher temperatures are required to overcome Van der Waal's forces. A higher degree of branching and consequent disorderly and looser arrangement of polymer chains results in polyethylenes of lower density, exhibiting lower melt temperatures.

Molecular Weight - The molecular weight (MW) is the average of all sizes of polymer chains produced during polymerization<sup>12</sup>. The higher the MW, the more energy (temperature, pressure) is required to process it. With an increase in MW, however, there is a corresponding increase in strength, toughness, and chemical resistance.

Molecular Weight Distribution - Polymers are made up of molecules of varying chain length, and the molecular weight distribution describes the range of chain sizes present. If most of the molecules fall within a very narrow range of MW, the product will have better mechanical properties than one with a broader range of MW. However, such materials are more difficult to process since they require higher extrusion temperatures.

Melt Index - The melt index is probably the most important consideration since it determines the ease with which the molten polyethylene flows under prescribed conditions of temperature and pressure. The melt index, as specified by ASTM D-1238, is a measure of the viscosity of the melt at 190°C and is expressed in units of grams/10 minutes. Values for the melt index can range from <1g/10 minutes for high molecular weight polyethylene to >4000g/10 minutes for some low molecular weight polyolefins. For typical applications however, polyethylenes have melt indices ranging between 1 and 60g/10 minutes.

Cross-Linking - Cross-linking of polyethylene, whether induced by the addition of a cross-linking agent or by exposure to radiation, leads to beneficial changes in many of its properties such as heat resistance, compressive strength and chemical resistance.

3.1.2 Low-Density Polyethylene (LDPE). The properties of low, medium, and high-density polyethylene (HDPE) are compared in Table 2. Properties such as the mechanical strength and chemical resistance of the HDPE might give it an advantage over LDPE for the solidification of LLW. However, because higher temperatures and pressures are required for the processing of HDPE than for LDPE, LDPE is preferred for the solidification of LLW<sup>6,7,8</sup>. A low processing temperature is more desirable for economic reasons, and also for ease of processibility and prevention of volatilization/decomposition of the waste components.

An experimental survey of several commercially available LDPEs established preferred materials based on processing parameters such as melt temperature, pressure, melt index (a measure of viscosity) and extrusion rate,

achievable waste loadings, and test results of waste form performance'. Polyethylenes with a density range of 0.917-0.924 g/cm<sup>3</sup>, melt indices of 2.0-55.0g/10 min., MW of 19,000-76,000, and MW distribution of 2.5-3.8 were evaluated. A LDPE with a density of 0.924g/cm<sup>3</sup>, melt index of 55g/10 min., MW of 55,000 and a MW distribution of 2.5 was determined to be most suitable for the encapsulation of salt wastes.

### 3.2 Polyester-Styrene

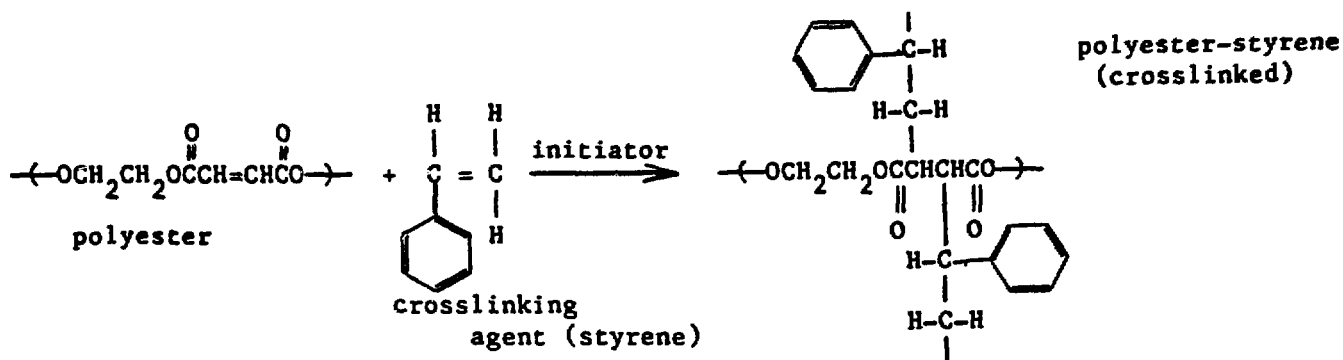
Polyester-styrene is a thermoset polymer. Once a thermoset is formed through application of certain chemicals or heat which create tightly cross-linked chains, it cannot be remelted or reformed. Thermosetting polymers such as epoxy, unsaturated polyester, and vinyl ester-styrene, have been applied in several countries for the solidification of low- and intermediate-level waste<sup>13, 14, 15</sup>.

The selection of polyester resin as a potential solidification agent for sodium nitrate waste was based on its commercial availability, cost, resistance to water permeation, chemical and mechanical properties, and ease of processibility. Thus far, several types of commercially available unsaturated polyester-based thermosetting resins have been investigated. These consist primarily of polyester-styrene resins which can be used to solidify dry wastes and those which can be used to immobilize aqueous waste by forming an emulsion with water. The latter type is known as a water-extendible polyester (WEP) and will immobilize either dry or wet (slurries, solutions) wastes, similar to the DOW vinyl-ester-styrene system.

3.2.1 Monomer-Polymer Conversion. The essential ingredients in commercial unsaturated polyester resins are a linear polyester resin, a cross-linking monomer, and inhibitors to prevent polymerization until the resin is ready for use. The usual cross-linking monomers are styrene, methyl methacrylate, vinyl toluene, alpha-methyl styrene, and diallyl phthalate. The cross-linking monomer most extensively used is styrene. Polyester-styrene mixtures are available from the manufacturer in varying proportions depending on their intended use. Manufacturers make other modifications of the basic resins such as the addition of promoters for initiating cures at room temperature, and incorporation of thixotropic agents such as pyrogenic silica for increasing the viscosity of the resin mixture. Other admixtures may be added for flame retardancy, improved chemical resistance, better weathering characteristics, and minimized shrinkage during the cure period.

The process of converting polyester resins from liquids to solids involves the chemical reaction between the polyester resin and the monomer in which the polyester is dissolved, to form a cross-linked, thermoset polymer. This chemical reaction is activated by a free radical mechanism which opens the unsaturated (double) bonds in the resin and monomer. Once a thermoset material is formed, the plastic cannot be reformed or remelted as can thermoplastic materials such as polyethylene.

A typical reaction of a catalyzed mixture of polyester resin and styrene monomer to form a cross-linked polyester-styrene is as follows:



This reaction continues until all the reactive monomer molecules are depleted.

Usually, the co-polymerization reaction is promoted by the addition of so-called catalyst bodies which decompose to form very reactive free radicals that become the centers for polymer growth. The principal materials used are organic peroxides such as methyl ethyl ketone peroxide (MEKP), cumene hydroperoxide, and benzoyl peroxide. The decomposition of the catalyst to form free radicals can be accomplished by heating the system to the temperature at which the catalyst decomposes at a fairly rapid rate; alternatively, promoters (accelerators) can be added to initiate the decomposition of the catalyst to form free radicals at room temperature or at temperatures considerably below those required when catalyst alone is used. Promoters consist of metallic salts (i.e., cobalt naphthenate), anilines (i.e., diethyl or dimethyl aniline), and mercaptans (i.e., dodecyl mercaptan), and therefore are characterized as reducing agents. The efficiency of a promoter is specifically related to the type of catalyst, so that it is generally best to follow the recommendations of suppliers in selecting catalyst-promoter systems.



Cure times depend on a combination of temperature, catalyst-promoter combinations, and admixtures which can retard or accelerate the rate of polymerization. The polymerization reaction is exothermic, resulting in increased temperatures, which in turn increase the free-radical activity. This unique situation is called autoacceleration and may result in an excessive temperature rise if not controlled.

3.2.2 Water-Extendible Polyester-Styrene (WEP). Water-extendible polyester-styrene resins form an emulsion with water rendering it compatible with wet wastes such as slurries or solutions of nitrate salts. There are two types of WEP: those that have a closed-pore structure and those that have open or interconnected pores. The interconnected pore system loses all water from the emulsion during curing, rendering the pores readily accessible to leaching waters. The closed pore system consists of small water droplets completely surrounded by polymer. In order for the encapsulated waste to be leached, water must first diffuse through this polymer barrier. The closed-pore system was considered for waste solidification studies.

#### 4. STUDIES OF WASTE-BINDER COMPATIBILITY

The chemical composition of a waste stream should be considered when developing a solidification system to avoid processing problems resulting from waste-binder interactions. Both SRP and RFP waste streams contain a substantial amount of sodium nitrate ( $\text{NaNO}_3$ ), however, there are notable differences in the types and concentration of other chemical constituents. Of particular interest in this study is the presence of sodium nitrite ( $\text{NaNO}_2$ ) in SRP waste and potassium nitrate ( $\text{KNO}_3$ ) in RFP waste. Both compounds, along with  $\text{NaNO}_3$ , are categorized as strong oxidizers and are capable of becoming unstable at elevated temperatures or in combination with organic materials.

Since the solidification agents considered for this study are organic materials (polyethylene, polyester-styrene), a series of experiments were conducted to determine the thermal stability of the major waste components in the presence of polymers at elevated temperatures. A differential scanning calorimeter (DSC) was used to characterize the thermal behavior of selected compounds and to determine the effects of oxidizing compounds on organic polymers. The self-ignition temperature of polymers, separately and in combination with several selected waste components, were also measured.

##### 4.1 Studies of Thermal Stability

A Perkin-Elmer Differential Scanning Calorimeter (DSC) was used to characterize the thermal behavior of the compounds listed in Table 5.

Table 5

## Compounds Tested for Thermal Stability Using a Differential Scanning Calorimeter

Polyethylene	Polyethylene + NaNO <sub>3</sub>
NaNO <sub>3</sub>	" + NaNO <sub>2</sub>
NaNO <sub>2</sub>	" + KNO <sub>3</sub>
KNO <sub>3</sub>	" + Na <sub>2</sub> SO <sub>4</sub>
Na <sub>2</sub> SO <sub>4</sub>	" + NaAlO <sub>2</sub>
NaAlO <sub>2</sub>	" + Na <sub>2</sub> CO <sub>3</sub>
Na <sub>2</sub> CO <sub>3</sub>	" + NaOH
NaOH	" + RFP Salt (simulated)
RFP Salt (simulated)	" + SRP Salt (simulated)
SRP Salt (simulated)	" + SRP Salt (real)
SRP Salt (real)	
Polyester-Styrene	Polyester-Styrene + NaNO <sub>3</sub>
NaNO <sub>3</sub>	" + NaNO <sub>2</sub>
NaNO <sub>2</sub>	

Aluminum or gold pans were used to encapsulate -0.1 g samples of the above compounds and compound-solidification agent mixtures (at equal amounts of each). The samples were heated in the DSC oven while the temperature was raised at the rate of 40°C/min over a range from 50°C to 400°C. Nitrogen was used as the carrier gas. The measured differences in heat (calories) intake or output between sample and reference pan represent endothermic and exothermic reactions, respectively. The thermograms, shown in Figures 4.1-4.11, describe the thermal behavior of the various compounds tested. All major peaks of the mixtures could be identified by matching them with the peaks in

the thermograms of the individual compounds. In each case, the thermal profiles depict endothermic reactions characteristic of the melting points and transition temperatures of the respective compounds. No exothermic reactions were observed over the temperature range used.

#### 4.2 Measurements of Self-Ignition Temperature

The self-ignition temperature of a material is defined by the American Society for Testing and Materials (ASTM) as "the lowest initial temperature of air passing around the specimen at which, in the absence of an ignition source, the self-heating properties of the specimen lead to ignition or ignition occurs of itself, as indicated by an explosion, flame, or sustained glow."

The measurements were made according to ASTM-D-1929, "Standard Methods for Ignition Properties of Plastics," on specimens of polyethylene and PES individually and in combination with  $\text{NaNO}_3$ ,  $\text{NaNO}_2$ , and actual SRP salt waste. The polyethylene specimens containing  $\text{NaNO}_2$  were tested at salt loadings of 30, 50, and 70 wt%. All other specimens tested contained 50 wt% salt. The SRP waste solution was vacuum dried at  $20^\circ\text{C}$  prior to incorporation into the polymer binders. The specimen (~3g) was placed in a hot-air ignition furnace with an air flow velocity of 152 cm/s, while the furnace temperature was increased at a rate of  $600^\circ\text{C}/\text{h}$ . The temperature of the specimen and the air passing around the specimen was monitored with chromel-alumel thermocouples. Both temperatures were recorded at the first indication of ignition.

The measured self-ignition temperatures for specimens containing varying concentrations of salt waste are presented in Table 6.

Differential Scanning Calorimeter (DSC) Thermograms

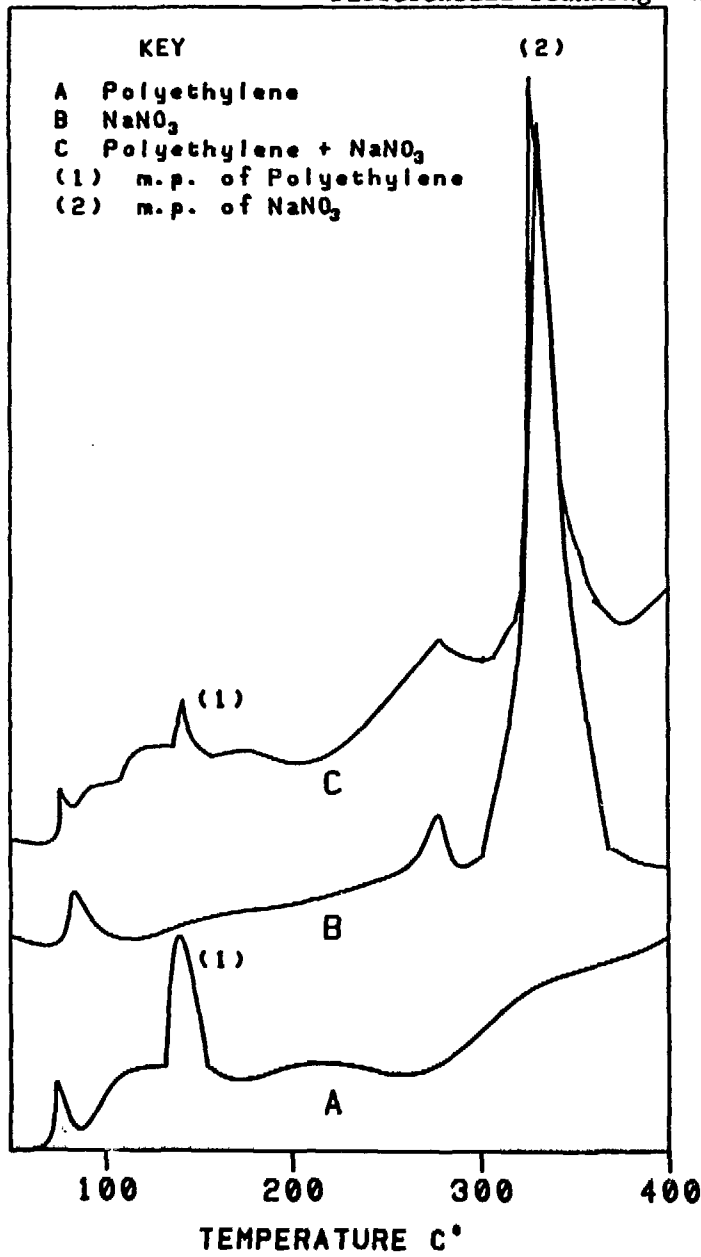


Figure 4.1

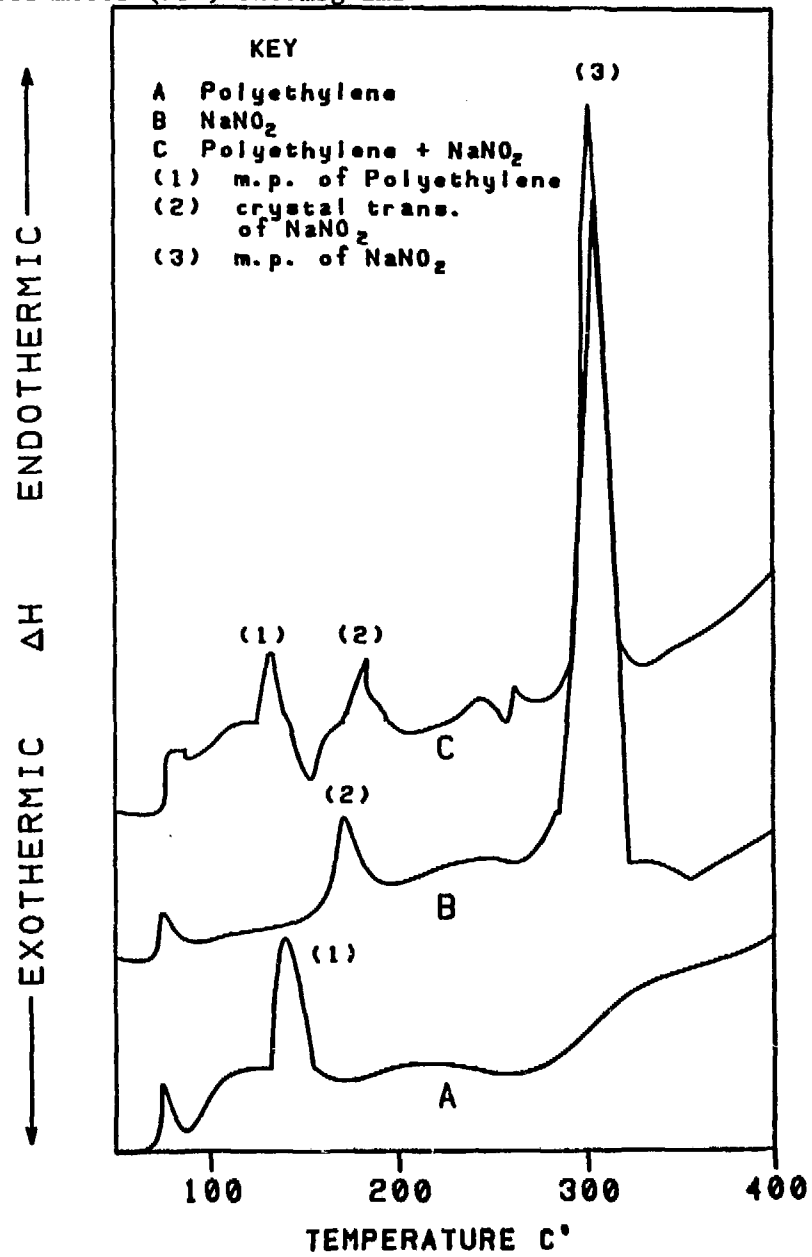


Figure 4.2

Differential Scanning Calorimeter (DSC) Thermograms

- 21 -

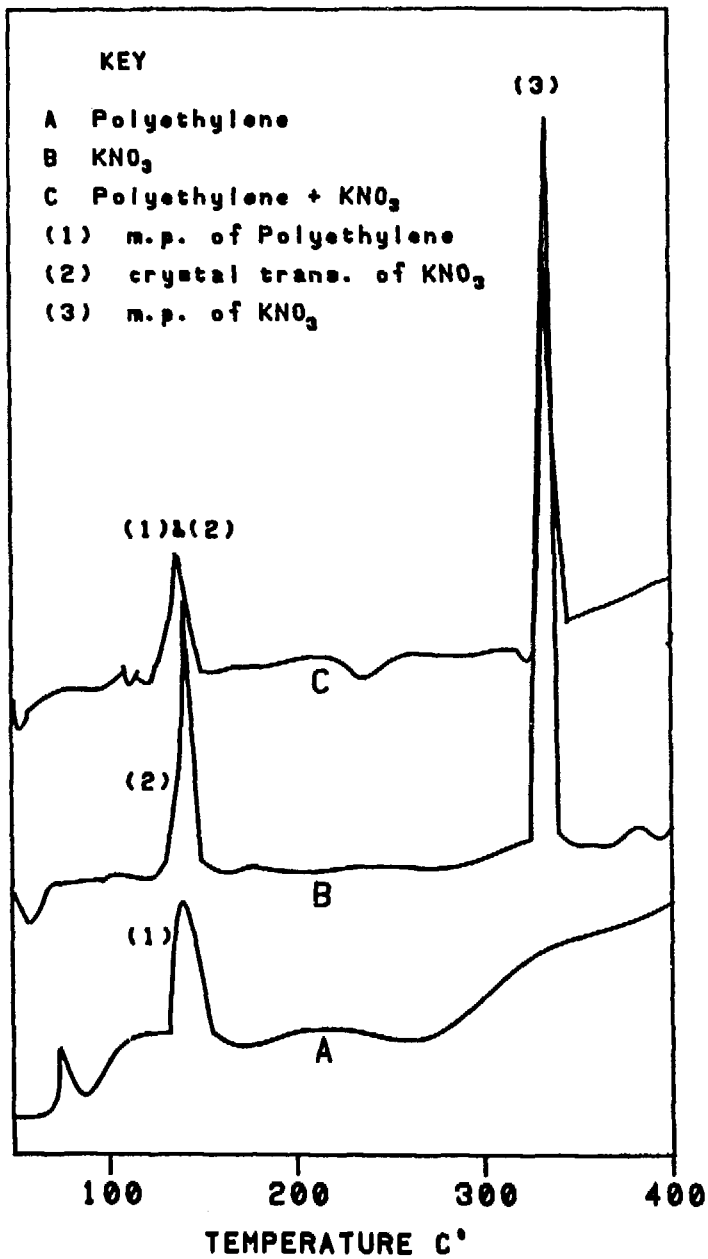


Figure 4.3

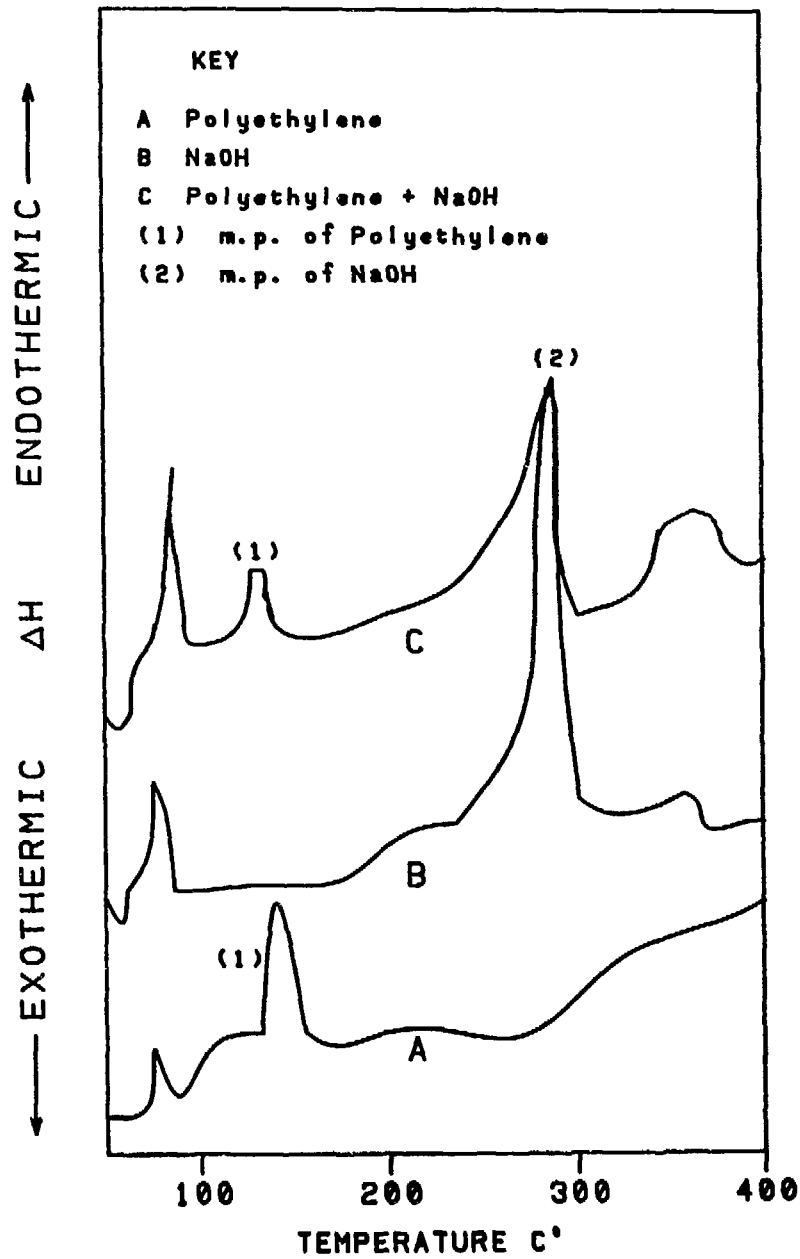


Figure 4.4

Differential Scanning Calorimeter (DSC) Thermograms

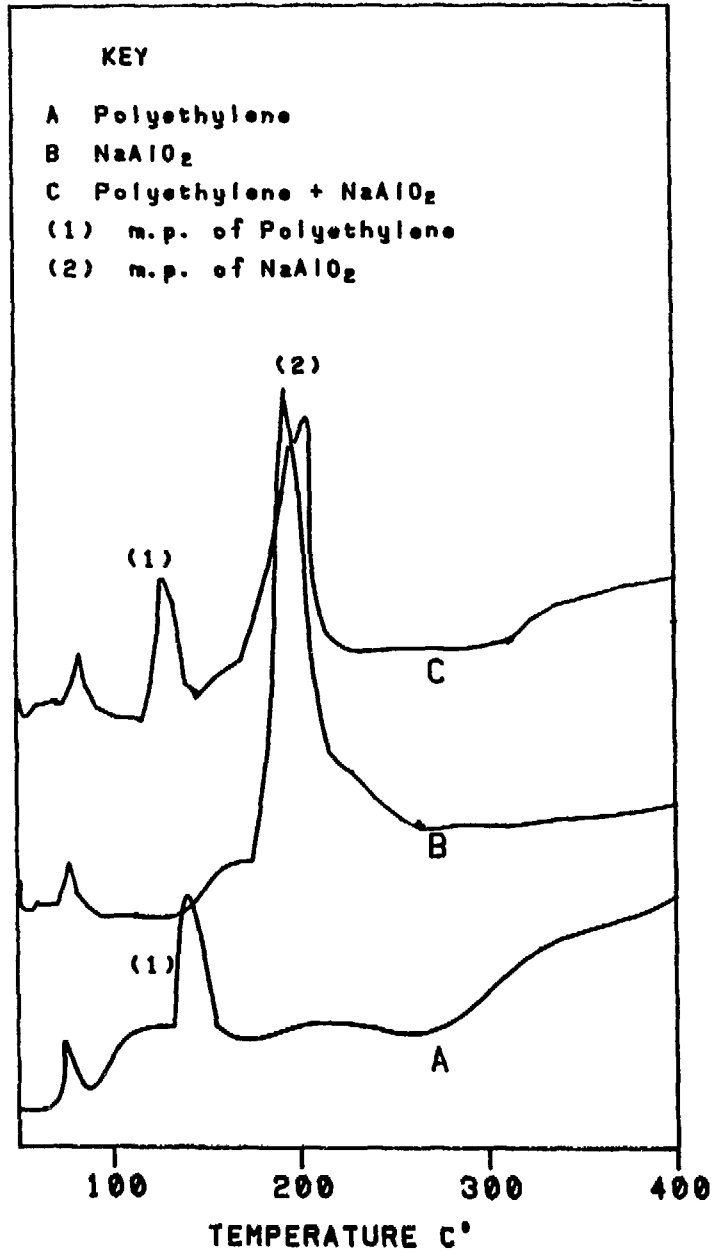


Figure 4.5

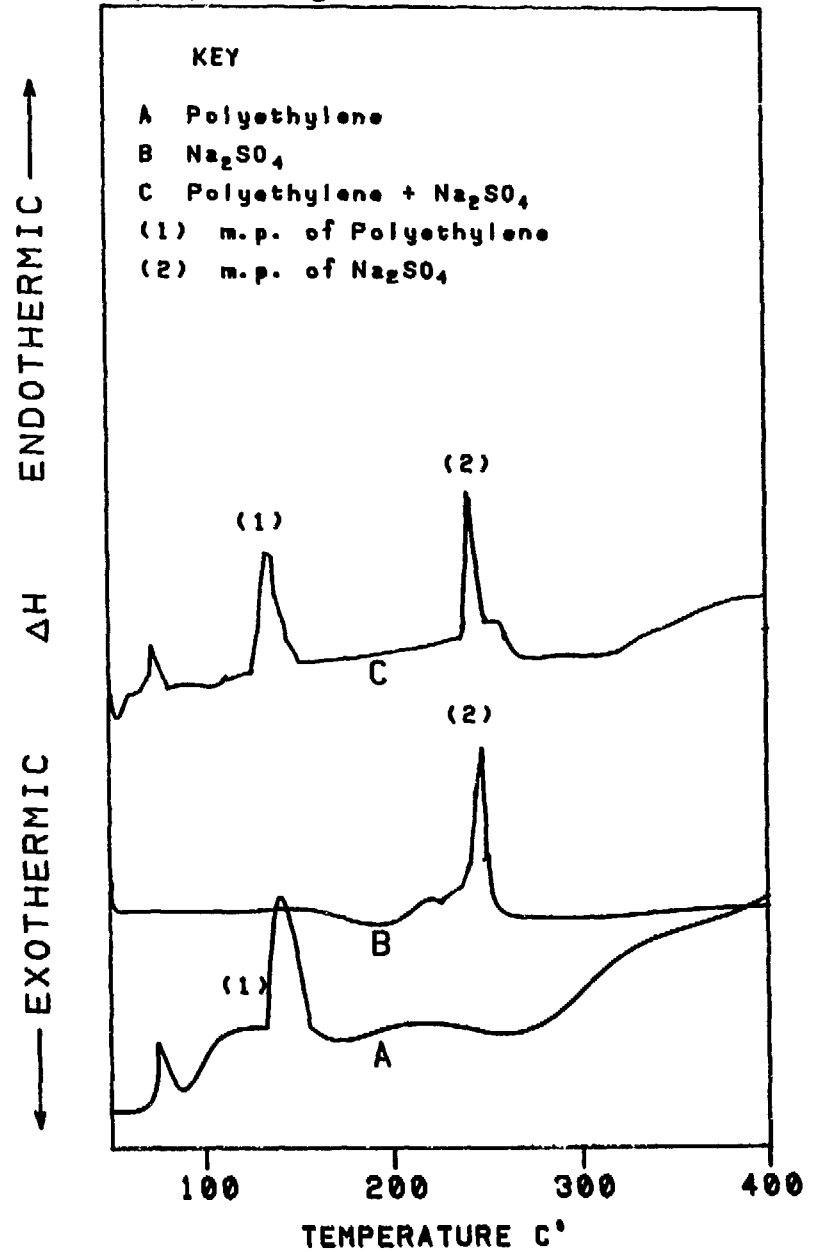


Figure 4.6

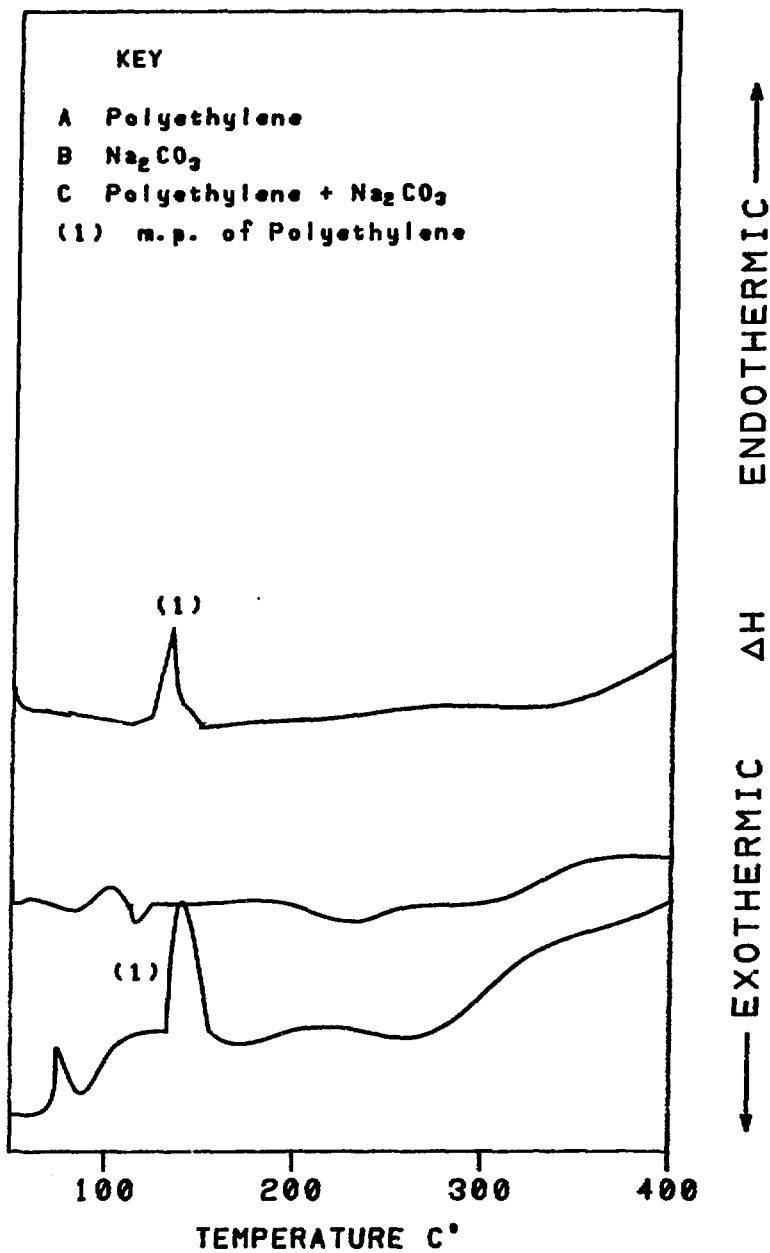


Figure 4.7 Differential Scanning Calorimeter (DSC) Thermogram



Differential Scanning Calorimeter (DSC) Thermograms

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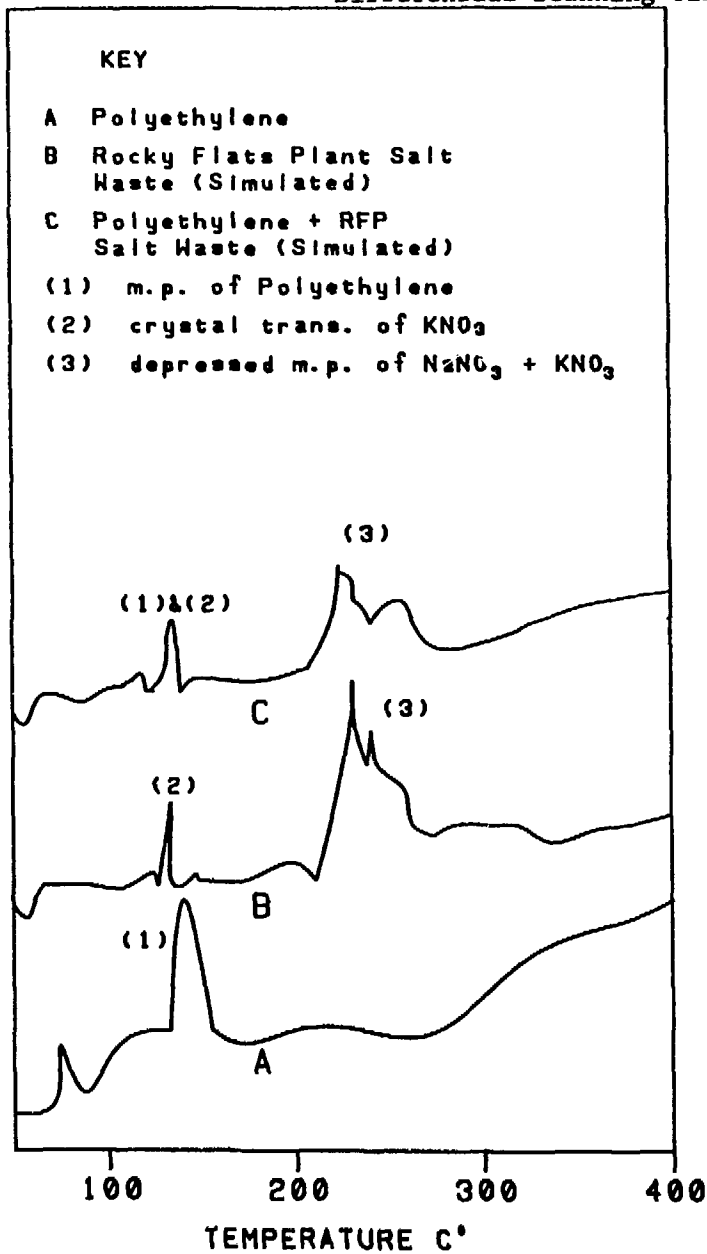


Figure 4.8

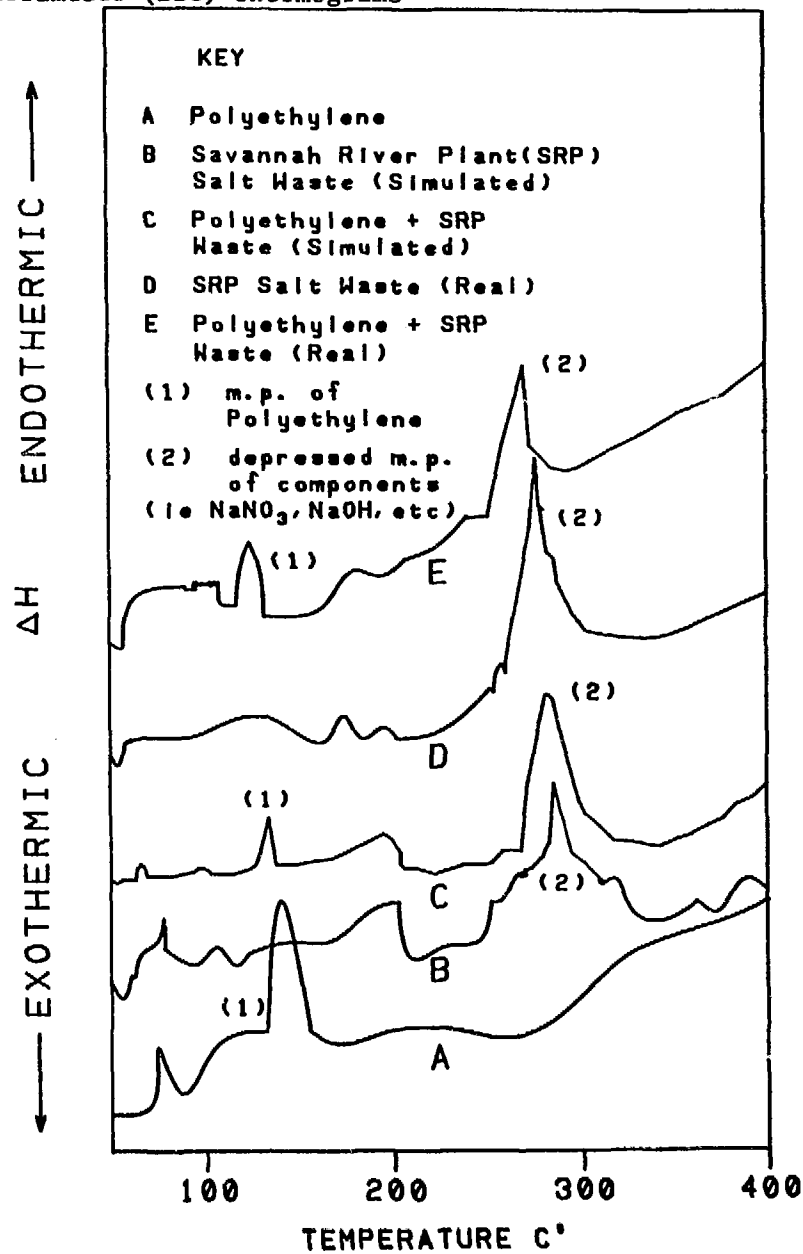


Figure 4.9

Differential Scanning Calorimeter (DSC) Thermograms

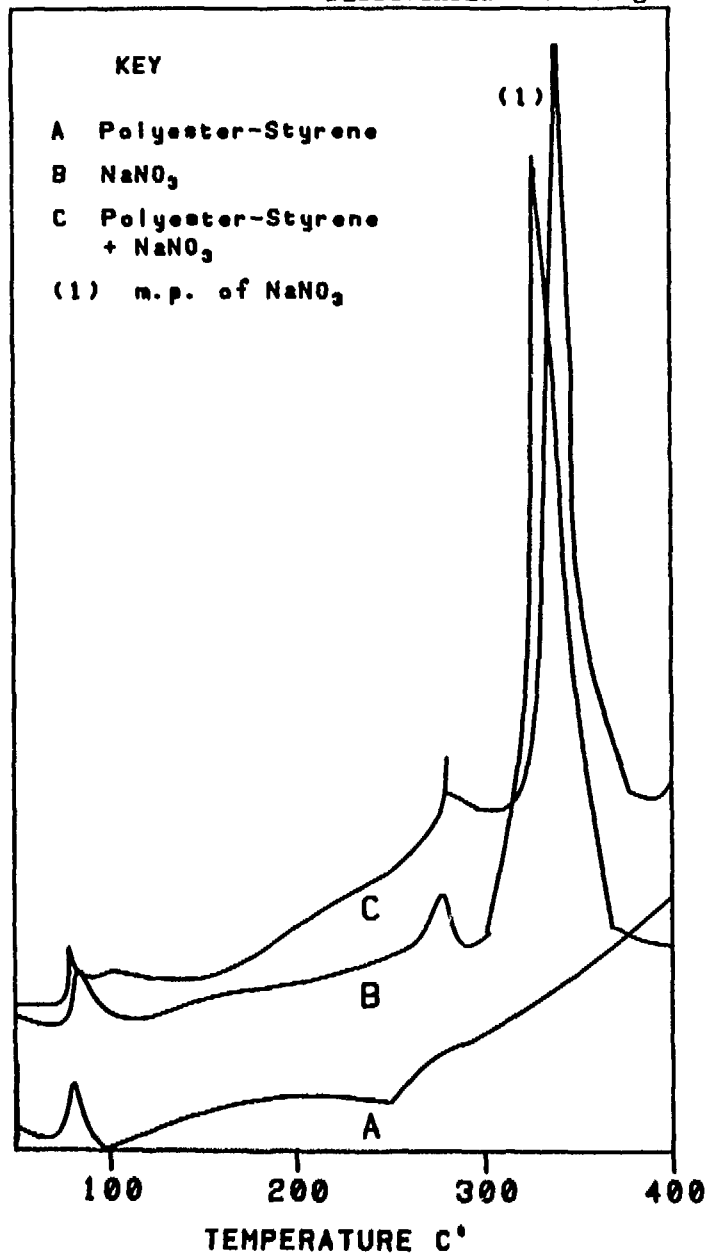


Figure 4.10

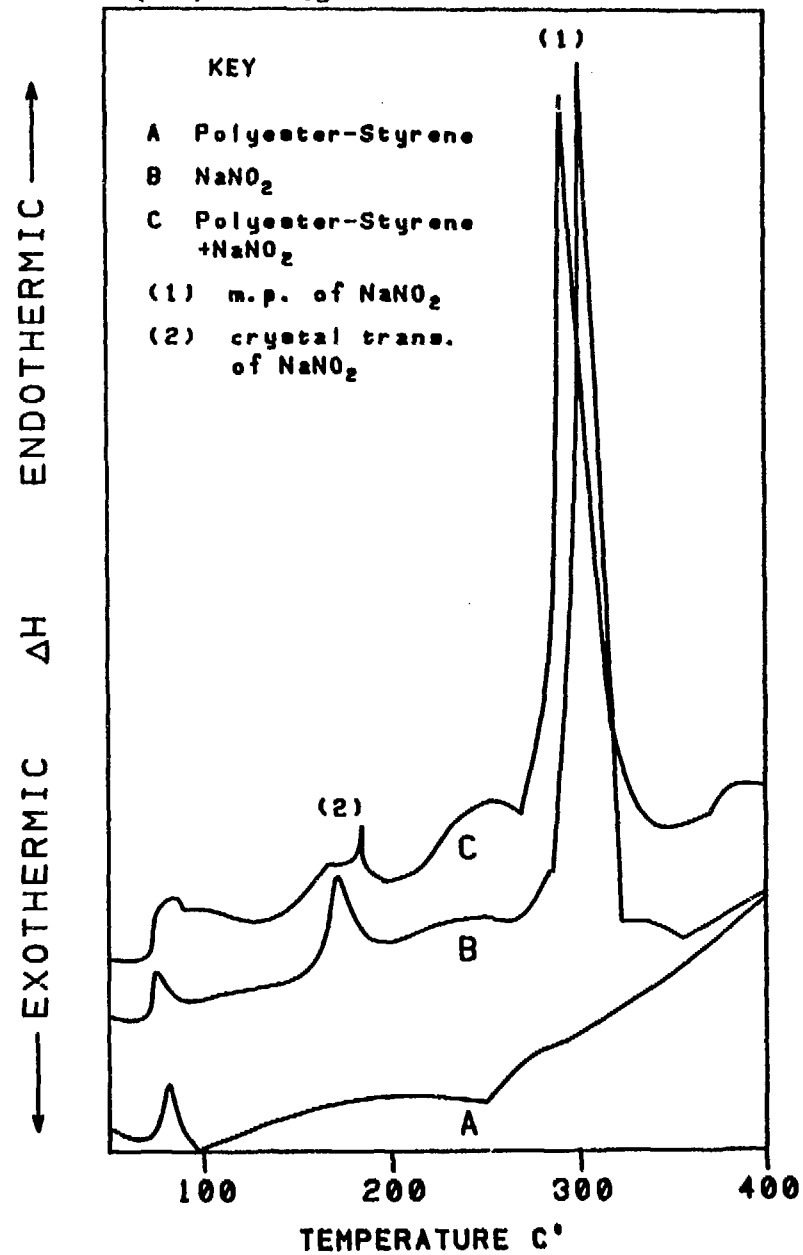


Figure 4.11

Table 6

**Self-Ignition Temperatures of Polyethylene and Polyester-Styrene with Nitrate  
Salt and Salt Waste Components**

Specimens	Specimen Temperature at Ignition Point (°C ± 5°C)	Air Temperature at Ignition Point (°C ± 5°C)
Polyethylene	434	433
Polyethylene/50 wt% NaNO <sub>3</sub>	380	381
Polyethylene/30 wt% NaNO <sub>2</sub>	362	360
Polyethylene/50 wt% NaNO <sub>2</sub>	360	358
Polyethylene/70 wt% NaNO <sub>2</sub>	363	359
Polyethylene/50 wt% SRP Real Waste	365	365
Polyester-Styrene (PES)	435	437
PES/50 wt% NaNO <sub>3</sub>	415	416
PES/50 wt% NaNO <sub>2</sub>	372	370
PES/50 wt% SRP Real Waste	374	374

## 5. IMMOBILIZATION PROCESSES

### 5.1 Polyethylene

Polyethylene is a thermoplastic resin which is thermally stable when used as a binder for nitrate salt waste. When heated, polyethylene softens and flows as a liquid; when cooled, it solidifies. Immobilization is accomplished by mixing the salt waste with molten polyethylene to form a homogeneous monolithic solid upon hardening of the binder.

Processes for mixing wastes with solidification agents can be divided into two categories: in-line and in-drum. The choice usually depends on the application of a specific solidification agent to various waste types. For polyethylene, in-line mixing is preferred to in-drum mixing since several mixing devices can be employed to overcome problems associated with the melt-viscosity behavior of thermoplastics.

**5.1.1 In-Line Mixing Solidification Systems.** Several techniques for in-line mixing were investigated for incorporating nitrate salt wastes into polyethylene. These included: (1) thin-film evaporator/dryers, (2) dual-action mixers, and (3) screw-type extruders. A description of these processes follows.

**5.1.1.1 Thin-Film Evaporator/Dryer.** A simplified flow diagram for a thin-film evaporator/dryer system is shown in Fig. 5.1 This type of system can be used to simultaneously dry and mix waste with polyethylene for immobilization and to reduce the volume of slurries and solutions of nitrate salt waste.

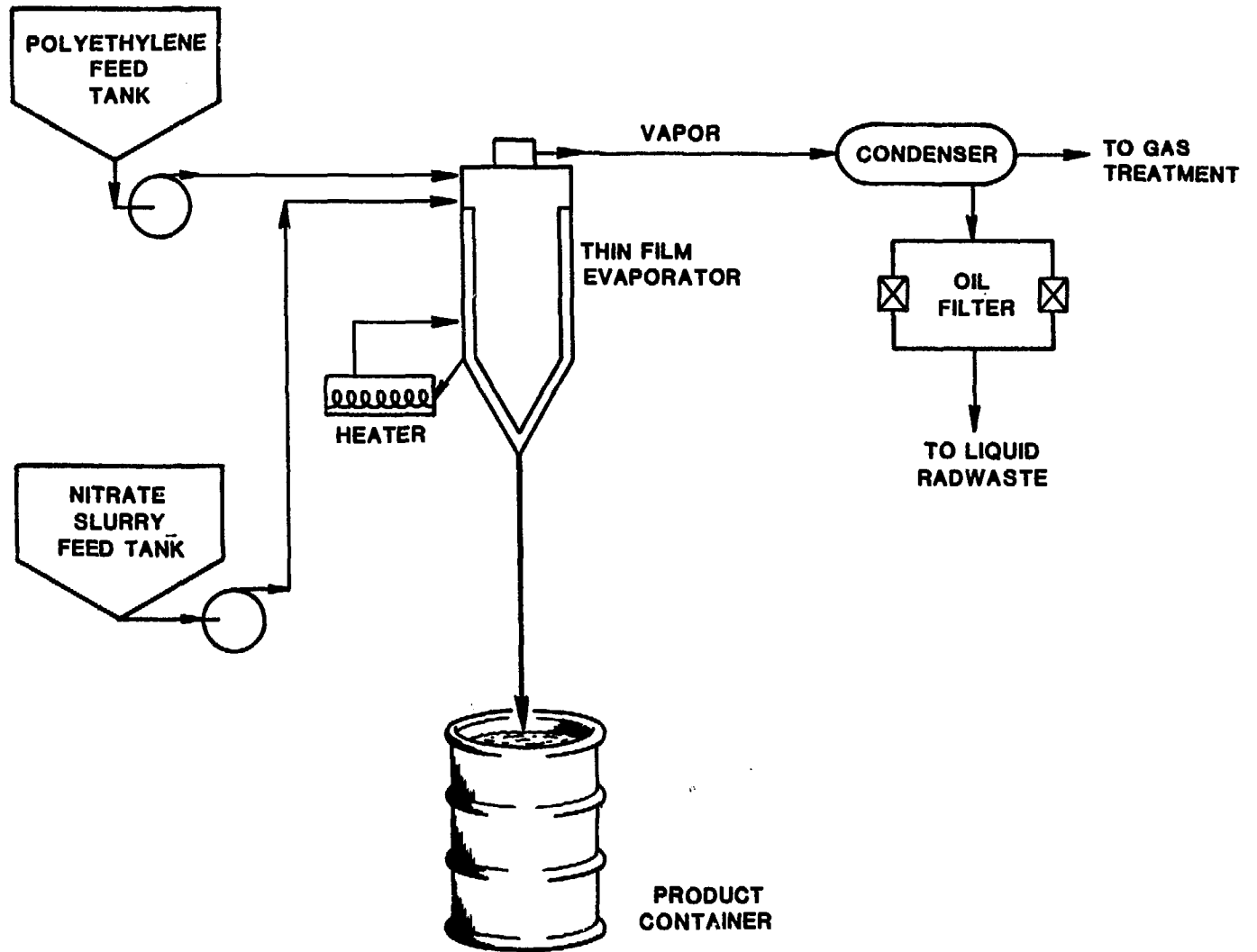


Figure 5.1 Schematic diagram of a thin-film evaporator/polyethylene system for the solidification and volume reduction of aqueous salt wastes.

The thin-film evaporator/dryer consists of a heated cylindrical vessel containing a rotating center element with blades. The blades may be in contact with the wall or may leave a small gap from the wall. The waste is continuously mixed with polyethylene on the wall of the vessel as it moves through the unit, and the mixture is discharged into a waste disposal container. This system is easily adapted to continuous or batch-type operation.

5.1.1.2 Dual-Action Mixer. A schematic of a dual-action mixer, developed and fabricated at BNL for immobilization of dried nitrate salt waste with polyethylene, is shown in Fig. 5.2. The dual-action mixer consists of a stainless steel vessel heated by a series of external resistance band heaters.

The waste-binder mixture is stirred by the dual mixing action of a combined impeller blade and teflon wiper-stirrer. The wiper blades are oriented at 180 degrees and are supported on the power shaft by a stainless steel plate which was drilled to promote internal flow. The wipers serve to stir the mixture and scrape the vessel's interior surface, reducing particle size and improving homogeneity. A standard 3-blade impeller at the base of the power shaft provides a vortex mixing action.

The mixer is powered by a high torque motor driven by compressed air. The molten mixture is delivered to a disposal container through a heated ball valve assisted by a pressurized inert gas.

The order in which waste and binder are added has an observable impact on the ease of processibility. Heating the combined waste-binder mixture led to clumping and segregation of ingredients. Pre-melting the polyethylene and gradually adding waste solids provided a more workable mixture, especially for higher waste loadings.

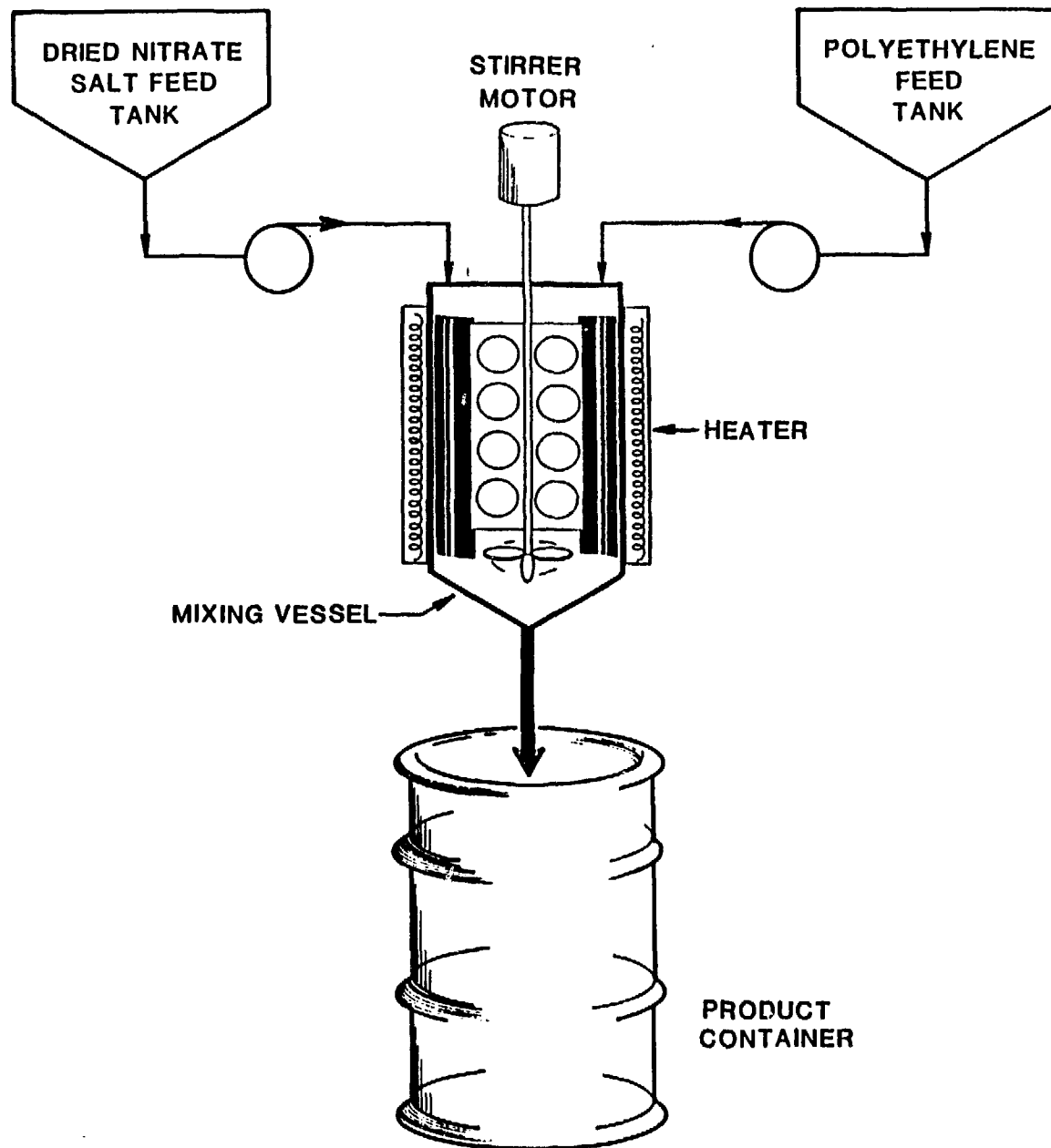


Figure 5.2 Schematic of dual-action heated mixing vessel.

A similar process was demonstrated at Oak Ridge National Laboratory for the solidification of wastes (initially aqueous wastes) in bitumen and polyethylene<sup>3</sup>.

5.1.1.3 Screw-Type Extruder. The use of screw-type extruders for transforming thermoplastic resins such as polyethylene into products is commonplace in the plastic industry. Fig. 5.3 is a simplified schematic of the extrusion process for polyethylene developed at BNL for immobilizing dry wastes<sup>7</sup>.

The four basic components of the extruder system are feed hoppers, a rotating auger-like screw, a heated cylinder in which the screw rotates, and an output die assembly to shape the final product. The extrusion process for solidification of radioactive waste in polyethylene involves heating, mixing and extruding of materials in one basic operation. The process is broken down into the following steps:

1. The polyethylene binder and dry waste materials are transferred from storage in either a single hopper or individual hoppers to the extruder feed throat. Metering of waste-to-binder ratios is accomplished at this step.
2. The mixture is conveyed through a heated cylinder by the motion of the rotating screw. The first part of the cylinder is kept at a temperature below the melting point of polyethylene. This gradually pre-heats the materials, but at the same time assures proper transport of the mixture.



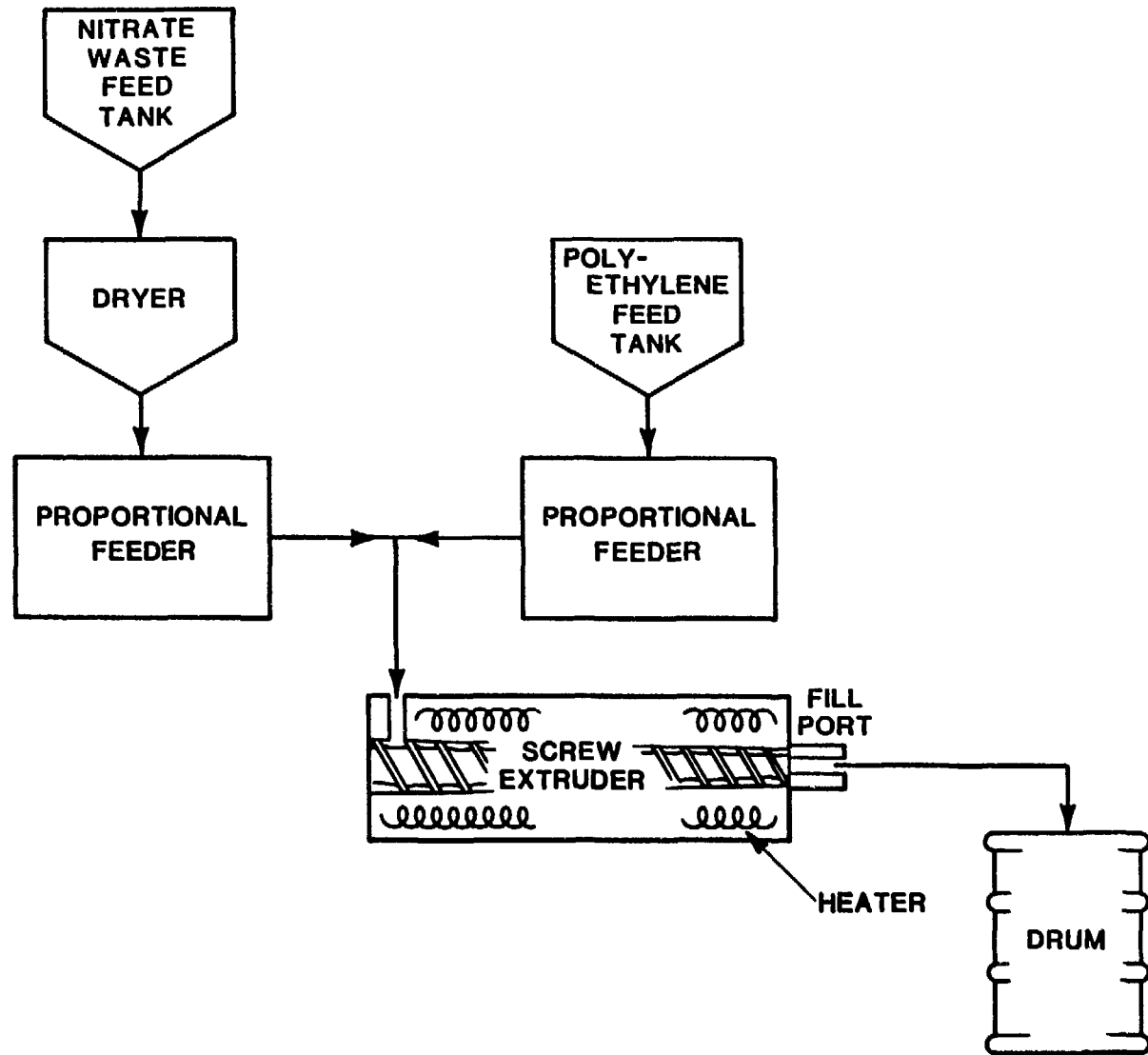


Figure 5.3 A schematic of the BNL continuous extruder process for immobilization of dry wastes in polyethylene.

3. As the waste-binder mixture moves forward past the initial pre-heating zone, it is masticated under pressure due to the compressive effects of a gradual reduction in the channel area between the screw and cylinder. The rotation of the screw also assists in mixing the materials into a homogeneous state.
4. The gradual transfer of thermal energy by the combined effects of the barrel heaters and frictional heat melts the polyethylene. The frictional heat input is difficult to control and must be compensated for by the regulation of the resistance band heaters. In some cases, the excessive heat must be removed by the use of external blowers.
5. The melted thermoplastic-waste mixture (120°C) is forced through an output die into a mold and is allowed to cool and solidify.

The process described above is especially well suited for incorporation of dry wastes resulting from advanced technologies in volume reduction. Pre-drying of aqueous wastes before incorporation into polyethylene results in improved packing efficiencies and homogeneity of the product. Homogeneity of the waste form also depends on the particle size of the dried waste, as do the leaching characteristics.

Commercial extruder-evaporator systems are available which simultaneously evaporate water from liquid wastes while encapsulating the residual solids in a binder such as asphalt<sup>16</sup>. Since the thermoplastic properties of polyethylene are similar to those of bitumen, it is highly feasible that polyethylene can easily be adapted to this system.

A simplified flow diagram of an extruder-evaporator system for volume reduction and waste solidification, presently in use at a commercially operated nuclear power plant, is shown in Fig. 5.4<sup>17</sup>.

An evaluation of the above processes indicates that the extrusion method is preferred for immobilizing dried nitrate salts in polyethylene. This process simultaneously mixes and heats the waste and binder material to produce a homogeneous mixture at extrusion temperatures in the range of 120°C. All test samples, as reported in this document, were produced using the extrusion process.

## 5.2 Polyester-Styrene

The polyester resins discussed in this report are unsaturated resins (dibasic acids, dehydric alcohols), dissolved in styrene monomer. Upon curing, the styrene cross-links with the polyester resin to form a thermoset co-polymer. Cross-linking is a permanent change; thus, once molded, a thermoset polymer cannot be reheated and molded again.

Although ordinary polyester-styrene mixtures are immiscible with water, special formulations are commercially available for the immobilization of aqueous nitrate salt wastes. These materials are known as water-extendible polymers and will form a water-in-oil emulsion with the aqueous waste. Solidification of the emulsion is initiated by addition of suitable combinations of catalysts and promoters.

Similar catalyst-promoter combinations can be used for solidification of dried salt wastes in ordinary polyester-styrene mixtures. Depending on the density of the dry waste, it may be necessary to adjust the viscosity of the base resin to prevent segregation of the waste during curing. This is

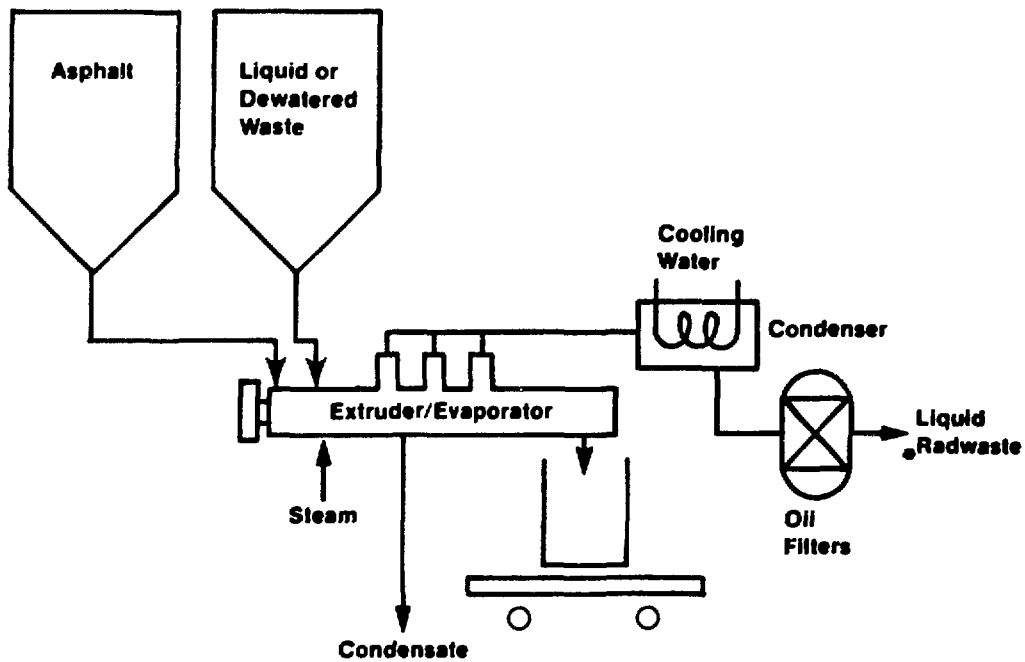


Figure 5.4 A simplified process flow diagram for an extruder/evaporator (Waste Chem. Corp).

accomplished by adding a thixotropic agent (thickening powder) to the base polyester-styrene resin. The thixotropic agent used in these studies is a manufactured siliceous powder; other materials such as zeolites or bentonites also can be used.

The solidification of aqueous wastes with a water-extendible polyester-styrene binder requires high-speed, high-shear mixing to obtain the requisite aqueous phase droplet size and dispersion to give stable emulsions. The pH of the aqueous waste should be adjusted to between 7 and 9 to facilitate and maintain the formation of an emulsion.

High-shear mixing is not necessary for the solidification of dry solid salt wastes since the solid particulates are suspended in the waste-polyester-styrene mixture and remain suspended during the curing stage.

Both types of mixing solidification processes, in-drum and in-line, can be used for incorporation of nitrate waste into polyester-styrene resin. Generally, the in-drum process is preferred since it eliminates the problems of premature polymerization that may occur in an in-line mixer, or the need for periodically flushing the system and for processing the flushed material. Examples of typical in-drum and in-line solidification processes are given below.

**5.2.1 In-Drum Mixing Solidification Process.** The in-drum process involves mixing the waste and polyester-styrene inside a disposal container. A simplified flow diagram for the WEP in-drum system is shown in Fig. 5.5.

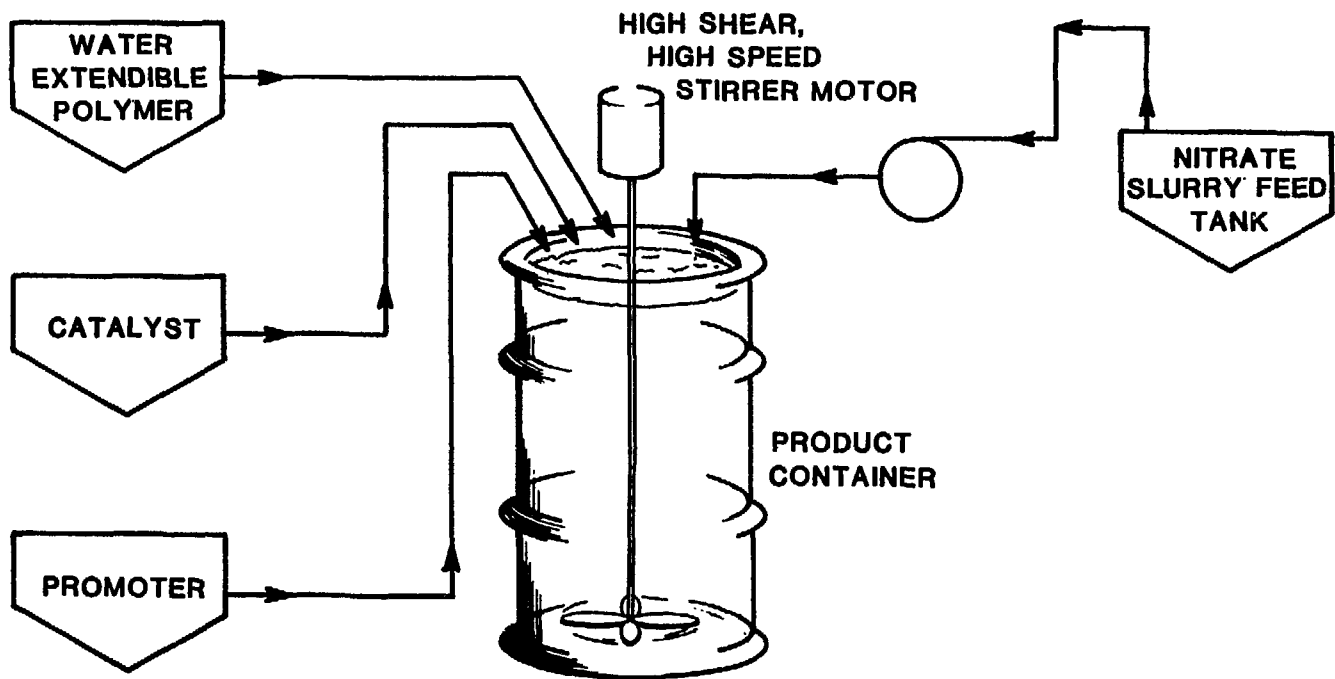


Figure 5.5 Simplified schematic of an In-drum Mixing System for the immobilization of aqueous nitrate waste with water-extensible polymers (WEP).

Mixing techniques used include:

1. Mixing with a reusable element that is removed before the mixture hardens.
2. Mixing with a disposable mixer that is left in the drum after stirring.

In this process, aqueous salt waste and the WEP (containing a promoter) are metered into a drum for emulsification at high speed mixing to produce a water-in-oil resin emulsion. The catalyst then is added to the emulsion causing the contents to gel and cure to a hard solid.

A similar in-drum process can be utilized for immobilization of dried nitrate salts with polyester-styrene. The polyester-styrene containing a promoter is placed in a drum and a metered amount of dried waste and catalyst are stirred in. The contents of the drum is then left to solidify.

**5.2.2 In-Line Mixing Solidification Process.** A simplified flow diagram for an in-line mixing system for immobilizing nitrate salts with polyester-styrene resin is shown in Fig. 5.6. The waste and immobilization agent are mixed and then transferred to the disposal container. This includes:

1. A batch mixing process, where the waste and immobilization agent are mixed in a process vessel and then transferred to the disposal container as a batch; and
2. A continuous mixing process, where the waste and immobilization agent are continuously metered into the mixer and the mixture is continuously transferred to the disposal container.

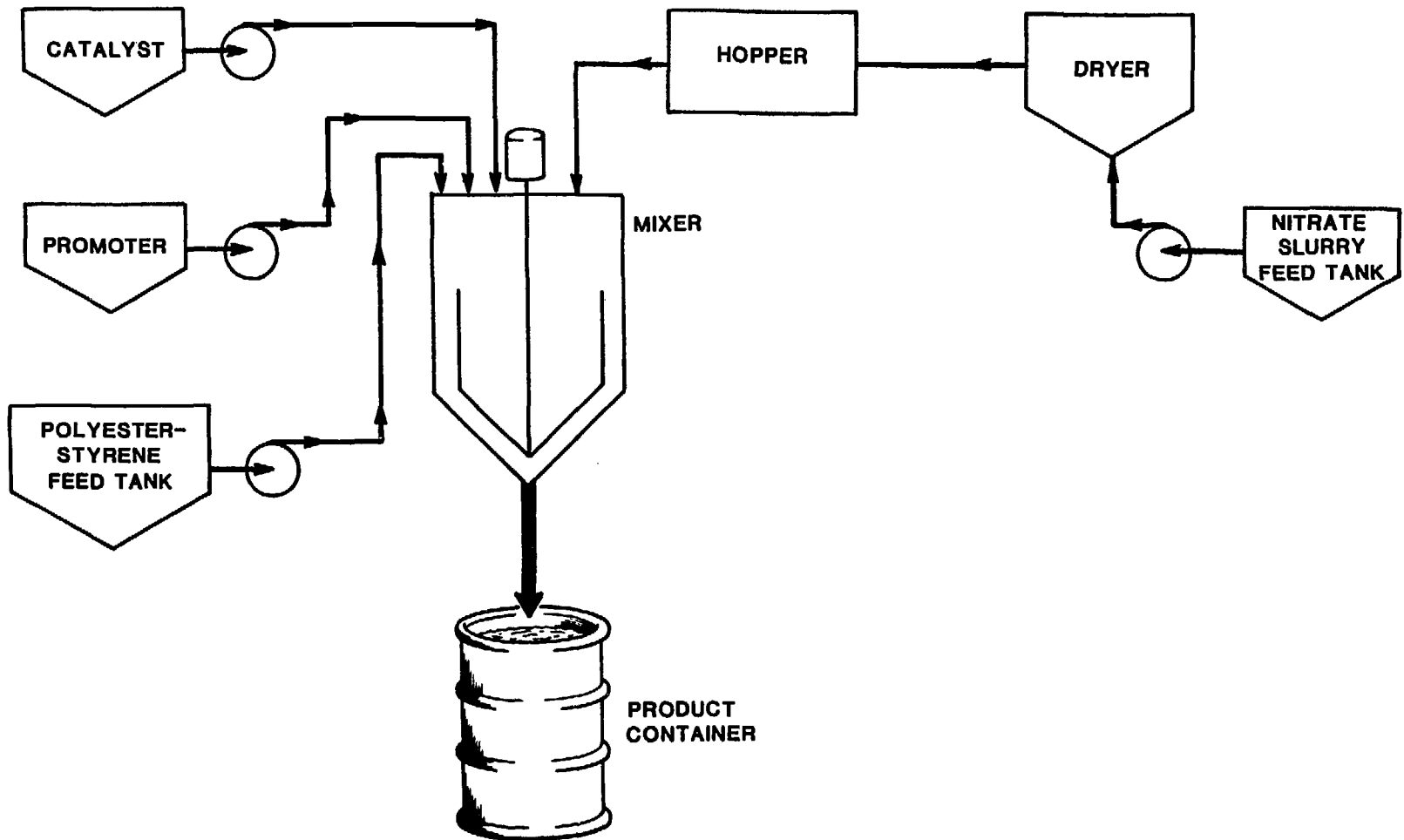


Figure 5.6 Simplified schematic of an In-line Mixing System for immobilization of dried nitrate salt waste with polyester-styrene.



## 6. FABRICATION AND EVALUATION OF WASTE FORMS

### 6.1 Polyethylene

6.1.1 Fabrication of Waste Forms. Laboratory-size samples containing 30, 50, 60 and 70 wt% sodium nitrate were prepared for evaluation using the extrusion process described in Section 5.1.1.3. The homogeneous mixtures were solidified in cylindrical containers, yielding samples which were approximately 4.8 cm in diameter and 9.0 cm in height. Samples exceeding 70 wt% waste loadings were not prepared because the leaching properties of 70 wt% loaded samples approached those of saltstone waste forms<sup>18</sup>.

To demonstrate the homogeneity of the waste forms, micrographs of typical polyethylene samples containing 30, 50 and 70 wt% sodium nitrate, are shown in Figures 6.1, 6.2 and 6.3, respectively. In the samples containing 30 and 50 wt% salt, the salt crystals are completely surrounded by the polyethylene, although the 50 wt% loaded sample contains some larger groups of salt crystals. In the 70 wt% loaded sample, many of the salt crystals are in contact with one another contributing to higher leach rates since there is insufficient polyethylene to encapsulate them.

6.1.2 Waste Form Evaluation. Stability of a waste form is an important factor in controlling the release of  $\text{NaNO}_3$  waste into the groundwater. A series of tests for evaluating waste forms were suggested by NRC in its Branch Technical Position Paper on Waste Form<sup>19</sup> in support of 10 CFR 61<sup>20</sup>. Two of these tests, the 90-day immersion in water and the ANS 16.1 Leach Test<sup>21</sup>, were used in this study to evaluate the polyethylene- $\text{NaNO}_3$  waste forms. The method outlined in ASTM D-695 was used to measure the compressive yield strength

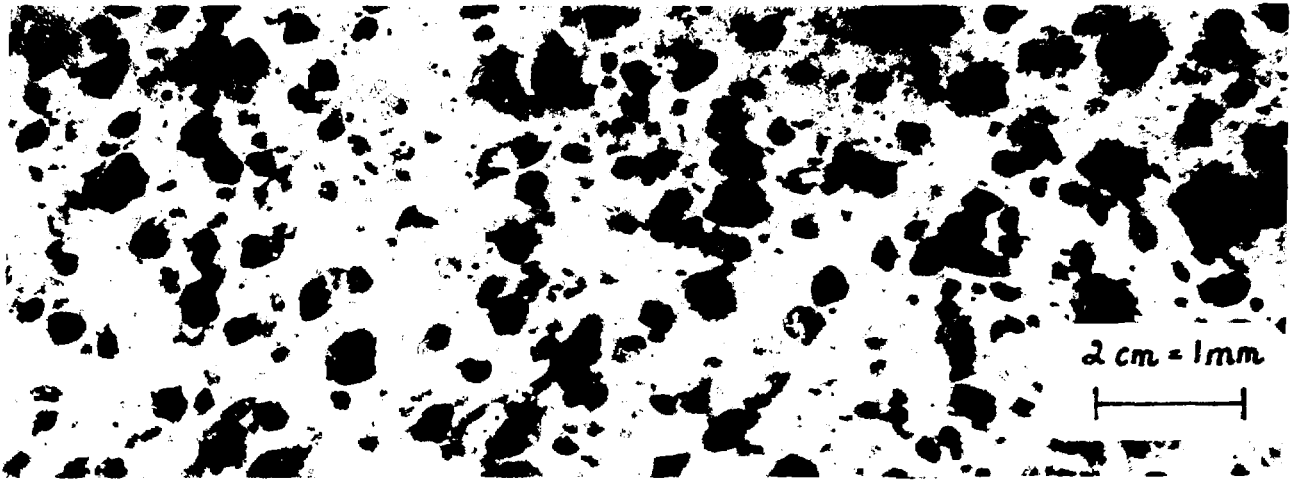


Figure 6.1 Micrograph of a cross section of polyethylene/ $\text{NaNO}_3$  (30 wt%) waste form.



Figure 6.2 Micrograph of a cross section of polyethylene/ $\text{NaNO}_3$  (50 wt%) waste form.

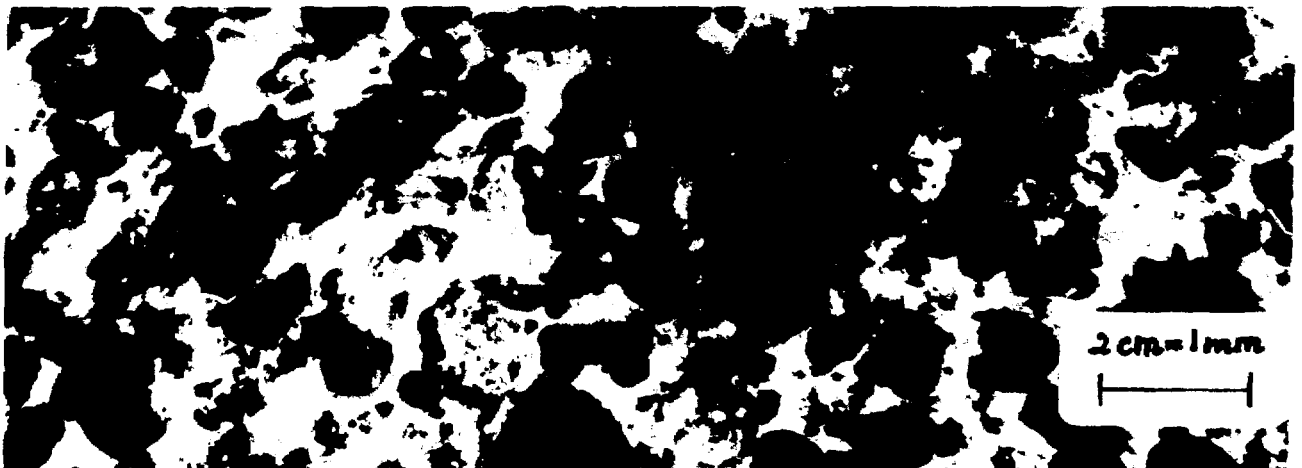


Figure 6.3 Micrograph of a cross section of polyethylene/ $\text{NaNO}_3$  (70 wt%) waste form.

before and after immersion testing. Since some states have adopted the EPA drinking water standards<sup>22</sup> as a guideline for the amount of nitrates that can be released into the environment which could eventually enter the groundwater, the EPA Extraction Procedure<sup>23</sup> was performed to determine compliance.

6.1.2.1. Compressive Yield Strength. To ensure that a waste form remains stable under the compressive loads inherent in a disposal environment, NRC has recommended that solidified waste forms should have compressive strengths of at least 50 psi when tested in accordance with ASTM C-39 "Compressive Strength of Cylindrical Concrete Specimens," or ASTM D-1074. Since polyethylene does not have a discrete brittle fracture yield point under compressive load, these standard compressive strength tests are not applicable. Instead, the ASTM D-695 "Standard Test Method for Compressive Properties of Rigid Plastics," was used. Prior to testing, the samples were machined so that the ends were parallel, flat, clean surfaces as prescribed in Section 6.1 of ASTM D-695. The compressive yield strength in pounds per square inch was determined by dividing the vertical load obtained at the plateau of the stress-strain curve (yield point) by the original cross-sectional area of the test specimen. Table 7 gives the compressive yield strength of polyethylene samples containing 30, 50, 60 and 70 wt%  $\text{NaNO}_3$ .

6.1.2.2 Water Immersion Test. Waste forms disposed by shallow land burial may sometimes be exposed to aqueous conditions in the form of percolate and/or groundwater. Depending upon the composition of the waste and solidification material, these conditions may cause swelling, cracking, dissolution or exfoliation of the waste form structure and subsequent deterioration of the disposal trench.

Table 7

**Compressive Yield Strengths Of Polyethylene Waste Forms Containing Sodium Nitrate Waste**

Waste Loading wt.% NaNO <sub>3</sub>	Compressive Yield Strength (psi)			
	Number of Samples Tested	Before Immersion	Number of Samples Tested	After 90-Day Immersion
30	3	2370±280	2	2550±370
50	3	1920±160	3	1920±350
60	3	2200±70	3	2310±380
70	1	1020	1	720

Waste forms were immersed for 90 days in deionized water, then the compressive yield strength of the specimens was measured. These data are also included in Table 7. Table 7 shows that the compressive yield strength of polyethylene waste forms remained essentially the same (within experimental error) when waste loadings were increased from 30 to 60 wt%, or when the samples were immersed in water for 90 days. Only the samples loaded with 70 wt% waste exhibited a noticeable reduction in strength. This is to be expected, judging by the micrograph in Figure 6.3, in which most of the salt crystals are in contact with one another, as compared to the micrographs of the 30 and 50 wt% waste forms which show the salt crystals to be encapsulated by the polyethylene.

6.1.2.3 Leach Testing. Leach testing was performed for a minimum duration of 90 days in accordance with the procedures in ANS 16.1 Standard, "Measurement of the Leachability of Solidified Low-Level Radioactive

Wastes"<sup>21</sup>. This test was designed to provide a standardized laboratory method for characterizing the leaching behavior of low-level waste forms. Although the procedures do not necessarily simulate leaching from a waste form under actual burial conditions, the test allows a comparison of the relative leachability of various combinations of waste and binder. This test addresses specifically the leaching of radionuclides, however, it also is applicable to non-radioactive chemical species and is used in these studies to determine the release rate of  $\text{NaNO}_3$  from the waste forms.

Replicate samples of polyethylene waste forms containing 30, 50, 60, and 70 wt%  $\text{NaNO}_3$  were leached in demineralized water. The volume of leachant employed ranged between 1500 and 1700 ml, as specified by the ratio of  $10 \pm 0.2$  cm of leachant volume to external geometric surface area of the waste form. The temperature was maintained at  $20 \pm 2^\circ\text{C}$ . After rinsing the specimens for an initial 30 seconds, the leachant was replenished at the following time intervals: 2 hour, 5 hour, 17 hour, 24 hour intervals for the next four days, 13 day, 28 day and 45 day, for a total of ten leachate samplings. Aliquots of the leachates were analyzed for sodium (Na) using atomic absorption spectrophotometry (AA). Since the only source of Na in the samples is  $\text{NaNO}_3$ , the amount of Na released from the samples corresponds directly to the amount of  $\text{NaNO}_3$  released.

The leaching data for  $\text{NaNO}_3$ -polyethylene waste forms are presented in terms of the incremental fraction leached (IFL), the cumulative fraction leached (CFL), the incremental leaching rate per second (ILR) and the leaching indices. These data are tabulated in Appendix A. The CFL is also given in Table 8 and presented graphically as a function of leaching time (days) in Figure 6.4. These data clearly demonstrate the dependence of leachability upon increased waste loadings of 30, 50, 60, and 70 wt%  $\text{NaNO}_3$ .

# Leaching of Sodium Nitrate from Polyethylene Waste Forms

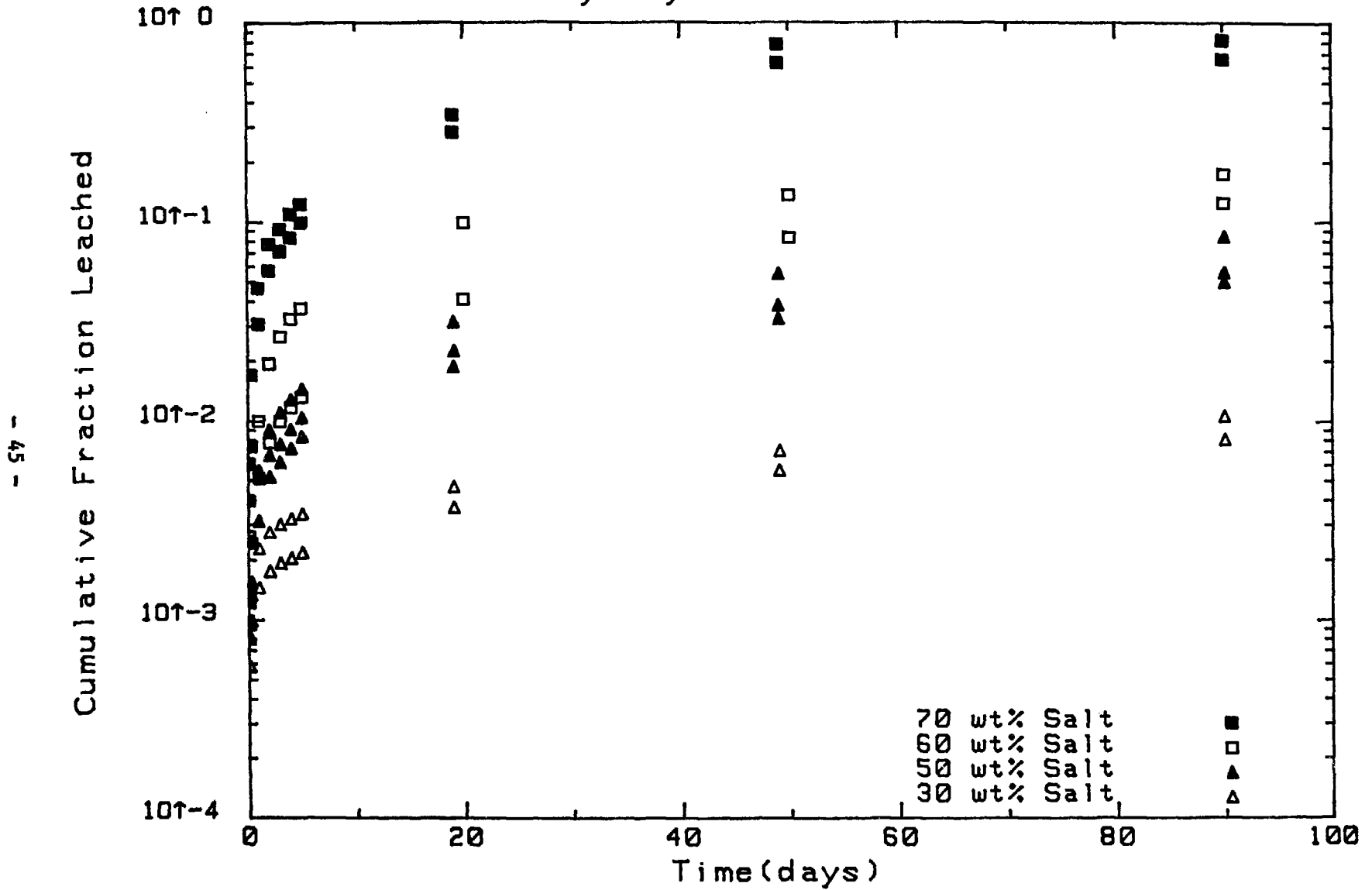


Figure 6.4 Cumulative fraction leached of sodium nitrate as a function of time from polyethylene waste forms.

Table 8

Average  $\text{NaNO}_3$  Leaching Indices and Cumulative Fraction Leached at the End of 90 Days for Polyethylene Waste Forms<sup>a</sup>

Waste Loading $\text{NaNO}_3$ (wt%)	Average Leaching Index	Cumulative Fraction Leached (CFL)
30	11.1	$9.0 \times 10^{-3}$
50	9.7	$6.3 \times 10^{-2}$
60	9.0	$1.5 \times 10^{-1}$
70	7.8	$7.3 \times 10^{-1}$

a. Calculated in accordance with ANS 16.1 Leach Test.

The leaching indices were calculated as prescribed in the ANS 16.1 Leach Test Method. This index is a dimensionless figure of merit which quantifies the relative leachability for a given waste form. It can thus be used as a basis for comparison of the retention capabilities for various waste components by different combinations of waste types and solidification agents.

The leachability index for a given species,  $i$ , is given by:

$$L_i = 1/10 \sum_{n=1}^{10} [\log (\beta/D_i)]n \quad (\text{Eq. 1})$$

where:

$\beta = 1 \text{ cm}^2/\text{sec}$  (defined constant)

$D_i = \text{effective diffusivity (cm}^2/\text{sec)}$

The effective diffusivity is calculated from the leach test data by application of the following expression:

$$D_i = \pi \left[ \frac{a_n/A_o}{(\Delta t)_n} \right]^2 (V/S)^2 T$$

where

$a_n$  = species of interest released from the specimen during the leaching period n

$A_0$  = initial amount of species of interest in the test specimen

V = volume of specimen ( $\text{cm}^3$ )

S = geometric surface area of specimen ( $\text{cm}^2$ )

$(\Delta t)_n$  = incremental leaching time (sec)

$T = [1/2 (t_n^{1/2} + t_{n-1}^{1/2})]^2$ , the leaching time (secs), representing the "mean time" of the leaching interval.

where:

$t_n$  = leaching time at the end of leaching interval n.

Average leaching indices were calculated for each of the replicate sets of polyethylene waste forms. These values are included in Appendix A. The average of the replicate sets of leaching indices is listed in Table 8. Since the leaching index is inversely proportional to effective diffusivity, higher values represent reduced leachability.

6.1.2.4 EPA Extraction Procedure (EP). The EPA Extraction Procedure (EP) for hazardous wastes was performed on polyethylene- $\text{NaNO}_3$  samples to determine nitrate releases. The EP, which is described in detail in 40 CFR Part 261, App. II<sup>23</sup>, was designed to characterize the leaching performance of hazardous waste packages under landfill conditions. The National Interim Primary Drinking Water Standards<sup>22</sup> set a limit for total nitrogen at 10 mg/l (44 mg of nitrate/l). In order to take into account the attenuation and dilution expected during migration of the leachate to the groundwater, the limit set in the standard is combined with a generic attenuation-dilution factor of 100 to



yield the regulatory limit ( $44 \text{ mg/l} \times 100 = 4400 \text{ mg/l}$ ). The amount of a contaminant released during the EP is compared to the regulatory limit for compliance.

Prior to leaching, samples (3.3 cm diameter x 7.1 cm length) containing 30, 50, 60 and 70 wt%  $\text{NaNO}_3$  were subjected to the EPA Structural Integrity Procedure (SIP) as prescribed in the EP<sup>23</sup>. The SIP involves dropping a 0.33 Kg weight fifteen times onto the face of the sample. This procedure tests the integrity of a monolithic sample. None of the samples subjected to the SIP showed any signs of damage.

The samples were leached in demineralized water, equal to sixteen times the sample's weight. The leachate was adjusted to a  $\text{pH} = 5.0 \pm 0.2$  with 0.5N acetic acid and monitored as prescribed in the test procedure to maintain the pH constant. After the initial correction, no further additions of acid were required. The leachate was agitated by tumbling at  $30 \pm 2 \text{ rpm}$ . At the end of the 24 hour leaching period the leachate was analyzed for sodium using AA. The release of nitrate was calculated to be 11, 40, 64, and 492 ppm ( $\text{mg/l}$ ) for 30, 50, 60, and 70 wt%  $\text{NaNO}_3$  loaded samples, respectively. Figure 6.5 shows nitrate release as a function of waste loading. These values are considerably lower than the regulatory limit discussed above.

## 6.2 Polyester-Styrene (PES)

6.2.1 Formulations and Fabrication of Waste Forms. As mentioned in Section 3.2, two types of PES resins were used in these studies, those which can be used to solidify dry wastes and those which can be used to immobilize aqueous waste by forming an emulsion with the water in the waste. The latter

Nitrate Release from Polyethylene/Sodium Nitrate  
Waste Forms Using the EPA Extraction Procedure

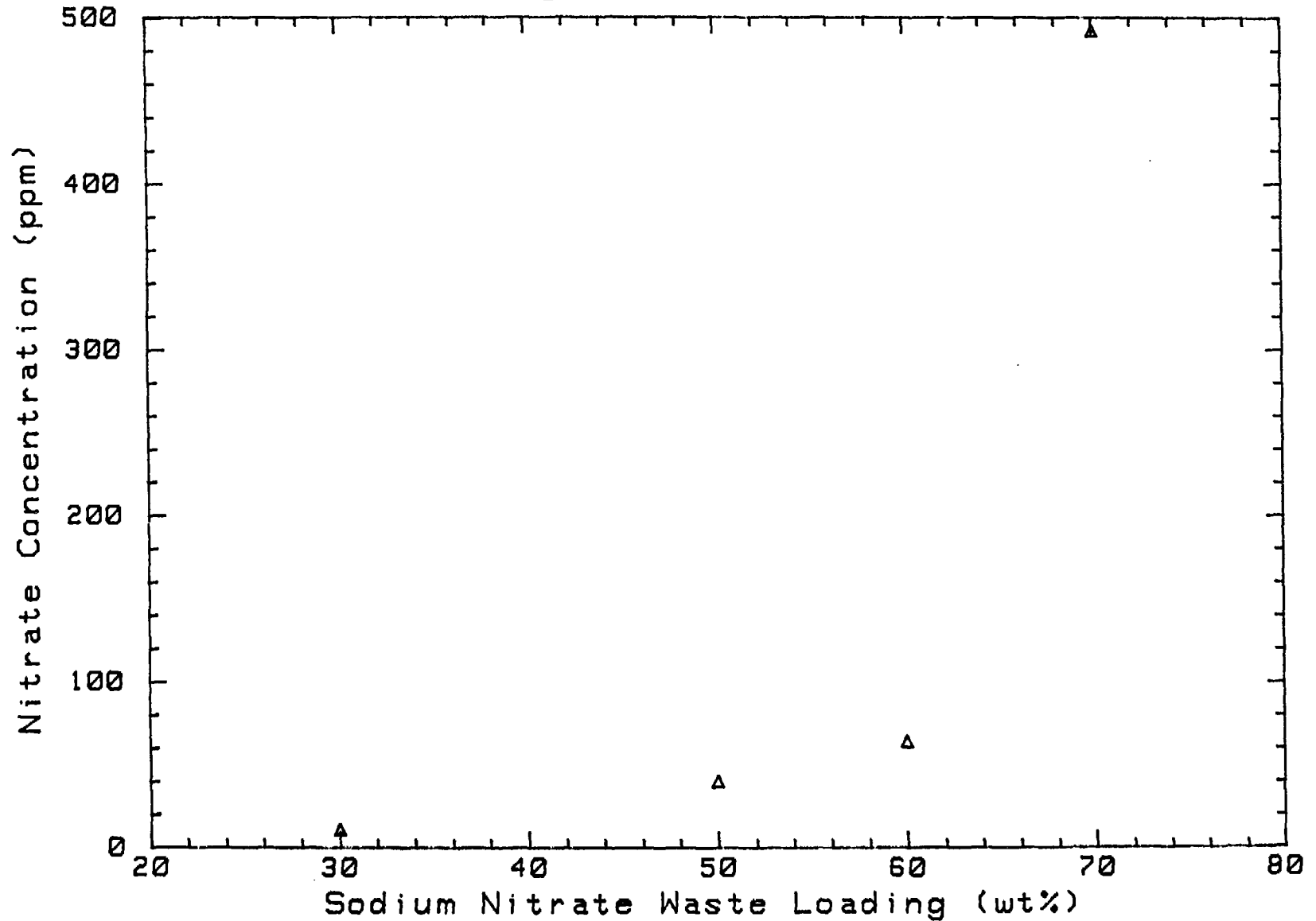


Figure 6.5 Concentration of nitrate released from polyethylene waste forms as a function of sodium nitrate waste loading.

is called a water extendible polyester (WEP) and is usually capable of immobilizing either dry or wet (slurries, solutions) wastes, similar to the DOW vinyl ester-styrene system.

6.2.1.1 Ordinary Polyester-Styrene. The PES resins used in these studies are Ashland 1289-2. Laboratory-scale samples (4.6 cm diameter x 9.0 cm length) containing 31 wt%, 49 wt%, and 70 dry wt%  $\text{NaNO}_3$  were prepared in batches using the method described in Section 5.2. The materials were combined in a mixing vessel in the following order: additional promoter, if needed, was added to the base polyester-styrene resin; a thixotropic agent was added to increase the viscosity of the resin to prevent segregation of the waste during curing; dry simulated  $\text{NaNO}_3$  waste was stirred in to form a homogeneous mixture; and the catalyst was mixed in to give an even distribution throughout the mixture. The mixture was then poured into cylindrical containers and left to cure. The most satisfactory combinations of resin, waste, and admixtures used are summarized in Table 9.

6.2.1.2 Water-Extendible Polyester-Styrene. Laboratory-scale samples (4.6 cm diameter x 9.0 length) containing simulated  $\text{NaNO}_3$  waste slurries and solutions were prepared with Ashland WEP-662 (resin A) and Altek 78-50 ERM (resin B) resins. The salt slurry waste was encapsulated in WEP resin A at 27 wt%  $\text{NaNO}_3$  loading and in WEP resin B at  $\text{NaNO}_3$  loadings of 25, 50, and 70 wt%. The salt solution waste was encapsulated in both types of WEP resins at  $\text{NaNO}_3$  loadings of 28 wt%. This is the maximum loading attainable with salt solution waste because it represents the maximum amount of water tolerated by the system. Resins used with salt slurries also contained 7-8 wt% (approximately 5

Table 9

Formulations for Solidification of  $\text{NaNO}_3$  Waste in Polyester-Styrene

POLYESTER STYRENE WT %	$\text{NaNO}_3$ WASTE		THIXO- TROPIC MATERIAL WT%	CATAYLST (Initiator) Benzoyl Peroxide (50%)	PROMOTER* (Accelerator) Dimethylaniline WT %
	$\text{NaNO}_3$ WT %	WATER WT %			
62.7	31.3	0	4.7	0.9	0.4
48.7	48.7	0	1.6	0.7	0.3
29.8	69.6	0	0	0.4	0.2

\*Additions to as-received resins.

wt% of total waste form) thixotropic materials. The formulations of the most satisfactory samples are summarized in Table 10. The method used to combine the materials is described Section 5.2. When WEP is used with wet wastes the pH of the waste has to be adjusted between 7-9 and high-speed, high-shear agitation is needed to form an emulsion between the aqueous waste and organic polymer.

**6.2.2 Waste Form Evaluation.** The same tests that were performed on polyethylene- $\text{NaNO}_3$  waste forms, as discussed in Section 6.1.2, were used to evaluate the stability of PES- $\text{NaNO}_3$  and WEP- $\text{NaNO}_3$  waste forms. These four tests are: a 90-day immersion in water, the ANS 16.1 Leach Test, measurement of compressive yield strength according to ASTM D-695, and the EPA Extraction Procedure.

Table 10

Formulations for Solidification of  $\text{NaNO}_3$  Waste in Water-Extendible Polyester-Styrene

RESIN TYPE	WT %	NaNO <sub>3</sub> WASTE		THIXO-TROPIC MATERIAL WT %	CATALYST (Initiator) Methyl Ethyl Ketone Peroxide WT %	PROMOTER* (Accelerator) Cobalt Naphthenate WT %
		NaNO <sub>3</sub> WT %	WATER WT %			
A	35.4	28.3	35.4	-	0.5	0.4
B	35.6	28.5	35.6	-	0.4	-
A	61.5	27.4	5.5	4.4	0.6	0.6
B	55.7	24.9	14.9	4.0	0.5	-
B	41.5	49.8	8.3	-	0.4	-
B	17.5	69.9	12.5	-	0.2	-

\*Additions to as-received resins.

6.2.2.1 Compressive Yield Strength. Samples taken from the same lots used in the leach test and water immersion test were destructively tested for compressive yield strength. Testing was performed according to ASTM D-695. The results outlined in Table 11 show that both PES and WEP waste forms have compressive strengths that exceed 2000 psi.

6.2.2.2 Water Immersion Test. Tests were performed on PES and WEP samples to determine their mechanical stability after immersion in water for 90 days. Those WEP samples which contained 50 and 70 wt%  $\text{NaNO}_3$  failed the immersion test. The 50 wt% loaded samples increased in volume by 25% by the 19th day

of immersion and showed signs of cracking. The 70 wt% loaded samples doubled in size in 2 hours. On the rest of the samples, compressive yield strength measurements were made according to ASTM D-695 immediately upon removal of the samples from the water. The compressive yield strength of the immersed samples containing ~30 wt% waste does not differ significantly from that of samples which were not exposed to water (Table 11). For PES samples at higher waste loadings (50 and 70 wt%) there is a difference between the controls and immersed samples, yet all of the samples tested exceed 1800 psi in compressive yield strength. These values are over a factor of ten higher than the NRC prescribed value of 50 psi for commercial waste forms.

Table 11

Compressive Yield Strengths of Polyester-Styrene/Sodium Nitrate Waste Forms

Resin Type	NaNO <sub>3</sub> Wt%	Water Wt%	Compressive Yield Strength (psi)			
			Number of Samples Tested	Before Immersion	Number of Samples Tested	After 90 Days Immersion
PES	33 <sup>a</sup>	—	3	7730 ± 490	3	7710 ± 200
PES	50 <sup>a</sup>	—	3	4920 ± 140	3	2900 ± 70
PES	70 <sup>a</sup>	—	2	5790 ± 820	3	1890 ± 90
WEP-A	25 <sup>b</sup>	5.5	3	3180 ± 130	3	3050 ± 270
WEP-B	27 <sup>b</sup>	14.9	3	5420 ± 40	2	4520 ± 450
WEP-A	28 <sup>c</sup>	35.4	3	2440 ± 10	3	2120 ± 10
WEP-B	28 <sup>c</sup>	35.6	3	2400 ± 40	3	2070 ± 170

a. Dry salt waste.

b. Salt solution.

c. Salt slurry.

6.2.2.3 Leach Testing. Samples containing 33, 50, and 70 wt%  $\text{NaNO}_3$  as dry salt were prepared using PES; for others containing 25 or 27 wt%  $\text{NaNO}_3$  as a slurry WEP was used as the solidification agent. WEP was also used for samples containing 28 wt%  $\text{NaNO}_3$  as solution waste. All samples were leached in demineralized water according to the ANS 16.1 Leach Test. Leachate volume was  $10 \pm 0.2$  times the surface area of the sample and ranged between 1500 and 1700 ml. Temperature was maintained at  $22 \pm 2^\circ\text{C}$ . Aliquots were analyzed for sodium by AA.

The resulting data are calculated as CFL, IFL, ILR, and average leaching indices, and are tabulated in Appendix B. The CFL at the end of 90 days of leaching and the average leaching indices are also listed in Table 12. The CFL is plotted as a function of leaching time and is shown in Figure 6.6. The data indicate that similar leaching characteristics pertain to waste forms containing dry salt, salt slurry or salt solution at a  $\text{NaNO}_3$  loading of approximately 30 wt%. Also, a dependence of leaching on increased waste loadings can be seen, as observed in the leaching of polyethylene waste forms.

6.2.2.4 EPA Extraction Procedure (EP)<sup>23</sup>. Polyester-styrene samples (3.3 cm diameter x 7.1 cm length), containing 33, 50, and 70 wt% simulated  $\text{NaNO}_3$  waste, were subjected to the EP as described in Section 6.1.2.4.

None of the samples were damaged during the EPA Structural Integrity Procedure (SIP) which was performed before leaching as prescribed in the EP. After leaching for 24 hours, the leachates were analyzed for Na using AA. The amount of nitrates released from samples containing 33, 50, and 70 wt%  $\text{NaNO}_3$  was 43, 148, and 671 ppm, respectively. These values are considerably lower than the EPA regulatory limit of 4400 ppm, which is derived by combining the

# Leaching of Sodium Nitrate from Polyester-Styrene Waste Forms

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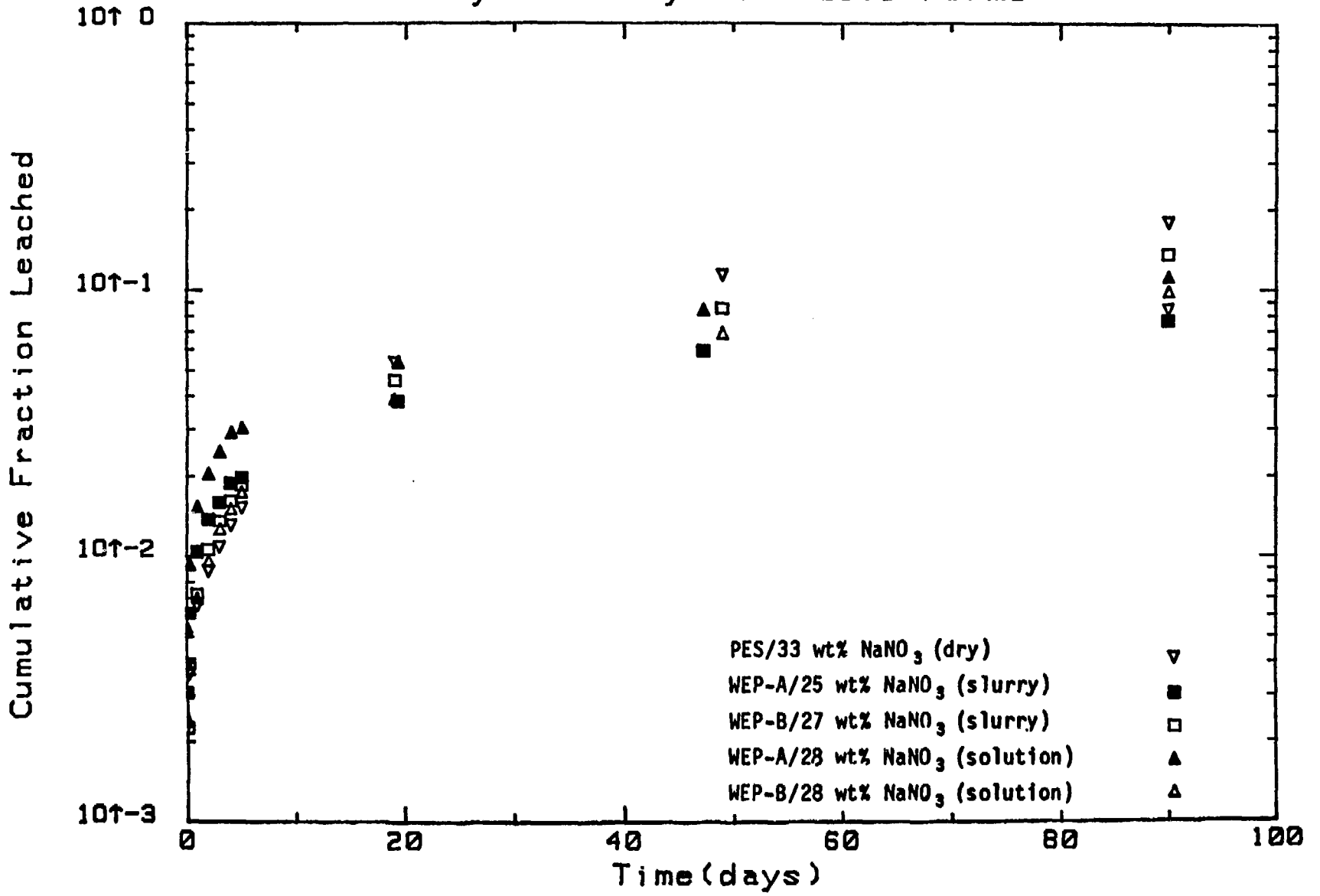


Figure 6.6 Cumulative fraction leached of sodium nitrate as a function of time from polyester-styrene waste forms.



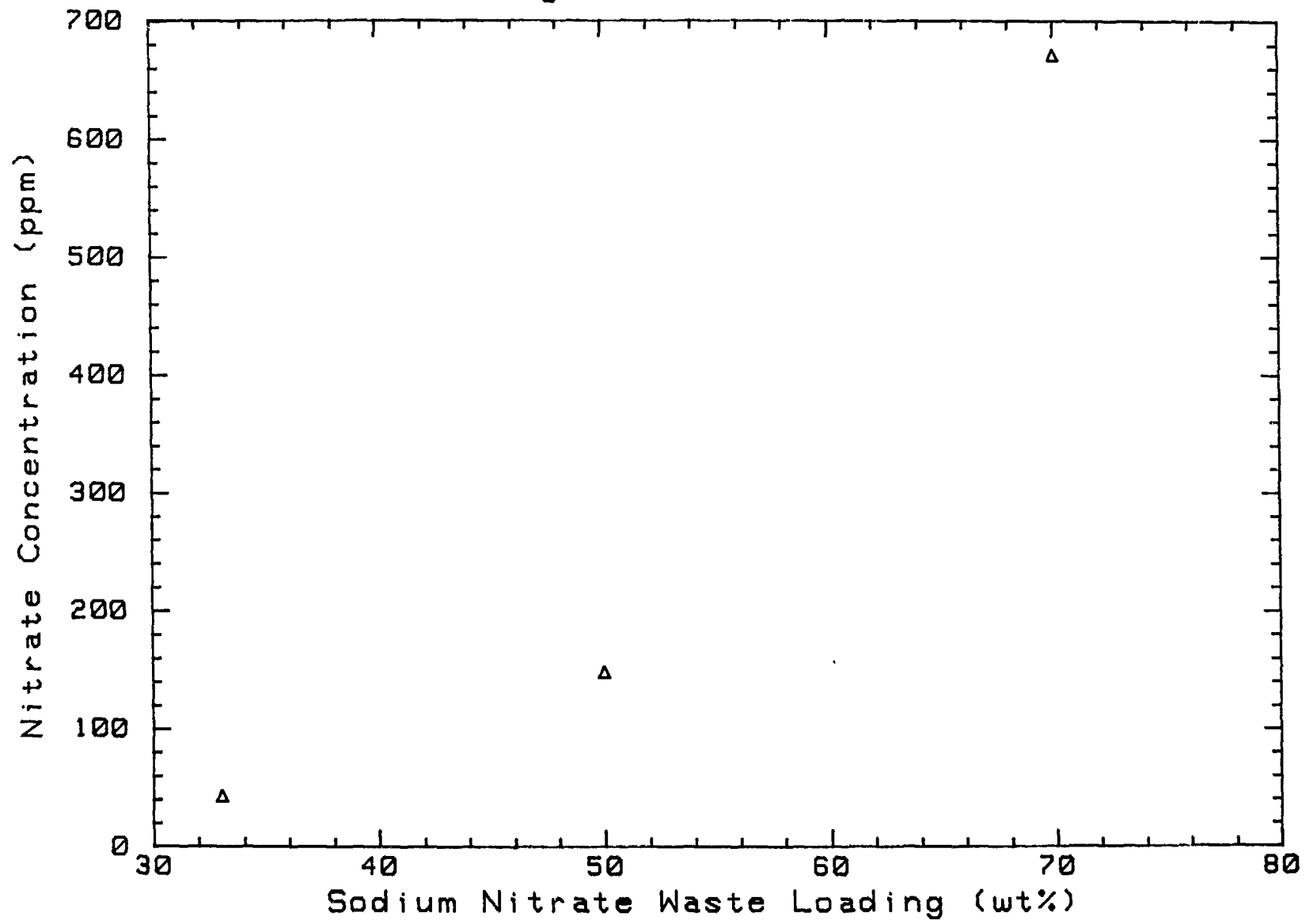
national drinking water standard of 44 ppm with an attenuation/dilution factor of 100 (see Section 6.1.2.4). A plot of nitrate release as a function of waste loading is shown in Figure 6.7.

Table 12

Average  $\text{NaNO}_3$  Leaching Indices and Cumulative Fraction Leached at the end of 90 Days for Polyester-Styrene Waste Forms

Resin Type	Waste Loading $\text{NaNO}_3$ (wt%)	Average Leaching Index	Cumulative Fraction Leached (CFL)
PES	33 (dry)	9.2	$1.8 \times 10^{-1}$
PES	50 (dry)	8.4	$2.6 \times 10^{-1}$
PES	70 (dry)	7.7	$5.6 \times 10^{-1}$
WEP-A	25 (as slurry)	9.3	$7.6 \times 10^{-2}$
WEP-B	27 (as slurry)	9.1	$1.4 \times 10^{-1}$
WEP-A	28 (as solution)	8.9	$1.1 \times 10^{-1}$
WEP-B	28 (as solution)	9.3	$9.8 \times 10^{-2}$

Nitrate Release from Polyester-Styrene/Sodium Nitrate Waste Forms Using the EPA Extraction Procedure



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Figure 6.7 Concentration of nitrate released from polyester-styrene waste forms as a function of sodium nitrate waste loading.

## 7. CONCLUSIONS

- The feasibility of polyethylene as a solidification agent for nitrate salt waste was demonstrated.
- The screw-extrusion method which blends the polyethylene and salt wastes to form a molten or semi-molten homogeneous product is preferred for optimum processing.
- The selection of polyethylene used in this process was based on properties such as density, melt index, and molecular weight distribution (MWD). A polyethylene requiring a processing temperature of  $-120^{\circ}\text{C}$  was selected as a compromise between the properties of the end product and the requirements of the processing equipment.
- High volumetric efficiencies (70 wt%) were obtained using a bench-top single-screw extruder. State-of-the-art drying and processing techniques would significantly improve waste loadings and the properties of the product.
- Leaching rates for polyethylene waste forms containing 70 wt% sodium nitrate salts (dry) were comparable to SRP saltstone waste forms containing approximately 13 wt% sodium nitrate.

- Polyethylene waste forms containing as much as 70 wt% nitrate salt waste greatly exceeded the NRC guideline for 50 psi minimum compressive strength, after being immersed in water for 90 days, implying that the waste forms remain stable after immersion in water for that period.
  
- The compatibility of polyethylene and nitrate waste salts at temperatures to 400°C was confirmed using differential scanning calorimetric techniques, implying that these materials remain chemically stable at temperatures greatly exceeding those required for processing.
  
- Self-ignition temperatures, measured according to ASTM D-1929, are 434°C for polyethylene, and between 365°C and 380°C for mixtures of polyethylene and nitrate wastes. This demonstrates the inherent safety of these materials at or above the temperatures (~120°C) required for processing.
  
- Results of the EPA Extraction Procedure indicate that the nitrate release from polyethylene waste forms containing 70 wt% sodium nitrate waste is approximately an order of magnitude lower than that of the regulatory limit.
  
- Polyester-styrene (PES) was shown to be a feasible solidification agent for nitrate salt waste.

- PES-NaNO<sub>3</sub> waste forms of high loading efficiencies (70 wt%) were demonstrated. Using the ANS 16.1 Leach Test and the EPA Extraction Procedure, nitrate releases were comparable to those of polyethylene waste forms at the same waste loading.
- The compressive yield strength of PES-NaNO<sub>3</sub> waste forms exceeded the NRC recommended minimum value of 50 psi by at least a factor of 30.
- The compatibility of PES-nitrate waste salts at temperatures up to 400°C was confirmed by using differential scanning calorimetric techniques.
- Measurement of self-ignition temperatures show an ignition point of 435°C for PES and a range from 370°C to 416°C for mixtures of PES and nitrate wastes. These values are similar to those obtained for polyethylene-nitrate waste mixtures.
- Water-extendible polyester-styrene (WEP) successfully solidified solutions and slurries of sodium nitrate salt. Waste forms containing 30 wt% salt had leaching rates for nitrate similar to PES waste forms. The compressive yield strength after immersion in water for 90 days remained at over 2000 psi.
- WEP waste forms containing 50 and 70 wt% salt waste as a slurry failed the immersion test. More work is needed to determine the maximum waste loading at which the waste forms would retain their physical integrity.

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**APPENDIX A**

**Leaching Data for Polyethylene Waste Forms**

**Key**

**Time = Cumulative Leaching Time, Days**

**IFL = Incremental Fraction Leached**

**CFL = Cumulative Fraction Leached**

**Rate = Incremental Leaching Rate Per Second**

**LI = Leaching Index**



Table A-1

30% wt% Sodium Nitrate in Polyethylene

1-29-86-30A  
LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/day)	LI
1	.08	5.79E-04	5.79E-04	6.95E-03	10.518
2	.29	4.05E-04	9.84E-04	1.95E-03	10.708
3	1.00	4.63E-04	1.45E-03	6.54E-04	11.117
4	2.00	3.01E-04	1.75E-03	3.01E-04	11.400
5	3.00	1.74E-04	1.92E-03	1.74E-04	11.648
6	4.00	1.16E-04	2.04E-03	1.16E-04	11.851
7	5.00	1.27E-04	2.17E-03	1.27E-04	11.659
8	19.00	1.51E-03	3.67E-03	1.08E-04	11.421
9	49.00	1.97E-03	5.64E-03	6.56E-05	11.378
10	90.00	2.43E-03	8.07E-03	5.93E-05	11.142

AVERAGE Na LI = 11.284

1-29-86-30B  
LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/day)	LI
1	.08	9.40E-04	9.40E-04	1.13E-02	10.092
2	.29	3.88E-04	1.33E-03	1.86E-03	10.741
3	1.00	9.40E-04	2.27E-03	1.33E-03	10.497
4	2.00	4.70E-04	2.74E-03	4.70E-04	11.008
5	3.00	2.59E-04	3.00E-03	2.59E-04	11.297
6	4.00	2.12E-04	3.21E-03	2.12E-04	11.323
7	5.00	1.76E-04	3.39E-03	1.76E-04	11.372
8	19.00	1.27E-03	4.65E-03	9.07E-05	11.565
9	49.00	2.41E-03	7.06E-03	8.03E-05	11.198
10	90.00	3.53E-03	1.06E-02	8.60E-05	10.815

AVERAGE Na LI = 10.991

Table A-2

50% wt% Sodium Nitrate in Polyethylene

1-29-86-50A

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/day)	LI
1	.08	8.24E-04	8.24E-04	9.89E-03	10.175
2	.29	7.06E-04	1.53E-03	3.39E-03	10.190
3	1.00	3.59E-03	5.12E-03	5.07E-03	9.302
4	2.00	1.59E-03	6.71E-03	1.59E-03	9.919
5	3.00	9.12E-04	7.62E-03	9.12E-04	10.171
6	4.00	1.47E-03	9.09E-03	1.47E-03	9.608
7	5.00	1.27E-03	1.04E-02	1.27E-03	9.629
8	19.00	1.21E-02	2.24E-02	8.62E-04	9.578
9	49.00	1.57E-02	3.81E-02	5.24E-04	9.538
10	90.00	1.77E-02	5.58E-02	4.31E-04	9.384

AVERAGE Na LI = 9.749

1-29-86-50B

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/day)	LI
1	.08	7.95E-04	7.95E-04	9.54E-03	10.265
2	.29	7.34E-04	1.53E-03	3.52E-03	10.214
3	1.00	1.59E-03	3.12E-03	2.24E-03	10.067
4	2.00	2.08E-03	5.20E-03	2.08E-03	9.743
5	3.00	9.78E-04	6.18E-03	9.78E-04	10.168
6	4.00	1.10E-03	7.28E-03	1.10E-03	9.917
7	5.00	1.07E-03	8.35E-03	1.07E-03	9.832
8	19.00	1.04E-02	1.87E-02	7.42E-04	9.765
9	49.00	1.39E-02	3.27E-02	4.65E-04	9.700
10	90.00	1.71E-02	4.98E-02	4.18E-04	9.469

AVERAGE Na LI = 9.914

Table A-2 (cont.)

50% wt% Sodium Nitrate in Polyethylene

1-29-86-50C

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/day)	LI
1	.08	1.23E-03	1.23E-03	1.47E-02	9.859
2	.29	1.23E-03	2.46E-03	5.90E-03	9.739
3	1.00	3.16E-03	5.62E-03	4.46E-03	9.443
4	2.00	3.39E-03	9.01E-03	3.39E-03	9.290
5	3.00	1.99E-03	1.10E-02	1.99E-03	9.524
6	4.00	1.81E-03	1.28E-02	1.81E-03	9.456
7	5.00	1.64E-03	1.44E-02	1.64E-03	9.434
8	19.00	1.70E-02	3.14E-02	1.21E-03	9.312
9	49.00	2.34E-02	5.48E-02	7.80E-04	9.222
10	90.00	2.92E-02	8.41E-02	7.13E-04	8.976

AVERAGE Na LI = 9.426

Table A-3

60 wt% Sodium Nitrate in Polyethylene

71686A

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.08	2.60E-03	2.60E-03	3.61E-07	9.204
2	.29	2.75E-03	5.34E-03	1.52E-07	9.035
3	1.00	4.64E-03	9.98E-03	7.58E-08	9.104
4	2.00	9.38E-03	1.94E-02	1.09E-07	8.401
5	3.00	7.04E-03	2.64E-02	8.15E-08	8.421
6	4.00	6.19E-03	3.26E-02	7.16E-08	8.384
7	5.00	4.29E-03	3.69E-02	4.97E-08	8.592
8	20.00	6.19E-02	9.88E-02	4.78E-08	8.227
9	50.00	3.74E-02	1.36E-01	1.44E-08	8.795
10	90.00	3.74E-02	1.74E-01	1.08E-08	8.731

AVERAGE Na LI = 8.690

71686E

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.08	1.21E-03	1.21E-03	1.68E-07	9.880
2	.29	1.21E-03	2.42E-03	6.72E-08	9.760
3	1.00	2.72E-03	5.15E-03	4.45E-08	9.580
4	2.00	2.62E-03	7.77E-03	3.04E-08	9.522
5	3.00	2.17E-03	9.94E-03	2.51E-08	9.457
6	4.00	1.77E-03	1.17E-02	2.04E-08	9.488
7	5.00	1.46E-03	1.32E-02	1.69E-08	9.541
8	20.00	2.77E-02	4.09E-02	2.14E-08	8.938
9	50.00	4.24E-02	8.33E-02	1.63E-08	8.701
10	90.00	4.14E-02	1.25E-01	1.20E-08	8.658

AVERAGE Na LI = 9.353

Table A-4

## 70% wt% Sodium Nitrate in Polyethylene

1-29-86-70A  
LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/day)	LI
1	.08	3.93E-03	3.93E-03	4.72E-02	8.833
2	.29	3.54E-03	7.47E-03	1.70E-02	8.805
3	1.00	2.32E-02	3.07E-02	3.27E-02	7.696
4	2.00	2.63E-02	5.70E-02	2.63E-02	7.495
5	3.00	1.41E-02	7.11E-02	1.41E-02	7.804
6	4.00	1.22E-02	8.33E-02	1.22E-02	7.786
7	5.00	1.53E-02	9.87E-02	1.53E-02	7.476
8	19.00	2.44E-01	3.42E-01	1.74E-02	6.982
9	49.00	4.32E-01	7.75E-01	1.44E-02	6.673
10	90.00	3.93E-02	8.14E-01	9.59E-04	8.704

AVERAGE Na LI = 7.825

1-29-86-70B  
LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/day)	LI
1	.08	6.06E-03	6.06E-03	7.28E-02	8.466
2	.29	1.09E-02	1.70E-02	5.24E-02	7.836
3	1.00	2.95E-02	4.65E-02	4.16E-02	7.497
4	2.00	3.07E-02	7.72E-02	3.07E-02	7.371
5	3.00	1.41E-02	9.13E-02	1.41E-02	7.814
6	4.00	1.78E-02	1.09E-01	1.78E-02	7.467
7	5.00	1.33E-02	1.22E-01	1.33E-02	7.607
8	19.00	1.58E-01	2.80E-01	1.13E-02	7.370
9	49.00	3.43E-01	6.24E-01	1.14E-02	6.883
10	90.00	3.03E-02	6.54E-01	7.39E-04	8.939

AVERAGE Na LI = 7.725

## APPENDIX B

### Leaching Data for Polyester-Styrene Waste Forms

#### Key

Time = Cumulative Leaching Time, Days

IFL = Incremental Fraction Leached

CFL = Cumulative Fraction Leached

Rate = Incremental Leaching Rate Per Second

LI = Leaching Index

Table B-1

33 wt% Sodium Nitrate (dry) in Polyester-Styrene

42386B

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.21	3.61E-03	3.61E-03	2.00E-07	9.386
2	1.00	2.91E-03	6.52E-03	4.26E-08	9.722
3	2.00	2.31E-03	8.83E-03	2.67E-08	9.689
4	3.00	2.01E-03	1.08E-02	2.32E-08	9.580
5	4.00	2.21E-03	1.30E-02	2.55E-08	9.349
6	5.00	2.11E-03	1.51E-02	2.44E-08	9.279
7	19.00	3.81E-02	5.33E-02	3.15E-08	8.672
8	49.00	6.02E-02	1.13E-01	2.32E-08	8.465
9	90.00	6.52E-02	1.79E-01	1.84E-08	8.343

AVERAGE Na LI = 9.165

Table B-2

## 50 wt% Sodium Nitrate (dry) in Polyester-Styrene

1121861A

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.08	5.11E-03	5.11E-03	7.10E-07	8.643
2	.29	4.30E-03	9.41E-03	2.39E-07	8.673
3	1.00	8.20E-03	1.76E-02	1.34E-07	8.638
4	2.00	8.89E-03	2.65E-02	1.03E-07	8.476
5	3.00	6.47E-03	3.30E-02	7.49E-08	8.523
6	4.00	6.81E-03	3.98E-02	7.89E-08	8.329
7	5.00	6.58E-03	4.64E-02	7.62E-08	8.249
8	19.00	6.32E-02	1.10E-01	5.22E-08	8.192
9	47.00	8.37E-02	1.93E-01	3.46E-08	8.089
10	90.00	7.03E-02	2.63E-01	1.89E-08	8.286

AVERAGE Na LI = 8.410

1121861B

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.08	5.37E-03	5.37E-03	7.46E-07	8.593
2	.29	4.94E-03	1.03E-02	2.75E-07	8.545
3	1.00	7.96E-03	1.83E-02	1.30E-07	8.655
4	2.00	1.14E-02	2.97E-02	1.32E-07	8.250
5	3.00	8.66E-03	3.84E-02	1.00E-07	8.261
6	4.00	8.02E-03	4.64E-02	9.28E-08	8.179
7	5.00	6.81E-03	5.32E-02	7.88E-08	8.211
8	19.00	6.04E-02	1.14E-01	5.00E-08	8.223
9	47.00	7.86E-02	1.92E-01	3.25E-08	8.136
10	90.00	6.41E-02	2.56E-01	1.73E-08	8.358

AVERAGE Na LI = 8.341



Table B-3

70 wt% Sodium Nitrate (dry) in Polyester-Styrene

112186A  
LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.08	1.18E-02	1.18E-02	1.64E-06	7.957
2	.29	1.50E-02	2.68E-02	8.35E-07	7.636
3	1.00	2.88E-02	5.56E-02	4.71E-07	7.595
4	2.00	2.68E-02	8.24E-02	3.10E-07	7.567
5	3.00	2.05E-02	1.03E-01	2.37E-07	7.570
6	4.00	1.83E-02	1.21E-01	2.12E-07	7.521
7	5.00	1.50E-02	1.36E-01	1.74E-07	7.583
8	19.00	1.34E-01	2.70E-01	1.10E-07	7.591
9	47.00	1.58E-01	4.28E-01	6.55E-08	7.584
10	90.00	1.25E-01	5.53E-01	3.36E-08	7.838

AVERAGE Na LI = 7.645

112186B  
LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.08	1.06E-02	1.06E-02	1.48E-06	8.048
2	.29	1.30E-02	2.37E-02	7.24E-07	7.752
3	1.00	2.73E-02	5.10E-02	4.46E-07	7.635
4	2.00	2.61E-02	7.71E-02	3.02E-07	7.581
5	3.00	2.16E-02	9.86E-02	2.50E-07	7.518
6	4.00	1.71E-02	1.16E-01	1.97E-07	7.573
7	5.00	1.48E-02	1.30E-01	1.71E-07	7.587
8	19.00	1.37E-01	2.68E-01	1.14E-07	7.559
9	47.00	1.67E-01	4.34E-01	6.89E-08	7.533
10	90.00	1.34E-01	5.69E-01	3.61E-08	7.765

AVERAGE Na LI = 7.655

Table B-4

25 wt% Sodium Nitrate (as slurry) in Water Extendible Polyester-Styrene Type A

61185B

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.08	3.06E-03	3.06E-03	4.43E-07	9.057
2	.30	3.06E-03	6.12E-03	1.61E-07	9.000
3	1.00	4.23E-03	1.03E-02	6.99E-08	9.184
4	2.00	3.35E-03	1.37E-02	3.88E-08	9.305
5	3.00	2.19E-03	1.59E-02	2.53E-08	9.450
6	4.00	2.91E-03	1.88E-02	3.37E-08	9.352
7	5.00	8.74E-04	1.97E-02	1.01E-08	9.988
8	19.30	1.84E-02	3.80E-02	1.49E-08	9.265
9	47.30	2.08E-02	5.89E-02	8.62E-09	9.278
10	90.00	1.75E-02	7.64E-02	4.74E-09	9.473

AVERAGE Na LI = 9.306

Table B-5

## 27 wt% Sodium Nitrate (as slurry) in Water Extendible Polyester-Styrene Type B

42586A

LEACHING DATA FOR Na

#	TIME(d)	IFL	OFL	RATE(1/sec)	LI
1	.08	2.25E-03	2.25E-03	3.13E-07	9.331
2	.30	1.69E-03	3.94E-03	9.15E-08	9.478
3	1.00	3.27E-03	7.21E-03	5.38E-08	9.403
4	2.00	3.38E-03	1.06E-02	3.91E-08	9.292
5	3.00	2.93E-03	1.35E-02	3.39E-08	9.187
6	4.00	2.59E-03	1.61E-02	3.00E-08	9.145
7	5.00	2.37E-03	1.85E-02	2.74E-08	9.114
8	19.00	2.70E-02	4.55E-02	2.23E-08	8.906
9	49.00	3.94E-02	8.49E-02	1.52E-08	8.768
10	90.00	5.07E-02	1.36E-01	1.43E-08	8.497

AVERAGE Na LI = 9.112

Table B-6

28 wt% Sodium Nitrate (as solution) in  
Water Extendible Polyester-Styrene Type A

6486B2

LEACHING DATA FOR Na

#	TIME(d)	IFL	CFL	RATE(1/sec)	LI
1	.08	5.32E-03	5.32E-03	7.70E-07	8.566
2	.30	3.93E-03	9.26E-03	2.07E-07	8.771
3	1.00	6.02E-03	1.53E-02	9.95E-08	8.867
4	2.00	5.21E-03	2.05E-02	6.03E-08	8.916
5	3.00	4.17E-03	2.46E-02	4.82E-08	8.880
6	4.00	4.40E-03	2.90E-02	5.09E-08	8.685
7	5.00	1.16E-03	3.02E-02	1.34E-08	9.734
8	19.30	2.31E-02	5.33E-02	1.87E-08	9.054
9	47.30	3.04E-02	8.38E-02	1.26E-08	8.939
10	90.00	2.78E-02	1.12E-01	7.53E-09	9.061

AVERAGE Na LI = 8.947

Table B-7

28 wt% Sodium Nitrate (as solution) in  
Water Extendible Polyester-Styrene Type B

32186A  
LEACHING DATA FOR Na

#	TIME(d)	IFL	OFL	RATE(1/sec)	LI
1	.08	2.38E-03	2.38E-03	3.31E-07	9.325
2	.30	1.37E-03	3.75E-03	7.41E-08	9.702
3	1.00	3.21E-03	6.96E-03	5.29E-08	9.459
4	2.00	2.62E-03	9.58E-03	3.03E-08	9.556
5	3.00	3.03E-03	1.26E-02	3.51E-08	9.197
6	4.00	2.38E-03	1.50E-02	2.75E-08	9.260
7	5.00	2.38E-03	1.74E-02	2.75E-08	9.150
8	19.00	2.14E-02	3.88E-02	1.77E-08	9.149
9	49.00	2.98E-02	6.85E-02	1.15E-08	9.054
10	90.00	2.98E-02	9.83E-02	8.40E-09	9.001

AVERAGE Na LI = 9.285