



# Technical Resource Document

## Solidification/ Stabilization and its Application to Waste Materials

**TECHNICAL RESOURCE DOCUMENT  
SOLIDIFICATION/STABILIZATION  
AND ITS APPLICATION TO  
WASTE MATERIALS**

by

**BATTELLE  
Columbus Division  
Columbus, Ohio 43201-2693**

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**Charles Mashni  
Technical Project Monitor:  
Waste Minimization, Destruction and  
Disposal Research Division  
Risk Reduction Engineering Laboratory  
Cincinnati, Ohio 45268**

**RISK REDUCTION ENGINEERING LABORATORY  
OFFICE OF RESEARCH AND DEVELOPMENT  
U.S. ENVIRONMENTAL PROTECTION AGENCY  
Cincinnati, Ohio 45268**

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## FOREWORD

Today's rapidly developing and changing technologies and industrial products and practices frequently carry with them the increased generation of materials that, if improperly dealt with, can threaten both public health and the environment. The U.S. Environmental Protection Agency (USEPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between improving the quality of life and minimizing the risks to the environment. These laws direct the USEPA to perform research to define our environmental problems, measure the impacts, and search for solutions.

The Risk Reduction Engineering Laboratory (RREL) is responsible for planning, implementing, and managing research, development, and demonstration programs in order to provide authoritative and reliable information that can be used by both regulators and the regulated in their common efforts to protect the environment from the hazards of industrial and municipal waste. In addition, RREL is also responsible for coordinating and disseminating the latest engineering and scientific technology developments aimed at mitigating the harmful effects of environmental contaminants.

This Technical Resource Document contains the latest information on the use of solidification/stabilization for the treatment of hazardous waste, assembled for EPA by Battelle in close consultation with a distinguished panel of experts eminently renowned in this field. It addresses several issues including such important questions as to when this technology is appropriate for a specific waste and when it is not. Our goal is to provide the user community with the most comprehensive information available to enable them to manage their waste in the most efficient, feasible, and safe manner and to maintain a harmonious relationship between man and his environment.

E. Timothy Oppelt, Director  
Risk Reduction Engineering Laboratory



## ABSTRACT

Stabilization/solidification (S/S) processes are effective in treating a variety of difficult to manage waste materials for reuse or disposal. S/S has been identified as the Best Demonstrated Available Technology for treating a wide range of Resource Conservation and Recovery Act (RCRA) non-wastewater hazardous waste subcategories. S/S has been selected as the treatment technology of choice for 26% of the remedial actions complete at Superfund sites through fiscal year 1992.

The standard bulk material handling and mixing equipment used in many S/S processes make the technology appear simple. However, there are significant challenges to the successful application of S/S processes. The morphology and chemistry of S/S-treated waste are complex. Selection of the binder requires an understanding of the chemistry of the bulk material, the contaminants, and the binder. The S/S user must be fully aware of the complex interactions among the various components to ensure efficient and reliable results.

Battelle, under the direction of the U.S. Environmental Protection Agency, has prepared this Technical Resources Document (TRD) as a resource for the S/S user community and a guide to promote the best future application of S/S processes. An extensive body of information is available describing the theory and practice of S/S processes. However, no one document existed combining theory, practice, and regulatory aspects of S/S application to RCRA, Superfund, and similar waste materials. This TRD pulls a diverse range of materials into one comprehensive reference.

The TRD is intended for site managers considering S/S as an option for treating hazardous wastes. It provides technology transfer to persons responsible for selection and design of S/S treatment methods. Information about S/S technology is presented in detailed text descriptions supported by summary tables, checklists, and figures. It gives the user a summary of current S/S technology. The technology areas covered are binders and their binding mechanisms, waste interferences with S/S processes, S/S treatment of organic contaminants, air emissions for S/S processes, leaching mechanisms, long-term stability, reuse and disposal of S/S-treated waste, and economics. Information is also provided to clarify the limitations of S/S technology and ongoing research to fulfill future development needs.

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## CONTENTS

	<u>Page</u>
NOTICE . . . . .	ii
FOREWORD . . . . .	iii
ABSTRACT . . . . .	iv
TABLES . . . . .	xv
FIGURES . . . . .	xvii
ACKNOWLEDGMENTS . . . . .	xviii
 1 INTRODUCTION . . . . .	 1-1
1.1 BACKGROUND . . . . .	1-2
1.1.1 Definition of Solidification and Stabilization . . . . .	1-2
1.1.2 Position of S/S in the U.S. EPA Environmental Management Options Hierarchy . . . . .	1-2
1.1.3 Application of Solidification/Stabilization . . . . .	1-4
1.2 PURPOSE AND SCOPE . . . . .	1-11
1.2.1 Objectives . . . . .	1-11
1.2.2 Scope . . . . .	1-13
1.2.2.1 Waste Types . . . . .	1-13
1.2.2.2. Processes . . . . .	1-13
1.2.3 Audience . . . . .	1-14
1.2.3.1 CERCLA Applications . . . . .	1-14
1.2.3.2 RCRA Applications . . . . .	1-15
1.3 REGULATORY CONSIDERATION . . . . .	1-15
1.3.1 Regulatory Framework . . . . .	1-15
1.3.2 RCRA Land Disposal Restrictions . . . . .	1-16
1.3.3 Application of Land Disposal Restrictions to CERCLA Sites . . . . .	1-19
1.3.4 Toxic Substances Control Act . . . . .	1-21
1.3.5 Other Environmental Regulations . . . . .	1-22
 2 SOLIDIFICATION/STABILIZATION (S/S) TECHNOLOGY SCREENING PROCEDURES .	 2-1
2.1 INTRODUCTION . . . . .	2-1
2.1.1 Overview . . . . .	2-1
2.1.2 The Need for Treatability Studies . . . . .	2-3

## CONTENTS (Continued)

2.2	SITE-SPECIFIC BASELINE INFORMATION REQUIREMENTS . . . . .	2-5
2.2.1	Waste Sampling . . . . .	2-5
2.2.1.1	Composites vs. Hot Spots . . . . .	2-5
2.2.1.2	Statistical Approaches . . . . .	2-9
2.2.2	Waste Acceptance . . . . .	2-12
2.2.3	Waste Characterization . . . . .	2-13
2.2.3.1	Regulatory Framework . . . . .	2-15
2.2.3.2	Contaminant Characteristics & Treatment Types . . . . .	2-16
2.2.3.3	Sampling and Analysis . . . . .	2-21
2.2.4	Site Characterization . . . . .	2-25
2.2.5	Quality Assurance/Quality Control . . . . .	2-27
2.2.6	Guidance for Site-Specific Information Requirements . . . . .	2-28
2.3	PERFORMANCE OBJECTIVES . . . . .	2-28
2.3.1	Regulatory Requirements . . . . .	2-33
2.3.1.1	CERCLA . . . . .	2-34
2.3.1.2	RCRA . . . . .	2-40
2.3.2	Technical and Institutional Requirements . . . . .	2-43
2.3.3	Approach for Setting Performance Criteria . . . . .	2-43
2.4	INITIAL TECHNOLOGY SCREENING . . . . .	2-46
2.4.1	Technology Screening/Feasibility Study Process . . . . .	2-47
2.4.1.1	CERCLA Technology Screening . . . . .	2-47
2.4.1.2	Technology Screening at RCRA TSD Facilities . . . . .	2-51
2.4.2	General Criteria for Not Using S/S . . . . .	2-51
2.4.3	Outcome of Technology Screening . . . . .	2-51
2.5	WASTE/BINDER COMPATIBILITY LITERATURE SCREENING . . . . .	2-56
2.5.1	Identify Available Binders . . . . .	2-56
2.5.2	Screening Process . . . . .	2-56
2.5.2.1	Interferences and Chemical Incompatibilities . . . . .	2-58
2.5.2.2	Metal Chemistry Considerations . . . . .	2-58
2.5.2.3	Organic Chemistry Considerations for Target Contaminants . . . . .	2-59
2.5.2.4	Compatibility with the Disposal or Reuse Environment . . . . .	2-59
2.5.2.5	Cost . . . . .	2-60
2.5.2.6	Process Track Record . . . . .	2-60

## CONTENTS (Continued)

2.6	LABORATORY BENCH-SCALE SCREENING OF THE WASTE/BINDER MIXTURES . . . . .	2-61
2.6.1	Purpose . . . . .	2-61
2.6.2	Approach . . . . .	2-62
2.6.2.1	Experimental Design . . . . .	2-62
2.6.2.2	Performance Testing . . . . .	2-64
2.6.3	Technical Guidance . . . . .	2-66
2.7	BENCH-SCALE PERFORMANCE TESTING/PROCESS OPTIMIZATION . . . . .	2-66
2.7.1	Purpose and Objectives . . . . .	2-66
2.7.2	How Much Performance Testing? . . . . .	2-70
2.7.2.1	Levels of Risk . . . . .	2-70
2.7.2.2	Levels of Performance Testing . . . . .	2-70
2.7.2.3	Tests for Specific Binding Agents . . . . .	2-74
2.7.2.4	Acceptance Criteria . . . . .	2-74
2.7.2.5	Process Optimization . . . . .	2-75
2.7.3	Technical Guidance . . . . .	2-76
2.8	PILOT-SCALE AND FIELD DEMONSTRATIONS . . . . .	2-76
2.8.1	The Need for Process Scale-Up . . . . .	2-76
2.8.2	Scale-Up Issues . . . . .	2-81
2.8.2.1	Waste Excavation and Handling . . . . .	2-82
2.8.2.2	Stabilizing Agent Storage . . . . .	2-83
2.8.2.3	Pretreatment of Waste . . . . .	2-83
2.8.2.4	Mixing and Curing . . . . .	2-83
2.8.2.5	Stabilized Waste Disposal . . . . .	2-85
2.8.3	Sampling and Analysis of the Treated Waste . . . . .	2-85
3	S/S PROCESS PERFORMANCE TESTS . . . . .	3-1
3.1	PHYSICAL TESTS . . . . .	3-1
3.1.1	General Property Tests . . . . .	3-2
3.1.1.1	Moisture Content . . . . .	3-2
3.1.1.2	Particle Size Analysis . . . . .	3-2
3.1.1.3	Specific Gravity . . . . .	3-2
3.1.1.4	Suspended Solids . . . . .	3-8
3.1.1.5	Paint Filter Test . . . . .	3-8
3.1.1.6	Liquid Release Test (LRT) . . . . .	3-8
3.1.1.7	Atterberg Limits . . . . .	3-8
3.1.1.8	Visual Observation . . . . .	3-9

## CONTENTS (Continued)

3.1.2	Bulk Density Tests . . . . .	3-9
3.1.3	Compaction Tests . . . . .	3-9
3.1.4	Permeability (Hydraulic Conductivity) Tests . . . . .	3-9
3.1.5	Porosity Tests . . . . .	3-10
3.1.6	Strength Tests . . . . .	3-10
3.1.6.1	Unconfined Compressive Strength (UCS) . . . . .	3-10
3.1.6.2	Immersion Compressive Strength . . . . .	3-11
3.1.6.3	Triaxial Compression . . . . .	3-11
3.1.6.4	Flexural Strength . . . . .	3-11
3.1.6.5	Cone Index . . . . .	3-11
3.1.7	General Concrete/Soil-Cement Tests . . . . .	3-12
3.1.8	Durability Testing . . . . .	3-12
3.2	LEACHING/EXTRACTION TESTS . . . . .	3-13
3.2.1	Toxicity Characteristic Leaching Procedure (TCLP) . . . . .	3-19
3.2.2	Extraction Procedure Toxicity (EP Tox) Test . . . . .	3-20
3.2.3	TCLP "Cage" Modification . . . . .	3-20
3.2.4	California Waste Extraction Test (Cal WET) . . . . .	3-20
3.2.5	Multiple Extraction Procedure (MEP) . . . . .	3-21
3.2.6	Synthetic Acid Precipitation Leach Test . . . . .	3-21
3.2.7	Monofilled Waste Extraction Procedure (MWEF) . . . . .	3-22
3.2.8	American Nuclear Society Leach Test (ANSI/ANS/16.1) . . . . .	3-22
3.2.9	Dynamic Leach Test (DLT) . . . . .	3-22
3.2.10	Shake Extraction Test . . . . .	3-23
3.2.11	Equilibrium Leach Test (ELT) . . . . .	3-23
3.2.12	Sequential Extraction Test (SET) . . . . .	3-23
3.2.13	Sequential Chemical Extraction (SCE) . . . . .	3-23
3.2.14	Static Leach Test Method (Ambient- and High-Temperature) . . . . .	3-24
3.2.15	Agitated Powder Leach Test Method . . . . .	3-24
3.2.16	Soxhlet Leach Test Method . . . . .	3-24
3.3	CHEMICAL TESTS AND ANALYSES . . . . .	3-25
3.3.1	pH . . . . .	3-25
3.3.2	Oxidation/Reduction Potential (Eh) . . . . .	3-25
3.3.3	Major Oxide Components . . . . .	3-30
3.3.4	Total Organic Carbon (TOC) . . . . .	3-30
3.3.5	Oil and Grease . . . . .	3-30
3.3.6	Electrical Conductivity . . . . .	3-30
3.3.7	Acid Neutralization Capacity (ANC) . . . . .	3-31
3.3.8	Alkalinity . . . . .	3-31
3.3.9	Total Dissolved Solids (TDS) . . . . .	3-31
3.3.10	Reactive Cyanide and Sulfide . . . . .	3-32
3.3.11	Reactivity of Silica Aggregates . . . . .	3-32
3.3.12	Metal Analysis . . . . .	3-32
3.3.13	Volatile Organic Compounds . . . . .	3-32
3.3.14	Base, Neutral, and Acid (BNA) Organic Compounds . . . . .	3-33

## CONTENTS (Continued)

3.3.15	Polychlorinated Biphenyls (PCBs)	3-33
3.3.16	Other Contaminant Analyses	3-34
3.3.17	Anion Measurements	3-34
3.3.18	Interferants Screen	3-34
3.4	BIOLOGICAL TESTS	3-34
3.5	MICROCHARACTERIZATION	3-36
3.5.1	X-Ray Diffraction	3-37
3.5.2	Fourier Transform Infrared (FTIR) Spectroscopy	3-37
3.5.3	Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Analysis (EDXA)	3-38
3.5.4	Nuclear Magnetic Resonance (NMR) Spectroscopy	3-38
3.5.5	Optical Microscopy	3-38
4	STATUS OF SOLIDIFICATION/STABILIZATION TECHNOLOGY	4-1
4.1	S/S PROCESSES AND BINDERS	4-2
4.1.1	Inorganic Binders	4-3
4.1.1.1	Cement Processes	4-4
4.1.1.2	Pozzolanic Processes	4-7
4.1.1.3	Ettringite Formation Effects	4-8
4.1.2	Organic Binders	4-9
4.1.2.1	Thermoplastic Processes	4-11
4.1.2.2	Thermosetting Processes	4-12
4.1.3	Additives	4-12
4.1.4	Pretreatment	4-14
4.1.4.1	Adjustment of Physical Characteristics	4-14
4.1.4.2	Pretreatment of Inorganic Constituents	4-14
4.1.4.3	Pretreatment of Organic Constituents	4-15
4.1.4.4	Treatment Trains Involving S/S	4-15
4.2	IMMOBILIZATION MECHANISMS	4-16
4.2.1	Physical Mechanisms	4-16
4.2.2	Chemical Mechanisms	4-18
4.2.2.1	Inorganic Wastes	4-18
4.2.2.1.1	Basic Chemical Equilibria	4-18
4.2.2.1.2	Effect of Alkaline Conditions	4-21
4.2.2.1.3	Effect of Redox Potential	4-22

## **CONTENTS (Continued)**

4.2.2.1.4	Metal Silicates . . . . .	4-25
4.2.2.1.5	Other Low Solubility Phases . . . . .	4-26
4.2.2.2	Organic Wastes . . . . .	4-27
4.2.3	Concept of Surface Sealing . . . . .	4-28
4.3	POTENTIAL INTERFERENCES . . . . .	4-29
4.3.1	Interferences with Solidification . . . . .	4-30
4.3.2	Interferences with Stabilization . . . . .	4-30
4.4	ISSUES DEALING WITH THE STABILIZATION OF ORGANIC WASTES AND OF MIXED ORGANIC AND INORGANIC WASTES . . . . .	4-39
4.4.1	Introduction . . . . .	4-39
4.4.2	S/S Additives Compatible with Organics . . . . .	4-40
4.4.3	Approach to Evaluating Feasibility of S/S for Wastes Containing Organics . . . . .	4-45
4.4.3.1	Destructive or Removal Technologies Versus S/S . . . . .	4-48
4.4.3.2	Volatile Organic Contaminants . . . . .	4-48
4.4.3.3	Nonpolar Organic Contaminants . . . . .	4-49
4.4.3.4	Degradation and By-Product Formation . . . . .	4-50
4.5	AIR EMISSIONS AND CONTROL . . . . .	4-50
4.5.1	Volatile Organic Compounds . . . . .	4-51
4.5.2	Particulates and Other Emissions . . . . .	4-52
4.5.3	Controlling Air Emissions . . . . .	4-52
4.5.4	Significance of the Amended Clean Air Act . . . . .	4-53
4.6	LEACHING MECHANISMS . . . . .	4-54
4.6.1	Leaching Associated with Inorganic S/S Processes . . . . .	4-55
4.6.2	Leaching Associated with Organic S/S Binders . . . . .	4-58
4.6.3	Leaching Models . . . . .	4-58
4.6.3.1	Dissolution/Diffusion Kinetics . . . . .	4-58
4.6.3.1.1	Nonporous Solid . . . . .	4-59
4.6.3.1.2	Porous Solid . . . . .	4-61
4.6.3.2	Examples of Existing Models . . . . .	4-63
4.6.3.3	The Moving Boundary or Shrinking Core Model . . . . .	4-66

## CONTENTS (Continued)

4.7	LONG-TERM PERFORMANCE . . . . .	4-66
4.7.1	Field Studies . . . . .	4-67
4.7.2	Laboratory Studies . . . . .	4-68
4.7.3	Modeling . . . . .	4-69
4.8	USE/REUSE VERSUS DISPOSAL . . . . .	4-70
4.8.1	Alternatives . . . . .	4-70
4.8.2	Limitations . . . . .	4-73
4.8.3	Compatibility With the Disposal Environment . . . . .	4-73
4.9	COST INFORMATION . . . . .	4-74
4.9.1	Treatability Study Costs . . . . .	4-75
4.9.1.1	Waste Characterization and Establishing Performance Objectives . . . . .	4-75
4.9.1.2	Bench-Scale Testing and Analysis . . . . .	4-75
4.9.2	Full-Scale Remediation Costs . . . . .	4-77
4.9.2.1	Planning . . . . .	4-77
4.9.2.2	Mobilization and Demobilization . . . . .	4-78
4.9.2.3	Treatment . . . . .	4-78
4.9.2.4	Final Disposal . . . . .	4-82
4.9.3	Estimates of Stabilization Costs . . . . .	4-82
4.9.4	Case Study . . . . .	4-84
5	TECHNOLOGY SHORTCOMINGS AND LIMITATIONS . . . . .	5-1
5.1	PROCESS/BINDER CONSIDERATIONS . . . . .	5-1
5.1.1	Hierarchy of Waste Management . . . . .	5-1
5.1.2	Scale-Up Uncertainties . . . . .	5-1
5.1.3	Proprietary Binders . . . . .	5-1
5.1.4	Binder "Overkill" . . . . .	5-2
5.2	WASTE FORM/CONTAMINANT ISSUES . . . . .	5-2
5.2.1	Complications of Physicochemical Form of the Target Contaminants . . . . .	5-2
5.2.2	Interferences and Incompatibilities . . . . .	5-2
5.2.3	Volatile Organic Contaminants . . . . .	5-3
5.2.4	Multicontaminant Wastes . . . . .	5-4



## CONTENTS (Continued)

5.2.5	Weaknesses of Cement-Based Waste Forms . . . . .	5-4
5.2.6	Sample Heterogeneity . . . . .	5-5
5.3	TREATABILITY AND PERFORMANCE TESTING ISSUES . . . . .	5-5
5.3.1	Testing Limitations . . . . .	5-5
5.3.2	Long-Term Performance . . . . .	5-7
5.3.3	Reproducibility . . . . .	5-8
5.3.4	Limitations in S/S Treatability Reference Data . . . . .	5-8
6	CURRENT RESEARCH AND FUTURE DEVELOPMENT NEEDS . . . . .	6-1
6.1	CURRENT RESEARCH . . . . .	6-1
6.1.1	Binders . . . . .	6-1
	Experimental Study of S/S Treatment of Hazardous Substances . . . . .	6-1
	Improvement in S/S Treatment of Hazardous Inorganic Wastes by Silica Fume (Microsilica) Concrete . . . . .	6-1
	Physical and Chemical Aspects of Immobilization . . . . .	6-1
	Evaluation of Solidification/Stabilization of RCRA/CERCLA Wastes . . . . .	6-2
6.1.2	Mechanisms . . . . .	6-2
	Review and Analysis of Treatability Data Involving S/S Treatment of Soils . . . . .	6-2
	Morphology and Microchemistry of S/S-Treated Waste . . . . .	6-2
	Fate of PCBs in Soil Following Stabilization with Quicklime . . . . .	6-3
	S/S Treatment of Salts of As, Cd, Cr, and Pb . . . . .	6-3
	The Nature of Lead, Cadmium, and Other Elements in Incineration on Residues and Their Stabilized Products . . . . .	6-3
6.1.3	Interferences . . . . .	6-4
	Factors Affecting the S/S Treatment of Toxic Waste . . . . .	6-4
	Effects of Selected Waste Constituents on S/S-Treated Waste Leachability . . . . .	6-4
6.1.4	Organics and Air Emissions . . . . .	6-4
	Roles of Organic Compounds in Solidification/Stabili- zation of Contaminated Soils . . . . .	6-4
	Measurement of Volatile Emissions from S/S-Treated Waste . . . . .	6-5

## CONTENTS (Continued)

Field Assessment of Air Emissions from Hazardous Waste S/S Processing . . . . .	6-5
S/S Treatment of Metal Wastes Contaminated with Volatile Organics . . . . .	6-5
Immobilization of Organics in S/S Waste Forms . . . . .	6-5
6.1.5 Test Methods . . . . .	6-5
Method Development . . . . .	6-5
Investigation of Test Methods for Solidified Waste . . .	6-6
Critical Characteristics of Hazardous S/S-Treated Waste . . . . .	6-6
Advanced Test Methods . . . . .	6-6
Assessment of Long-Term Durability of Solidified/Stabilized Hazardous Waste Forms – Lab Component and Field Component . . . . .	6-7
6.1.6 Leaching and Transport Models . . . . .	6-7
Contaminant Profile Analysis . . . . .	6-7
The Binding Chemistry and Chemical Leaching Mechanism of Hazardous Substances in Cementitious S/S Binders . . . . .	6-7
Development of a Numerical Three-Dimensional Leaching Model . . . . .	6-8
Acid Leaching Rate and Advancement of Acid Front in S/S-Treated Waste . . . . .	6-8
Leaching Test Methods and Models . . . . .	6-8
Review and Analysis of Treatability Data Involving Solidification/Stabilization of Soils . . . . .	6-9
6.1.7 Compatibility with Disposal or Reuse . . . . .	6-9
Assessment of Long-Term Durability of S/S-Treated Waste . . . . .	6-9
Effect of Curing Time on Leaching . . . . .	6-9
Field Performance of S/S-Treated Waste . . . . .	6-10
Utilization and Disposal . . . . .	6-10
6.1.8 Treatability Tests and S/S Process Application . . . . .	6-10
Superfund Innovative Technology Evaluation (SITE) Program . . . . .	6-10
Municipal Waste Combustion Residue S/S Program . . . . .	6-10
Leaching Mechanisms and Performance of S/S-Treated Hazardous Waste Substances in Modified Cementitious and Polymeric Matrices . . . . .	6-12
Stabilization Potential of Lime Injection Multistage Burner (LIMB) Product Ash	

## **CONTENTS (Continued)**

	Used with Hazardous Distillation Residues . . .	6-12
	Stabilized Incinerator Residue in a Shore Protection Device . . . . .	6-12
6.2	FUTURE DEVELOPMENT . . . . .	6-13
6.2.1	Binders . . . . .	6-13
6.2.2	Mechanisms . . . . .	6-13
6.2.3	Interferences . . . . .	6-14
6.2.4	Organics and Air Emissions . . . . .	6-14
6.2.5	Test Methods . . . . .	6-14
6.2.6	Leaching and Transport Models . . . . .	6-15
6.2.7	Compatibility with Disposal or Reuse . . . . .	6-15
6.2.8	Treatability Tests and S/S Application . . . . .	6-16
7	REFERENCES . . . . .	7-1
APPENDIX A:	SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS . . . . .	A-1
APPENDIX B:	DRAFT REPORT: SAMPLING AND ANALYTICAL PROCEDURES . . . . .	B-1
APPENDIX C:	GLOSSARY . . . . .	C-1

## CONTENTS (Continued)

### TABLES

TABLE 1-1.	RCRA WASTES FOR WHICH SOLIDIFICATION/STABILIZATION IS IDENTIFIED AS BEST DEMONSTRATED AVAILABLE TECHNOLOGY (BDAT) . . . . .	1-5
TABLE 2-1.	GENERAL INDUSTRIAL WASTE CATEGORIES . . . . .	2-14
TABLE 2-2.	EXAMPLES OF SOME METAL WASTES TESTED FOR SOLIDIFICATION/STABILIZATION TREATMENT . . . . .	2-17
TABLE 2-3.	EXAMPLES OF SOME METAL AND ORGANIC MIXED WASTES TESTED FOR SOLIDIFICATION/STABILIZATION TREATMENT . . . . .	2-19
TABLE 2-4.	EXAMPLES OF SOME ORGANIC MIXED WASTES TESTED FOR SOLIDIFICATION/STABILIZATION TREATMENT . . . . .	2-20
TABLE 2-5.	EXAMPLES OF OTHER INORGANIC WASTES TESTED FOR SOLIDIFICATION/STABILIZATION TREATMENT . . . . .	2-22
TABLE 2-6.	GUIDANCE FOR COLLECTING BASELINE INFORMATION . . . . .	2-29
TABLE 2-7.	ALTERNATIVE TREATMENT LEVELS FOR SOIL AND DEBRIS CONTAMINATED WITH RESTRICTED RCRA HAZARDOUS WASTES . . . . .	2-36
TABLE 2-8.	EXAMPLES OF REGULATORY ARARs . . . . .	2-40
TABLE 2-9.	TOXICITY CHARACTERISTIC CONSTITUENTS AND REGULATORY LEVELS . . . . .	2-41
TABLE 2-10.	EXAMPLES OF TREATABILITY PERFORMANCE OBJECTIVES BASED ON NONREGULATORY FACTORS . . . . .	2-44
TABLE 2-11.	APPLICABILITY OF SOLIDIFICATION/STABILIZATION TO SITE-SPECIFIC WASTE . . . . .	2-54
TABLE 2-12.	BENCH-SCALE BINDER SCREENING GUIDANCE . . . . .	2-67
TABLE 2-13.	RISK FACTORS FOR EVALUATING LEVELS OF PERFORMANCE TESTING . . . . .	2-71
TABLE 2-14.	LEVELS OF PERFORMANCE TESTING AND EXAMPLE TESTING REQUIREMENTS . . . . .	2-73
TABLE 2-15.	GUIDANCE FOR BENCH-SCALE PERFORMANCE TESTING . . . . .	2-77
TABLE 3-1.	PHYSICAL TESTS . . . . .	3-3
TABLE 3-2.	LEACHING/EXTRACTION TESTS . . . . .	3-14

## CONTENTS (Continued)

TABLE 3-3.	EXTRACTION CONDITIONS . . . . .	3-16
TABLE 3-4.	CHEMICAL TESTS . . . . .	3-26
TABLE 3-5.	BIOLOGICAL TESTS . . . . .	3-35
TABLE 3-6.	MICROCHARACTERIZATION TESTS . . . . .	3-37
TABLE 4-1.	$pK_{sp}$ VALUES FOR SELECTED METAL PRECIPITATES . . . . .	4-19
TABLE 4-2.	$pK_{sp}$ VALUES FOR SELECTED As AND Se PRECIPITATES . . . . .	4-24
TABLE 4-3.	SUBSTANCES THAT MAY AFFECT CEMENT REACTIONS: INHIBITION AND PROPERTY ALTERATION . . . . .	4-31
TABLE 4-4.	SUMMARY OF FACTORS THAT MAY INTERFERE WITH SOLIDIFICATION/STABILIZATION PROCESSES . . . . .	4-33
TABLE 4-5.	POTENTIAL CHEMICAL INCOMPATIBILITIES BETWEEN BINDER AND WASTE CONSTITUENTS . . . . .	4-38
TABLE 4-6.	S/S PROCESSES TESTED ON OR APPLIED TO ORGANIC-CONTAINING WASTES . . . . .	4-41
TABLE 4-7.	COSTS OF TYPICAL ANALYTICAL TESTS OF UNTREATED AND TREATED WASTES . . . . .	4-76
TABLE 4-8.	COSTS OF TYPICAL STABILIZATION CHEMICALS . . . . .	4-79
TABLE 4-9.	COMPARISON OF MAJOR COST ELEMENTS OF SOLIDIFICATION/STABILIZATION WITH CEMENT . . . . .	4-80
TABLE 4-10.	ESTIMATED TREATMENT COSTS MENTIONED IN THE RODs FOR SUPERFUND SITES WHERE STABILIZATION HAS BEEN SELECTED AS A COMPONENT OF THE REMEDIAL ACTION . . . . .	4-83
TABLE 4-11.	STABILIZATION COSTS FOR AN 1,800-CUBIC-YARD SITE CONTAMINATED WITH LEAD . . . . .	4-85
TABLE 6-1	SUPERFUND INNOVATIVE TECHNOLOGY EVALUATION PROGRAM: SOLIDIFICATION/STABILIZATION TECHNOLOGIES . . . . .	6-11
TABLE A-1	SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS . . . . .	A-4
TABLE A-2	SUMMARY SHEET . . . . .	A-23

## CONTENTS (Continued)

### FIGURES

FIGURE 1-1. U.S. ENVIRONMENTAL PROTECTION AGENCY'S HIERARCHY OF HAZARDOUS WASTE MANAGEMENT . . . . .	1-3
FIGURE 2-1. S/S TECHNOLOGY SCREENING . . . . .	2-2
FIGURE 2-2. INFORMATION COLLECTION STEPS IN THE TECHNOLOGY-SCREENING PROCESS . . . . .	2-6
FIGURE 2-3. GENERAL TECHNOLOGY SCREENING PROCEDURE . . . . .	2-50
FIGURE 2-4. DETERMINING WHETHER S/S IS APPLICABLE AT A RCRA TSD FACILITY . . . . .	2-52
FIGURE 2-5. S/S DECISION TREE AT A RCRA TSD FACILITY . . . . .	2-53
FIGURE 2-6. WASTE/BINDER COMPATIBILITY LITERATURE SCREENING . . . . .	2-57
FIGURE 2-7. LABORATORY SCREENING OF WASTE/BINDER MIXTURES . . . . .	2-63
FIGURE 2-8. BENCH-SCALE PERFORMANCE TESTING OF SELECTED WASTE/BINDER MIXTURES . . . . .	2-68
FIGURE 2-9. PILOT-SCALE TEST SCREENING . . . . .	2-80
FIGURE 4-1. PROGRESS OF CEMENTATION REACTIONS . . . . .	4-10
FIGURE 4-2. GENERAL DECISION TREE FOR S/S APPLIED TO ORGANIC CONTAMINANTS. . . . .	4-46
FIGURE 4-3. SCHEMATIC ILLUSTRATION OF CONCENTRATION PROFILES, $C(x)$ , CHARACTERISTIC OF SPECIES DISSOLVING FROM A NONPOROUS SOLID INTO AN AQUEOUS MEDIUM, WITH $x$ BEING THE DISTANCE INTO THE SOLUTION MEASURED FROM THE SOLID/LIQUID INTERFACE. THE TWO RATE-LIMITING CASES AND AN INTERMEDIATE CASE ARE SHOWN . . . . .	4-62
FIGURE 4-4. ILLUSTRATION OF SPECIES DISSOLUTION WITHIN A POROUS SOLID. DISSOLUTION ACROSS A PORE WALL IS SHOWN, COUPLED WITH TRANSPORT THROUGH THE SOLUTION-FILLED PORE TO THE EXTERNAL SURFACE . . . . .	4-64

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## **1 INTRODUCTION**

Solidification/stabilization (S/S) processes are effective in treating a variety of difficult-to-manage waste materials for reuse or disposal. They are flexible enough to accommodate mixtures of contaminants and economical enough to be used for large volumes of waste. Some common S/S applications are incinerator ash, wastewater treatment sludge, and low-level waste from nuclear power plants. S/S has been identified as the Best Demonstrated Available Technology (BDAT) for treating a wide range of Resource Conservation and Recovery Act (RCRA) nonwastewater listed and characteristic wastes. S/S has also been the treatment technology of choice for 26% of the remedial actions completed at Superfund sites through fiscal year 1992 (U.S. EPA, 1992).

This document is a technical resource for the S/S user community and a guide to promote the best future applications of S/S processes. The standard bulk materials handling and mixing equipment processes used in S/S processes make the technology appear simple. However, there are significant challenges to the successful application of S/S processes. This Technical Resources Document (TRD) describes S/S process screening procedures and summarizes the status of S/S processes to assist users and reviewers in their selection, planning, and application of S/S technology.

S/S is frequently the technology of choice for immobilizing soils and sludges containing one or more metal contaminants. S/S is often chosen also for waste with poor handling quality (e.g., a dense, viscous sludge) or for large volumes of waste that are difficult to treat using other technologies (e.g., power plant desulfurization sludge).

The morphology and chemistry of S/S-treated waste are complex. Therefore, selection of the binder requires an understanding of the chemistry of the bulk material, the contaminants, and the binder, as well as of the complex interactions among the various components, to ensure efficient and reliable results. Although there is no sure prescription for selecting a successful binder, a well-structured testing program guided by an understanding of the mechanisms involved in S/S systems will reduce uncertainty in the selection process.



## **1.1 BACKGROUND**

### **1.1.1 Definition of Solidification and Stabilization**

The term "solidification/stabilization" (S/S) refers to a category of waste treatment processes that are being used increasingly to treat a wide variety of wastes—both solid and liquid. Generally, S/S processes are designed and used to accomplish one or more of the following objectives:

- Reduce contaminant/pollutant mobility or solubility
- Improve the handling and physical characteristics of the waste by producing a solid with no free liquid
- Decrease the exposed surface area across which transfer or loss of contaminants may occur.

Numerous other terms, such as "immobilization" and "fixation," have been used to refer to S/S technology. "Solidification" and "stabilization" are preferred here because they encompass the variety of mechanisms that may contribute to contaminant immobilization by this technology. "Solidification" refers to a process in which materials are added to the waste to produce a solid. This may or may not involve a chemical bonding between the toxic contaminant and the additive. "Stabilization" refers to converting a waste to a more chemically stable form. This conversion may include solidification, but it almost always includes use of a physicochemical reaction to transform the contaminant to a less mobile or less toxic form. Note that biological processes such as bioremediation are not included in this definition of S/S (Wiles, 1987).

### **1.1.2 Position of S/S in the U.S. EPA Environmental Management Options Hierarchy**

The U.S. Environmental Protection Agency's (U.S. EPA) hierarchy of hazardous waste management is shown in Figure 1-1. The hierarchy lists, in descending order of emphasis, technical alternatives for the management of hazardous waste. Pollution prevention and waste minimization programs should be instituted to reduce the volume of waste at the source or to recover, reuse, or recycle the waste. If the waste cannot be eliminated or reduced, destructive treatment methods should then be examined. For degradable contaminants, treatment technologies that destroy the contaminant are preferred.

**First Choice (Pollution Prevention):**

**Reduce/Eliminate Waste Products at the Source**

- Design Long-Lived, Low-Impact Products
- Use Less-Hazardous Input Materials
- Minimize Use of Non-Recoverable Input Materials and of Water
- Conserve Energy in Production Operations and Facility Operation
- Improve Process Technology and Practices

**Second Choice (Pollution Prevention):**

**Reuse (Closed-Loop Recycling)**

- Recover Chemicals
- Reuse Water
- Recover Waste Heat

**Third Choice:**

**Recycle Off-Site**

- Ensure Safe Transport to Recycling Operation
- Select Environmentally-Sound Recycling Technology

**Fourth Choice:**

**Treat and Dispose of Unavoidable Wastes Safely**

- Minimize Volume and Toxicity of Wastes
- Dispose of Safely

**FIGURE 1-1. U.S. ENVIRONMENTAL PROTECTION AGENCY'S HIERARCHY OF HAZARDOUS WASTE MANAGEMENT**

However, S/S processes have an important place in the hierarchy because of their ability to treat otherwise intractable wastes.

### **1.1.3 Application of Solidification/Stabilization**

S/S processes are used to manage numerous types of wastes, such as those covered by Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) remediation projects. As shown in Table 1-1, S/S processes have been identified as the Best Demonstrated Available Technology (BDAT) for a variety of Resource Conservation and Recovery Act (RCRA) non-wastewater wastes. S/S processes have been applied to a variety of wastes, such as nuclear, municipal ash, and wastewaters and slurries.

In the case of contaminated soils and debris, S/S is a relatively inexpensive and versatile method of treating large amounts of material with a variety of contaminants. For example, a review of 487 Records of Decision (RODs) from the 1980s showed that 53 sites (11%) documented S/S as at least one component of the source control remedy (U.S. EPA, 1989a). In fiscal year (FY) 1988, S/S processes were used at 25% of the active Superfund sites (U.S. EPA, 1989b). Waste types treated in these projects included soil, sediment, sludges, liquids, and debris. Contaminant types included volatile organic compounds (VOCs) at 21 sites, polychlorinated biphenyls (PCBs) at 19 sites, and inorganics, including metals, asbestos and cyanide, at 43 sites (U.S. EPA, 1989a). It should be noted that more than one type of contaminant may have been present at a given site.

The ROD analysis indicated that, while wastes containing some VOC contamination are treated by S/S processes, the VOCs were not the prime target. Low levels of VOCs can be incorporated coincidentally in a waste treated to immobilize inorganic contaminants (see Section 4.4.3). However, whenever VOCs are present, the possibility of their release as air emissions during treatment needs to be considered. Sites contaminated with high levels of VOCs required pretreatment prior to S/S treatment. Of the sites using S/S processes on wastes with VOC contamination, 33 percent reported using pretreatment; of those without VOCs, only 3 percent used pretreatment.

As shown in Table 1-1, S/S processes can be used for a number of types of sludge that contain inorganic contaminants and, in some cases, inorganics mixed with organics. In cases where high levels of organics are present, the waste is typically incinerated initially. S/S processes can be

**TABLE 1-1. RCRA WASTES FOR WHICH SOLIDIFICATION/STABILIZATION IS IDENTIFIED AS BEST DEMONSTRATED AVAILABLE TREATMENT TECHNOLOGY (BDAT)**

Code	Waste Description	BDAT Treatment/ Treatment Train	Reference
D001	Ignitable (40 CFR 261.21(a)(2))	S/S (one alternative)	55 FR 22714
D002	Other corrosives (40 CFR 261.22 (a)(2))	S/S (one alternative)	55 FR 22714
D003	Reactive sulfides (40 CFR 261.23 (a)(5))	S/S (one alternative)	55 FR 22714
D005	Barium	S/S (one alternative)	55 FR 22561
D006	Cadmium	S/S (except batteries)	55 FR 22562
D007	Chromium	S/S (one alternative)	55 FR 22563
D008	Lead	S/S	55 FR 22565
D009	Mercury (subclass)	S/S (<260 mg/kg total Hg)	55 FR 22572
D010	Selenium	S/S	55 FR 22574
D011	Silver	S/S	55 FR 22575
F006	Some wastewater treatment sludges	Alkaline Chlorination + Precipitation + S/S	54 FR 26600
F007	Spent cyanide plating bath solutions	Alkaline Chlorination + Precipitation + S/S	54 FR 26600
F008	Plating sludges from cyanide processes	Alkaline Chlorination + Precipitation + S/S	54 FR 26600

**TABLE 1-1. RCRA WASTES FOR WHICH SOLIDIFICATION/STABILIZATION IS IDENTIFIED AS BEST DEMONSTRATED AVAILABLE TREATMENT TECHNOLOGY (Continued)**

Code	Waste Description	BDAT Treatment/ Treatment Train	Reference
F009	Spent stripping and cleaning solutions from cyanide processes	Alkaline Chlorination + Precipitation + S/S	54 FR 26600
F011	Spent cyanide solutions from salt bath cleaning	Electrolytic Oxidation + Alkaline Chlorination + Precipitation + S/S	54 FR 26600
F012	Quenching wastewater treatment sludges from cyanide processes	Electrolytic Oxidation + Alkaline Chlorination + Precipitation + S/S	54 FR 26600
F019	Wastewater treatment sludges from coating of aluminum except for some zirconium phosphating processes	S/S	55 FR 22580
F024	Process wastes from the production of certain chlorinated aliphatic hydrocarbons	Incineration + S/S	55 FR 22589
F039	Leachates from listed wastes	S/S (metals)	55 FR 22607
K001	Bottom sediment sludge from the treatment of waste-waters from wood preserving processes that use creosote and/or pentachlorophenol	Incineration + S/S	54 FR 31153
K006	Wastewater treatment sludge from the production of chromium oxide green pigments (anhydrous or hydrated)	S/S (hydrated form only)	55 FR 22583

**TABLE 1-1. RCRA WASTES FOR WHICH SOLIDIFICATION/STABILIZATION IS IDENTIFIED AS BEST DEMONSTRATED AVAILABLE TREATMENT TECHNOLOGY (Continued)**

Code	Waste Description	BDAT Treatment/ Treatment Train	Reference
K015	Still bottoms from distillation of benzyl chloride	Incineration + S/S	55 FR 22535
K022	Distillation bottom tars from the production of phenol/acetone from cumene	Incineration + S/S	53 FR 31156
K028	Spent catalyst from the hydrochlorinator reactor in the production of 1,1,1-trichloroethane	Incineration + S/S	55 FR 22589
K046	Wastewater treatment sludges from the manufacturing, formulation, and loading of lead-based initiating compounds	Reactive - Deactivation Stabilization Nonreactive - Stabilization	55 FR 22593
K048	Dissolved air flotation float from the petroleum refining industry	Incineration + S/S	53 FR 31160 55 FR 22595
K049	Slop oil emulsion solids from the petroleum refining industry	Incineration + S/S	53 FR 31160 55 FR 22595
K050	Heat exchanger bundle cleaning sludge from the petroleum refining industry	Incineration + S/S	53 FR 31160 55 FR 22595
K051	API separator sludge from the petroleum refining industry	Incineration + S/S	53 FR 31160 53 FR 22595
K052	Tank bottoms (leaded) from the petroleum refining industry	Incineration + S/S	53 FR 31160 55 FR 22595
K061	Emission control dust/sludge from primary steel production in electric furnaces	S/S (<15% Zn)	55 FR 22599

**TABLE 1-1. RCRA WASTES FOR WHICH SOLIDIFICATION/STABILIZATION IS IDENTIFIED AS BEST DEMONSTRATED AVAILABLE TREATMENT TECHNOLOGY (Continued)**

Code	Waste Description	BDAT Treatment/ Treatment Train	Reference
K069	Emission control dust/sludge from secondary lead smelting	S/S	55 FR 22568
K083	Distillation bottoms from aniline production	Incineration + S/S	55 FR 22588
K087	Decanter tank tar sludge from coking operations	Incineration + S/S	53 FR 31169
K100	Waste leaching solution from acid leaching of emission control dust/sludge from secondary lead production	Precipitation + S/S	55 FR 22568
K115	Heavy ends from the purification of toluenediamine in the production of toluenediamine via hydrogenation of dinitrotoluene	S/S	55 FR 26601
U051	Creosote	Incineration + S/S	55 FR 22582
U144	Lead acetate	S/S	55 FR 22565
U145	Lead phosphate	S/S	55 FR 22565
U146	Lead subacetate	S/S	55 FR 22565
U204	Selenious acid	S/S	55 FR 22574
U205	Selenium disulfide	S/S	55 FR 22574
U214	Thallium (I) acetate	S/S or Thermal Recovery	55 FR 3891
U215	Thallium (I) carbonate	S/S or Thermal Recovery	55 FR 3891
U216	Thallium (I) chloride	S/S or Thermal Recovery	55 FR 3891

**TABLE 1-1. RCRA WASTES FOR WHICH SOLIDIFICATION/STABILIZATION IS IDENTIFIED AS BEST DEMONSTRATED AVAILABLE TREATMENT TECHNOLOGY (Continued)**

Code	Waste Description	BDAT Treatment/ Treatment Train	Reference
U217	Thallium (I) nitrate	S/S or Thermal Recovery	55 FR 3891
P074	Nickel cyanide	Electrolytic Oxidation + Alkaline Chlorination + Precipitation + S/S	55 FR 26600
P099	Argenate (1-), bis(cyano-C)-potassium	Electrolytic Oxidation + Alkaline Chlorination + S/S	54 FR 26600
P013	Barium cyanide	S/S (one alternative)	55 FR 22561
P103	Selenourea	S/S	55 FR 22574
P104	Silver cyanide	Electrolytic Oxidation + Alkaline Chlorination + Precipitation + S/S	54 FR 26600
P110	Tetra ethyl lead	Incineration + S/S	55 FR 22568
P113	Thallic oxide	S/S or Thermal Recovery	55 FR 3888
P114	Thallium (I) selenite	S/S	55 FR 22574
P115	Thallium (I) sulfate	S/S or Thermal Recovery	55 FR 3888
P119	Ammonium vanadate	S/S	55 FR 3888
P120	Vanadium pentoxide	S/S	55 FR 3889



applied to decrease contaminant mobility in incinerator ash, if necessary. S/S is, in many cases, the only technology that can be applied to a difficult waste form. S/S processes can treat contaminated soil or lagoon sludge either in situ or after the material is excavated and have been successfully applied in the field to treat waste. S/S processes generally use simple, relatively inexpensive equipment and are cost-competitive with other treatment options. Availability of services from a number of vendors and an established record of field performance help minimize management and regulatory barriers to acceptance of the technology.

Laboratory experiments and field experience have demonstrated the ability of the S/S matrix to decrease contaminant mobility by a combination of physical and chemical mechanisms. The exact nature of these mechanisms is, however, not well understood. Long-term testing is difficult because environmental factors affecting the wastes are not defined. The measurement of long-term environmental exposure is cumbersome at best. Accelerated tests, if available, are not calibrated against real environmental effects. Methods need to be developed for measuring the combined effects of environmental factors. However, the main difficulties are the broad variety of wastes to be treated and the commercial secrecy surrounding some of the binder systems available on the market. Without an understanding of the mechanisms and chemistry involved, it is difficult to predict the long-term performance of a binder/waste combination.

Despite its flexibility and broad appeal, S/S treatment is not appropriate for all wastes. It is generally appropriate as a treatment alternative for material containing inorganics, semivolatile and/or non-volatile organics. S/S treatment is typically not the preferred choice in technologies for treating wastes containing only volatile organics (see Section 4.4.3). Selection of S/S treatment for waste containing semivolatile and nonvolatile organics requires a site-specific treatability study or non-site-specific treatability study data generated on waste which is very similar (in terms of type of contaminant, concentration, and waste matrix) to that to be treated. The use of an aqueous leaching methodology such as the TCLP is clearly not a meaningful indication of the degree of immobilization for low-solubility organic contaminants. Therefore, the use of a nonpolar solvent extraction (e.g., the Total Waste Analysis (TWA)) has been recommended. However, this recommendation is still under consideration by EPA because it is

unclear how the results of a solvent extraction relate to the environmental mobility of a contaminant in groundwater. Also, there are few if any data that demonstrate that the chemical interaction between an S/S binder and an organic contaminant is strong enough to resist leaching by an aggressive nonpolar extractant. Therefore, one of the potential pitfalls of using S/S technology to treat waste with significant nonpolar organic contaminants is the inability to adequately assess the extent of contaminant immobilization caused by S/S treatment.

A careful treatability testing program, guided by expert knowledge, is typically required to formulate, test, and apply an S/S treatment system. The need for treatability study data and the importance of conducting appropriate leachability tests as part of the study, are mandatory if organics are present in the waste.

## **1.2 PURPOSE AND SCOPE**

### **1.2.1 Objectives**

This Technical Resources Document (TRD) is intended to be a user's guide, emphasizing technology transfer and promoting the best possible future uses of S/S processes. It addresses the following questions:

- When are S/S processes the preferred treatment technology?
- How do I evaluate alternative S/S processes to select the correct one?
- What are the correct and incorrect ways of using S/S processes?
- How do I design the correct process?

The specific details and approach of each waste treatment project vary, depending on the needs and circumstances of the specific project. It is not possible to prescribe the details of a specific S/S project because there are so many variables. However, some generalized procedures for S/S implementation can be defined. Applying these procedures will enhance uniformity and consistency, thus helping to overcome difficulties sometimes encountered during the application of S/S technology. As the phrase "Technical Resources Document" implies, this document is a technical resource for the S/S user community. Technical information relating to S/S is summarized throughout the

text. Where the information is lengthy, references are provided to other documents to allow the reader access to more detailed background and technical information pertaining to S/S.

The document provides guidance in conducting S/S treatability studies in Chapter 2. High-quality treatability studies are an important step in the selection and optimization of an S/S treatment technology. Chapter 2 addresses the following aspects of each phase of an S/S treatability study, starting with the sampling and waste characterization phase and ending with the field demonstration phase:

- Information requirements
- Acceptance criteria
- Technology screening and testing procedures
- Sequence of activities
- Decision points

Chapter 3 is a review of analysis and test methods. Chapter 4 is a compilation of technical resources information on S/S processes, divided into 10 different sections. Chapter 5 is a discussion of S/S technology shortcomings and limitations. Chapter 6 is a description of ongoing research and a discussion of fruitful areas for further research. Chapter 7 provides bibliographic data for the references cited in the text. Appendix A consists of information checklists to provide users with guidance in planning and conducting S/S treatability studies.

Overall, the TRD gives an appraisal of S/S technology, with a "how-to" theme for technology screening. It does not address design issues or provide detailed instructions, because these are project-specific and cannot be prescribed based on generic information. For example, the TRD describes the options for pretreating waste to develop material with particle size distribution and other properties suitable to S/S treatment. However, selection of the pretreatment approach is site specific.

### **1.2.2 Scope**

This section broadly characterizes the categories of wastes and the types of processes covered in this document.

#### **1.2.2.1 Waste Types**

As stated in Section 1.1, S/S processes have been applied to a wide variety of wastes, both hazardous and nonhazardous, nuclear and nonnuclear, inorganic and organic, liquid and solid.

The primary wastes of interest in this document are wastes regulated under CERCLA, RCRA, and other environmental laws or acts. CERCLA soils and sludges are emphasized because CERCLA technology screening and performance requirements are the most detailed. RCRA is discussed because S/S treatment is identified as BDAT for many RCRA wastes (Table 1-1).

Because the principal aim of this document is to provide information on materials covered by environmental regulation, some classes of wastes are not addressed. Aqueous wastes contaminated with organics and/or metals are not covered. Nuclear wastes, which are regulated by the U.S. Nuclear Regulatory Commission (NRC) rather than the U.S. EPA, are not specifically addressed in the TRD. However, the large body of literature on nuclear S/S technology provides an important resource (Kibbey et al., 1978), and much of the S/S technology developed by the nuclear community is applicable to EPA-regulated wastes. Mixed wastes are not specifically discussed in this document; however, S/S technologies may be applicable to these wastes. For example, liquid radioactive and hazardous tank wastes have been stabilized with a cement-based system that satisfies EPA's hazardous waste regulations and U.S. Department of Energy long-term performance criteria (Peek and Woodrich, 1990).

#### **1.2.2.2 Processes**

S/S technology includes many classes of immobilization systems and applications; example classes include inorganic binders or organic binders, low-temperature processes (e.g., pozzolanic) or high-temperature processes (e.g., vitrification), in situ applications or ex situ applications, and S/S as a sole treatment technology or as a component of a treatment train. The scope of this TRD specifically excludes only vitrification and the formation of ceramics, which involve the application of very high temperatures

(>1,500-°F). Vitrification is discussed in a separate U.S. EPA guidance document currently under preparation [bibliographic citation needed].

### **1.2.3 Audience**

This document is intended for persons planning or applying S/S processes to hazardous waste management. The document describes the treatability testing and project planning approach leading to selection of an effective S/S technology and gives technical background on S/S treatment methods. It is intended to provide technology transfer to persons responsible for selection and design of S/S treatment methods. Information about S/S technology is presented in detailed text descriptions supported by summary tables, checklists, and figures to introduce users who are unfamiliar with S/S technology to the key concepts. The tables, checklists, and figures also serve as a ready reference for experts.

#### **1.2.3.1 CERCLA Applications**

For CERCLA projects, the users of the TRD may include responsible parties (RPs), Remedial Project Managers (RPMs), contractors, and technology vendors. Each has a different role in designing, conducting, and evaluating S/S process testing and selection under CERCLA, as described below.

Currently, RPs plan and manage clean up at approximately half of the Superfund sites. At enforcement sites, RPs are responsible for planning and executing S/S process testing and evaluation under federal or state oversight.

RPMs perform planning and oversight of the remediation. Their role in treatability investigations depends on the designated lead organization (federal, state, or private). Their activities generally include scoping the treatability study, establishing the data quality objectives, selecting a contractor, issuing a work assignment, overseeing the execution of the study, and informing or involving the public as appropriate.

Treatability studies for S/S process testing and evaluation are generally performed by remedial contractors or technology vendors. Their roles in treatability investigations include preparing work plans and other supporting documents, complying with regulatory requirements, executing the study, analyzing and interpreting the data, and reporting the results.

The RPs, RPMs, contractors, and vendors participate in identification of proposed response action, technology screening, development of remedial action alternatives, and evaluation of remedial action alternatives. The TRD provides S/S process-specific information to assist users through the CERCLA planning process.

#### **1.2.3.2 RCRA Applications**

Technology screening at RCRA treatment facilities is driven by the regulations, the specific technologies available at the facility, and the permit conditions. A treatment facility probably has one or more specific immobilization technologies in place with a menu of permitted treatment options available (U.S. EPA, 1989b). Consequently, screening at a RCRA TSD facility means determining whether each proposed waste is treatable by the available permitted immobilization technology. The criterion for satisfactory treatability is the ability of the treated waste to pass all the required tests for acceptance for disposal. The TRD will help RCRA TSD facility operators and engineers match wastestreams to S/S treatment options, design treatability studies, and select test methods. It also will help generators of characteristically hazardous waste who treat their waste to remove the requirements for Subpart C disposal.

### **1.3 REGULATORY CONSIDERATIONS**

This section is intended to provide a brief introduction to the major regulatory considerations for S/S. Due to the complexity of the regulations, this discussion does not attempt to be comprehensive, but rather provides an overview of the regulatory framework within which S/S is generally applied. It is very important for anyone considering the use of S/S treatment to consult the regulatory agencies that have authority over that waste. State and local regulations may vary widely, and implementation of regulatory requirements is often developed on a site-specific basis, particularly in the case of Superfund sites.

#### **1.3.1 Regulatory Framework**

Cleanup and disposal of hazardous wastes are regulated primarily by two federal laws and their amendments.

First is the Resource Conservation and Recovery Act of 1976 (RCRA), as amended by the Hazardous and Solid Waste Amendments of 1984 (HSWA). These give EPA authority to regulate disposal of hazardous waste and set standards for treatment.

The second major law regulating hazardous waste is the Comprehensive Environmental Response Compensation and Liability Act (CERCLA) of 1980, as amended by the Superfund Amendments and Reauthorization Act (SARA) of 1986. CERCLA regulates the cleanup of spilled materials and abandoned hazardous waste sites.

Generally, CERCLA sites are not regulated by RCRA directly. However, CERCLA Section 121(d)(2) requires that Superfund response actions comply with other environmental laws that are applicable or relevant and appropriate requirements (ARARs) (U.S. EPA, 1989c). Determination of ARARs is site-specific. If portions of RCRA regulations constitute ARARs, then these regulations apply to the Superfund sites.

### **1.3.2 RCRA Land Disposal Restrictions**

The part of RCRA that most affects the use of S/S is that related to the Land Disposal Restrictions (LDRs), also referred to as "landban." The LDRs were included in RCRA as part of the Hazardous and Solid Waste Amendments (HSWA) of 1984 following a growing concern that hazardous waste being disposed in the ground (such as in a landfill) would eventually be released into the environment despite containment efforts. Under HSWA, land disposal of hazardous waste is prohibited unless it has been treated first. U.S. EPA is required to establish treatment standards for each type of RCRA hazardous waste. The RCRA definition of "land disposal," or "placement," includes but is not limited to:

any "placement" of hazardous waste in a landfill, surface impoundment, waste pile, injection well, land treatment facility, salt dome formation, salt bed formation, underground mine or cave, and concrete bunker or vault.  
(RCRA 3004(k))

LDRs apply only to wastes that are land-disposed after the effective date of the restrictions. That is, the LDRs do not require that wastes land-disposed prior to the date of the restrictions be removed and treated.

However, wastes being treated under CERCLA remedial response actions may still fall under the land disposal restrictions if RCRA regulations apply as ARARs.

As discussed above, U.S. EPA has established three types of LDR treatment standards (U.S. EPA, 1989c), specified in 40 CFR Part 268:

- a. A concentration level to be achieved prior to disposal of the waste or treatment residual (the most common type of treatment standard)
- b. A specified technology to be used prior to disposal, or
- c. A "no land disposal" designation when the waste is no longer generated, is totally recycled, is not currently being land disposed, or no residuals are produced from treatment.

Treatment standards are established on the basis of the Best Demonstrated Available Technology (BDAT) rather than on risk-based or health-based standards. "Best" is defined as the technology that offers the greatest reduction of toxicity, mobility, or volume of the waste. To be "demonstrated," a treatment technology must be demonstrated to work at a full-scale level, as opposed to bench-scale or pilot-scale. "Available" means that a technology is commercially available. S/S has been identified as BDAT for a variety of waste codes. These waste codes are listed in Table 1-1.

The majority of LDR treatment standards promulgated to date specify concentration levels. For wastes with treatment standards expressed as concentrations, any technology that can achieve the required concentration-based levels may be used (i.e., the BDAT used by U.S. EPA to set the standards is not the required technology). To establish a concentration level(s) for a specific waste code, U.S. EPA selects a subset of the hazardous constituents found in the waste (known as "BDAT constituents") and sets treatment standards for each of these constituents. Although the waste may contain additional constituents, only the treatment standards for the "BDAT constituents" must be met before the wastes can be land-disposed. The residues from treatment of an originally listed waste (e.g., ash or scrubber water) are also listed RCRA hazardous wastes (because of the "derived from" rule), and are therefore also prohibited from land disposal unless they meet the treatment standards for the waste code of the original listed waste from which they derive (U.S. EPA,



1989d). Separate standards are established for wastewaters and nonwastewaters.

If a treatment standard is promulgated as a specified technology, that technology must be used to treat the waste unless an Equivalent Treatment Method Petition is approved by U.S. EPA. To be granted, the petition must demonstrate that the alternative technology achieves an equivalent measure of performance.

Sometimes, both a concentration standard and a treatment standard apply to the same waste code. When this is the case, the two standards usually address different contaminants in that waste. Generally, the technology-based treatment is applied first, then the waste is tested for the concentration and further treatment is applied if necessary to meet the concentration-based standard.

U.S. EPA recognized that not all wastes can be treated to the LDR treatment standards and that alternative treatment standards and methods of land disposal may provide significant reduction in the toxicity, mobility, or volume of wastes and may be protective of human health and the environment. The LDRs therefore provide the following compliance options to meeting the restrictions discussed above:

- **Treatability Variance:** This option is available when U.S. EPA has set a treatment standard as a concentration level, but because a generator's waste differs significantly from the waste used to set the standard, the promulgated treatment standards cannot be met or the BDAT technology is inappropriate for that waste. (For the purposes of the LDRs, CERCLA site managers are considered generators of hazardous waste.) Under a treatability variance, U.S. EPA approves an alternative treatment standard that must be met before that waste can be land-disposed.
- **Equivalent Method Petition:** This option is available when U.S. EPA has set a treatment standard that specifies a technology (e.g., incineration). Generators may use a different technology (e.g., chemical treatment) if they can demonstrate that this technology will achieve a measure of performance equivalent to that of the specified technology.
- **No Migration Petition:** This option may be used to meet any of the four types of LDR restriction. Generators may land-dispose of wastes that do not meet the LDR restriction if they can demonstrate that no

hazardous constituents above health-based levels will migrate from the disposal unit or injection zone for as long as the wastes remain hazardous.

- **Delisting:** This option may be used to demonstrate that a waste is nonhazardous and therefore not subject to any of the RCRA subtitle C hazardous waste regulations, including the LDRs. Delisting only applies when the CERCLA waste is a listed RCRA hazardous waste. Characteristic wastes need not be delisted, but they must be treated to no longer exhibit the characteristic before they can be considered nonhazardous. Generators must demonstrate that (1) the waste does not meet any of the criteria for which the waste was listed as a hazardous waste; and (2) other factors, including additional constituents, do not cause the waste to be hazardous.

### **1.3.3 Application of Land Disposal Restrictions to CERCLA Sites**

CERCLA Section 121(d)(2) specifies that on-site Superfund remedial actions shall attain "other Federal standards requirements, criteria, limitations, or more stringent State requirements that are determined to be legally applicable or relevant and appropriate (ARAR) to the specified circumstances at the site" (U.S. EPA, 1989d). In addition, the National Oil and Hazardous Substances Contingency Plan (NCP) requires that on-site removal actions attain ARARs to the extent practicable. Off-site removal and remedial actions must comply with legally applicable requirements.

For LDRs to be applicable to a CERCLA response, the action must constitute placement of a restricted RCRA hazardous waste. Therefore, the CERCLA site manager must answer these three questions:

1. Does the response action constitute placement?
2. Is the CERCLA substance being placed also a RCRA hazardous waste?
3. Is the RCRA waste restricted under the LDRs?

With respect to the first question, if the waste is transported off site and placed in a land disposal unit as defined by RCRA (landfill, surface impoundments, waste pile, injection well, land treatment facility, salt dome formation, underground mine or cave, concrete bunker, or vault), placement

occurs. On-site disposal of wastes is often less well defined. U.S. EPA uses the concept of "areas of contamination" (AOCs), which are viewed as the equivalent of RCRA units to determine if LDRs apply. An AOC is delineated by areal extent of contiguous contamination. Such contamination must be continuous, but may contain varying types and concentrations of hazardous substances (for example, a waste source such as a waste pit, landfill, or pile, and the surrounding contaminated soil). For on-site disposal, placement occurs when wastes are moved from one AOC into another. Examples of placement include consolidation of wastes from different AOCs into a single AOC, or excavation from an AOC for treatment in a separate unit such as an incinerator or tank that is within the AOC followed by redeposit into the same AOC. Placement does not occur when wastes are left in place or moved within a single AOC (for example, treatment in situ, capping in place, or processing within the AOC — but not in a separate unit such as a tank — to improve structural stability).

The second question entails determining whether the CERCLA substance is a RCRA hazardous waste. Site managers are not required to presume that a substance is a RCRA hazardous waste unless there is affirmative evidence to support such a finding. There are two types of RCRA wastes: listed wastes (those waste types or compounds specifically listed in 40 CFR Part 261) and characteristic wastes (wastes exhibiting the characteristics of ignitability, corrosivity, reactivity, or toxicity, as defined in 40 CFR Part 261). Information on the source, prior use, and process type is usually required and can be obtained from facility business records or examination of processes used at the facility.

In addition to the two categories of RCRA wastes, three principles may apply:

- The "derived from" rule
- The "mixture rule"
- The "contained in" interpretation

First, the "derived from" rule (40 CFR 261.3(c)(2)) states that any solid waste derived from the treatment, storage, or disposal of a listed RCRA waste is also a listed waste, regardless of the concentration of hazardous constituents. For example, ash and scrubber water from incineration of a listed waste

are hazardous on the basis of the derived-from rule. However, wastes derived from a characteristic waste are hazardous only if they exhibit the characteristic.

Another principle is the "mixture rule" (40 CFR 261.3(a)(2)). Under this rule, when any solid waste and a listed hazardous waste are mixed, the entire mixture is a listed hazardous waste. Mixtures of solid wastes and characteristic hazardous wastes are hazardous only if the mixture exhibits a characteristic.

The third principle is the "contained in" interpretation (Office of Solid Waste Memorandum dated November 13, 1986). Under this interpretation, any mixture of a nonsolid waste and a RCRA-listed hazardous waste must be managed as a hazardous waste as long as the material contains (i.e., is above health-based levels of) the listed hazardous waste. For example, if soil or groundwater contains a listed hazardous waste, that soil or groundwater must be managed as a RCRA hazardous waste as long as it "contains" the waste.

If a waste is a RCRA-listed hazardous waste, a "derived from" waste, or a mixture of a listed waste and a solid waste, the waste must be delisted in order to be exempted from the RCRA system. Characteristic wastes need not be delisted, only treated to no longer exhibit the characteristic. A "contained in" waste also does not have to be delisted; it only has to no longer "contain" the hazardous waste.

If the answers to the first two questions determined that placement will occur and that the waste is a RCRA hazardous waste, the third step is to determine applicability of the landbans as specified by the treatment standards promulgated in 40 CFR Part 268. If treatment standards have been promulgated for the waste in question, the landbans apply and the waste must be treated in accordance with these standards. For several of these standards the BDAT used to derive the standard is S/S.

#### **1.3.4 Toxic Substances Control Act**

The Toxic Substances Control Act (TSCA) regulates numerous toxic chemicals, many of which are not commonly encountered in hazardous waste. However, one group of compounds that is regulated under TSCA — polychlorinated biphenyls (PCBs) — is a fairly common type of contaminant at Superfund sites. PCB-containing wastes (other than the California List Wastes) — for example, liquids that contain both PCBs above 50 ppm and RCRA hazardous wastes —

generally require cleanup when the total PCB levels are greater than 50 ppm. However, 40 CFR 761.120(a)(1) excludes spills that occurred prior to May 4, 1987, from the scope of the U.S. EPA's PCB Spill Policy. The U.S. EPA recognizes that old spills require site-by-site evaluation because of the likelihood that the site involves more pervasive PCB contamination than fresh spills, and because old spills are generally more difficult to clean up than fresh spills (particularly on porous surfaces such as concrete). Therefore, spills that occurred before May 4, 1987, are to be decontaminated to requirements established at the discretion of the U.S. EPA, usually through its regional offices.

#### **1.3.5 Other Environmental Regulations**

In addition to RCRA, CERCLA and TSCA, other environmental legislation may be applicable to the use of S/S:

- The Clean Water Act regulates the discharge of liquid effluents to waters of the U.S.
- The Clean Air Act regulates the release of pollutants into the air.
- The Safe Drinking Water Act controls levels of pollutants in drinking water and regulates underground injection wells.
- The Occupational Safety and Health Act regulates exposure of workers to toxic substances and harmful work practices.
- State and/or local regulations pertaining to hazardous wastes, which may be more stringent than the federal regulations.

In the event that S/S produces effluents or conditions which fall under the jurisdiction of one or more of these acts, compliance would be required. As noted at the beginning of this section, consultation with all cognizant regulatory officials responsible for a particular waste or site is advised before undertaking treatment.

## **2 SOLIDIFICATION/STABILIZATION (S/S) TECHNOLOGY SCREENING PROCEDURES**

### **2.1 INTRODUCTION**

#### **2.1.1 Overview**

The process of technology selection, evaluation, and optimization is frequently referred to as "technology screening." A treatment technology that has been properly screened prior to full-scale implementation has the highest probability of success in the field.

This chapter provides guidance on the S/S technology screening process and the steps needed to select and test an appropriate S/S process for each waste type. Figure 2-1 shows the major steps in the technology screening process and their order of implementation. Sections 2.2 through 2.8 correspond to each of these major steps.

Sections 2.2 through 2.4 describe activities that must be undertaken before conducting treatability studies. Section 2.2 discusses the fundamental information requirements for characterizing the waste, including guidance on waste sampling. An example of a Sampling and Analysis Plan is provided in Appendix B. Section 2.3 addresses the need for, and issues related to, establishing S/S treatability performance objectives or acceptance criteria. Regulatory, technical, and institutional requirements are discussed, and an approach for setting performance criteria is presented. Section 2.4 overviews the generic technology screening process leading to the selection of S/S rather than other types of technologies and references documents offering more detail on this subject.

Sections 2.5 through 2.8 describe in detail each of the tiers of treatability testing for S/S processes. Section 2.5 addresses waste/binder compatibility screening. Section 2.6 discusses laboratory screening of waste/binder mixtures, including binder screening and optimization. Section 2.7 addresses bench-scale performance testing, and Section 2.8 discusses pilot-scale testing. During each sequential tier of treatability testing, the testing becomes more specific to the individual waste form.

Three points relating to the technology screening process (Figure 2-1) are emphasized:

- The screening process often requires several iterations through some or all of the steps.

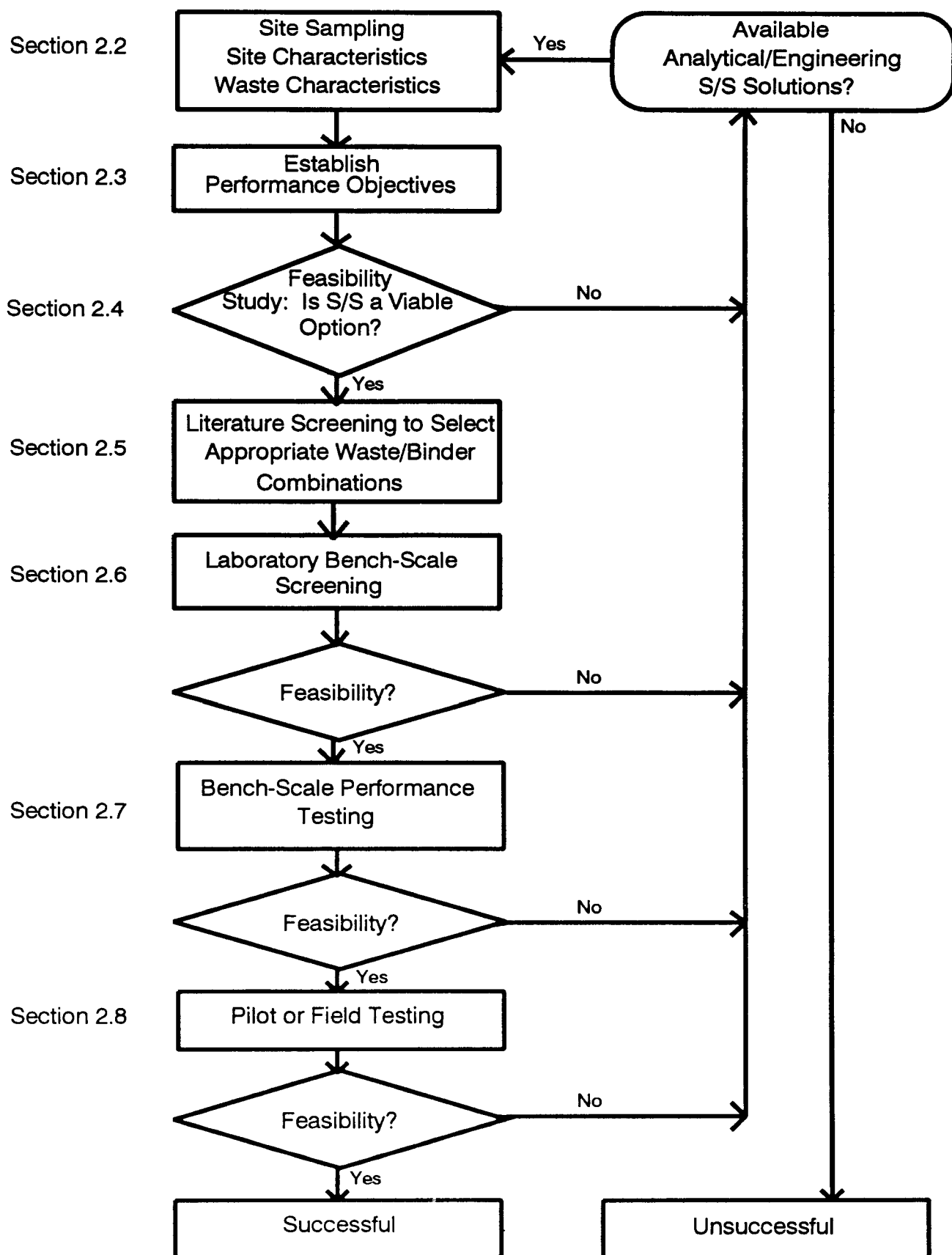


FIGURE 2-1. SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING

A decision point occurs at the end of each step, and, depending on the outcome of the analysis, it may be necessary to return to an earlier stage of the screening process, modify the approach, and repeat one or more steps.

- The screening process must be flexible. Project-specific circumstances require a flexible approach because not all projects have the same set of needs and resources. Under certain circumstances it may be prudent to skip steps or entire sequences of steps. For example, minimal or even no treatability testing might be required for a well-developed S/S process applied to a simple waste. Project-specific resource limitations may also indicate the need to eliminate certain steps. In designing each treatability study, procedural decisions will have to be made based on the trade-offs of the various alternatives. Eliminating various steps in the technology screening procedure can reduce the likelihood of successful technology application; the party responsible for the treatability study must evaluate the risk associated with eliminating such steps.
- In the event that, during treatability testing, S/S appears not to be feasible (i.e., certain critical performance goals are not being achieved), then it may be advisable to return to an earlier step in the screening process and repeat the screening procedure using a different approach or a different set of assumptions. For example, perhaps a completely different binder type should be tested, or the waste should be pretreated prior to S/S. Unsuccessful S/S treatability studies are not uncommon, but technical deficiencies can frequently be overcome by testing different binders or by modifying the S/S process.

### **2.1.2 The Need for Treatability Studies**

Treatability studies provide valuable site-specific data needed to select and implement the appropriate remedy. The Remedial Investigation/Feasibility Study (RI/FS) interim final guidance document (U.S. EPA, 1988a) specifies nine evaluation criteria for use in analyzing alternatives.

Treatability studies can address seven of these criteria:

- Overall protection of human health and the environment
- Compliance with applicable or relevant and appropriate requirements (ARARs)



- Implementability
- Reduction of toxicity, mobility, or volume
- Short-term effectiveness
- Cost
- Long-term effectiveness

The other two criteria affecting the evaluation and selection of the remedial alternative – community and state acceptance – can influence the decision to conduct treatability studies on a particular technology.

Treatability studies should be conducted by individuals or groups with the proper expertise and training. These may include research laboratories, universities, S/S vendors, or treatability vendors. EPA (1990a) provides a compilation of vendors qualified to perform S/S treatability studies and indicates the types of media and contaminant groups in which the firms are experienced.

Several documents provide varying levels of guidance on the design and conduct of treatability studies. For example, U.S. EPA (1989e) provides generic guidance for conducting treatability studies under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) in the context of the RI/FS process and the preparation of the Record of Decision (ROD). The guidance, which is not specific to any technology, includes a discussion of planning documentation and data quality objectives. A related draft document (U.S. EPA, 1990b) provides generic treatability study guidance under CERCLA on S/S technology for inorganic contaminants. Other technology-specific treatability guides have been or are in the process of being published for soil washing, aerobic biodegradation, soil vapor extraction, chemical dehalogenation, solvent extraction, and thermal desorption. An example of a facility-specific guidance document is Barth and McCandless (1989), which outlines S/S treatability testing procedures for U.S. EPA's Center Hill Research Facility. All of these documents supplement information contained in this chapter and should be consulted for appropriate levels of guidance.

## **2.2 SITE-SPECIFIC BASELINE INFORMATION REQUIREMENTS**

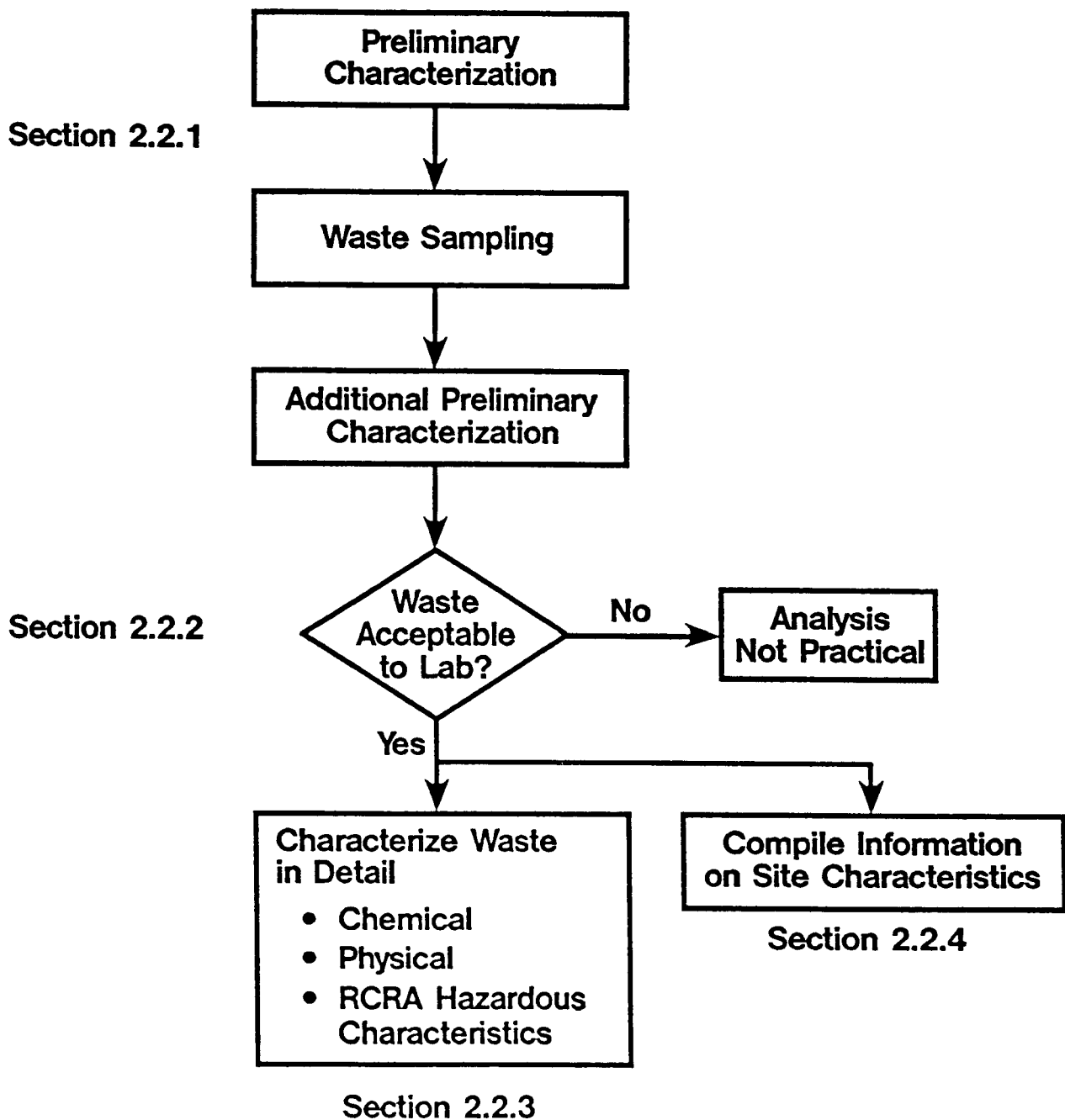
The purpose of this section is to discuss the information requirements for technology screening, which are presented in five subsections: Waste Sampling, Waste Acceptance (the acceptability of the waste at the treatability or analytical laboratory in terms of compliance with applicable permits and other requirements), Waste Characterization, Site Characterization, and Quality Assurance/Quality Control (Sections 2.2.1 through 2.2.5). Figure 2-2 presents the sequence of information collection steps. Initially, preliminary characterization of the waste is needed to support preliminary decisions about the use of S/S and waste acceptability at the test facility. This information is also used to determine appropriate worker protection provisions for waste sampling. Information for preliminary characterization is usually available from remedial investigation (RI) studies if the waste is from a CERCLA site or from other historical records or testing. The RI studies generally do not provide enough information to determine appropriateness of S/S; therefore, additional waste sampling is required to support a waste-specific determination of the appropriateness of various treatability approaches. If the waste is not acceptable at the testing facility, the project cannot proceed until the problem is resolved. In Section 2.2.6, Table 2-6 briefly outlines guidance on site-specific baseline information needs.

### **2.2.1 Waste Sampling**

The principal objective of waste sampling is to obtain waste samples for analysis and treatability testing that are representative both of the waste as a whole, and of the extremes of waste composition ("hot spots"), which can be used for worst-case testing. This can be accomplished in several ways, as described in Section 2.2.1.1. It is also important to obtain a sufficient number of samples and volume of sample to satisfy the analytical and bench-scale testing requirements, because repeat sampling can be expensive and undesirable.

#### **2.2.1.1 Composites vs. Hot Spots**

Many factors affect site sampling. This document is not intended to provide complete coverage of the many reports that should be referred to for guidance regarding sampling strategies and collection and preservation



**FIGURE 2-2. INFORMATION COLLECTION STEPS IN THE TECHNOLOGY-SCREENING PROCESS**

requirements. Such documents include an EPA soil sampling quality assurance document (U.S. EPA, 1989f), EPA's Solid Waste Test Method Manual, commonly referred to as SW-846 (U.S. EPA, 1986a), Conner (1990, Chapter 17), and U.S. EPA (1989e). A sampling technique developed by U.S. EPA Region 10 especially for S/S treatability studies has been shown to be very effective (U.S. EPA Region 10, 1200 Sixth Avenue, Seattle, WA, (206) 442-5810). The discussion that follows emphasizes several issues applicable to sampling for S/S treatability studies.

Prior to detailed sampling, historical records or a grab sample should be used to determine whether the waste can be sampled safely. The waste material should be surveyed to determine the necessary sampling apparatus and the procedures that must be used. Also, some analytical data should be available at this point to determine the appropriate level of personal protective equipment.

As indicated in Section 2.2.1, the principal objective of the sampling activity is to obtain waste samples that are representative of the waste as a whole (in terms of both chemical and physical characteristics) and that are collected in sufficient quantity to permit all the necessary analytical tests to be conducted. Representativeness is crucial but difficult to quantify (U.S. EPA, 1989f). The two approaches to achieving representativeness are as follows:

- Combine samples from a wide range of sampling locations both vertically and spatially to produce a single composite sample that represents the "overall average." A variation of this approach would include compositing the subset of samples with the highest target contaminant levels to produce a "worst-case composite" for bench-scale testing. However, if S/S treatment is applied in batches, combining samples would not represent high-concentration areas that could occur in a particular batch.
- Collect samples from a wide range of locations but do not composite. Analyze samples individually and select the "hot spots" for subsequent bench-scale testing.

Both approaches have advantages and disadvantages. Compositing samples may be more appropriate when (1) a batch-mixing system is to be used

in the field or treated samples are to be composited prior to analysis or (2) the primary purpose in conducting the treatability study is to compare stabilization with some other completely different treatment process. In the latter case, the waste needs to be uniform to ensure comparability. Also, wastes that are already contained in barrels are usually sampled by compositing.

The "hot spot" approach may be more appropriate when a continuous flow-through mixing system such as a pug mill is employed, or when the process will be applied to in situ waste. The composite approach risks overlooking the zones of unusually elevated contaminant or interferant concentrations that may cause the process to fail to satisfy its performance criteria. On the other hand, the "hot spots" may be difficult to define for complex waste forms and may lead one in the direction of an unnecessarily expensive S/S process. The issue is sufficiently complex that an expert system would be needed to sort out all the variables and point to the preferred approach for each individual case. The logic used in selecting samples for treatability studies will be examined by the regulatory authority before accepting test results.

The amount of sample collected should be adequate to satisfy the needs of the waste acceptance, waste characterization, bench-scale screening, and performance testing activities and should include a suitable quantity to be archived for possible later use. One RCRA-permitted facility typically uses 130 kg as the rule of thumb (Barth and McCandless, 1989). This includes about 110 kg for testing and an additional 20% safety margin.

Nonpermitted facilities can perform treatability tests under the treatability study exemption (40 CFR 261.4). However, these facilities are limited to a total of 1000 kg of waste in the facility at one time. Therefore, the testing facility may be reluctant to accept unnecessarily large quantities of sample, particularly if they are performing treatability studies for more than one client.

One possible solution that allows collection of larger quantities of sample is to hold the sample at the site and ship batches to the test facility as needed. Generally, at least 10 kg of sample is needed to provide enough sample to test; however, it is important to be sensitive to the 1000 kg limit.

In practice, sample quantity needs will vary from project to project, depending on the size of the waste material, the complexity of waste

chemistry, QA/QC requirements, and the binder to be used. Other factors affecting sample volume requirements cannot be known beforehand.

#### **2.2.1.2 Statistical Approaches**

It should be emphasized that sampling in support of S/S treatability studies encompasses more than the usual soil or waste sampling undertaken in RI studies at a Superfund site. It is important that the samples are adequately sized and representative. Since wastes may be found in diverse locations and physical states, each sampling routine should be designed to fit the waste and the situation. Wastes to be treated with S/S may occur as nonhomogeneous mixtures in stratified layers or as poorly mixed conglomerates. For such wastes it is particularly important to have a carefully assessed, well-planned, and well-executed sampling routine to ensure that samples are representative. For example, wastes stored in surface impoundments with stratified sludges and covered by wastewater, would probably require samples of the wastewater, the sludges, and the soil beneath the sludges. Additional information on sampling plans can be found in the ASTM Standard Guide for General Planning of Waste Sampling (ASTM-D-4687-87).

Cost is an important factor in determining the extent of sampling. Involvement of a statistician knowledgeable in sample design can help to minimize cost by ensuring that the samples are collected in the most efficient way so as to provide adequate information for statistical analysis of the results.

Sampling for S/S must address four areas, depending on the specific needs of the treatability study and regulatory requirements:

- Chemical composition of the untreated waste
- Physical properties of the untreated waste
- Process control sampling (U.S. EPA, 1990b)
- Quality assurance/quality control (QA/QC) representativeness and accuracy

The first two areas of sampling apply to all S/S treatability studies. However, sampling for process control applies only to pilot-scale studies and to the actual S/S remedial operation.

Assessment of the chemical composition and physical properties of wastes in S/S treatability studies typically is based on a limited number of field measurements. However, the variability of field measurements can be quite complex. This variability is compounded by several factors such as measurement uncertainty, field heterogeneities (e.g., in soil and water properties), and sampling variability. In cases where decisions must be made from highly variable data, it is crucial that the information upon which the decisions are based be obtained from samples that are selected through the use of statistical sampling design procedures. There are at least three important purposes for statistical sampling design:

- to ensure that the sampling is representative
- to provide numerical estimates for decision making that have quantifiable error limits
- to improve sampling efficiency (i.e., to provide estimates that are precise enough at the lowest possible cost)

The design steps for selecting field sampling locations, measurements, and data analyses for S/S treatability studies are similar to those described by other authors for environmental monitoring of chemicals (Keith, 1988; Gilbert, 1987). These five steps can be summarized as follows:

1. Define the sampling zones, sampling frames, and variables(s) of interest.
2. Define a general sample collection strategy for each sampling zone.
3. Develop a statistical model and statistical sampling objectives for each sampling zone.
4. Specify the estimation and/or testing procedures to be employed and their desired statistical properties.
5. Select the sampling design parameters to achieve the desired statistical properties.

The "sampling zone" refers to the specific waste area that must be characterized, typically a contaminated soil body or waste accumulation. The

"sampling frame" then refers to the complete set of potential sampling units (e.g., soil grab samples or core samples) that make up the sampling zone. Each sampling objective must be related to a specific variable that can be measured on every sampling unit (e.g., waste sample, soil sample, water sample). In this way, each objective can be stated in terms of the measured variable and some summary value across the entire sampling zone, such as an average value or a maximum value. Generally, a variety of physical, chemical, and biological properties (e.g., soil moisture, pH, and chemical concentrations) can be measured on each collected sample.

The "sampling strategy" specifies the general method, such as systematic, random, or stratified random, by which sampling locations will be selected. However, establishing the sampling strategy for a particular zone describes the final sampling plan only in general terms. To lay out the specific sampling plan in each zone, the number and locations of samples need to be clearly defined in terms of several sampling design parameters. The "statistical properties" of the sampling design, such as estimation precision, are then a function of these parameters. Examples of design parameters for a monitoring program are as follows: number of sampling locations, number of replications, grid configuration and orientation, sampling times, and measurement precision. If wastes are present in stratified layers such as in a lagoon or waste pit, the depths at which samples are taken will be important to the sample design.

After establishing the sampling frame and variable(s) of interest for each sampling zone, an appropriate mathematical model should be selected to describe the anticipated statistical properties of the measured values. It is important that a knowledgeable statistician be involved in both sample design and model selection. The sampling objectives for each zone can then be refined and restated in terms of the variables and parameters of the statistical model. For every sampling objective, the estimation and inference procedures to be employed must be stated clearly and referenced. Generally, these procedures will involve either estimating of parameter values for the statistical model or testing a statistical hypothesis about the parameter values.

Some examples of mathematical models commonly used in environmental assessments and S/S treatability studies are listed below:



- Gaussian (Normal) Model - used to estimate the average of some characteristic of the waste (e.g., average concentration in soil of a specified contaminant); estimator is the arithmetical average of the measured data.
- Lognormal Model - used to estimate the median of some characteristic of the waste; this model is less sensitive to outlier data than the Gaussian model; estimator is the antilog of the average of the log-transformed data.
- Binomial Model - used to estimate proportions of some characteristic of the waste (e.g., fraction of the waste where the concentration of a contaminant is above a specified threshold); estimator is a sample proportion calculated by comparing measured data to the specified threshold.

Data quality objectives can then be established at levels that make possible reliable decision-making about the chemical and physical properties of the waste from the sampling results. From a sampling design point of view, determining the desired quality of the data amounts to setting requirements for the statistical performance of the selected estimation and inference procedures. Once the data quality objectives have been determined, the specific sampling plan can be established by setting the number of samples, replications, etc. required to satisfy the data quality objectives.

For example, a data quality objective for a particular study might be to assess the waste for the average concentration of a toxic metal (e.g., mercury) in the waste to within an error of plus-or-minus 20%. Using the properties of the mathematical model, the statistician can easily determine the minimum number of samples required to satisfy the data quality objective. It is often useful to have the statistician prepare a table relating different sample sizes to the corresponding statistical confidence levels, so that sampling costs can be controlled by trading off resources available against confidence required.

### **2.2.2 Waste Acceptance**

Waste acceptance involves analyzing a representative subsample to determine compliance with existing facility permits for the laboratory where subsequent analytical and bench-scale testing is to occur and to screen waste for the safety of facility personnel. The primary issue here is that S/S

treatment in the field usually involves close contact between workers and the waste, and there are types of waste that may be too toxic to permit either the laboratory or field operations to be conducted safely. Such wastes are screened from further consideration as a candidate for S/S treatment at this point. Less toxic materials can be handled by nonpermitted facilities if they have a treatability study exemption (40 CFR 261.4 (f)(4)).

A representative subsample of the untreated waste must undergo chemical analysis before being shipped to the analytical or bench-scale testing facility (or facilities) to meet U.S. Department of Transportation (DOT) shipping requirements and to demonstrate compliance with existing facility permits, permit exclusions for treatability studies, and/or Health and Safety Plans. Problematic constituents include dioxins, furans, radio-nuclides, and excessive levels of PCBs or cyanide. In addition, there may be applicable DOT pre-shipment requirements and hazardous waste manifest or driver certification requirements that must be satisfied during shipping. In addition, even if the waste does not present an unacceptable degree of hazard at a permitted laboratory or test facility, it may present health or safety problems for workers in the field during the full-scale S/S treatment. The potential for this type of situation should also be assessed (U.S. EPA, 1990b).

### **2.2.3 Waste Characterization**

The purpose of this section is to provide a brief overview of the various waste types and contaminants and their suitability for treatment with S/S technology. Industrial wastes include a wide variety of materials, both hazardous and nonhazardous. The wastes may come from various types of industries such as manufacturing, chemical production, petroleum refineries, or power production. These wastes typically include materials such as sludges, spent cleaning materials, pickle liquors, plating wastes, and combustion residues. Many of these wastes are complex mixtures that cannot be categorized easily. Table 2-1 lists generic wastes under broad industrial groupings. These generic waste types are not all amenable to S/S treatment but are presented to illustrate the types of industrial wastes encountered in practice. S/S processes are generally used to treat sludges or contaminated soils. Major producers of hazardous sludge include private industries,

TABLE 2-1. GENERAL INDUSTRIAL WASTE CATEGORIES

Industry	Waste category or source
Automobile	Automobile assembly wastes, foundry plant wastes, neutralized pickle liquors, treated plating wastes, treatment plant wastes
Chemical	Acids, alkalies, metal-containing sludges, treatment plant sludge
Chemical cleaning	Spent cleaning solutions
Dredging	Contaminated dredge spoils
Food processing	Biological treatment sludges
Leather tanning and finishing	Biological treatment sludges, metal-containing sludges
Metal finishing and major appliance	Dissolved metal solutions, pickle liquors, rinse water neutralization sludge, treatment plant sludge
Municipal	Sewage sludges, water treatment sludges
Nonferrous metals	Air pollution control (APC) dust and sludges, lime/limestone wet scrubber sludge, waste pickle liquors, water treatment sludge
Paint and painting	Metal pickling and cleaning wastes, paint sludges
Pharmaceutical	Biological treatment sludge, filter cake, spent carbon
Plastic and rubber	Biological treatment sludge, metal-containing sludge
Pollution control	APC sludges, general spent activated carbon, spent resins, water treatment plant sludges
Power	Fly ash, lime/limestone scrubber sludges, boiler cleaning solutions
Pulp and paper	Biological treatment sludges, spent clay and fibers
Refinery and petrochemical	American Petroleum Institute oil/water/sludge mixtures, biological treatment sludge, spent lime sludges
Sanitary landfill	Landfill leachates
Steel	APC dust and sludges, metal fines, scale pit sludge, waste pickle liquors, water treatment sludge
Textile	Biological treatment sludges, metal-containing sludges

Reprinted from: Conner, J. R. 1990. *Chemical Fixation and Solidification of Hazardous Wastes*. Van Nostrand Reinhold, New York. pp. 267-268. Used by permission of Van Nostrand Reinhold.

utility companies, and water/wastewater treatment plants. Waste types can be broadly categorized under a variety of hazardous waste regulations.

#### **2.2.3.1 Regulatory Framework**

One major waste type considered in this document is wastes covered by CERCLA (see Section 1.3.) Hazardous substances under CERCLA are broadly defined and include a wide variety of materials. The concept of "hazardous substances" under CERCLA is defined with reference to all of the major federal environmental statutes. Approximately 700 elements, compounds, and waste streams are designated as "hazardous substances" under CERCLA (40 CFR 302.4) by virtue of their regulation under one or more of these other environmental statutes. However, petroleum, natural gas, natural gas liquids, liquefied natural gas, and synthetic gas usable for fuel are excluded from the definition of "hazardous substances" under CERCLA.

Hazardous wastes that are covered by the Resource Conservation and Recovery Act (RCRA) are defined in the regulations specified in 40 CFR Part 261. Such wastes are either "listed wastes" or "characteristic wastes," as discussed in the following paragraphs (see also Section 1.3).

"Listed wastes" are specific chemicals or specific types of wastes listed in 40 CFR Part 261 Subpart D. Each listed waste is assigned a hazardous waste identification number. Hazardous wastes from nonspecific sources (e.g., spent halogenated solvents used in degreasing) are listed in 40 CFR 261.31. Hazardous waste from specific sources (e.g., distillation bottoms from the production of acetaldehyde from ethylene) are listed in 40 CFR 261.32. Discarded commercial chemical products, off-specification materials, container residues, and spill residue (i.e., specific chemicals) are listed in 40 CFR 261.33.

Wastes that are not specifically listed may be considered hazardous because they have one or more of the four characteristics defined in 40 CFR 261 Subpart C. These hazardous characteristics – ignitability, corrosivity, reactivity, and toxicity – are defined in 40 CFR Part 261 Subpart C.

The Toxic Substances Control Act (TSCA) provides a regulatory framework for dealing comprehensively with risks posed by the manufacture and use of chemical substances. Under TSCA, U.S. EPA is authorized to regulate the manufacture, processing, distribution, use, and disposal of a chemical or a mixture of chemicals. The U.S. EPA can place restrictions on specific

compounds or groups of compounds if they pose an unreasonable risk to health or the environment. Polychlorinated biphenyls (PCBs) are one group of compounds the U.S. EPA has chosen to regulate under TSCA. The disposal requirements for PCBs given in 40 CFR 761.60 apply to cleanup of PCB-contaminated wastes or soils at CERCLA sites.

#### **2.2.3.2 Contaminant Characteristics and Treatment Types**

Contaminant classes in wastes include metals and metal compounds, organics of various types, and other constituents such as anions. The class of contaminants in a waste will influence the type of S/S treatment that can be applied to the waste.

Metal and metal compounds include native metal, salts of metals, and metal anions such as arsenate, molybdate, or selenate. Metal contaminants cannot be destroyed by chemical or thermal methods. Therefore, they are either extracted from the waste and concentrated into a more manageable form via a soil washing/extraction technology or are immobilized via S/S. Although immobilization is theoretically possible for most metals, the difficulty and cost of such treatment varies greatly according to numerous factors, such as form, speciation, quantity, and concentration of the metal. Some examples of metals and groups of metals tested for S/S treatment are listed in Table 2-2.

Certain organic-contaminated wastes, such as heavy sludges or soil contaminated with organics, are also amenable to S/S treatment. This is true particularly if the organics are present with metals or anions, are minor components of the waste, or are nonvolatile and/or viscous (see Table 2-3). Given the wide variety of organic compounds, it is not possible to prepare a comprehensive list of organic compounds amenable to S/S treatment. However, Table 2-4 lists some organic wastes that have been considered as candidates for S/S treatment. Solidification/stabilization, either directly or following incineration, has been identified as the Best Demonstrated Available Technology (BDAT) for some organic wastes (see Table 1-1). However, wastes with significant quantities of organic material, particularly volatile organic material, typically are treated better with other types of treatment technology. Organic materials can frequently be extracted or destroyed by chemical or thermal processes. Organics can be difficult to stabilize with inorganic S/S binders and can, in fact, interfere with the setting reactions (see

**TABLE 2-2. EXAMPLES OF SOME METAL WASTES TESTED FOR SOLIDIFICATION/  
STABILIZATION TREATMENT**

Contaminant	Waste Type
Aluminum	Metal finishing
Aluminum (and other metals)	Aluminum anodizing sludge
Antimony (and other metals)	Battery manufacturing flue dust
Arsenic	Phosphoric acid filter cake
Arsenic	Fly ash
Arsenic	Herbicide waste
Arsenic	Phosphoric acid filter cake
Barium	Various
Cadmium (and zinc)	Salt slurry
Cadmium (and other metals)	Battery plant sludge
Cadmium (and other metals)	Contaminated soil
Chromium (and other metals)	Chromium plating sludge
Chromium (and other metals)	Aluminum anodizing sludge
Chromium (and other metals)	Chromic acid rinse
Chromium (and other metals)	Contaminated soil
Copper	Catalyst
Copper	Catalyst substrate
Copper (and zinc)	Filter press cake
Copper (and tin)	Foundry sand
Copper (and other metals)	Metal finish
Copper (and other metals)	Clarifier sludge
Lead	Portland cement kiln dust
Lead (and other metals)	Battery plant sludge
Lead (and other metals)	Battery manufacturing flue dust
Lead (and other metals)	Contaminated soil
Mercury	Chlor-alkali mercury cell
Nickel (and other metals)	Battery plant sludge
Nickel (and other metals)	Metal finishing sludge

**TABLE 2-2. EXAMPLES OF SOME METAL WASTES TESTED FOR SOLIDIFICATION/  
STABILIZATION TREATMENT (Continued)**

Contaminant	Waste Type
Nickel (and metals)	Contaminated soil
Silver	Various
Sodium	Metal finishing salt sludge
Tin (and metals)	Battery manufacturing flue dust
Zinc (and cadmium)	Metal salt slurry
Zinc (and copper)	Clarifier sludge
Zinc (and copper)	Filter press cake
Zinc (and metals)	Battery plant sludge
Zinc (and metals)	Contaminated soil
Mixed metals	Paint sludge
Mixed metals	Foundry sludge
Mixed metals	Ore processing leaching residue
Mixed metals	Printing wastewater treatment sludge
Mixed metals	Printing wastewater treatment filter cake
Mixed metals	Paint waste incinerator ash
Mixed metals	Electrochemical machining waste
Mixed metals	Biosludge from chemical process waste treatment
Mixed metals	Clarifier sludge
Mixed metals	Lagoon sludge
Mixed metals	Wastewater treatment filter cake
Mixed metals	Neutralized acids
Mixed metals	Foundry and baghouse dust

Note: Degree of solidification/stabilization achieved was not reported.  
Sources: Conner, 1990, pp. 269-271; and U.S. EPA, 1989g.

**TABLE 2-3. EXAMPLES OF SOME METAL AND ORGANIC MIXED WASTES  
TESTED FOR SOLIDIFICATION/STABILIZATION TREATMENT**

Contaminant	Waste Type
Aluminum, paraffins, and water	Waste lubricant
Barium and organics	Coke dust
Chromium and organics	Tannery waste
Chromium and organics	Drilling mud
Oil, cadmium, chrome, and lead	Refinery sludge
Oil, lead, chromium, and arsenic	Refinery sludge
Oil, lead, PCB, and arsenic	Contaminated soil
PAH and organics	Contaminated soil
PCB and VOC	Contaminated soil
Metals and oil	Spent oil re-refining bleach clay
Metals and oil	Metal finishing buff wash
Metals, oil, and sulfur	Synthetic oil sludge
Metals and organics	Weathered oil waste
Metals and organics	Coating manufacture waste sludge
Metals and organics	Coating manufacture wastewater treatment sludge
Metals and organics	Wastewater treatment plant sludge
Metals and organics	Hazardous waste landfill leachate
Metals and organics	Landfill leachate
Metals and organics	Mixed lagoon sludge
Metals and organics	Printing waste sludge
Metals and organics	Solder stripping solution
Metals and organics	Wire manufacture vinyl waste
Metals and organics	Tannery lagoon biosludge

Note: Degree of solidification/stabilization achieved was not reported.  
Sources: Conner, 1990, pp. 269-271; U.S. EPA, 1989g.



TABLE 2-4. EXAMPLES OF SOME ORGANIC WASTES TESTED FOR  
SOLIDIFICATION/STABILIZATION TREATMENT

Contaminant	Waste type
Carbon tetrachloride and carbon disulfide	Waste sludge
Chlorinated hydrocarbons	Petrochemical manufacturing waste
Creosote	Waste sludge
Kepone	Contaminated soil
Naphthalene compounds	Waste sludge
Oil and grease	Contaminated soil
Oil and grease	Oil, soap, and grease in water
Oil and grease	Oil sludge
Pesticides	Sludge
PCB	PCB oil
PCB	Contaminated soil
Silicones	Silicone waste
Solvents	Rubber waste
Solvents	Paint waste
Synthetic rubber	Rubber waste
Vinyl chloride and ethylene chloride	Sludge
Organics	Paint wastewater treatment sludge
Organics	Paint waste sludge
Organics	Acrylic/epoxy paint wash
Organics	Mixed lagoon sludge
Organics	Oil refining caustic waste

**TABLE 2-4. EXAMPLES OF SOME ORGANIC WASTES TESTED FOR  
SOLIDIFICATION/STABILIZATION TREATMENT (Continued)**

Contaminant	Waste type
Organics	Tall oil resin waste
Organics	Organic phase of landfill leachate
Organics	Lacquer solvent still bottoms
Organics	Synthetic resin waste
Organics	Tannery waste
Organics	Phenolic resin waste

Note: Degree of solidification/stabilization achieved was not reported.  
Sources: Conner, 1990, pp. 269-271; U.S. EPA, 1989g.

Section 4.4.3 for a detailed discussion of the issues concerning the stabilization of organic contaminants).

On the other hand, fluid oil- and solvent-based wastes, such as used solvents, distillation bottoms, and refinery wastes, are candidates for S/S treatment only in specialized applications where solidification is required temporarily for safety in transportation or storage, or in spill control work. These wastes are normally incinerated if they are hazardous.

Other constituents of concern in S/S include several additional nonmetal inorganic species. Table 2-5 lists examples of some inorganic species tested for S/S treatment.

#### **2.2.3.3 Sampling and Analysis**

Waste characterization for S/S treatability studies goes beyond the requirements of the RI and is usually done after the RI has been completed. This characterization phase involves analyzing untreated waste samples for chemical, physical, and hazardous characteristics. The minimum amount of waste characterization for CERCLA sites is screening for substances on the

**TABLE 2-5. EXAMPLES OF OTHER INORGANIC WASTES TESTED FOR  
SOLIDIFICATION/STABILIZATION TREATMENT**

Contaminant	Waste type
Acid waste	Metal finishing solution
Acid (and metals)	Sludge
Acid waste (and organics)	Sludge
Boron fluoride	Pilot plant waste
Caustic waste	Aluminum drawing waste
Cyanide (and metals)	Plating sludge
Fluoride (and metals)	Calcium fluoride sludge
Fluoride (and organics)	Mixed petroleum refining wastes
Oxalates, sulfides (and organics)	Spent pulping liquor

Note: Degree of solidification/stabilization achieved was not reported.  
Sources: Conner, 1990, pp. 269-271; and U.S. EPA, 1989g.

Hazardous Substances List. Actual chemical analysis for each of these compounds may not be necessary if site records clearly show certain substances to be absent. However, some confirmation analyses may be necessary. The objective is to determine with confidence the primary target contaminants and any waste substrates or characteristics that may interfere significantly with the S/S process.

Two additional objectives for collecting waste characterization data are that such data are useful in selecting the most suitable binding agent for the waste and in predicting the ultimate performance of the waste/binder mixture. While at present these objectives are not always achievable, they underscore the need for an accurate and statistically designed database of waste characteristics information for each waste type being evaluated.

The amount of new data that must be generated as part of the S/S treatability study can frequently be minimized by examining waste and site history and any characterization data that may have been already generated. If data exist and are reliable, they may eliminate or reduce the need for additional testing. At a minimum, background information on waste history will allow the subsequent analytical activities to be more focused, emphasizing target contaminants and problem constituents.

Types of characterization data that may be required for the untreated waste include chemical, physical, or physicochemical (i.e., relating to the form of the contaminant as opposed to its bulk concentration). A number of frequently used testing methodologies are compiled in Chapter 3, and their applicability to untreated waste is indicated. The reasons for generating characterization data include:

- To gather information on substances that interfere with common S/S processes.
- To establish baselines for comparison with chemical data on the treated waste.
- To gather information on U.S. EPA hazard characteristics.
- To establish the target contaminants and their physicochemical form.

One of the primary reasons for collecting characterization data is to establish the target contaminants in the waste, in terms of both identity and concentration. At a minimum, the waste should be characterized using a "total waste analysis" or the equivalent, including:

- Elemental analysis (metals)
- Volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs)
- Base, neutral, and acid compounds (BNAs) (see Chapter 3 for methods)

The minimum analysis should also include leaching data to define the soluble portion of the contaminant in the waste, yielding an understanding of

contaminant partitioning in the waste. If possible, it is desirable also to have some information on physicochemical form. This is true particularly for metals, whose reactivity with various binding agents can vary significantly depending on the species present. Because detailed microcharacterization can be expensive, the analysis program should be thought out carefully. Examples of microcharacterization data include valence state information for elements such as arsenic (As) or chromium (Cr), solid phase characterization, elemental analysis, and structural characterization. Section 3.5 provides a brief overview of procedures for microcharacterization. Detailed micro-characterization is typically used only in research and development projects.

Characterization of wastes from CERCLA sites should include at least substances on the Hazardous Substances List (both organics and metals). Also, if not collected as part of the baseline data discussed above, data on the soluble (leachable) contaminants in the waste need to be generated to establish the target contaminants whose leachabilities must be reduced during the S/S process. Also needed are data on the RCRA hazard characteristics of the waste. The four types of hazard characteristics are toxicity, ignitability, reactivity, and corrosivity. If present, the hazard characteristics for ignitability, reactivity, and/or corrosivity may preclude stabilization or at least indicate the need for pretreatment.

Baseline data can include a variety of parameters and, by definition, are needed to assess how the parameters change during S/S treatment. Such data may be either chemical (e.g., pH, Eh, total and leachable contaminants) or physical (e.g., specific gravity, permeability, physical state, total solids, particle size distribution, presence of debris, dustiness, viscosity, etc.). Perhaps the most important baseline data at this stage are data that demonstrate the hazardous nature of the waste and thus constitute the basis for the S/S treatment. The hazardous classification may be based upon either soluble (e.g., Toxicity Characteristic Leaching Procedure [TCLP]) or total (acid-digestible) contaminant concentrations. If the waste is not legally hazardous and if there is no other regulatory-driven need to stabilize the waste, there may be no need to proceed with the S/S project.

Another characterization data category is constituents that may interfere with the S/S process. These include a great variety of constituents, depending on the binding agent contemplated. Examples are oil and

grease and soluble salts such as halide for cement-based technologies (see Section 4.3).

Along with the chemical data, there is a need for physical properties and textural characteristics data, because heterogeneous wastes containing large blocks or boulders may be difficult to process without pretreatment. Also included in this category are other parameters that will aid in the selection of the binding agent or the design of the S/S process. Examples are particle size and water content.

#### **2.2.4 Site Characterization**

Information on site characteristics is an important aspect of the technology screening process. The following types of information are highly useful:

- Baseline information on the geology, hydrology, weather, etc., may constrain the design of the field treatment system, influence project timing, and have other effects.
- Site layout and proximity to needed resources also affect engineering design and, therefore, project cost.
- Information on site history may provide valuable insight about the waste, including the types of chemicals that were used at the site and the general location where they were released or disposed of. Knowledge of site operations can also suggest metal speciation (e.g., presence of anionic forms of metal).

Overall site-specific concerns with regard to a remedial action project are geared toward evaluating waste containment potential. Important site parameters in this regard include the following (modified from Colonna et al., 1990).

- Area of the site
- Permeability of the area soils, both for a review of leaching capabilities and for possible liner/cap material
- Amount and type of rocks and debris

- Existing groundwater contamination
- Baseline information on uncontaminated or upgradient groundwater
- Groundwater flow regimes
- Velocity and direction of both groundwater and ambient air
- Site drainage
- Site meteorology
- Proximity to populated areas
- Location and sensitivity of receptors
- Access routes to and from the site, including any United States Department of Transportation (U.S. DOT) restrictions
- Available work area/stockpiling area on the site
- Final disposal options and their site-specific implications
- Postremediation use of the site
- Sensitive environmental areas within the work site, such as floodplains or marshes
- Waste product volume increase and its implications for the capacity of the site to contain final product if on-site disposal is required/preferred
- Potential for fugitive dust
- Ability to mix the materials adequately on the site
- Availability of the binder materials and additives in the amounts required for the entire site

Most of the site information needs can be categorized as relating to water table, climate, soil characteristics, site layout, or logistics (U.S. EPA, 1989b).

In some cases, the waste site cannot provide sufficient area for the expected processing, binder stockpiling, and temporary or final waste disposal. Some kinds of processing require stockpiling of untreated excavated wastes, the processed wastes, and the binder. These materials may have to be

covered to reduce exposure to wind and precipitation. Binders increase the volume of the waste product, and this added volume could present difficulties if the S/S product is buried in the original waste site excavation. Solutions to problems posed by limited area must be developed on a site-specific basis. Delivery of preweighed amounts of the binder directly to the process site is a possible solution. The binder then can be added directly to the mixing area rather than being stockpiled in bulk containers.

The presence of an elevated water table extending into the potential disposal zone in the waste area creates four problems:

1. A water table poses the possibility of existing groundwater contamination.
2. Excess water (especially flowing water) can cause excavation difficulties.
3. A water table creates the potential need for dewatering a saturated waste material prior to its processing.
4. Also, if on-site disposal is selected, there is a higher potential for leaching of the disposed waste, and there probably will be a requirement for a permanent groundwater monitoring system and collection of leachate.

All four of these problems have significant cost implications and must be resolved before the final technology selection is made (Colonna et al., 1990).

#### **2.2.5 Quality Assurance/Quality Control**

Quality assurance/quality control (QA/QC) is an important aspect of waste sampling and characterization. The results of the chemical analyses must be valid and statistically significant.

The U.S. EPA's quality assurance policy requires that every monitoring and measurement program have a written and approved Quality Assurance Project Plan (QAPjP). These requirements are specified in Costle (1979a and 1979b). The specified QA/QC requirements apply to all environmental data collection, monitoring, and measurement efforts authorized or supported by the U.S. EPA. It is important that anyone undertaking an S/S treatability study



understand U.S. EPA QA/QC objectives and requirements in order to achieve data quality.

Another objective of the QA/QC program is to assess and identify measurement errors that may enter the data collection and measurement system at various phases of the project during sampling, sample handling/ preparation, and analysis. The U.S. EPA Superfund Treatability Study Protocol (U.S. EPA, 1990b) and the documents cited therein provide an overview of U.S. EPA QA/QC guidelines for treatability studies, including a discussion of the following:

- Preparation of the Quality Assurance Project Plan (QAPjP)
- Data quality objectives (DQO)
- The need to identify the sources and types of errors that may occur during the sampling, analysis, and treatability measurement process
- The need for quality control samples
- Data quality indicators, measurement errors, and documentation

#### **2.2.6 Guidance for Site-Specific Information Requirements**

Table 2-6 lists several guidelines pertaining to the sampling and analysis activities that support the S/S technology screening process, as discussed in Section 2.2. For many remedial action projects involving S/S, particularly those involving relatively simple sites, not all of the guidance in Table 2-6 will necessarily apply. For large, complex projects, there may be additional issues and concerns not listed in Table 2-6.

### **2.3 PERFORMANCE OBJECTIVES**

Treatability performance objectives or performance standards are specified values of the properties of S/S-treated wastes as determined by specific tests or measurements. The properties tested are those that are legally mandated and/or considered crucial for predicting the efficacy and long-term reliability of S/S. Every remedial action project needs a clearly defined set of measurable performance objectives. The success or failure of

TABLE 2-6. GUIDANCE FOR COLLECTING BASELINE INFORMATION

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Sampling Guidelines

1. Consistent with agency guidance (see section 2.2.1). Issues such as sampling techniques, sample preservation and storage, holding times, chain-of-custody, etc.
2. Sampling locations statistically randomized for representativeness.
3. Samples composited prior to analysis for representativeness.
4. Debris, large rock fragments, vegetative material, etc., removed, unless they are not to be separated from the waste prior to treatment in the field.
5. "Hot spot" samples collected for worst-case analysis.

Waste Acceptance Criteria

1. Waste complies with transportation and facility (bench-scale treatability testing and/or analytical laboratory) permits as well as with health and safety plans.

Waste Characterization

1. Total waste analysis for target contaminants.
  2. TCLP and other appropriate leaching data on untreated waste for establishing baseline leaching data and determining the presence of RCRA toxicity characteristic.
  3. RCRA and other hazard characteristic tests as appropriate including the following:
    - ignitability
    - corrosivity
    - reactivity
    - toxicity
    - infectivity
    - (radioactivity)
  4. Other chemical analyses to establish baselines and possible S/S interferences, for example
    - pH
    - redox potential
    - salt content
    - oil and grease content
    - leaching tests
-

TABLE 2-6. GUIDANCE FOR COLLECTING BASELINE INFORMATION (Continued)

- 
5. Total contaminant analysis at the same time as a soluble (leachable) contaminant analysis, on the same subsample. This is to ensure that the subsample used to generate the soluble data does not contain a low contaminant level because of sample heterogeneity (avoid false negatives).
  6. Baseline physical characteristics of the untreated waste:
    - physical state
    - dustiness
    - paint filter test and/or liquid release test
    - bulk density
    - specific gravity
    - phase separation
    - permeability
    - moisture content
    - particle size
    - porosity
    - health hazards
  7. Other data on physicochemical form of the target contaminants — X-ray diffraction, scanning electron microscopy, optical microscopy, valence states of redox-sensitive contaminants such as As and Cr, organometallics (e.g., tetraethyl lead, butyl tin compounds), nickel-carbonyl, etc.
  8. Total waste volume measured or calculated.
  9. Presence and amount of debris that may interfere with S/S.
  10. Textural characteristics of the waste:
    - oily, liquid
    - clayey
    - dry granular
    - hard massive, etc.
    - sludge
  11. Heterogeneity of target contaminant distribution in the waste.
-

TABLE 2-6. GUIDANCE FOR COLLECTING BASELINE INFORMATION (Continued)

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QA/QC

1. Suitable QA/QC program, with built-in mechanisms to define data quality objectives, to evaluate sources of error, and to provide suitable documentation.
2. Analytical laboratories should possess appropriate qualifications or certifications.
3. Sufficient amount of analytical replication to permit a statistical analysis of the results (e.g., confidence intervals to address sample heterogeneity).
4. Use of a second analytical laboratory for interlaboratory verification on a portion of the more critical analytical measurements.

Baseline Site Characteristics

1. Fundamental site characterization data:
    - geology
    - hydrology, surface water and groundwater
    - geochemistry, soils
    - climatology, meteorology (especially temperature, wind, and rainfall)
  2. Knowledge of the proportion of waste that occurs above the groundwater table.
  3. Compatibility of site with heavy field equipment, for example
    - topography, slope, presence of obstacles
    - ability to excavate
    - available space
    - storage areas
    - characteristics consistent with any special requirements such as dikes, berms, and groundwater diversion or suppression systems
    - surface water drainage, etc.
-

TABLE 2-6. GUIDANCE FOR COLLECTING BASELINE INFORMATION (Continued)

- 
4. Proximity of site to necessary resources, for example
    - water
    - supplies
    - chemicals
    - electricity
    - equipment rentals
    - access routes
    - disposal facility
    - waste to be tested
  5. Proximity of site to possible receptors, for items such as
    - noise
    - fugitive dust
    - volatiles
    - odors
  6. Proximity of site to sensitive environmental areas, for example
    - floodplains
    - wetlands
    - protected species breeding grounds
    - populated areas
  7. Measurement of baseline contaminant levels in various media (air, water, soil, etc.) to determine if contaminants were released during the field demonstration.
  8. Availability of backfill, if necessary.
- 

the project depends to a large degree upon the ability to satisfy these objectives.

Performance objectives are a function of the compliance requirements selected for the site, the test methods used to evaluate the performance of the stabilized waste, and the analytical procedures (models) used to relate test data to performance objectives (Barich and Mason, 1992). The performance objectives are established early in the process of planning the treatability study. Specifying performance objectives goes hand-in-hand with selecting the tests to conduct because the objectives are expressed as results for specific tests. The performance objectives constitute acceptance criteria: if treatment by S/S cannot meet these criteria at the bench scale, S/S alone

probably cannot provide sufficient treatment to meet site cleanup goals. Once test methods and performance objectives are determined, the criteria to be used in interpreting test results can be derived readily (U.S. EPA, 1990b).

Before specific treatability performance objectives are set, the data quality needs of the project must be defined (Section 2.2.5). The early implementation of an appropriate QA/QC program and the establishment of DQOs will ensure that data of known and documented quality are generated. For a detailed discussion of DQOs, see U.S. EPA (1987a). Guidance on DQOs in the treatability study process can be obtained in U.S. EPA (1989e).

Treatability performance objectives can be grouped into two general types. Regulatory performance objectives (Section 2.3.1) are those based on applicable or relevant and appropriate requirements (ARARs) for the site. All other performance objectives may be classified as technical/institutional (Section 2.3.2). These relate to the characteristics of the S/S-treated waste for which explicit regulatory standards do not exist. Examples include cost effectiveness, a requirement for the S/S-treated waste to support vehicular traffic, and resistance of the treated waste to biodegradation. U.S. EPA, 1989e, Chapter 3, provides additional guidance.

### **2.3.1 Regulatory Requirements**

The regulatory requirements pertinent to treatability testing of S/S are those standards that the remedial alternative will have to meet when implemented at full scale. The regulatory framework for RCRA wastes is clearly defined in the regulations. The CERCLA regulatory framework is derived from site-specific ARARs about which general guidance is given below. An ARAR search needs to be conducted early on in the conduct of the feasibility study and well before the onset of the treatability testing. ARARs can be numerous, and a process has been established by the U.S. EPA to identify ARARs for Superfund projects (Section 121, Superfund Amendments and Reauthorization Act [SARA] of 1986, Public Law 99-499). The various ARARs often have different goals. Multiple goals make it increasingly expensive and increasingly difficult to comply with all the goals.

### 2.3.1.1 CERCLA

There are several types of ARARs under CERCLA: action-specific, chemical-specific, and location-specific. Action-specific ARARs are technology- or activity-specific requirements or limitations related to various activities. Chemical-specific ARARs are usually numerical values that establish the amount or concentration of a chemical that may be in or discharged to the ambient environment. Location-specific requirements are restrictions placed on the concentrations of hazardous substances or the conduct of activities solely because they occur in a special location.

Detailed guidance on the ARAR search is given in U.S. EPA (1988b). Some aspects of ARAR identification that apply to S/S treatability standards are discussed here.

Most federal laws that contain location-specific ARARs are institutional or administrative in nature. These laws regulate the types of activities that may take place in particular types of locations such as seismic fault zones, floodplains, or critical habitats for endangered species. State and local regulations are more likely to provide location-specific ARARs for treatability testing. Pertinent regulations would include discharge limits or nondegradation standards for particular water bodies and basin-wide air quality standards (U.S. EPA, 1990b, Chapter 3).

Relevant technology (action)-specific ARARs must be identified. At present, there are few explicit performance standards for S/S-treated wastes. The U.S. Nuclear Regulatory Commission (NRC) has established performance standards for stabilized nuclear wastes (both high-level and low-level), but these are not applicable to nonnuclear materials. Hazardous wastes that are disposed of on land may be regulated under RCRA, and standards for treatment of such wastes are currently being promulgated. Wastes that are stabilized by in situ techniques, such as deep mixing, may not fall under the purview of RCRA rules. Wastes that are excavated, treated, and land-disposed of either on or off the site (i.e., they undergo "placement") may be regulated by RCRA rules. Land-disposed RCRA wastes usually need to demonstrate a minimum unconfined compressive strength of 50 psi (U.S. EPA, 1986b), but the actual target value for a specific site may be higher or lower depending on site-specific requirements. In addition, technology- and action-specific treatment standards for a number of RCRA waste classes are named in the RCRA land disposal restrictions (LDRs).

For many waste classes, including inorganics and some organic contaminants, treatment standards are expressed as percent reduction in contaminant leaching, as measured by pre- and post-treatment TCLP tests. Note, however, that there has been a tendency in RODs to express treatment standards, even for metal contaminants, in terms of reduction of total contaminant levels. This poses complications for the application of S/S technology because, under normal circumstances, S/S neither destroys nor removes the contaminant, but instead immobilizes it. These standards are directly applicable to laboratory screening and bench-scale testing of these waste classes; they can be used to gauge the efficiency of S/S treatment during treatability studies. For many organic contaminants, RCRA treatment standards are expressed as destruction-removal efficiency (DRE), where the efficiency of the treatment technology is measured by pre- and post-treatment total (as opposed to soluble) contaminant concentrations. (U.S. EPA, 1990b, Chapter 3). Results of TCLP tests on post-treatment samples may be influenced by dilution of waste due to binder addition. Reduced contaminant concentration in leachate may not reflect reduced mobility of the contaminant unless results have been corrected for dilution effects.

At many CERCLA sites, the materials requiring treatment cannot be assigned to specific RCRA waste classes. Contaminated soil and debris are often the materials of concern. For such sites, an appropriate regulatory performance standard can be derived by the procedure used to establish a treatability variance under RCRA (U.S. EPA, 1989b). The U.S. EPA has set target cleanup ranges for wastes contaminated by the principal classes of organic and inorganic contaminants (Table 2-7). For an organic contaminant, the appropriate treatability performance objective is determined as follows: If the total concentration for the contaminant in the untreated waste falls below the "threshold concentration," then the total concentration of the contaminant in the S/S-treated waste must fall within the "concentration range." If the original total concentration of the contaminant exceeds the threshold value, then the difference between the total concentrations of the contaminant in the treated and untreated wastes must fall within the "percent reduction range." The relevance of these guidelines when treatment is by S/S is unclear, however, because S/S neither destroys nor removes the contaminant, but instead immobilizes it. The same logic applies for metallic contaminants, but the criteria are based on the contaminant concentration in the TCLP



**TABLE 2-7. ALTERNATIVE TREATMENT LEVELS FOR SOIL AND DEBRIS  
CONTAMINATED WITH RESTRICTED RCRA HAZARDOUS WASTES<sup>a</sup>**

Structural Functional Groups	Concentration Range (ppm)	Threshold Concentration (ppm)	Percent Reduction Range	Technologies that Achieved Recommended Effluent Concentration Guidance <sup>b</sup>
Organics	Total Waste Analysis <sup>c</sup>	Total Waste Analysis <sup>c</sup>		
Halogenated nonpolar aromatics	0.5-10	100	90-99.0	Biological treatment, low-temperature stripping, soil washing, thermal destruction
Dioxins	0.00001-0.05	0.5	90-99.9	Dechlorination, soil washing, thermal destruction
PCBs	0.1-10	100	90-99.9	Biological treatment, dechlorination, soil washing, thermal destruction
Herbicides	0.002-0.02	0.2	90-99.9	Thermal destruction
Halogenated phenols	0.5-40	400	90-99	Biological treatment, low-temperature stripping, soil washing, thermal destruction
Halogenated aliphatics	0.5-2	40	95-99.9	Biological treatment, low-temperature stripping, soil washing, thermal destruction
Halogenated cyclics	0.5-20	200	90-99.9	Thermal destruction
Nitrated aromatics	2.5-10.0	10,000	99-99.99	Biological treatment, soil washing, thermal destruction

TABLE 2-7. ALTERNATIVE TREATMENT LEVELS FOR SOIL AND DEBRIS  
CONTAMINATED WITH RESTRICTED RCRA HAZARDOUS WASTES<sup>a</sup> (Continued)

Structural Functional Groups	Concentration Range (ppm)	Threshold Concentration (ppm)	Percent Reduction Range	Technologies that Achieved Recommended Effluent Concentration Guidance <sup>b</sup>
Organics	Total Waste Analysis <sup>c</sup>	Total Waste Analysis <sup>c</sup>		
Heterocyclics	0.5-20	200	90-99.9	Biological treatment, low-temperature stripping, soil washing, thermal destruction
Polynuclear aromatics	0.5-20	400	95-99.9	Biological treatment, low-temperature stripping, soil washing, thermal destruction
Other polar organics	0.5-10	100	90-99.9	Biological treatment, low-temperature stripping, soil washing, thermal destruction
Inorganics	TCLP	TCLP		
Antimony	0.1-0.2	2	90-99	Immobilization
Arsenic	0.27-1	10	90-99.9	Immobilization, soil washing
Barium	0.1-40	400	90-99	Immobilization
Chromium	0.5-6	120	95-99.9	Immobilization, soil washing
Nickel	0.5-1	20	95-99.9	Immobilization, soil washing
Selenium	0.005	0.08	90-99	Immobilization
Vanadium	0.2-22	200	90-99	Immobilization

TABLE 2-7. ALTERNATIVE TREATMENT LEVELS FOR SOIL AND DEBRIS  
CONTAMINATED WITH RESTRICTED RCRA HAZARDOUS WASTES<sup>a</sup> (Continued)

Structural Functional Groups	Concentration Range (ppm)	Threshold Concentration (ppm)	Percent Reduction Range	Technologies that Achieved Recommended Effluent Concentration Guidance <sup>b</sup>
Inorganics	TCLP	TCLP		
Cadmium	0.2-2	40	95-99.9	Immobilization, soil washing
Lead	0.1-3	300	99-99.9	Immobilization, soil washing
Mercury	0.0002-0.008	0.06	90-99	Immobilization

<sup>a</sup> Source: U.S. EPA 1990c (Superfund LDR Guide No. 6A-2nd Edition).

<sup>b</sup> Other technologies may be used if treatability studies or other information indicates that they can achieve the necessary concentration or percent-reduction range.

<sup>c</sup> TCLP also may be used when evaluating waste with relatively low levels of organics that have been treated through an immobilization process.

leachate, rather than the total contaminant concentration (U.S. EPA, 1990b, Chapter 3).

Finally, an ARAR search may identify chemical-specific ARARs that should be evaluated during treatability testing. Numerical standards that may be ARARs have been promulgated under several federal laws. These include the Clean Water Act (water quality criteria for protection of human health and ambient water quality criteria), the Safe Drinking Water Act (maximum contaminant levels [MCLs] and MCL goals), and RCRA. If the S/S-treated wastes may be disposed of off-site, then the TCLP test and the RCRA characteristic tests should be specified; their acceptance criteria will constitute one set of performance objectives. Chemical-specific air quality standards may also apply and the ARARs cannot be exceeded. Because materials processing and the potential for volatilization are much different between bench- and full-scale, air quality standards are unlikely to form the basis for quantitative bench-scale tests (U.S. EPA, 1990b, Chapter 3).

For most S/S projects, resource limitations dictate that the treatability testing program be restricted to a subset of the contaminants present on the site. The contaminants to be evaluated should be selected according to the following characteristics (U.S. EPA, 1990b, Chapter 3):

- Toxicity or carcinogenicity - select the most harmful contaminants.
- Mobility - select the most soluble contaminants.
- Geochemistry - select a representative contaminant from each of the major functional types present.
- Concentration - all factors being equal, select the contaminants present at the highest concentrations.

Generally, if the number of contaminants being evaluated in treatability testing exceeds four or five at any one time, it becomes increasingly difficult to satisfy the performance objectives for all of the contaminants. If the ROD has been signed and site cleanup goals have been specified, the contaminants named therein should be monitored throughout the treatability study. Examples of regulatory performance objectives for CERCLA S/S studies are summarized in Table 2-8.

**TABLE 2-8. EXAMPLES OF REGULATORY ARARs**

- 
1. Total contaminant treatment standards for disposal
  2. Soluble contaminant treatment standards for disposal
    - a. routine leaching procedure (e.g., TCLP)/
    - b. other leaching procedure (e.g., ANSI/ANS/16.1)
  3. Mobility criteria from geochemical transport model
  4. Land activity restrictions (e.g., in seismic fault zones, flood-plains, critical habitats of endangered species)
  5. "Placement" restrictions (e.g., 50 psi unconfined compressive strength criterion)
  6. Air emissions standards
  7. Noise restrictions
  8. Compliance with the Clean Water Act or Safe Drinking Water Act
  9. Compliance with state and local regulations and laws.
- 

#### **2.3.1.2 RCRA**

The factors for accepting stabilized waste at a treatment, storage, and disposal (TSD) facility under RCRA are much less complex than for CERCLA. The principal criteria (U.S. EPA, 1989b) are as follows:

- Paint Filter Test (PFT) for free liquid
- Adherence to TCLP maximum concentration limits (see Table 2-9)
- Screens for hazardous waste characteristics
  - ignitability
  - corrosivity
  - reactivity
  - radioactivity
- Compliance with LDRs (see Section 1.3 for a discussion of the nature and applicability of RCRA Land Disposal Restrictions).

**TABLE 2-9. TOXICITY CHARACTERISTIC CONSTITUENTS  
AND REGULATORY LEVELS**

EPA HW No. <sup>a</sup>	Constituent (mg/L)	Regulatory Level
D004	Arsenic	5.0
D005	Barium	100.0
D018	Benzene	0.5
D006	Cadmium	1.0
D019	Carbon tetrachloride	0.5
D020	Chlordane	0.03
D021	Chlorobenzene	100.0
D022	Chloroform	6.0
D007	Chromium	5.0
D023	o-Cresol	200.0 <sup>b</sup>
D024	m-Cresol	200.0 <sup>b</sup>
D025	p-Cresol	200.0 <sup>b</sup>
D026	Cresol	200.0 <sup>b</sup>
D016	2,4-D	10.0
D027	1,4-Dichlorobenzene	7.5
D028	1,2-Dichloroethane	0.5
D029	1,1-Dichloroethylene	0.7
D030	2,4-Dinitrotoluene	0.13 <sup>c</sup>
D012	Endrin	0.02
D031	Heptachlor (and its hydroxide)	0.008
D032	Hexachlorobenzene	0.13 <sup>c</sup>
D033	Hexachloro-1,3-butadiene	0.5
D034	Hexachloroethane	3.0
D008	Lead	5.0
D013	Lindane	0.4

**TABLE 2-9. TOXICITY CHARACTERISTIC CONSTITUENTS  
AND REGULATORY LEVELS (Continued)**

EPA HW No. <sup>a</sup>	Constituent (mg/L)	Regulatory Level
D009	Mercury	0.2
D014	Methoxychlor	10.0
D035	Methyl ethyl ketone	200.0
D036	Nitrobenzene	2.0
D037	Pentachlorophenol	100.0
D038	Pyridine	5.0 <sup>c</sup>
D010	Selenium	1.0
D011	Silver	5.0
D039	Tetrachloroethylene	0.7
D015	Toxaphene	0.5
D040	Trichloroethylene	0.5
D041	2,4,5-Trichlorophenol	400.0
D042	2,4,6-Trichlorophenol	2.0
D017	2,4,5-TP (Silvex)	1.0
D043	Vinyl chloride	0.2

<sup>a</sup> Hazardous waste number

<sup>b</sup> If o-, m-, and p-cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level for total cresol is 200 mg/L.

<sup>c</sup> Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

### **2.3.2 Technical and Institutional Requirements**

In addition to regulatory requirements, other factors may shape the treatability performance objectives. Technical/institutional objectives are developed from constraints imposed by administrative factors, by the site itself, or by the waste to be treated. These objectives address special problems that may detract from the implementability of the S/S process or from the long-term performance of S/S-treated waste at the site. For successful remediation, developing such objectives and solving these problems may be as important as meeting applicable regulatory requirements.

Table 2-10 lists potential types of performance objectives that fall outside of the regulatory domain. For some objectives, such as cost-effectiveness and controlling the production of hazardous vapors, quantitative acceptance criteria may not exist. For many nonregulatory tests, quantitative performance standards for particular site conditions can be developed.

Some of the performance objectives listed in Table 2-10 for S/S-treated wastes have been studied in depth. Tests for these properties are widely performed and have been applied successfully in evaluating S/S-treated wastes. Examples of such properties are waste volume increase, sulfate or sulfide content, and leachability, as measured by various tests (see Chapter 3). The importance of other properties in maintaining the integrity of S/S-treated wastes is not well understood. The corresponding tests may be considered research tests and their results subject to various interpretations (U.S. EPA, 1990b, Chapter 3).

### **2.3.3. Approach for Setting Performance Criteria**

The laboratory tests to be performed and performance criteria for these tests to meet are chosen at the same time. One should not begin the testing program without a clear definition of what results will constitute success and failure. The available physical, leaching, chemical, biological, and microcharacterization tests and their typical applications are discussed in Chapter 3.

Every bench-scale treatability study should consider tests of leaching, unconfined compressive strength, and free liquids. These tests are likely to form at least a portion of the basis for any regulatory evaluation of the S/S-treated waste.



TABLE 2-10. EXAMPLES OF TREATABILITY PERFORMANCE OBJECTIVES  
BASED ON NONREGULATORY FACTORS<sup>a</sup>

Objective	Potential generic test(s)
<u>Qualitative Objectives</u>	
Demonstrate basic feasibility	Leaching test One or more physical tests
Reagent costs not to exceed a given amount	Optimize mix; calculate binder cost per volume stabilized
Assay for off-gassing of volatile compounds	Measure temperature of fresh mixture Monitor air with organic vapor detector while mixing
Ensure thorough mixing	Microscopy; visual examination of fractured monoliths Observation of binder miscibility, wetting during mixing
Identify soil containing interfering minerals	X-ray diffraction
Treat a minimum proportion of contaminated material on site	Assay site for debris and large particles; determine handling needs
<u>Quantitative Objectives</u>	
Prevent unfavorable reactions between waste and binder	Potential reactivity of aggregates Petrographic examination of aggregates for concrete
Create a pumpable mix	Liquid waste consistency/ classification (see Table 3-3)
Ensure complete microencapsulation of contaminants	Collect, analyze any bleed water
Volume increase not to exceed a threshold value	Calculate volume change from treated, untreated waste bulk densities

**TABLE 2-10. EXAMPLES OF TREATABILITY PERFORMANCE OBJECTIVES  
BASED ON NONREGULATORY FACTORS (Continued)**

Objective	Potential generic test(s)
Ensure sufficient long-term structural integrity	Compressive strength Resistance to wet/dry and freeze/thaw stressing
Determine ability of S/S-treated waste to support heavy equipment soon after placement	Trace development of bearing capacity with cone penetrometer
Determine ability of cured S/S monolith to support vehicular traffic	Flexural strength test California bearing ratio Proctor compaction of subbase (and stabilized material, if it is a friable soil-cement)
Assure resistance to sulfate attack on S/S monolith	SO <sub>4</sub> content of waste
Prevent fracturing of S/S monolith	Shrink/swell potential of subbase material
Curtail fugitive dust emissions during full-scale fixation	Particle size analysis Moisture content of wastes
Minimize contaminant leaching	Leaching tests
Determine long-term leach behavior	Acid neutralization capacity Resistance to redox change Chemistry of surrounding soil and groundwater Accelerated aging/weathering tests
Minimize leachate toxicity	Aquatic bioassays
Resist biodegradation of organic contaminants or asphaltic binders	Biodegradation tests
Reduce contaminant load or concentration at the receptor to below threshold value	Leaching tests Permeability Transport modeling

<sup>a</sup> U.S. EPA, 1990b

The currently accepted version of the TCLP leaching test is usually required. However, depending on the anticipated disposal setting and environmental or human health risks, TCLP may not be adequate and additional leaching tests may be needed. The types of contaminants and their level of hazard and concentration, the planned disposal or reuse scenario, and the S/S approach used all influence the selection of leaching tests. Additional leaching tests are particularly important if there is a need to characterize the fundamental mechanisms involved (e.g., for risk analysis to receptor populations).

Beyond these basic regulatory requirements, further testing is initially projected on the basis of site hydrologic conditions. If an aqueous driving force for leachate production exists, additional leach testing should be considered. In addition, it may be necessary to evaluate contaminant transport using a modeling approach. Additional background and guidance on this issue is provided in U.S. EPA (1989e, Section 3.3). An aqueous driving force may not exist. For example, the final remedial design may specify that the S/S-treated waste be placed above the seasonal high-water table and an impervious cap and runoff controls be constructed. In such cases, leaching and physical integrity tests will usually suffice to demonstrate whether the S/S process can be considered reliable for the site. Attenuation by engineering controls or natural processes is not usually considered in this case (U.S. EPA, 1989e, Chapter 3).

Strength and freedom from free liquids are two other frequently applied performance criteria. Other types of measurements should be planned based on site-specific factors such as those listed in Tables 2-8 and 2-10. Selection of testing depends on waste characteristics, disposal or reuse scenario, type of S/S progress, and scientific objectives of the program.

## **2.4 INITIAL TECHNOLOGY SCREENING**

After the performance objectives for a treatability project have been identified, it is necessary to determine what treatment technology or technologies have the potential of complying with those performance objectives. This section briefly discusses the screening process whereby S/S is compared with other treatment alternatives and the most appropriate technology or technologies are selected for further evaluation to determine compliance with the performance objectives. Various terms have been applied to this technology screening process, including "feasibility study" (FS) for remedial

actions and "economic evaluation/cost analysis" (EE/CA) for removal actions. The screening process is described in other publications, such as U.S. EPA (1988c), and it is beyond the scope of this TRD to describe the process in detail. Therefore, an overview of the basic elements of the process is given in Section 2.4.1 and rules of thumb for screening problematic waste types for S/S technology are provided in Section 2.4.2.

#### **2.4.1 Technology Screening/Feasibility Study Process**

In the broadest sense, the majority of wastes are potentially treatable with S/S. Pretreatment can be used to transform an untreatable waste form to a form that can be treated with S/S. However, for certain wastes, the pretreatment requirements may make the technology impractical based on cost or other criteria, and there clearly are situations where a different type of technology will be more effective or appropriate.

##### **2.4.1.1 CERCLA Technology Screening**

The first step in the technology screening process is to identify candidate remedial alternatives (treatment/removal technologies or treatment trains). A number of different technologies have been developed. Many technologies are applicable only to certain types of wastes. For example, U.S. EPA (1988c) lists the following broad categories of treatment technologies:

- Fluidized bed incineration
- Rotary kiln incineration
- Infrared thermal treatment
- Wet air oxidation
- Pyrolysis-incineration
- Vitrification (in situ, ex situ)
- Chemical extraction
- Glycolate dechlorination
- Solidification/stabilization

- Chemical reduction/oxidation
- Biodegradation
- In situ biodegradation

Treatment technologies continually are being developed, modified and refined. In selecting a remedial alternative (which includes selecting the treatment technologies), an analysis is performed with respect to a number of different evaluation criteria. The process described in the National Contingency Plan entails a detailed analysis of each remedial alternative with respect to nine different evaluation criteria in three main categories. These criteria are presented below. All selected remedies should provide the best trade-offs among the Primary Balancing Criteria and must, at a minimum, attain the Threshold Criteria. The Modifying Criteria are evaluated following the public comment period.

#### Threshold Criteria:

- Overall Protectiveness of Human Health and the Environment. This criterion evaluates the adequacy of protection that the remedy provides while describing how risks are eliminated, reduced, or controlled through treatment, engineering controls, and/or institutional controls.
- Compliance with Applicable or Relevant and Appropriate Requirements. This criterion addresses whether a remedy would meet all of the ARARs of federal and state environmental statutes and/or provide grounds for invoking a waiver.

#### Primary Balancing Criteria:

- Reduction of Toxicity, Mobility, or Volume Through Treatment. This criterion addresses the anticipated treatment performance of the remedy.
- Short-Term Effectiveness. This criterion refers to the speed with which the remedy achieves protection, as well as the remedy's potential to create adverse impacts on human health and the environment during the remedial action.
- Long-Term Effectiveness and Permanence. This criterion evaluates the magnitude of residual risk and

the ability of the remedy to maintain reliable protection of human health and the environment over time once the remedial action has been completed.

- Implementability. This criterion examines the technical and administrative feasibility of executing a remedy, including the availability of materials and services needed to implement the chosen solution.
- Cost. This criterion includes the capital and operation and maintenance costs of the remedy.

#### Modifying Criteria:

- State Acceptance. This criterion indicates whether, based on its review of the planned remedial alternative, the state concurs with, opposes, or has no comment on the preferred alternative.
- Community Acceptance. This criterion evaluates the reaction of the public to the remedial alternatives and to the U.S. EPA's Proposed Plan.

A similar approach is employed using EPA's Engineering Evaluation/Cost Analysis (EE/CA) for selecting CERCLA removal actions/approaches. In this case, the technology screening takes place in two stages, as shown in Figure 2-3. First, all alternative remedial actions are compared based on timeliness and effectiveness to protect human health and the environment. Then, the smaller subset of remedial actions that satisfy these criteria are evaluated based on (a) technical feasibility, (b) cost, and (c) administrative and managerial feasibility. The process may be iterative and may have several different possible outcomes, which are discussed further in Section 2.4.3.

Another factor considered during technology screening is the position of the remedial action/technology in terms of the hierarchy of hazardous waste management (Section 1.1.2). Remedial actions that allow recycling, reuse, or recovery of the waste or some portion of the waste are preferable to treatment and disposal. For example, all other factors being equal, smelting or soil washing would be preferable to S/S for wastes containing appropriately high metal contents because some contaminants would be recovered and could then be recycled. However, it is important to consider the full system effects when making the comparison. One example is the need for pretreatment and the residuals generated, such as liquid waste produced during soil washing.

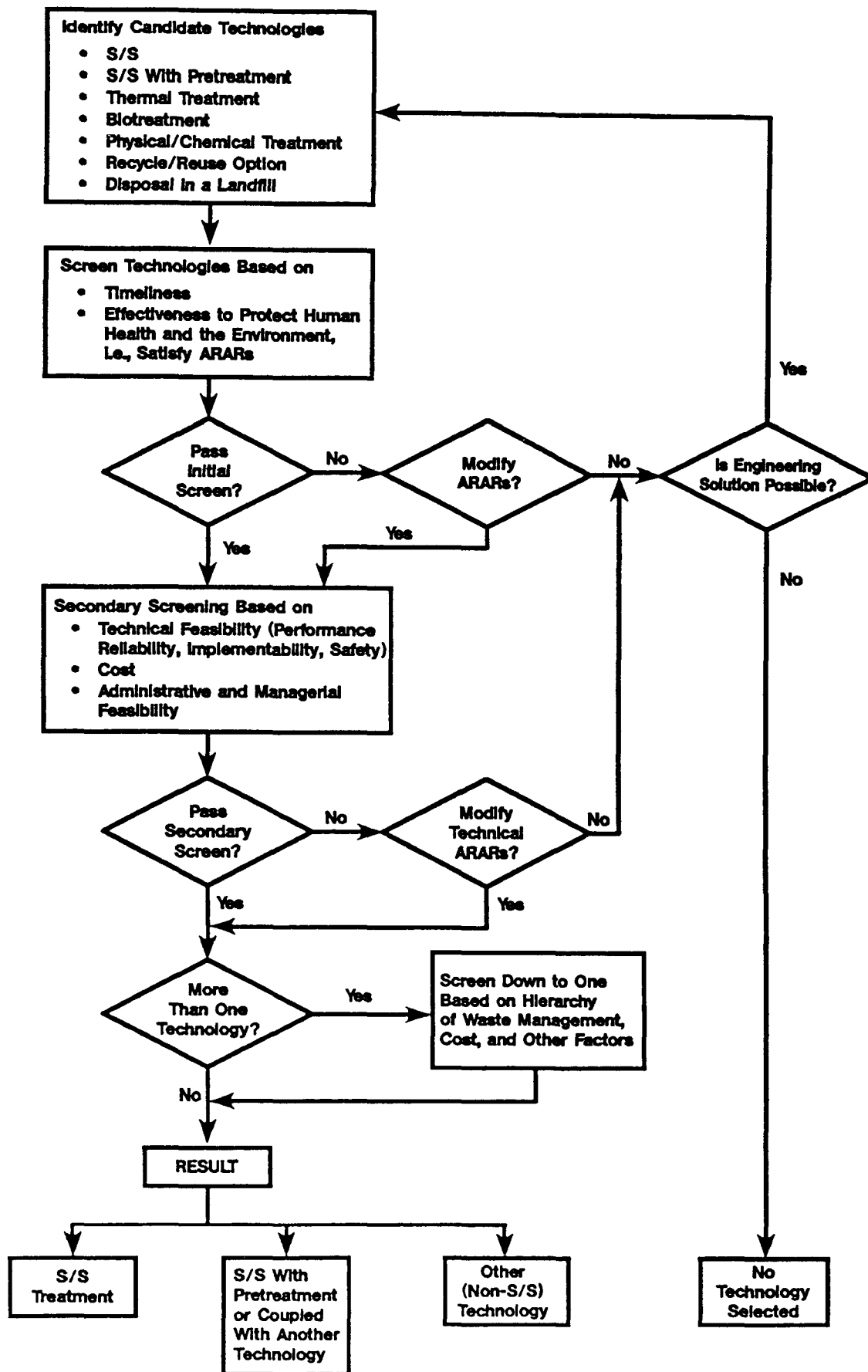


FIGURE 2-3. GENERAL TECHNOLOGY SCREENING PROCEDURE

#### **2.4.1.2 Technology Screening at RCRA TSD Facilities**

Remediation under CERCLA and RCRA corrective action are driven primarily by regulations, waste characteristics, and site characteristics and have the full range of available treatment technologies as options. In contrast, RCRA treatment, storage and disposal (TSD) facilities are driven by the regulations and by the specific permitted technologies available at the facility. Any one treatment facility probably has one or more specific immobilization technologies in place with a limited menu of pretreatment options available (U.S. EPA, 1989b).

The RCRA TSD facility personnel need to select, screen, and test treatment technologies to process waste streams while complying with permit conditions. Some flexibility may be gained by using pretreatment options or process modifications.

The criterion for satisfactory treatability is the ability of the treated waste to pass all the required tests for disposal. A flowchart for determining the potential suitability of S/S for waste treatment at a RCRA TSD facility is provided in Figure 2-4. Once the waste has been found to be potentially suitable for S/S, the approach for bench-scale screening is as outlined in Figure 2-5. This approach is basically a simplified version of the tiered approach for S/S treatability testing described in Sections 2.5 through 2.7.

#### **2.4.2 General Criteria for Not Using S/S**

Because the applicability of S/S processes to site-specific wastes depends on several variables, specifying criteria for not using S/S is difficult or impossible without site-specific data. However, it is possible to generalize about criteria that indicate potential S/S inapplicability. Table 2-11 summarizes the types of waste that are generally not amenable to S/S processes or that could pose problems.

#### **2.4.3 Outcome of Technology Screening**

The outcome of the technology screening process is a determination of one of the following (Figure 2-3):

- Waste can be treated with S/S without pretreatment.



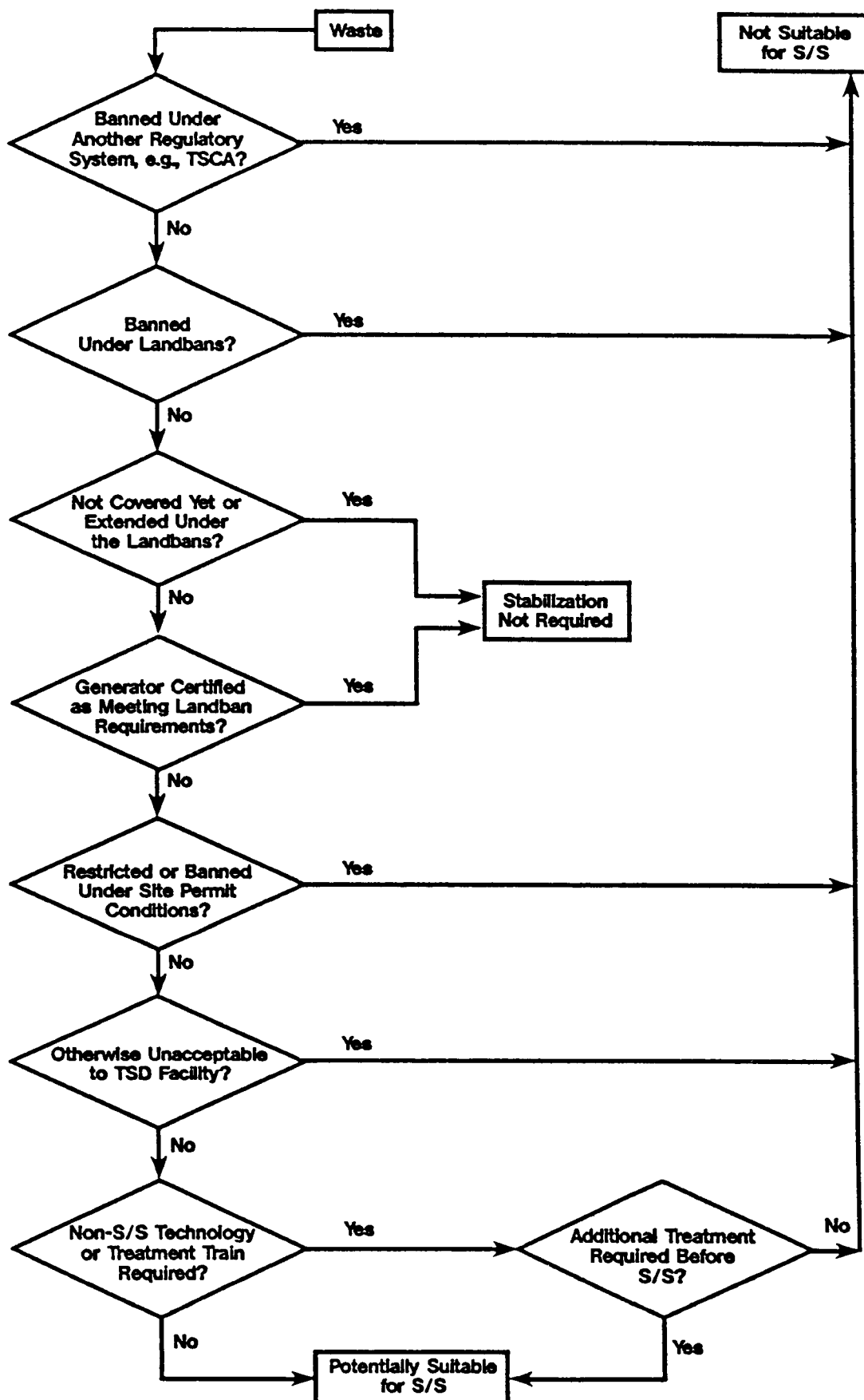


FIGURE 2-4. DETERMINING WHETHER S/S IS APPLICABLE AT A RCRA TSD FACILITY  
(from U.S. EPA, 1989b)

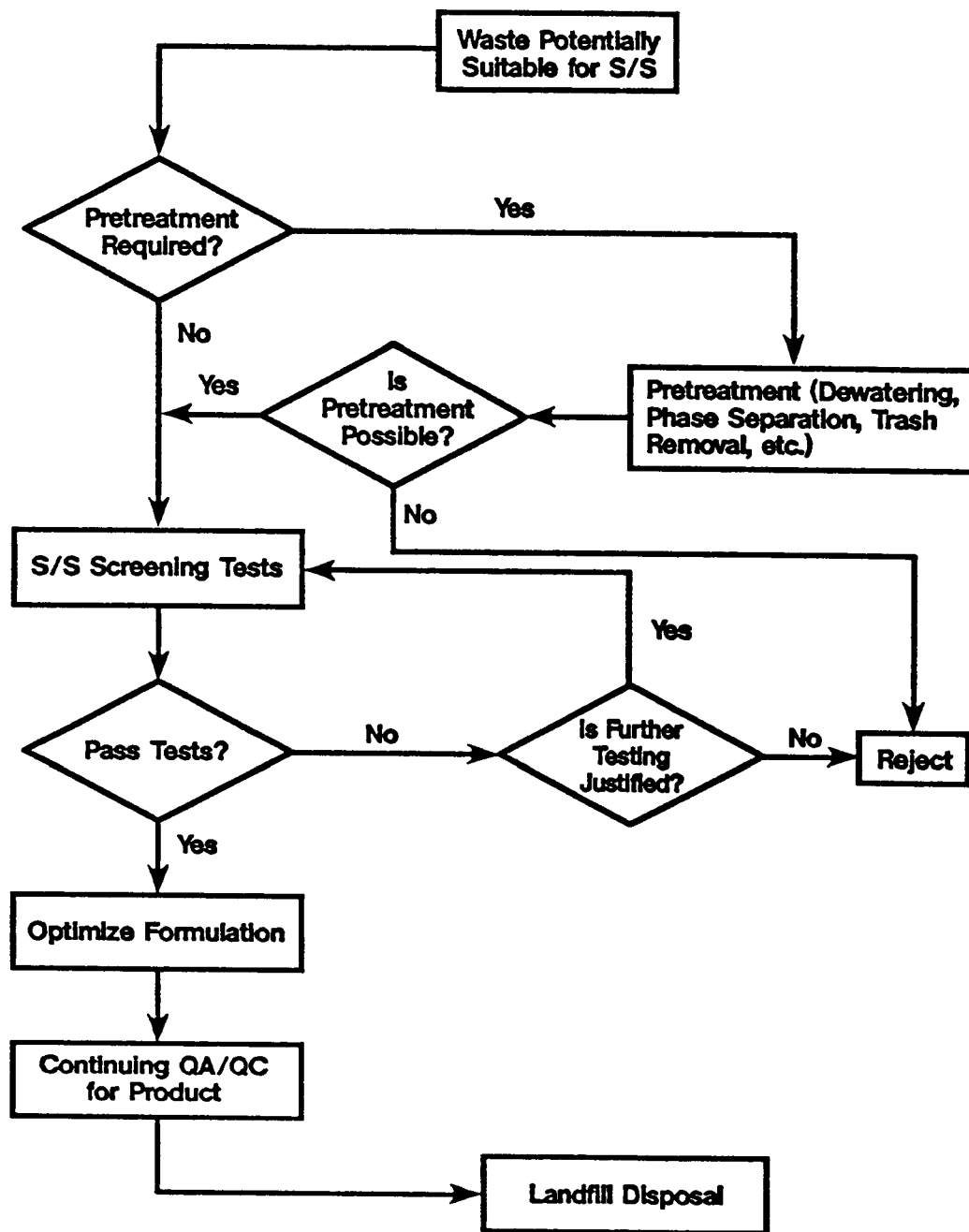


FIGURE 2-5. S/S DECISION TREE AT A RCRA TSD FACILITY (from U.S. EPA, 1989b)

TABLE 2-11. APPLICABILITY OF SOLIDIFICATION/STABILIZATION  
TO SITE-SPECIFIC WASTE

---

A. WASTES THAT ARE UNSUITABLE FOR S/S<sup>a</sup>

1. Wastes that are readily treatable by recycling, reuse, or recovery technology, all other factors being equal.
2. Wastes that are treatable using a destructive technology, all other factors being equal.
3. Wastes that contain land-banned constituents (unless land disposal can be avoided) and other high-hazard materials (e.g., dioxins, high levels of PCBs, pesticides, etc.).
4. Waste for which the ARARs cannot be satisfied with existing S/S technology (unless ARARs are modified).
5. Wastes that have unacceptable physical characteristics such as being too solid or viscous to mix or handle.
6. Wastes where waste volume expansion would exceed reuse space constraints.
7. Wastes that are treatable using a much less expensive technology, all other factors being equal.

B. WASTES THAT POSE COMPLICATIONS FOR S/S

1. Wastes with volatile organics (pretreatment is usually required).
2. Wastes that contain a large number of different types of contaminants.
3. Wastes that are situated such that field S/S will be difficult or expose local receptors to unacceptable risk.
4. Wastes with large amounts of interfering/incompatible constituents (pretreatment necessary).
5. Wastes that contain organics as the primary contaminants.

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<sup>a</sup> S/S is not recommended for these wastes unless no other option exists.

- Waste can be treated with S/S with pretreatment or coupled with a different technology (treatment train).
- Waste can be treated with some type of technology other than S/S.
- No treatment technology is currently available for the waste.

Wastes that can be treated "as is" with S/S are those whose target contaminants are expected to respond favorably to S/S using at least one known binding agent. Such wastes should not have properties that would interfere with the S/S process.

Wastes requiring pretreatment include materials that are hazardous by virtue of ignitability, corrosivity, reactivity, infectiousness, presence of radionuclides, or some other property that would normally preclude secure land burial. Such wastes cannot be solidified or stabilized and disposed of in landfills without adequate pretreatment. Wastes that present specific problems, such as excessive escape of volatile organics of concern during treatment, may also fall into this category and require either pretreatment to reduce the volatiles or the use of additives in the S/S treatment formulation to inhibit emissions during S/S processing. Another example of a reason to pretreat a waste prior to S/S would be to remove an interference with the S/S technology. Conversely, S/S may itself also be the pretreatment step, for example, to improve material handling characteristics prior to treatment by a different technology.

Finally, wastes for which S/S is currently not a practical option include highly hazardous materials (because S/S does not convert metals or break organics down into basic chemicals), wastes containing excessive interferants that will not respond to treatment, and mixed wastes with complex chemistries that require several pretreatment steps prior to S/S. Such wastes become too expensive to process when compared with the cost for transportation and secure land burial in a RCRA-permitted facility. In many cases, these types of wastes will be treatable using a different type of technology. In rare cases, the waste will simply be untreatable. If allowed within regulations such as landban, the waste may be disposed of. If the disposal option is foreclosed, additional research will be needed.

## **2.5 WASTE/BINDER COMPATIBILITY LITERATURE SCREENING**

After it has been determined that S/S is a potentially applicable technology for a specific waste (Section 2.4), surveying the technical literature to identify applicable binder types is a good starting point for the treatability study. The literature screening should result in the selection of two to four candidate binders for further evaluation at the laboratory bench-scale screening tier (Section 2.6).

The literature-screening step basically conforms to the remedy screening step, as outlined in U.S. EPA (1989e). Technical information resources, including information from reports, guidance documents, vendor information, and electronic databases, are useful reference materials. Any available performance and cost information should also be obtained for all binders being considered. A flowchart illustrating the waste/binder compatibility literature screening processes, as well as the organization of this section, is presented in Figure 2-6. The objective of this screening step is to identify, as simply and as inexpensively as possible, those binder types most suited for the site-specific waste and its contaminants and for the related waste disposal scenario.

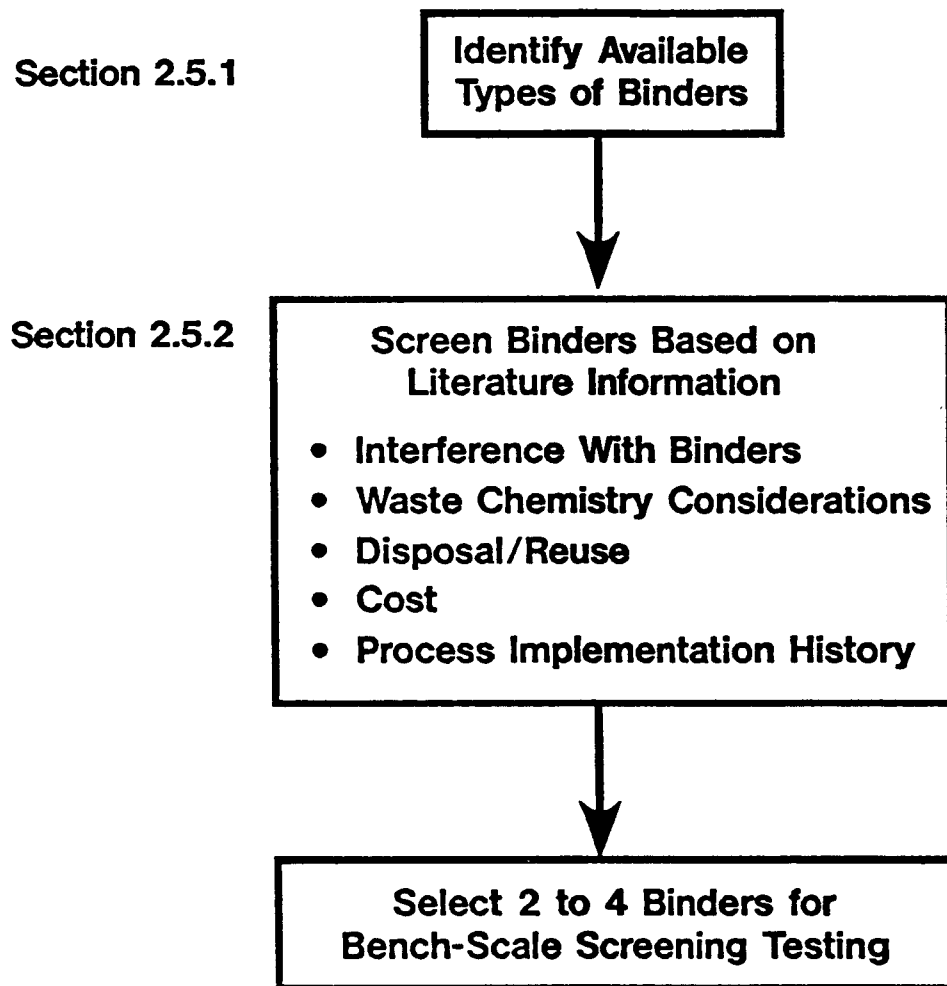
### **2.5.1 Identify Available Binders**

The selection of two to four binders for further evaluation is not a requirement, but is recommended because it improves the probability of a successful treatability study and requires minimal additional time and cost. This literature screening step is also intended to minimize potentially expensive trial-and-error bench-scale testing in the laboratory.

If a single binder or binder system has been preselected for bench-scale testing, then the literature screening step is no longer relevant. However, if that binder system proves to be ineffective in bench-scale screening (Section 2.6), then it will be necessary to select and test additional binder systems before it can be concluded that S/S is an inappropriate treatment technology.

### **2.5.2 Screening Process**

The principal criteria for waste/binder compatibility literature screening are to determine (a) interferences and chemical incompatibilities,



**FIGURE 2-6. WASTE/BINDER COMPATIBILITY LITERATURE SCREENING**

(b) metal chemistry considerations, (c) compatibility with the disposal or reuse environment, (d) cost, and (e) process track record. Ideally, an expert system should be developed to provide systematic evaluation of these issues. However, a fully usable system for complex waste forms has yet to be developed. The literature screening criteria are summarized below.

#### **2.5.2.1 Interferences and Chemical Incompatibilities**

Proper S/S treatment using pozzolanic binders may be inhibited in the presence of certain chemical constituents, such as high concentrations of oil, grease, and other organics, as well as chlorides and other soluble salts. Certain S/S processes will not function properly if the chemical environment is not adequately controlled. For example, sodium sulfide is incompatible with acids, which not only impair S/S but also may result in the release of toxic hydrogen sulfide gas. These and other types of waste/binder incompatibilities are summarized in Section 4.3 and in references cited therein. Material Safety Data Sheets (MSDSs) for individual waste components provide another potentially useful source of data on interferences and chemical incompatibilities.

#### **2.5.2.2 Metal Chemistry Considerations**

Metal chemistry is complex and has not been examined in any systematic manner as it pertains to S/S treatment and the chemical mechanisms of immobilization of contaminated soils. Section 4.2.2 and U.S. EPA (1990b, Appendix D) summarize some of the relevant chemical reactions. When alkaline binding agents such as cement, pozzolanic binders, or soluble silicates are used, the formation of metal hydroxides, oxides, and possibly silicates will be an important S/S mechanism. When sodium sulfide is used, extremely insoluble sulfide salts may form with numerous metals. In addition, metal carbonates, phosphates, and sulfates occasionally can be important in some systems.

Numerous chemical complexities exist. Chemical conditions, such as high pH (see Section 4.1.1) that are favorable for the immobilization of certain metals (e.g., Ni and Zn) actually may be detrimental to others. For example, As and Cr form soluble anionic species at high pH. Also, the solubility of many metal hydroxides is affected by their amphoteric behavior

(solubility increases at both high and low pH). The minimum solubility for one metal may be several pH units different from the minimum solubility for another. Geochemical equilibrium modeling may be necessary to resolve issues related to complex waste chemistries.

#### **2.5.2.3 Organic Chemistry Considerations for Target Contaminants**

If organic contaminants are present, the binder selection must also be based upon compatibility with the organic contaminants. Section 4.2.2.2 discusses some of the types of binders and additives that are used frequently for immobilizing organic contaminants. These include such materials as activated carbon and modified clays. In general, generic binders such as Portland cement do a poor job of immobilizing organics, with the exception of highly polar compounds in low-to-moderate concentrations.

When evaluating the feasibility of applying S/S technology to wastes containing significant concentrations of organic contaminants, there are a number of issues that should be examined, as discussed in detail in Section 4.4. First and foremost is whether a destruction or extraction technology is available and applicable to the waste. All other factors being approximately equal, destruction or extraction technologies are preferred to S/S because they eliminate or remove the contaminant as opposed to just immobilizing it. Other issues that should be considered before concluding that S/S is the preferred approach for wastes containing organic contaminants are: (a) the volatility of the organics and whether air emissions may occur during excavation, mixing, and/or curing; (b) the solubility of the organics in water and the meaningfulness of conducting aqueous leach tests as a measure of the degree of immobilization of the organics by S/S treatment; and (c) whether the organic contaminants may degrade or transform to other by-products during S/S treatment and the toxicity of those by-products.

#### **2.5.2.4 Compatibility with the Disposal or Reuse Environment**

The ultimate planned use of the S/S-treated waste has a bearing on binder selection. Although many treated wastes may be disposed of in a municipal landfill, monofill, or some other subsurface burial site, others may be reused as fill, road base, or construction material. For still others, the



method of treatment will be direct incorporation of the untreated waste, such as sandblasting grit, into a composite, such as asphalt. Numerous disposal and/or reuse options exist, but these are constrained by legal and institutional concerns. Reuse options, as opposed to disposal options, are nonroutine and subject to intense scrutiny to demonstrate environmental protection. Ultimate use options need to be anticipated and factored into the binder screening process along with product compatibility considerations.

For example, if the waste disposal location lies in the saturated zone, binder selection must consider the probability of water reaching the waste. Low permeability, adequate compressive strength, and stability in the groundwater geochemical environment will be important criteria. Also, engineering controls of disposal site hydrogeology may be incorporated to supplement binder performance criteria. Waste disposal site and waste performance considerations all relate to the protection of public health and the environment.

#### **2.5.2.5 Cost**

Cost is an additional binder screening criterion, although this criterion should be applied only after the interference, chemistry, and disposal/reuse environment issues have been considered. Because economic considerations are secondary to performance considerations, cost should be used only to screen binders that are significantly less economical or whose benefits clearly do not justify the added expenditure.

#### **2.5.2.6 Process Track Record**

Finally, process track record may be a discriminating factor in the selection of binders for bench-scale testing. Several databases have been developed that may be referred to as sources of information on successful treatability studies. Conner (1990) contains numerous tables of performance data from previous treatability studies, organized by metal. Means et al. (1991a) contains, on a disk in PC-DOS spreadsheet format, a tabulation of more than 2,500 performance data from S/S treatability studies. The database can be sorted by metal, waste type, binder type, or other delineators. Note, however, that published performance data from previous treatability studies generally are of limited value in designing future treatability studies

because seldom do those publications provide the level of detail necessary to permit replication of the experiment. Also, subtle variations in waste chemistry can lead to very different treatability results.

Although process track record may be one of the factors used to select binders for bench-scale testing, its inclusion here is not intended to discourage the use of innovative or experimental binders or S/S technology, which may prove very useful in certain circumstances.

## **2.6 LABORATORY BENCH-SCALE SCREENING OF THE WASTE/BINDER MIXTURES**

### **2.6.1 Purpose**

The result of the waste/binder compatibility literature screening described in Section 2.5 will be a list of binders or binder additive systems that are promising candidates for S/S treatment. If only one binder is identified, then it should be tested as described in this section to determine whether it has merit; otherwise it will be necessary to identify an alternative binder.

Because the technology screening to this point has been based on the literature review and generic information from previous S/S projects, the analysis now needs to be made specific to the actual waste being studied. Waste/binder mixes should be tested in the laboratory to determine relative performance. Because analytical testing is expensive, it is impractical to conduct a full set of performance tests on all of the waste/binder mixtures. Therefore, the testing at this stage takes the form of "screening" as opposed to detailed performance testing and is limited to the minimum required to indicate process applicability.

The bench-scale screening process described in Section 2.6 essentially equates to the "remedy selection" screening step in U.S. EPA's guidance for treatability testing under CERCLA (U.S. EPA, 1989e). Note that for certain S/S projects, where there is a high level of confidence that a given binder will easily satisfy the project's performance goals, this bench-scale screening step may be deemed unnecessary. This might be the case in situations where waste properties are simple and straightforward, and where the selected binder has a demonstrated track record for the waste being stabilized. However, because of the numerous possible subtleties in S/S process implementation and the possible effects of site-specific water properties on

binder performance, it is highly recommended that bench-scale screening be conducted whenever possible.

The general steps of the bench-scale screening process are summarized in Figure 2-7. As this figure indicates, several iterations may be necessary. Candidate binders identified from Section 2.5 are screened using simple bench-scale treatability tests. If the performance data do not discriminate sufficiently among binders, then additional screening criteria, such as ease of implementation in the field and cost, also may be considered at this stage. The binder or binder system that is ultimately selected will undergo more thorough bench-scale performance testing as described in Section 2.7.

### **2.6.2 Approach**

Bench-scale screening entails mixing relatively small amounts of waste with binders for testing individual parameters or indicators of S/S technology performance. These laboratory tests, which are used to determine whether the "chemistry" of the process works, are usually performed in batch (e.g., "jar tests") with treatment parameters varied one at a time. Because small volumes and inexpensive reactors such as bottles or beakers are used, bench-scale screening tests can be an economical way to test a relatively large number of performance and chemistry variables. It is also possible to evaluate a treatment train made up of several technologies and to generate limited amounts of residuals for evaluation.

#### **2.6.2.1 Experimental Design**

At the screening stage a large number of treatment options are possible. For this reason, it is important to efficiently design the laboratory experiments. The important experimental questions to be answered can generally be expressed as hypotheses that are supported or disproved based on the experimental data. Decisions about how many and what kinds of data to measure are made most reliably on the basis of statistical experimental design procedures used to reduce the effects of experimental errors in the measured data. The area of experimental design has been well developed (e.g., Cochran and Cox, 1957; Hicks, 1973). The six fundamental steps in developing a statistical experimental design are as follows:

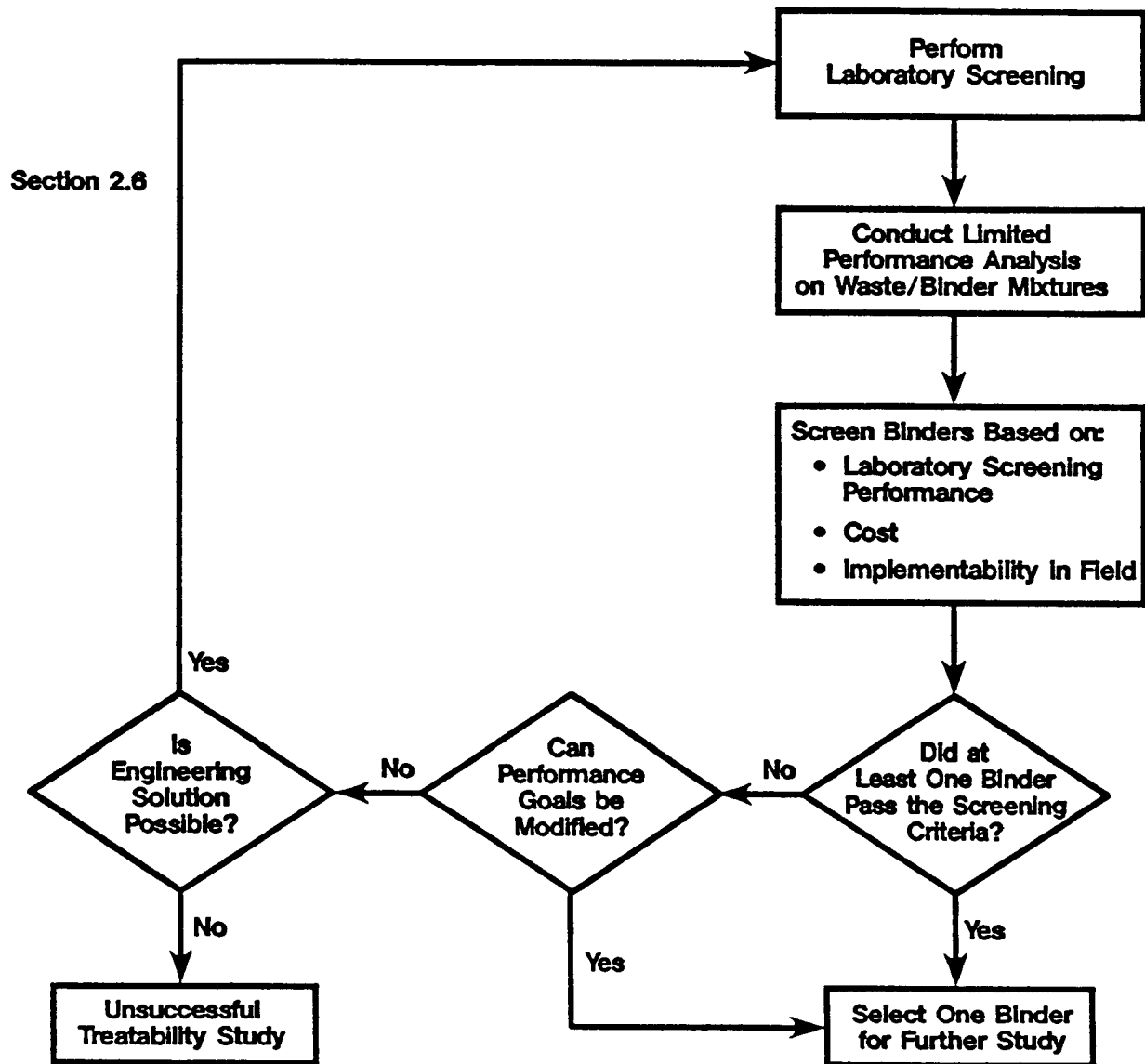


FIGURE 2-7. LABORATORY SCREENING OF WASTE/BINDER MIXTURES

1. Clearly define the experimental objectives along with the tests to be performed.
2. Define the experimental factors to be controlled, as well as the levels and combinations of these factors to be investigated.
3. Establish the method of randomization to be used.
4. Select a statistical model to describe the experiment.
5. Specify the data analysis procedures to be employed as well as the desired statistical properties.
6. Select the experimental design parameters to achieve the desired statistical properties.

#### **2.6.2.2 Performance Testing**

Bench-scale screening is performed at this stage to comparatively evaluate the candidate binding agents. As previously indicated, extensive analytical data are not needed. Depending on the performance criteria of concern, one or two simple performance tests, such as the frequently recommended TCLP and unconfined compressive strength (UCS) tests, should suffice for screening. The TCLP is recommended because of its regulatory status and because, compared to other leaching tests, it is relatively simple and inexpensive to perform. The UCS test is recommended because most disposal and reuse options for S/S-treated waste will have some level of UCS performance standards. For example, 50 psi is typical guidance per U.S. EPA (1986b). However, situations may be encountered where the use of other screening tests is justified. Testing methods are discussed in Chapter 3.

It may be appropriate at this stage to test the effectiveness of different binder/waste ratios, because an optimal ratio cannot be determined a priori. If the binder/waste ratio is not treated as a variable, some useful binder may be rejected from further consideration because it was tested at the wrong proportion(s). One test facility typically uses binder/waste ratios of 0.1, 0.3, and 0.6 based on dry weight (Barth and McCandless, 1989). These are probably appropriate for most generic binders. However, specialty binders may operate optimally at other ratios. If the binder/waste ratio is treated as a variable at this stage, then three variations will yield the necessary data

for most cases. More or fewer binder/waste ratios may be needed depending on factors such as waste complexity and toxicity. However, for CERCLA remedial actions, it rarely is worthwhile to test at binder:waste ratios greater than 1.0, because of chemical costs and the disposal complications presented by the volume expansion of the waste at the higher ratios. Higher ratios may be useful if blast furnace slag or kiln dust are available or if higher water contents require higher binder addition.

Whatever performance criteria are chosen for testing, the waste should satisfy the criteria with some margin of safety because the laboratory is a more controlled environment than the field for testing. In the field, ingredient proportions and the thoroughness of mixing are more variable. Typical guidance for the extent of this margin of safety is that the performance criteria should be satisfied by at least a factor of 2. For example, TCLP-tested Pb should be  $\leq 2.5$  mg/L, versus the U.S. EPA threshold of 5.0 mg/L. This is technical guidance, not policy.

If screening tests fail to discriminate sufficiently among the binders (i.e., they perform similarly), then it may be appropriate to screen the binders based on other factors, such as ease of field application (implementability) or cost. Ease of application in the field refers to process complexity or sensitivity of performance to process parameters. Highly complex processes, such as numerous sequential steps and processes that are extremely sensitive to process parameters, such as exact ingredient proportions and thorough mixing, may be very difficult to implement in the field and probably should not be attempted unless preceded by a pilot- or full-scale demonstration. Health and safety considerations for workers and nearby inhabitants also affect the ease of using a particular S/S process at a particular site. Both the S/S field equipment necessary and treatment chemicals used should be conducive to safe and efficient application under actual field conditions.

A final factor affecting binder screening is cost. If all other factors (performance and implementability) are equal, then cost may be used to select a binder. The most significant cost items are usually chemicals, equipment rentals or use rates, and labor. The latter two categories of cost information are difficult to estimate at this stage. However it should be possible to develop a sense for the overall process complexity and maximum possible processing rate. Additional information pertaining to the cost of S/S treatment is provided in Section 4.10.

### **2.6.3 Technical Guidance**

Guidance for bench-scale binder screening is summarized in Table 2-12. This information is provided to assist in planning and implementing valid bench-scale screening tests for S/S.

## **2.7 BENCH-SCALE PERFORMANCE TESTING/PROCESS OPTIMIZATION**

### **2.7.1 Purpose and Objectives**

At this stage in the S/S treatability study, limited treatability testing has been conducted and a promising binder has been identified. Now it is necessary to demonstrate that the binder will achieve all relevant project performance goals and to optimize the S/S process in terms of design, field implementability, and cost performance. This step in the treatability study is referred to herein as "bench-scale performance testing/process optimization" and equates to the "remedy design testing" step in U.S. EPA's guidance for performing treatability studies under CERCLA (U.S. EPA, 1989e). Bench-scale performance or remedy design testing is frequently performed soon after the Record of Decision in CERCLA projects, prior to implementing the remedy.

A descriptive performance testing protocol that will satisfy the requirements of all S/S projects cannot be specified because site-specific projects have different performance goals and because the response of individual wastes to S/S technology can be unpredictable. In the absence of extensive regulatory requirements for S/S treatment projects, acceptance criteria must be determined largely on a case-by-case basis. The approach summarized here and illustrated in Figure 2-8 advocates that the level of performance testing be set by the potential level of risk posed to human health and the environment. That is, the testing program should be based upon the guiding principles derived from the ultimate risk posed by the waste in its planned disposal or reuse environment.

Four principal factors affect risk in this context:

- Waste volume
- Type and concentration of contaminants (metals, organics, or both)

**TABLE 2-12. BENCH-SCALE BINDER SCREENING GUIDANCE**

- 
1. Test the effectiveness of any pretreatment system.
  2. Screen at least two to four binders at two or more binder/waste ratios.
  3. Ensure there are no binder/waste incompatibilities that could pose a safety hazard (release of toxic gas, etc.).
  4. Use process, waste, and binder information to determine whether to base testing on composited waste samples, worst-case samples, or both.
  5. Carefully monitor, control, and record binder additions, order and sequence of additions, timing, and other procedural information.
  6. Conduct several rounds of bench-scale testing to optimize binder performance.
  7. The chemical compositions of the binder and binder additives should be known or chemically analyzed to ensure that these ingredients do not contain hazardous constituents or properties. Consult MSDSs at a minimum.
  8. New ARARs may be developed as a result of the binder and/or binder additives (e.g., dust emissions, corrosivity [pH] limits, etc.).
  9. Have the treatability study witnessed by an independent third party or regulatory agency for impartiality.
  10. Simulate anticipated field conditions during curing as closely as possible (e.g., do not necessarily put the treated waste immediately into a sample jar).
  11. Allow the sample to cure properly before chemical and physical analyses.
  12. Calculate the percent reduction in TCLP contaminant concentration caused by stabilization both with and without the effects of waste dilution by binder ingredients.
  13. Test the most critical ARARs (e.g., leaching characteristics and critical chemical/physical properties).
  14. Assess air emissions if volatile organics are present.
  15. Send splits of a few samples to a second laboratory for interlaboratory verification.
  16. Conduct the bench-scale screening project under a proper QA/QC program, including statistical design, replication, blind controls, compliance with laboratory certification requirements, etc.
  17. Calculate or measure waste volume increase from binder/water additions.
-



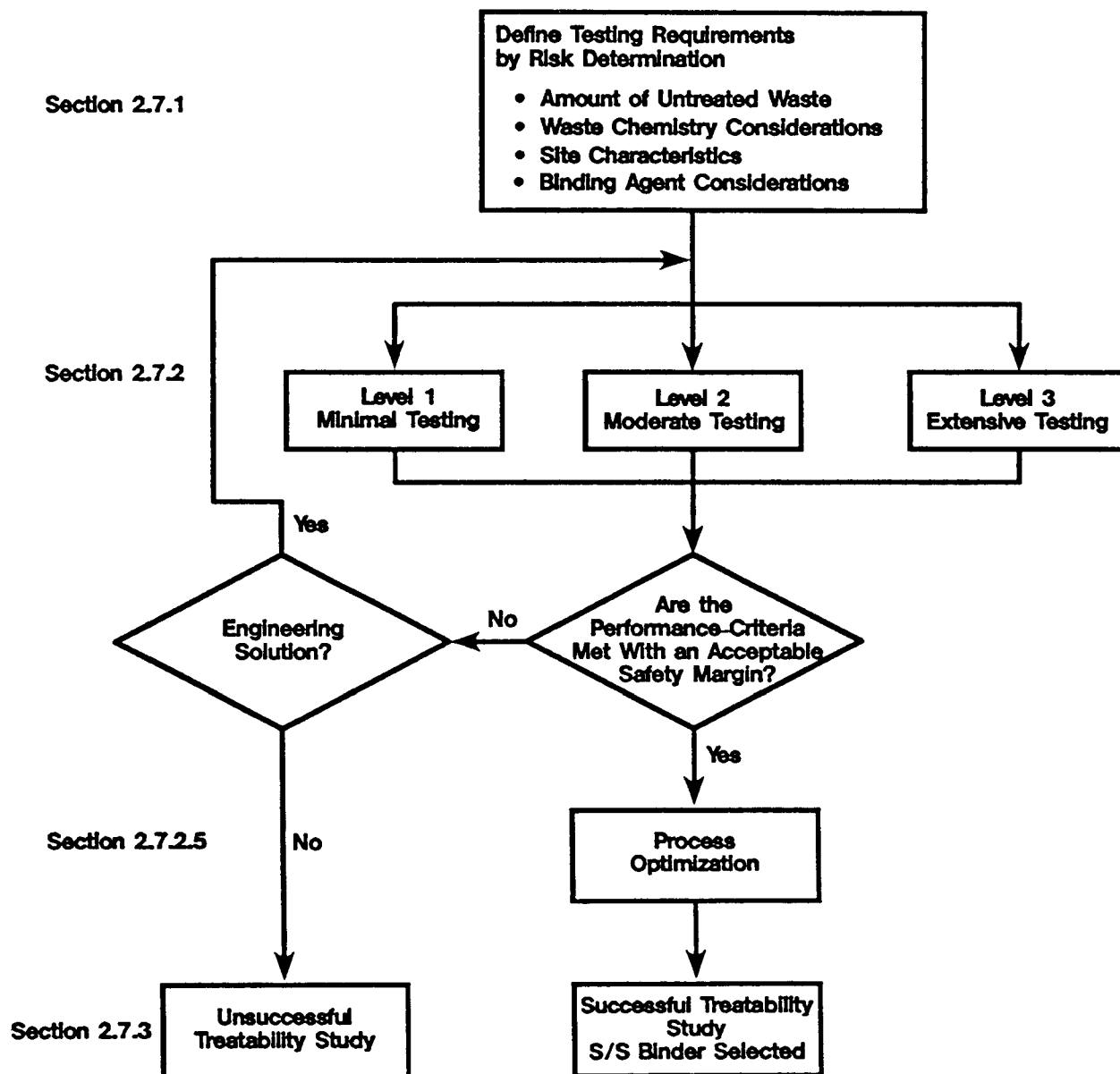


FIGURE 2-8. BENCH-SCALE PERFORMANCE TESTING OF SELECTED WASTE/BINDER MIXTURES

- Site characteristics of the planned disposal or reuse environment
- Demonstrated performance of the S/S process selected

This section provides qualitative guidance for determining the level of risk based on the above categories of factors. The level of risk then determines the general extent of recommended performance testing. More extensive testing requirements are required for projects that present greater risk in order to increase the level of confidence that the treated waste will remain stable for the long term. One type of testing requirement that is not derived from risk pertains to specific binders. Testing related to binder evaluation is discussed briefly in Section 2.7.2.3.

The goals of bench-scale performance testing and process optimization are to demonstrate that the S/S-treated waste is:

- Chemically and physically stable (i.e., no free liquids as determined by the paint filter test, low leaching rates)
- Compatible with its disposal or reuse environment (e.g., possesses adequate compressive strength, is nonbiodegradable, and has sufficiently low permeability)
- In conformance with the ARARs by an adequate margin of safety
- Cost-effective compared with other possible treatment technologies
- Demonstrated effective and readily implementable in the field

Generalized procedures and rationales for determining the level of performance testing are provided in the following sections. Please note that this approach applies mainly to projects under CERCLA remediation and RCRA placement. As indicated in Section 2.3, the testing requirements of a RCRA TSD facility are more specific and include the Paint Filter Test for free liquids, the TCLP for leachable metals, and the other three tests for hazardous waste characteristics (i.e., ignitability, corrosivity, and reactivity).

## **2.7.2 How Much Performance Testing?**

### **2.7.2.1 Levels of Risk**

Risk determination is probably equivocal in most cases, and exceptions to any approach will always be identified. No expert system exists yet for determining risk as it relates to S/S projects. One approach, however, is based on the principal risk factors identified in Table 2-13. This simplified approach is provided as rule-of-thumb guidance only. As indicated previously, numerous exceptions are likely to exist.

The categories of risk in Table 2-13 are (a) waste volume, (b) type and quantity of metal contaminants, (c) type and quantity of organic contaminants, (d) site (disposal or reuse) characteristics, and (e) demonstrated effectiveness of the S/S process. Each of these risk categories is subdivided into low, medium, or high risk levels. Examples of each are given in Table 2-13. The trends are straightforward. Larger volumes of waste, higher hazard contaminants, site conditions promoting possible exposure to human or ecological receptors, and undemonstrated S/S processes are all associated with higher risk and therefore higher levels of performance testing. Metals and organics are considered separately, because a waste containing both is more difficult to treat and therefore poses greater risk than a waste containing only one or the other. Table 2-13 shows where a project falls among the five risk factors and is used to determine the necessary level of performance testing, which is explained further in Section 2.7.2.2. Identifying the level of risk is a subjective determination on the part of the participants in the treatability study.

### **2.7.2.2 Levels of Performance Testing**

Three levels of performance testing correspond to the three levels of risk from Table 2-13. Table 2-14 describes some typical testing requirements (leaching, physical, and other chemical tests) for each of the three levels. The tests to be run cannot be specified exactly, as they will depend upon the needs of the individual S/S project. For example, a freeze/thaw test may not make sense for an S/S-treated waste placed entirely below the frost line. Permeability would be of little consequence for disposal in the desert far above the groundwater table. Thus, Table 2-14 provides guidance on the overall magnitude or level of effort associated with the testing program as opposed to specific testing requirements.

TABLE 2-13. RISK FACTORS FOR EVALUATING LEVELS OF PERFORMANCE TESTING

Risk factors	Risk levels		
	I. Low risk	II. Medium risk	III. High risk
A. Waste volume	• <1,000 cu yd	• >1,000 to <10,000 cu yd	• >10,000 cu yd
B. Metal contaminants	<ul style="list-style-type: none"> <li>• Noncarcinogenic</li> <li>• Low toxicity (e.g., Cr[III], Ba, Zn, Mo, Cu)</li> <li>• Low to moderate concentrations</li> </ul>	<ul style="list-style-type: none"> <li>• Noncarcinogenic</li> <li>• Moderate toxicity (e.g., Pb, Se, Sb)</li> <li>• Low to moderate concentrations</li> </ul>	<ul style="list-style-type: none"> <li>• Known or suspected carcinogen</li> <li>• High toxicity (e.g., Cr[VI], Cd, Hg, As, Be)</li> <li>• Very high concentrations</li> </ul>
C. Organic contaminants	<ul style="list-style-type: none"> <li>• Low total organic carbon content</li> <li>• Low hazardous organics</li> <li>• Low-interference organics</li> </ul>	<ul style="list-style-type: none"> <li>• Same general criteria as Level II metals (e.g., organic priority pollutants other than those under III, high-risk organics such as cresols, xylenes, and aldehydes)</li> </ul>	<ul style="list-style-type: none"> <li>• Same general criteria as Level III metals (e.g., PAHs, PCBs, dioxins, furans, certain pesticides, chlorophenols)</li> <li>• High-interference organics</li> </ul>
D. Site characteristics	<ul style="list-style-type: none"> <li>• Vadose zone disposal or depth to groundwater &gt;25 ft</li> <li>• Dry climate</li> <li>• Low population density</li> <li>• Distant from drinking water source</li> <li>• RCRA-permitted disposal facility</li> </ul>	<ul style="list-style-type: none"> <li>• Conditions intermediate between I and III</li> </ul>	<ul style="list-style-type: none"> <li>• Saturated zone disposal or depth to groundwater &lt;5 ft</li> <li>• Wet climate</li> <li>• High population density</li> <li>• Close to drinking water source</li> <li>• Windy conditions coupled with aboveground disposal or reuse</li> </ul>

TABLE 2-13. RISK FACTORS FOR EVALUATING LEVELS OF PERFORMANCE TESTING (Continued)

Risk factors	Risk levels		
	I. Low risk	II. Medium risk	III. High risk
E. History of process effectiveness	<ul style="list-style-type: none"> <li>Well-established, frequently used process with generic binders and contaminants that stabilize readily</li> </ul>	<ul style="list-style-type: none"> <li>Field-demonstrated, but not as frequently used</li> </ul>	<ul style="list-style-type: none"> <li>Innovative or complex process for contaminants that are more difficult to stabilize (e.g., As, Cr[VI], phenol)</li> </ul>

TABLE 2-14. LEVELS OF PERFORMANCE TESTING AND EXAMPLE TESTING REQUIREMENTS

Testing level	Example testing requirements <sup>ab</sup>		
	Leaching	Physical	Other chemical
I. Low	<ul style="list-style-type: none"> <li>One short-term test (e.g., 5-day ANSI/ANS/16.1)</li> </ul>	<ul style="list-style-type: none"> <li>Minimum number of parameters needed to demonstrate compatibility with disposal or reuse environment</li> </ul>	<ul style="list-style-type: none"> <li>As needed; pH usually required to demonstrate noncorrosiveness</li> </ul>
II. Medium	<ul style="list-style-type: none"> <li>One short-term test (e.g., 5-day ANSI/ANS/16.1) and one medium or long-term test (e.g., MEP)</li> </ul>	<ul style="list-style-type: none"> <li>Several physical parameters (e.g., UCS, permeability, specific gravity)</li> <li>Freeze/thaw and wet/dry tests if aboveground use is planned</li> </ul>	<ul style="list-style-type: none"> <li>As needed (e.g., pH, acid neutralization capacity)</li> </ul>
III. High	<ul style="list-style-type: none"> <li>Several tests, including one long-term test (e.g., MEP or 90-day ANSI/ANS/16.1); geo-chemical and/or transport modeling may be advisable</li> <li>A solvent extraction test (total waste analysis) to demonstrate the chemical stabilization of non-TCLP-list organics (see Section 4.4.3)</li> <li>Nonroutine analytical procedures as required to indicate chemical bonding</li> </ul>	<ul style="list-style-type: none"> <li>Applicable properties from Level II plus standard factor test, California bearing ratio, freeze/thaw and wet/dry tests, and others as appropriate</li> <li>Biodegradation tests if pertinent to the binder</li> </ul>	<ul style="list-style-type: none"> <li>pH, acid neutralization capacity, Eh, volatile emissions as appropriate to show chemical stability and compatibility</li> <li>Bioassays as appropriate</li> <li>Speciation of contaminant metals to show potential for long-term stability</li> </ul>

<sup>a</sup> Assumes TCLP levels and UCS have been determined previously in laboratory screening. If the process formulation has been modified, TCLP and UCS should be rerun.

<sup>b</sup> These are candidate tests given as examples only.

As Table 2-14 indicates, high-risk projects require more rigorous levels of testing to establish a higher degree of confidence that the S/S-treated waste will attain and maintain the required levels of performance. For most high-risk S/S projects, this also means that the potential for long-term leaching should be assessed. For final placement close to natural waterways, the need for acute bioassay testing may also be considered. A large number and wide variety of performance tests may be conducted. Chapter 3 discusses a selection of the many available physical, leaching, chemical, biological, and microcharacterization tests. Chapter 3 may be consulted for information about the types of tests available, the information they provide, and any existing acceptance criteria.

#### **2.7.2.3 Tests for Specific Binding Agents**

Binder selection is an additional consideration in designing the performance testing program. Certain types of tests relate more to the specific properties of the binder than to the risk associated with waste chemistry and site characteristics. Examples include the following:

- When sulfide is used as a treatment chemical, pH and reactive sulfide analyses (or sulfide reactivity, the so-called "Claussen test") should be conducted to ensure that the waste meets the RCRA corrosivity (pH less than 12.5) and reactive sulfide (less than 500 mg/kg) guidelines.
- When thermoplastics or other organic binders are used, biodegradation tests may be required.

In some situations, a test method may need to be modified to accommodate a specific S/S-treated waste. For example, because the oils and bitumens in asphalts would probably lead to filter plugging, the filtration procedures may need to be modified or eliminated completely.

#### **2.7.2.4 Acceptance Criteria**

The success of the treatability study will be measured in terms of whether the tests satisfy predetermined performance objectives. Some of these criteria are regulatory limits, such as the metal thresholds that have been established for the TCLP, EP Tox, and California Waste Extraction Test (WET).

However, most criteria are not strict regulatory limits and must be determined on a case-by-case basis. For example, the target permeability and UCS criteria will vary with the site characteristics of the disposal environment. Permeability requirements will vary with the water flux through the disposal zone and the proximity to the groundwater table. The UCS criteria should be based on an engineering calculation of the load under which the waste will be placed plus a safety factor.

The approach for determining acceptance criteria generally emphasizes designing to the needs of the individual project. It is not cost-effective to design overly restrictive criteria. However, the criteria need to be adequate to ensure, with an acceptable degree of probability, that the S/S-treated waste will perform satisfactorily in the field.

If the treatability study is unsuccessful (i.e., if some performance objectives are not satisfied), then several options are available, for example:

- Revise the performance objectives within regulatory limitations (for example, exception to ARARs)
- Modify the formulations
- Investigate a completely different binder system
- Add more engineering controls to the final placement location

Most performance defects identified in treatability studies can be corrected by process or binder modifications. However, the resulting S/S treatment system may be complex or expensive. If performance is so unsatisfactory that S/S is not a viable option, then the S/S treatability study is concluded.

#### **2.7.2.5 Process Optimization**

The bench-scale treatability environment offers an excellent opportunity to fine-tune the S/S process for site-specific waste. Process optimization includes the following types of activities:



- Determining the trade-offs between reducing the binder:waste ratio and associated cost savings versus process performance
- Determining the optimal sequence of binder or additives in terms of processing rate and process performance
- Evaluating the sensitivity of the S/S process to slight variations in binder amounts, curing conditions, and/or mixing efficiency
- Evaluating the sensitivity of the S/S process to expected variations in waste properties (average vs. worst-case contaminant concentrations, variable matrix properties, etc.)

Process optimization is an important step in maximizing cost-effectiveness and determining process sensitivities.

### **2.7.3 Technical Guidance**

Guidance for conducting bench-scale performance testing is provided in Table 2-15. The guidance provided in Section 2.6 (Table 2-12) is also applicable.

## **2.8 PILOT-SCALE AND FIELD DEMONSTRATIONS**

### **2.8.1 The Need for Process Scale-Up**

Bench-scale treatability testing ends when a suitable binder and binder:waste ratio is selected. The user must then determine whether a pilot test or field demonstration test of the stabilization process is necessary prior to a full-scale cleanup. A pilot test generally refers to an intermediate-scale simulation (often in the laboratory) of a full-scale operation. Field demonstration generally refers to a simulation of the full-scale operation conducted on-site with actual full-scale (or close to full-scale) equipment. A pilot or field test may be needed to build confidence in the binder selection or to gather data for design of the full-scale system. Pilot-scale studies are typically directed at resolving equipment sizing, selection, or scale-up issues. Usually in S/S technology, the field test is a dry-run of the full-scale treatment equipment under carefully monitored conditions prior to proceeding with full-scale treatment. The expense of a

TABLE 2-15. GUIDANCE FOR BENCH-SCALE PERFORMANCE TESTING

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1. The same guidelines concerning procedures for conducting bench-scale treatability tests provided under section 2.6 (Table 2-11) also apply here.
  2. Performance tests are needed for all ARARs, for example:
    - a. If subsurface disposal is planned, appropriate tests should be conducted (e.g., unconfined compressive strength and permeability, etc).
    - b. If surface or near-surface disposal is planned, appropriate tests should be conducted (e.g., wet/dry, freeze/thaw, etc.).
    - c. Long-term stability needs to be ensured. The TCLP is not sufficient evidence of long-term stability. Alternative leaching tests should be conducted that better address long-term stability (see Section 3.2) and/or the TCLP should be conducted on treated waste after different curing periods (Section 4.7).
    - d. For wastes having organic contaminants with low aqueous solubilities, leaching with an organic solvent may be appropriate (see Section 4.4.3).
    - e. For wastes containing organic contaminants, conduct a mass balance to account for the fractions of contaminants that are leachable, immobile, and released due to volatilization.
    - f. For a suspected colloidal contaminant transport mechanism, consider substituting larger pore-size filter medium for the standard filtration medium or using centrifugation instead of filtration.
    - g. Leach tests using site-specific groundwater (as opposed to generic leachate or distilled water) may be appropriate.
    - h. If the binder is biodegradable, a biodegradation performance test should be conducted.
    - i. If the disposal site could leach into an aquatic system, leachate bioassay may be appropriate.
    - j. Note that the binders themselves may contain contaminants such as metals; these should be taken into consideration in performance testing.
  3. A total contaminant analysis should generally be performed on the same subsample used for leach tests to eliminate false negatives.
  4. The leaching performance data should be corrected for the effect of dilution to determine the actual extent of stabilization due to binding.
-

TABLE 2-15. GUIDANCE FOR BENCH-SCALE PERFORMANCE TESTING (Continued)

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5. Simulate field conditions as closely as possible during curing.
  6. Allow the waste to cure for an appropriate period of time before analysis.
  7. The entire performance testing program should be conducted under an appropriate QA/QC program, including statistical design, replicates, analytical methods, blind controls, and other controls.
  8. There should be a safety margin in the performance data relative to the numerical thresholds because the S/S process may not work as well as in the field.
  9. The S/S process developed and demonstrated at this stage must be implementable in the field (i.e., not too complex).
  10. The volumetric expansion of the waste during treatment must conform to the disposal space constraints.
  11. The cost should be realistic for an S/S treatment option; depending on the circumstances, a realistic cost is usually less than \$150/ton.
  12. Splits of some proportion of the samples should be sent to a second analytical laboratory for interlaboratory comparison.
  13. It is advisable for bench-scale testing to be observed by an independent third party or regulatory agency for impartiality.
- 

pilot-scale (intermediate-scale) test is usually not warranted, except for very complex S/S projects.

The decision whether to do a pilot or field test hinges mainly on how widely a particular waste/binder system has been demonstrated in the past. Other factors such as regulatory requirements, full-scale equipment design, and cost estimation are also considered. If treatability testing shows that the waste contains common forms of contaminants that respond well to stabilization in a matrix that contains no significant amounts of interferants, and if the binder system is well-demonstrated and commonly used on these contaminants, then a pilot or field demonstration may not be necessary. If the contaminant species is complexed in the waste matrix, if the waste contains interferants, or if a not-so-well-understood binder system is being used, a pilot or field-scale demonstration is advisable to ensure the effectiveness of

the process. As indicated above, a field demonstration can be conducted simply as a discrete part of the full-scale cleanup, with a pause after the demonstration to evaluate effectiveness and/or allow for regulatory review. This is a useful step for calibrating material flow rates and for determining optimal processing rates. Any deficiencies in the field equipment can be identified and corrected, and field personnel can be trained in the safe operation of the full-scale equipment. Once the S/S process has been demonstrated in the field, the cleanup can continue with the same equipment.

Safety problems can also be identified during pilot/field testing. For example, the *Handbook for Stabilization/Solidification of Hazardous Waste* (U.S. EPA, 1986c) describes how rapid addition of a reactive stabilization agent (e.g., unhydrated lime) can cause rapid volatilization of lower boiling-point organics, leading to flash fires.

A specific case history demonstrates the advisability of a field test prior to full-scale treatment. Physical conditions during full-scale cleanup may vary from those in the laboratory so as to alter or prevent the desired reactions of the stabilization process. A case in point is described by Means et al. (1991b) for a field demonstration stabilizing sand blasting grit containing copper and lead as contaminants. A laboratory-proven binder system composed of sulfide and fly ash was used during the initial demonstration. The treated waste was stored in the open on plastic sheets for curing. Samples of the cured waste showed that the waste at the top of the pile was not as well stabilized as the waste at the bottom of the pile. During further treatability testing, it was discovered that when the waste was cured in a jar, stabilization was effective. When the waste was cured on a gentle incline in the open air, simulating the field waste material, some excess stabilization reagent was observed draining off the waste material. It was concluded that environmental conditions caused by piling were preventing the reaction between the sulfide and the metal ions from reaching completion. Thus, the field system was shown to be not as effective as the bench-scale system for this stabilization project. Fortunately, the problem was identified and corrected at an early stage of field treatment.

All the factors mentioned above should be taken into consideration in determining the need for a field demonstration before full-scale cleanup. Once a decision is made to proceed with the demonstration, the steps in the flowchart of Figure 2-9 may be followed. Two to four small batches of waste

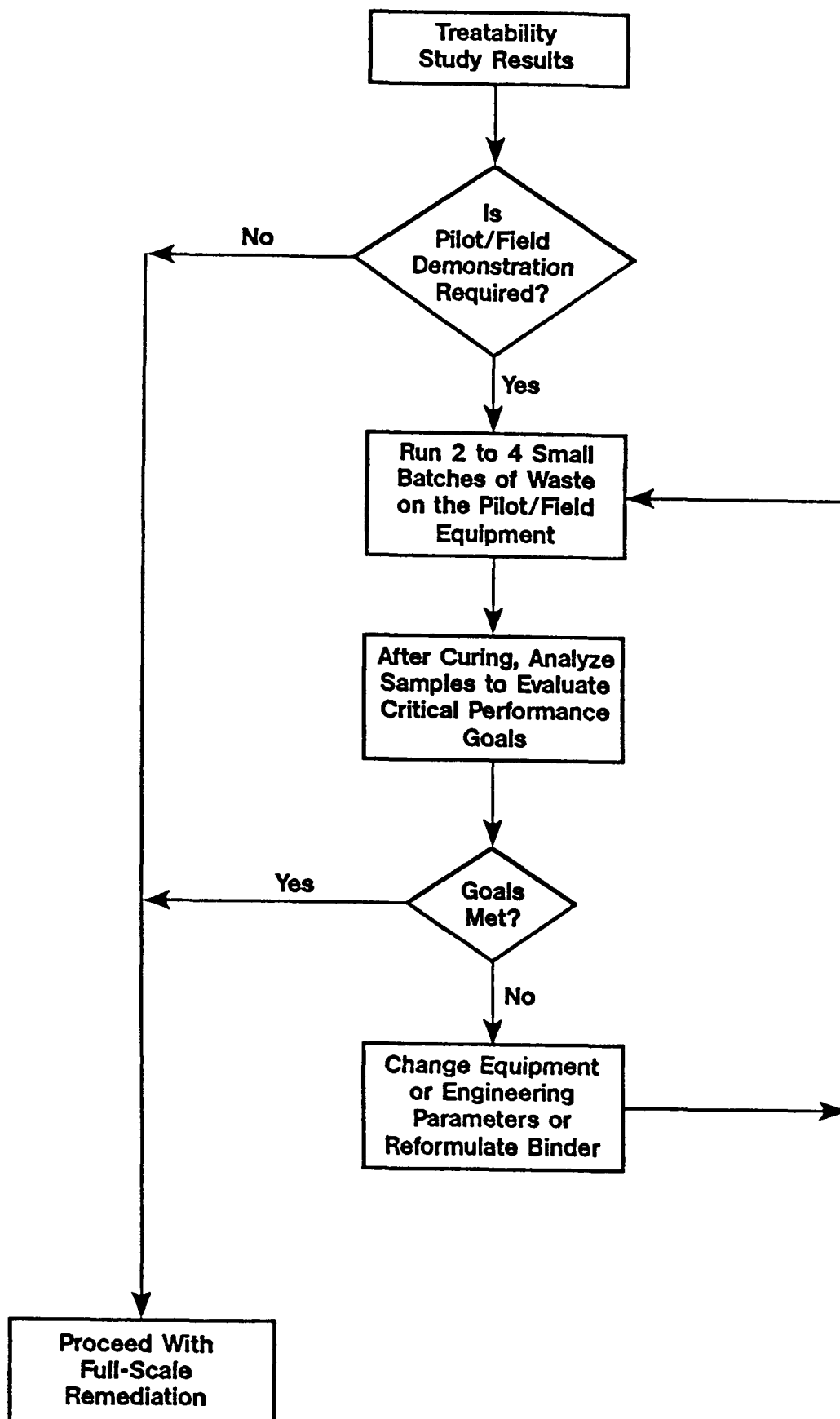


FIGURE 2-9. PILOT-SCALE TEST SCREENING

are typically run, with 1 to 15 cubic yards of untreated waste material per batch generally used depending on the size of the available equipment. Statistically significant samples are taken and analyzed by the tests described in Chapter 3 to demonstrate effectiveness. Adequate quality assurance procedures are followed during sample collection and analyses to ensure reliability.

After allowing the treated waste to cure, the samples can be analyzed and evaluated for critical performance goals as determined at an earlier stage. If the samples meet these performance objectives, the user may proceed with the full-scale cleanup. If the samples fail the performance objectives, the user has to determine whether the field-scale equipment, the binder formulation, and/or other engineering parameters (e.g., flow rates, storage environment) are at fault. Further testing may be necessary to identify the cause of the deviation between bench-scale and field-scale results.

### **2.8.2 Scale-Up Issues**

Scale-up from a bench-scale to field demonstration or full-scale process generally focuses on the materials handling aspects of the process since the chemistry already has been addressed in the bench-scale tests. Scale-up plans should address each of the following wherever applicable:

- Waste excavation for ex situ processes
- Waste handling
- Equipment selection & sizing
- Chemical reagents (binder) storage
- Pretreatment of waste
- Presence of debris
- Materials balance
- Mixing and curing
- Stabilized waste disposal

The most common methods of stabilization are plant mixing and in situ mixing. Plant mixing involves removing the waste from its location and

transferring it to a treatment plant. The waste is mixed with the stabilization agents in the fixed or mobile treatment plant. During in situ mixing, the waste remains in place, and the stabilization agents are injected or mixed with specialized augers or other equipment.

Another method, area mixing, is used mainly for treating oily sludges or semisolid wastes. In this method, a layer of waste is placed in the disposal area and covered with a layer of stabilization agents. The layers are lifted and turned over repeatedly and then dried and compacted. A top layer of clean soil is then added as a cap. Yet another method, in-drum mixing, is generally used for highly toxic wastes in drums. If there is enough headspace above the waste in the drum, stabilization agents may be added and mixed with the waste.

The U.S. EPA published several Technology Evaluation Reports on its SITE demonstrations of stabilization techniques such as plant mixing (U.S. EPA 1989h and 1989i) and in situ stabilization (U.S. EPA, 1989j). These reports contain important information on field operation and performance. The *Handbook for Stabilization* (U.S. EPA, 1986c) is also a good reference, describing operating characteristics and cost of large-scale equipment.

A discussion of some commonly used full-scale stabilization equipment follows.

#### **2.8.2.1 Waste Excavation and Handling**

Traditional earth-moving equipment (e.g., backhoes, draglines, bulldozers, front-end loaders) is used for this process. If free liquid is present on top of the waste, it may have to be pumped out and treated as a separate waste stream. The equipment operator may have to be completely enclosed or provided with breathing apparatus if air hazards are generated during excavation.

Depending on the nature of the waste and the site, the excavated waste can be transported to the treatment plant by a fixed system (conveyor or screw auger), dump truck (for soil), pump and hose (for liquids and sludges), or, if the waste is particularly hazardous, in drums. Spillage should be avoided during transport.

#### **2.8.2.2 Stabilizing Agent Storage**

For cost-effective operation, it is important that sufficient amounts of chemicals be available to avoid project shut-down for restocking. Amounts required are determined from treatability testing results, specifically the binder:waste ratio. Bins, hoppers, and silos are used for storage of dry chemicals. If liquid chemicals are being used, liquid storage tanks or drums may be necessary. Unless the waste volume is small, chemicals generally need to be replenished on a continuous basis during the project.

#### **2.3.2.3 Pretreatment of Waste**

Pretreatment may be necessary for (a) improving the material handling characteristics of the waste, (b) improving waste/binder compatibility, and (c) removing constituents that either interfere with or are not affected by S/S processing. (See Section 4.1.4). Pretreatment can sometimes also reduce the quantity of stabilization agents during mixing.

Pretreatment may include screening and/or size-reduction equipment such as crushers (to remove large rocks or debris that may clog up the mixing equipment), drying or dewatering, blending and homogenization, pH adjustment, or heating to drive off volatiles. If volatiles are being driven off, some capture mechanism for the vapors may be necessary. Oversize materials from screening may have to be treated separately or disposed of appropriately.

Pretreatment is important from a materials handling point of view, especially at sites where the waste is difficult to handle with standard earth-moving equipment. There have been instances where the entire remediation operation had to be temporarily abandoned because of problems at the pretreatment stage. Screens and crushers can easily get clogged, especially with wet, sticky, or fine materials such as clay. Use of vibratory screens or special crushers may be necessary.

#### **2.8.2.4 Mixing and Curing**

Mixing is a critical step in ensuring good S/S process performance. All precautions must be taken to ensure that the waste and binder chemicals are mixed thoroughly and allowed to cure adequately. A wide range of mixing equipment is suitable for this application. The choice of equipment depends



on the type of waste/binder system and method of stabilization. In the most simple and inexpensive situation, area mixing can be done with a backhoe.

For in situ mixing, special augers and drills are used to inject the stabilization agents into the soil and to cause agitation and mixing. Backhoes can be used as in situ mixers, but the mixing is not reliable. Another in situ process is grouting, whereby fluids (usually water and cement) are injected into the ground, where they are allowed to set in place.

Plant mixing provides the maximum control on the mixing process. A range of equipment, including pug mills, extruders, ribbon blenders, sigma mixers, muller mixers, and screw conveyors is available. Standard construction-type cement or concrete mixers and transit-mix trucks have also been used. Mixing can be done as either a batch or a continuous process. Known volumes or weights of waste and chemicals can be added with reasonable accuracy into the mixer by front-end loaders or conveyors. Water or slurries can be metered and pumped in.

If continuous operation is desired, all materials must be introduced at a carefully controlled rate. This may require specialized material-handling equipment such as live-bottom feeders. Equipment such as pug mills can frequently be operated in either batch or continuous mode. Thus, it is possible to use a pug mill in batch mode during pilot or field demonstration and then change to continuous mode with several minor modifications: changing the angles on the paddles or knives on the pug-mill shaft(s), changing the level of the discharge gate, and/or changing the speed of rotation of the screws. However, when mixers are switched from batch to continuous mode, they must be recalibrated to ensure that the desired residence time and mixing are being achieved.

Mixing options also depend on the type of waste being mixed. Certain clay-type soils can become extremely sticky and adhere to the shaft and sides of the mixer, leading to poor mixing. Obtaining good mixing can also be problematic if the viscosity of the mix changes rapidly during setting. Mixer performance needs to be evaluated in order to confirm the amounts of stabilization agents needed. During bench-scale testing, the amounts of chemicals required for full-scale operation can be underestimated because less than ideal mixing efficiency was not accounted for.

The size of the mixer generally determines the maximum throughput for the entire stabilization process. Mixers vary widely in size, with

achievable throughputs between 1 and 200 tons per hour. Continuous processing usually provides a greater throughput but at the possible expense of mixing efficiency. Two mixers can be used to improve mixing in high throughput continuous processes.

Curing of the waste can occur in either containers, pits, or free-standing piles. Controls should be implemented both to protect the surrounding environment from possible runoff or leaching from the curing waste and to protect the curing waste from wind and precipitation.

#### **2.8.2.5 Stabilized Waste Disposal**

If the stabilized waste is to be used as fill, the use of standard earth-moving equipment (e.g., graders, bulldozers, front-end loaders) will usually suffice. After replacement, the waste is compacted. The moisture content of the compacted material should be controlled to give the maximum density for a given material. The moisture-density relationship can be determined by the Proctor test (ASTM D698). Too much or too little moisture can be detrimental.

Stabilization generally results in a volume increase. This volume increase can be underestimated during bench-scale testing and should be re-established in the field.

Post-treatment controls (e.g., capping, slurry wall, soil cover) frequently accompany stabilization to effectively mitigate site-specific threats. Performance standards for caps are mentioned in 40 CFR 264.310 but may not always be appropriate. Final selection of capping materials and cap design depends on several factors such as climate, site hydrogeology, availability of materials, and regulatory requirements.

#### **2.8.3 Sampling and Analysis of the Treated Waste**

The guidance on sampling and analysis in Sections 2.2.1.2 and 2.2.3.3 has general applicability to the pilot or field demonstration as well. In situ projects pose special complications for verification testing. For example, drilling or coring is required and homogeneity and setting rates are more difficult to assess. Analyses must be conducted to determine compliance with the performance goals of ARARs (Section 2.3) in a statistically significant manner.

### **3 S/S PROCESS PERFORMANCE TESTS**

Many different tests can be applied to measure the performance of S/S processes. Some of these tests are mandated by federal, state, or local regulations, whereas others can be employed to provide additional assurance that a given S/S process is appropriate for its intended use. Testing can be expensive, especially when applied to a large number of samples and replicates to ensure statistical validity. On the other hand, only adequate testing can ensure attaining the data quality objectives (DQO). Less than necessary testing may lead to an unacceptable S/S-treated waste in a difficult form to reprocess.

This chapter provides a comprehensive review of the types of tests applied to S/S-treated waste. A given test program normally would use only a small subset of the tests mentioned here, depending on the specific performance goals of that test program (Section 2.3).

The purpose of this section is not to describe all possible tests in detail but rather to present an overview of example tests organized by type of test: physical, leaching/extraction, chemical, biological, and microcharacterization. For each category of test, a table lists and briefly describes the representative tests. Where possible, reference has been made to one possible example method. Some of the tests are most applicable to untreated soil or sludge samples, S/S-treated waste, or liquid samples. These material applications are denoted as U, S, or L in the column titled material application. The test may be required by regulation, provide general information about S/S processing, or support an experimental program. These testing applications are noted as R, I, or E in the column titled testing application. Both columns provide general guidance but specific uses will vary depending on testing program design.

U.S. EPA (1989g and 1990b) provides two sources of additional information on performance tests. The test descriptions offered in Sections 3.1 through 3.5 are taken primarily from U.S. EPA (1990b).

#### **3.1 PHYSICAL TESTS**

For the testing of S/S-treated waste, many existing physical and construction tests were adapted. Thus, caution should be exercised when using them to evaluate stabilized wastes. Such tests can be used to differentiate

among the attributes of different binders, to determine compliance with performance objectives, to establish material handling characteristics, and to select large-scale equipment. Table 3-1 gives an overview of physical tests, described below. The testing program should select appropriate tests based on the waste and site conditions and test program objectives (Section 2.7).

### **3.1.1 General Property Tests**

General property tests provide information relating physical characteristics of treated and untreated waste to various process and operational parameters. These tests are often performed to determine the suitability of the wastes to stabilization, to help select binders, or to help design treatability studies.

#### **3.1.1.1 Moisture Content**

Moisture content refers to "free" or "pore" water, not water of hydration. On untreated wastes, moisture content is used to determine the materials handling properties and to determine whether pretreatment (e.g., drying, dewatering) is needed. Moisture content is also used to estimate the need to add water to the S/S binder and to convert waste weights to a dry basis to improve reporting consistency. Moisture content may include some volatiles lost under the conditions of the test.

#### **3.1.1.2 Particle Size Analysis**

The size distribution of the particles in the waste or soil often indicates the potential for water movement through the material and the compressibility. Also, very fine-grained materials have been shown to produce poorly stabilized materials (U.S. EPA, 1986c). Presence of large particles may require the use of size reduction equipment. The best material for forming a strong interlocking matrix is well graded, with few particles in extreme sizes.

#### **3.1.1.3 Specific Gravity**

Specific gravity is the ratio of the mass of the dry solid portion of the waste to the mass of an equivalent volume of water. Specific gravity data are necessary to understand the weight-to-volume (e.g., tons to cubic

TABLE 3-1. PHYSICAL TESTS

Test Procedure	Method	Material Application <sup>(a)</sup>			Purpose	Testing Application <sup>(b)</sup>		
		U	S	L		R	I	E
<u>General Property Tests</u>								
Moisture (water content)	ASTM D 2216-85	X	X		To determine the percentage of free water in a material.		X	
Particle-size analysis (grain size)	ASTM D 422-63	X	X		To determine the particle-size distribution of material.		X	
Specific gravity	ASTM D 854-83	X	X	X	To determine the specific gravity of waste material or S/S-treated waste.		X	
Suspended solids	Standard Method 2540D			X	To determine the amount of solids that do not settle from a column of liquids.		X	
Paint Filter Test	EPA SW-846 Method 9095		X		To determine the presence of free liquids.	X	X	
Liquid Release Test	51 FR 46828		X		To determine the presence of free liquids released under pressure.	X	X	
Atterberg limits (liquid limit, plastic limit, and plasticity index)	ASTM D 4318-84	X	X		To define the bearing capacity and critical slope of a material as a function of its water content.		X	
Visual Observation	U.S. EPA, 1990d		X		To define general condition of the S/S-treated waste.		X	

TABLE 3-1. PHYSICAL TESTS (Continued)

Test Procedure	Method	Material Application <sup>(a)</sup>			Purpose	Testing Application <sup>(b)</sup>		
		U	S	L		R	I	E

<u>Bulk Density Tests</u>								
Bulk density by drive-cylinder method	ASTM D 2937-83	X			To determine in-place density and moisture content.			X
Bulk density by sand cone method	ASTM D 1556-90	X			To determine bulk density.			X
Bulk density by nuclear method	ASTM D 2922-81	X			To determine total, in-place wet density.			X
<u>Compaction Tests</u>								
Moisture-density relationship of soils and soil-aggregate mixtures (Proctor test)	ASTM D 698-78 ASTM D 558-82 ASTM D 1557-78	X			To determine the relation between moisture content and density of a material.			X
<u>Permeability Tests</u>								
Constant-head permeability	EPA SW-846 Method 9100	X	X		To measure the rate at which water will pass through a soil-like material.			X
Falling-head permeability	EPA SW-846 Method 9100	X	X		To measure the rate at which water will pass through a soil-like material.			X

TABLE 3-1. PHYSICAL TESTS (Continued)

Test Procedure	Method	Material Application <sup>(a)</sup>			Purpose	Testing Application <sup>(b)</sup>		
		U	S	L		R	I	E
<u>Porosity</u>								
Mercury intrusion	ASTM C 493-86	X	X		To measure total porosity and pore distribution.			X
Water or mineral spirit displacement	ASTM C 830-88	X	X		To measure apparent porosity and apparent specific gravity.			X
Helium displacement	Hannak and Liem, 1986	X	X		To measure porosity.			X
<u>Strength Tests</u>								
Unconfined compressive strength of cohesive soils	ASTM D 2166-85	X	X		To evaluate how cohesive soil-like materials behave under mechanical stress.	X	X	
Immersion compressive strength test	Kasten et al., 1989, p. 22	X	X		To evaluate a material's strength when wet.		X	X
Unconfined compressive strength of cylindrical cement specimen	ASTM D 1633-84		X		To evaluate how cement-like materials behave under mechanical stress.	X	X	
Compressive strength of hydraulic cement mortars	ASTM C 109-90		X		To measure the compressive strength of hydraulic cement mortars.	X	X	

TABLE 3-1. PHYSICAL TESTS (Continued)

Test Procedure	Method	Material Application <sup>(a)</sup>			Purpose	Testing Application <sup>(b)</sup>		
		U	S	L		R	I	E
Triaxial compressive strength	ASTM D 2850-87	X	X		To measure strength of an unconsolidated laterally-confined material.		X	
Flexural strength	ASTM D 1635-87		X		To evaluate a material's elastic deformation under stress.		X	X
Cone index	ASTM D 3441-86	X	X		To evaluate a material's hardness and setting.		X	
<u>General Concrete/Soil-Cement Tests</u>								
Heat of hydration	ASTM C 186-86		X		To measure temperature changes during mixing in order to predict VOC emissions.			X
Making and curing concrete test specimens in the field	ASTM C 31-90		X		To prepare S/S-treated waste under field conditions.		X	X
Making and curing concrete specimens in the laboratory	ASTM C 192-90		X		To prepare S/S-treated waste under laboratory conditions.		X	X
Bulk density of S/S-treated waste	Stegemann and Côté, 1991		X		To determine bulk density of a monolithic S/S-treated waste.		X	X



TABLE 3-1. PHYSICAL TESTS (Continued)

Test Procedure	Method	Material Application <sup>(a)</sup>			Purpose	Testing Application <sup>(b)</sup>		
		U	S	L		R	I	E
<u>Durability Tests</u>								
Wet/dry weathering (WDW)	ASTM D 4843-88	X			To determine how materials behave or degrade after repeated wet-dry cycles.	X	X	
Freezing and thawing of soil-cement mixtures	ASTM D 4842-90	X			To determine how materials behave or degrade after repeated freeze-thaw cycles.	X	X	
Thermal cycling	ASTM B 553-79	X			To determine the effect of thermal cycling.	X	X	

<sup>(a)</sup> Material Application Guide:

U = Untreated sample  
 S = S/S-treated sample  
 L = Liquid sample

<sup>(b)</sup> Testing Application Guide:

R = Regulatory requirement  
 I = Information for S/S process  
 E = Experimental program

yards) conversion factor for the waste. Specific gravity measurements on waste before and after treatment can be used to calculate the extent of waste volume expansion due to treatment. Specific gravity of insoluble materials can be determined by a water displacement method in which the volume of a waste sample is determined by water displacement in a volumetric flask.

#### **3.1.1.4 Suspended Solids**

The quantity of suspended solids in a mixture is one factor in determining the pumpability of liquid wastes. The decrease in volume of the waste that can be achieved by dewatering also can be estimated based on the suspended solids content.

#### **3.1.1.5 Paint Filter Test**

The Paint Filter Test is mentioned under RCRA in 40 CFR 264.314 and 265.314. This test is used to determine the presence of free liquids in the waste. The Paint Filter Test can be performed before treatment to help determine the degree of treatment needed or after stabilization to determine if the waste may be disposed of in a RCRA-authorized landfill. If the material fails the test, further treatment is required.

#### **3.1.1.6 Liquid Release Test (LRT)**

The Liquid Release Test is also devised to measure free water content. This method uses gas pressure to force a piston against a sample to squeeze any releasable liquid from the material. A special liquid release test apparatus or the zero headspace extraction apparatus, also used in the TCLP test (Section 3.2.1), may be used for the LRT. The U.S. EPA has proposed the LRT as a supplement to the Paint Filter Test (51 FR 46833, December 24, 1986).

#### **3.1.1.7 Atterberg Limits**

Atterberg limits are the boundaries of liquid and plastic consistency states for a soil-like material. Another parameter is the plasticity index, which is the difference in the moisture contents at the liquid and plastic limits. The Atterberg limits indicate general civil engineering

properties of a soil-like material and are used to estimate handling and storage characteristics.

#### **3.1.1.8 Visual Observation**

Careful observation and recording of the general condition of S/S-treated waste give a good indication of the performance of the S/S process. Characteristics to check include surface spalling, grain exfoliation, crack development, color, salt efflorescence, and surface pore size and condition.

#### **3.1.2 Bulk Density Tests**

In situ unit weight, void ratio, and degree of saturation are soil parameters used in most phase relationship, soil pressure, settlement, and stability problems. These parameters help to define the condition or physical makeup of a soil. The unit weight, defined as the ratio of the weight of the mass to the volume of the mass, may be expressed as either a dry, moist, or saturated unit weight. The void ratio equals the ratio of the volume of voids to the volume of solid materials. The porosity of a material, discussed in Section 3.1.5, is related to bulk density. The degree of saturation equals the ratio of the volume of water to the volume of voids, also expressed as a percent.

#### **3.1.3 Compaction Tests**

Moisture-density relationships define the compaction characteristics of a soil. The laboratory compaction test, generally referred to as the Proctor test, identifies the maximum dry unit weight that is achieved by using a specified compactive energy. Compaction tests also identify the optimum moisture content to achieve the maximum dry unit weight.

#### **3.1.4 Permeability (Hydraulic Conductivity) Tests**

Permeability is a measure of flow of a fluid through the tortuous pore structure of the waste or S/S-treated waste. Typical values of stabilized wastes range from  $10^{-4}$  to  $10^{-8}$  cm/s (U.S. EPA, 1989g). This can be compared to clay (used for liners), which is typically less than  $10^{-6}$  cm/s. A value of  $<10^{-5}$  cm/s is recommended for stabilized wastes planned for land burial (U.S. EPA, 1986c). However, high permeability is not as great a

problem if the contaminants in the waste do not easily leach to water. High permeability can also be addressed through engineering solutions (U.S. EPA, 1989g). It may be advisable in some cases to perform a permeability test on samples that have already undergone durability testing to determine whether this property changes under environmental stresses.

### **3.1.5 Porosity Tests**

The porosity indicates the void space in the solid that may or may not be available to retain liquids. The methods available for measuring porosity are based on determining the volume of fluid that can be forced into the pores. Each fluid has unique strengths and weaknesses. Mercury (ASTM C 493) is unlikely to dissolve the solid, but high pressure is needed to push mercury into the pores, possibly altering the pore size. Water- or mineral oil-based methods (ASTM C 830) use lower pressure, but the fluid may dissolve part of the solid. Using helium as the displacement fluid (Hannak and Liem, 1986) avoids both high pressure and dissolution. However, helium is a more penetrating fluid than water, so helium intrusion can overestimate the effective water porosity.

### **3.1.6 Strength Tests**

Strength testing indicates how well a material will hold up under mechanical stresses caused by overburden or earth-moving equipment. Strength testing is usually done on the stabilized waste, although testing the untreated waste can provide a baseline. A common mistake in S/S is to equate treated waste strength with the degree of contaminant stabilization. A correlation between strength testing and contaminant leachability has not been established. However, in general, better strength provides better physical barriers for the containment of contaminants.

#### **3.1.6.1 Unconfined Compressive Strength (UCS)**

The UCS test measures the shear strength of a material without lateral confinement. It is applicable to cohesive soil-like materials that do not release water during loading (ASTM D 2166) or to molded cylinders (ASTM D 1633 or C 109). It is not applicable to crumbly or fissured materials. The

ASTM D 1633 or C 109 tests for various binder mixes can also indicate the optimum water/additive ratios and curing times for the setting reaction.

For ASTM D 2166, the U.S. EPA generally considers a stabilized material as satisfactory if it has a compressive strength of at least 50 psi. However, the minimum required strength should be determined from the design loads to which the material may be subjected. Overburden pressures are generally around 1 psi per foot of depth.

Variations of these methods, such as the one-dimensional stability test (ASTM D 2435) and compressive strength of hydraulic cement mortars (ASTM C 109), are sometimes used.

#### **3.1.6.2 Immersion Compressive Strength**

Soil and soil-like materials can exhibit good strength when dry and yet become unconsolidated when saturated with liquid. In the immersion compressive strength test, a sample is soaked in water prior to compressive loading to simulate performance in a saturated disposal environment (Kasten et al., 1989, p. 22).

#### **3.1.6.3 Triaxial Compression**

The triaxial compression test determines the strength of a specimen encased in an impervious membrane and axially loaded to failure in compression. Triaxial compression testing is applied to unconsolidated soil and granular S/S-treated waste.

#### **3.1.6.4 Flexural Strength**

In contrast to the UCS, in the flexural strength test, loads are applied on the short axis of the sample. This test gives a measure of a material's ability to withstand tension or its resistance to cracking due either to settlement of the underlying fill or to surface loads (U.S. EPA, 1989g).

#### **3.1.6.5 Cone Index**

The cone index test is a quick screening evaluation for compressive strength (Myers, 1986). This test involves forcing a standard cone- or needle-shaped device into the stabilized waste and measuring the penetration

resistance. Three types of cones are available: the U.S. Army, pocket, or ASTM. Selection depends on the strength of the material and the application. The cone index test can be used instead of the UCS sometimes, but not exclusively, if results are required quickly. This test indicates the stability and load-bearing capacity of the stabilized waste (Cullinane and Jones, 1992). It can be used to determine the kind of earth-moving equipment needed to move the stabilized waste and the curing time required before other construction equipment can move over the stabilized waste (U.S. EPA, 1989g).

### **3.1.7 General Concrete/Soil-Cement Tests**

The test methods used to determine the heat of hydration and other factors involved in making and curing concrete test specimens can be used to evaluate the performance of cement/waste mixtures. Heat of hydration can be a useful measurement, particularly when the waste contains volatile organic compounds (VOCs). Although the standard test intervals for industrial applications are 7 and 28 days, it is recommended that the heat of hydration be measured at more frequent intervals for S/S testing.

In addition to the heat of hydration procedure, it also can be useful to prepare and cure samples of the S/S-treated waste under both field and laboratory conditions. In this way, the S/S-treated waste can be measured for physical parameters after having been subjected to realistic environmental variables. In the laboratory, process variables can be varied and controlled to simulate a wide variety of environmental conditions. Preparing and curing S/S specimens under a variety of potential environmental conditions makes it possible to apply the durability tests described in Section 3.1.8 and to assess the effects of both the curing process and the environmental factors in relation to the ultimate integrity of the S/S waste.

### **3.1.8 Durability Testing**

Durability testing evaluates the ability of a material to withstand environmental stresses such as freezing and thawing (ASTM D 4842) or wetting and drying (ASTM D 4843). Weight loss or the number of such cycles that the material can withstand without failing is an indication of its physical stability. Other performance tests such as UCS, flexural strength, and permeability can be conducted on the material after each cycle to determine

the change in performance due to climatic stresses. No standards have been established for determining acceptance after durability testing, in part because the tests are accelerated and calibration to real disposal environments has not yet been achieved. Hence, the test is more useful for comparing one stabilization process with another (U.S. EPA, 1989g). Engineering design can be used to address stabilized wastes with poor durability.

### **3.2 LEACHING/EXTRACTION TESTS**

The performance of stabilized wastes is generally measured in terms of leaching and extraction tests. A number of different leaching tests are available, and one or more may be required for regulatory approval. However, no single test program would use more than two or three of the leaching/extraction tests described below.

Leaching tests measure the potential of a stabilized waste to release contaminants to the environment. In all tests, the waste is exposed to a leachant and the amount of contaminant in the leachate (or extract) is measured and compared to a previously established standard, which may be a regulatory standard or baseline leaching data for the untreated waste. When using leaching tests to evaluate immobilization performance of S/S-treatment, potential effects of the reduction in contaminant concentration per unit mass of waste due to binder addition should be considered. The treated waste may give reduced contaminant concentration in the leachate due to waste dilution independent of any immobilization mechanism. Table 3-2 lists a number of the leaching tests that can be done to evaluate stabilization, along with information about the standard method, regulatory requirement, and purpose of each test. The extraction conditions of the leaching/extraction tests are summarized in Table 3-3 and Sections 3.2.1 through 3.2.16. Note that none of the tests described in this section have actually been field validated to verify the prediction of contaminant release.

As illustrated in Figure 4-1, the physical strength of cement-based S/S-treated waste develops over a period of several days. Many of the chemical reactions that cause immobilization occur more rapidly. Therefore, curing a sample for 28 days is not as critical for leaching tests, particularly those that require sample size reduction.

Many leaching tests require sample size reduction. The major issues in selecting a size reduction approach are (1) avoid contamination of the

TABLE 3-2. LEACHING/EXTRACTION TESTS

Test Procedure	Method	Material Application <sup>(a)</sup>			Purpose	Testing Application		
		U	S	L		R	I	E
Toxicity Characteristic Leaching Procedure (TCLP)	EPA SW-846 Method 1311	X	X	X	To compare toxicity data with regulatory level. Includes VOCs. RCRA requirement.	X	X	
Extraction Procedure Toxicity (EP Tox) Test	EPA SW-846 Method 1310	X	X	X	To evaluate leachate concentrations. RCRA requirement.	X	X	
TCLP "Cage" Modification	53 FR 18792		X		Adds qualitative evaluation of stability to TCLP test. Proposed RCRA requirement.		X	X
California Waste Extraction Test (Cal Wet)	California Code Title 22, Article 11, pp. 1800.75-1800.82	X	X	X	To provide a more stringent leaching test for metals than TCLP. California requirement.	X	X	
Multiple Extraction Procedure (MEP)	EPA SW-846 Method 1320	X	X	X	To evaluate waste leaching under acid conditions.		X	X
Synthetic Acid Precipitation Leach Test	EPA SW-846 Method 1312	X	X		For waste exposed to acid rain. For comment as RCRA requirement.		X	X
Monofilled Waste Extraction Procedure (MWEPP)	SW-924		X		For waste disposed in low-velocity saturated zone.		X	X
American Nuclear Society Leach Test	ANSI/ANS-16.1		X		To establish a diffusion coefficient for comparison of S/S-treated waste. NRC requirement.	X	X	



TABLE 3-2. LEACHING/EXTRACTION TESTS (Continued)

Test Procedure	Method	Material Application <sup>(a)</sup>			Purpose	Testing Application		
		U	S	L		R	I	E
Dynamic Leach Test	WTC, 1991, p. 17		X		To estimate diffusion coefficient for an S/S-treated waste.	X	X	
Shake Extraction Test	ASTM D 3987-85	X	X		To provide a rapid means of obtaining an aqueous extract.	X	X	
Equilibrium Leach Test (ELT)	WTC, 1991, p. 16	X	X		To evaluate maximum leachate concentrations.	X	X	
Sequential Extraction Test (SET)	Bishop, 1986, p. 240	X	X		To evaluate buffering capacity with multiple extractions.	X	X	
Sequential Chemical Extraction (SCE)	WTC, 1991, p. 17	X	X		To evaluate bonding nature of metals and organics in the S/S-treated waste.	X	X	
Static Leach Test (Ambient or High Temperature)	MCC-1P, MCC-2P		X		To evaluate the leach resistance of a bulk specimen in static fluid.	X	X	
Agitated Powder Leach Test	MCC-3S	X	X		To evaluate the leach resistance of a powdered specimen in agitated fluid.	X	X	
Soxhlet Leach Test	MCC-5S		X		To evaluate the leach resistance of a bulk specimen in constantly refreshed pure leachant, typically at elevated temperature.	X	X	

<sup>(a)</sup> Material Application Guide:  
 U = Untreated samples  
 S = S/S-treated sample  
 L = Aqueous sample

<sup>(b)</sup> Testing Application Guide:  
 R = Regulatory requirement  
 I = Information for S/S process  
 E = Experimental program

TABLE 3-3. EXTRACTION CONDITIONS

Test Method	Leaching Medium	Liquid:Solid Ratio	Particle Size	Number of Extractions	Time of Extractions
TCLP	Acetate buffer <sup>(a)</sup>	20:1	< 9.5 mm	1	18 hours
EP Toxicity	0.04 M acetic acid (pH = 5.0)	20:1	< 9.5 mm	1	24 hours
TCLP "Cage" Modification	Acetate buffer <sup>(a)</sup>	20:1	<sup>(b)</sup>	1	18 hours
Cal WET	0.2 M sodium citrate (pH = 5.0) or water for hexavalent chromium	10:1	< 2.0 mm	1	48 hours
Multiple Extraction Procedure	Same as EP Tox, then with sulfuric acid: nitric acid in 60:40 weight ratio adjusted to pH 3.0	20:1	< 9.5 mm	9 (or more)	24 hours per extraction
Synthetic acid precipitation leach test	<sup>(c)</sup>	20:1	< 9.5 mm	1	18 hours
Monofilled Waste Extraction Procedure	Deionized water or other for specific site	10:1 per extraction	< 9.5 mm or monolith	4	18 hours per extraction
ANSI/ANS/16.1	Deionized water	Volume-to-surface ratio of 10 cm	Monolith length-to-diameter ratio between 0.2 and 5.0	12	Leachant renewed at 2.7 hours; 1, 2, 3, 4, 5, 14, 28, 43, and 90 days

TABLE 3-3. EXTRACTION CONDITIONS (Continued)

Test Method	Leaching Medium	Liquid:Solid Ratio	Particle Size	Number of Extractions	Time of Extractions
Dynamic leach test	Deionized water	(d)	Monolith length- to-diameter ratio between 0.2 and 5.0	(d)	Leachant renewed at 0, 1, 4, 7, 24, 31, 48, 72, 79, and 100 hours; or for more immobile species, at 0, 4, 24, 31, 72, 104, 168, and 196 hours
Shake extraction test ASTM D 3987-85	Deionized water	20:1	Particulate or monolith as received	1	18 hours
Equilibrium leach test	Deionized water	4:1	< 150 $\mu\text{m}$	1	7 days
Sequential extraction test	0.04 M acetic acid	50:1	< 9.5 mm	15	24 hours per extraction
Sequential chemical extraction	Five leaching solutions increasing in acidity	Varies from 16:1 to 40:1	< 45 $\mu\text{m}$	5	Varies from 2 to 24 hours
Static leach test MCC-1P and MCC-2P	(e)	(f)	Monolith	1	Samples for each of 3, 7, 14, 28, 56, 91, 182, and 364 days plus optional 12-month intervals

TABLE 3-3. EXTRACTION CONDITIONS (Continued)

Test Method	Leaching Medium	Liquid:Solid Ratio	Particle Size	Number of Extractions	Time of Extractions
Agitated powder leach test MCC-3S	(e)	10:1	50% <0.044 mm 50% between 0.074 and 0.149 mm	1	Samples for each of 28, 56, 91, 182, 273, and 364 days plus optional 12-month intervals
Soxhlet leach test MCC-55	Deionized water	Continuous flow of redistilled water	Monolith	1	Samples for each of 3, 7, and 14 days

- (a) Either an acetate buffered solution with pH  $\approx$  5 or acetic acid with pH  $\approx$  3.0.
- (b) Monolith tumbled in wire cage during TCLP type extraction.
- (c) Sulfuric acid:nitric acid in 60:40 weight percent mix. pH adjusted with deionized water to 4.2 or 5.0 for site east or west of the Mississippi River, respectively.
- (d) The renewal frequencies are selected based on a known diffusion coefficient. The surface-to-volume ratio must be selected to ensure the contaminant is detected. The renewal frequency must be selected to ensure nonequilibrium conditions prevail.
- (e) Silicate water, deionized water, brine, or repository water.
- (f) The volume of leachant is based on the measured geometric surface area of the sample. The volume-to-surface ratio must be between 10 and 200 cm.

sample, (2) avoid partitioning of contaminants into a specific size fraction, and (3) avoid loss of contaminants, particularly volatile organics. The typical steps in size reduction are sample fragmentation, grinding, and sizing. Fragmentation is best done with a hammer and anvil and should be minimized to avoid metal contamination of the waste. Grinding can be done with agate, dense alumina or tungsten-carbide equipment. Mortar and pestle or mechanical grinder can be selected based on the sample throughput of the laboratory. Sizing should be done with nylon or other nonmetal screens.

### **3.2.1 Toxicity Characteristic Leaching Procedure (TCLP)**

In the TCLP test, waste samples are crushed to particle size less than 9.5 mm and extracted with an acetate buffer solution with a pH of 5 or an acetic acid solution with a pH of 3, depending on the alkalinity of the waste. Note, however, that the TCLP leachate is poorly buffered and that pH of the leachate upon contact with the waste may be much greater, as high as pH 10-11 or more, depending on the initial alkalinity of the waste. The acetate buffer is added only once at the start of the extraction. A liquid-to-solid ratio of 20:1 is used for an extraction period of 18 hours. The leachate is filtered prior to conducting the contaminant analyses. This test is used to evaluate the leaching of metals, volatile and semivolatile organic compounds, and pesticides from wastes that are categorized under RCRA as characteristically toxic and can be used on other wastes as well.

The TCLP test has been most commonly used by U.S. EPA and state agencies to evaluate the leaching potential of stabilized wastes, and TCLP is the test required by RCRA implementing regulations (40 CFR Part 261) for determining toxicity. Measurement of pH in the extract can help elucidate the pH-dependence of contaminant leaching.

The TCLP does not provide data on long-term stability (see Section 4.7). In fact, recent studies show a significant effect of curing time on both TCLP results and the chemical structure of the stabilized waste, as evidenced by spectroscopic analyses (Akhter and Cartledge, 1971; Cartledge, 1992). These observations underline the limitations of the TCLP test as an indicator of the long-term leaching of stabilized waste and emphasize the need for other types of leaching data.

### **3.2.2 Extraction Procedure Toxicity (EP Tox) Test**

The EP Tox test is the precursor of and is similar to the TCLP. Only one concentration of acetic acid solution (pH of 5) is used. The liquid-to-solid ratio starts at 16:1 and may increase as additional acid solution is added as needed to adjust the pH during the 24-hour test duration. Results of the EP Tox test are generally comparable to results of TCLP tests at pH 5 but may differ significantly at pH 3. As with the TCLP, the measurement of pH in the extract can help determine the pH-dependence of contaminant leaching. EP Tox cannot be used to assess volatiles.

### **3.2.3 TCLP "Cage" Modification**

The standard TCLP (Section 3.2.1) requires that all samples be passed through a 9.5-mm screen (or meet surface area requirements) before leaching. However, this requirement may not be appropriate for S/S-treated wastes that have been solidified to withstand the environmental stresses encountered in a landfill. Studies in 1988 (53 FR 18792) using a modification wherein the S/S-treated waste was tumbled in a cage indicated that well-stabilized wastes may remain more or less intact, whereas poorly stabilized wastes are significantly degraded. TCLP "cage" modification, proposed as a modified TCLP, requires no preliminary size reduction of samples. The resulting leachate can be used for analytical determinations of organics and metals.

The TCLP "cage" modification is still under development and currently has no regulatory status.

### **3.2.4 California Waste Extraction Test (Cal WET)**

The Cal WET is used by the State of California to classify hazardous wastes. The leachate is a sodium citrate buffer, the liquid-to-solids ratio is 10:1, and testing lasts 48 hours. The Cal WET test applies a soluble threshold limit concentration (STLC) as the regulatory standard. STLC standards for metal concentrations in the leachate are similar to those for the TCLP. However, California regulates several additional metals, such as copper, beryllium, nickel, and zinc, and a number of organic compounds, such as PCBs and pesticides. The Cal WET test also develops a Total Threshold Limit Concentration (TTLC) which is equivalent to a Total Waste Analysis

(TWA). The TWA gives the concentration of priority pollutants, organics, metals, and other substances of interest in the waste.

The Cal WET is a much more aggressive test than either the TCLP or EP Tox and almost always extracts higher levels of contaminants. This aggressive characteristic of the Cal WET has led to the development of a category of hazardous waste specific to the State of California, referred to as "California-only" hazardous waste. This specifically refers to a waste that fails the Cal WET but passes the TCLP. If the waste fails both the Cal WET and the TCLP, then the requirements of both California and the U.S. EPA must be met.

### **3.2.5 Multiple Extraction Procedure (MEP)**

Like the EP Tox, the MEP involves a first extraction with acetic acid, followed by at least eight extractions with a synthetic acid rain solution (sulfuric/nitric acid adjusted to pH 3). The MEP is intended to simulate leaching in an improperly designed landfill where the waste could come into contact with large volumes of acidic leachate. One advantage of the MEP over the TCLP is that the MEP gradually removes excess alkalinity in the waste over time. Thus, the leaching behavior of the contaminants (particularly metal contaminants) can be evaluated as a function of decreasing pH, where the solubility of most metals increases.

The MEP has been used in the regulatory environment for delisting U.S. EPA-listed wastes.

### **3.2.6 Synthetic Acid Precipitation Leach Test**

The TCLP (Section 3.2.1) and the EP Tox test (Section 3.2.2) apply to disposal in a sanitary or municipal landfill, a scenario that does not match the disposal setting of many S/S-treated wastes. A sanitary landfill environment is characterized by large concentrations of low-molecular-weight organic acids, such as acetic acid, that result from anaerobic fermentation of organic waste. The Synthetic Acid Precipitation Leach Test is similar to the TCLP, but the initial liquid-solid separation step has been eliminated and the acetate buffer extraction fluid has been replaced by a dilute nitric acid/sulfuric acid mixture. The Synthetic Acid Precipitation Test simulates acid rain as opposed to simulating a leachate in a sanitary or municipal landfill.

### **3.2.7 Monofilled Waste Extraction Procedure (MWEP)**

The MWEP involves multiple extractions of a monolith or of crushed waste with distilled/deionized water. The sample is crushed to less than 9.5 mm, or it can be left intact if it passes the U.S. EPA SW-846 Structural Integrity Test. The liquid-to-solid ratio is 10:1, and the sample is extracted with water four times at 18 hours per extraction. The MWEP is intended to derive leachate compositions in monofilled disposal facilities or to obtain leachate for testing the compatibility of lining materials with the leachate. Note that this procedure has not yet been approved by EPA.

### **3.2.8 American Nuclear Society Leach Test (ANSI/ANS/16.1)**

The ANSI/ANS/16.1 leaching test is intended mainly to develop a figure-of-merit for comparing the leaching resistance of S/S-treated waste. The results of the leaching tests are recorded in terms of cumulative fraction leached relative to the total mass of the waste sample. Then, results can be used to derive an effective diffusion coefficient and a leachability index, or figure-of-merit. The ANSI/ANS/16.1 is conducted over a period of 90 days and is intended to indicate contaminant release rate, unlike the batch tests described in preceding sections. Typically, the leachant is distilled water, but other solutions, such as simulated groundwater, may also be used.

### **3.2.9 Dynamic Leach Test (DLT)**

The DLT is a modified version of the ANSI/ANS/16.1 test (Section 3.2.8). The renewal frequency of the leaching solution and the leaching volume-to-solid ratio are adjusted based on an estimated or calculated diffusion coefficient and results from batch extraction tests such as the Equilibrium Leach Test (ELT) (Section 3.2.11). The solution renewal frequency is chosen to ensure that equilibrium has not been reached. The leaching volume-to-solid ratio is chosen to ensure that the contaminant can be detected. Data from the DLT can be used to determine a diffusion coefficient that can be used to predict long-term leaching performance (Stegemann and Côté, 1991). Like all the tests described in this section, field validation has not yet been done to verify the leaching prediction.



### **3.2.10 Shake Extraction Test**

The shake extraction test is applicable only to inorganic compounds. It involves the extraction of a solid waste with Type IV reagent water in a rotary agitator for 18 hours. The procedure is intended as a rapid means of obtaining an aqueous extract and is not intended to simulate site-specific leaching conditions.

### **3.2.11 Equilibrium Leach Test (ELT)**

The ELT involves static leaching of hazardous constituents by distilled water. The particle size of the crushed sample ( $\leq 150 \mu\text{m}$ ) is much smaller than that for TCLP and EP Tox to allow greater contact surface area and to reduce the time needed to achieve equilibrium. Water is added once at a liquid-to-solid ratio of 4:1, and the sample is agitated for 7 days. Like MWEF (Section 3.2.7), ELT can be used to determine equilibrium leachate concentrations under mild leaching conditions.

### **3.2.12 Sequential Extraction Test (SET)**

The SET is used to evaluate the waste buffering capacity and alkalinity of cement-based S/S-treated waste. Unlike acid neutralization capacity (Section 3.3.7), the SET involves 15 sequential extractions of one sample of crushed waste with particle sizes between 2.0 and 9.5 mm. Each extraction is performed on a shaker table for 24 hours with the same type of extraction solution (0.04 M acetic acid solution) and liquid-to-solid ratio of 50:1. With each extraction, 2 meq/g of acid is added to the ground waste. The pH is measured and the leaching solution is filtered. After the fifteenth extraction, the remaining solids are digested with three more extractions in which more concentrated acid solutions are used. These last three extractions are combined for analysis.

### **3.2.13 Sequential Chemical Extraction (SCE)**

The objective of the SCE test is to evaluate the nature and bonding strength of metals and organics in S/S-treated waste. This test was originally developed for sediments and adapted to evaluate inorganic waste constituents in a stabilized matrix. Like SET, the test involves sequential extraction of a sample. Unlike SET, however, the leaching solution increases in

acidity from neutral to very acidic with each sequential extraction. The particle size of the sample is also very small (less than 45  $\mu\text{m}$ ).

#### **3.2.14 Static Leach Test Method (Ambient- and High-Temperature)**

The Materials Characterization Center (MCC) at Pacific Northwest Laboratory (PNL), under a project for the United States Department of Energy (U.S. DOE) developed the static ambient- and high-temperature leach tests as part of a series of standard methods designed to evaluate the chemical durability of S/S-treated nuclear waste. The static leach tests use representative, monolithic specimens of the S/S-treated waste. Specimens of known geometric surface area are immersed in a reference leachant held at a specified temperature. The immersion period can vary from 3 days to many years. Temperatures ranging from 40°C to 190°C are used. The leachant is not agitated during the immersion period. This test is used to evaluate the leach resistance of monolithic S/S-treated waste.

#### **3.2.15 Agitated Powder Leach Test Method**

The MCC also developed the agitated powder leach test as part of a series of standard methods designed to evaluate the chemical durability of nuclear waste forms. The agitated leach test uses representative powdered waste specimens of the waste form. The powder is immersed in a reference leachant at a constant ratio of leachant volume to specimen mass of 10 ml/g. Test temperatures range from 40°C to 190°C. The powder and leachant are agitated by constant rolling of the specimen holder. The test is used to determine the maximum concentration of chemical elements in solution from the waste form under steady-state conditions, in closed, agitated systems.

#### **3.2.16 Soxhlet Leach Test Method**

The MCC developed the Soxhlet leach test as part of a series of standard methods designed to evaluate the chemical durability of S/S-treated nuclear waste. Although designed primarily for glass and ceramic waste forms, the Soxhlet leach test is applicable to any monolithic S/S-treated waste and the individual components of macroscale physical composite S/S-treated waste. Monolithic specimens of known geometric surface area are suspended in a continuously flowing stream of redistilled water. The precise test temperature

is determined by the barometric pressure in the laboratory but is near 100°C. The test measures the normalized mass losses from the specimen due to a constant flow of redistilled water at its boiling point under local conditions.

### **3.3 CHEMICAL TESTS AND ANALYSES**

Treatability testing usually involves collecting chemical data to define waste compositions and to assess binder performance. Table 3-4 describes a number of these chemical parameters and their applicability to evaluating untreated waste, S/S-treated waste, and aqueous samples. Total waste analyses of metals, VOCs, and BNAs can be applied to characterize untreated waste, S/S-treated waste, or leachate. Other chemical tests may be needed for optional information or to support research. For example, it may be necessary to screen for chemicals that interfere with S/S treatment, if historical information or other sources of information indicate that such chemicals may be present. The chemical test program should be developed based on specific waste and site characteristics.

#### **3.3.1 pH**

The pH is a measure of the hydrogen ion activity and indicates the acid-to-base balance of a material. The pH of untreated and S/S-treated wastes, waste leachates, or soils from the intended disposal site can be analyzed by U.S. EPA SW-846 Method 9045. Equal weights of soils/solids and deionized water are mixed and allowed to settle for 1 hour. The pH of the supernatant liquid is then measured electrometrically. The leachability of many metals is a function of the pH. The pH may also affect the leachability of some base, neutral, and acid (BNA) fraction compounds.

#### **3.3.2 Oxidation/Reduction Potential (Eh)**

The oxidation/reduction potential, Eh, characterizes the electrochemical state of the media being measured. Data on the Eh of the untreated or treated waste, waste leachates, or soils from the intended disposal site can be very useful. The Eh can be determined by ASTM D 1498. Numerous metals can exist in multiple oxidation states. For example, chromium can exist as Cr(III) or Cr(VI) and arsenic as As(III) or As(V). The leachability of these

TABLE 3-4. CHEMICAL TESTS

Test Procedure	Method	Material Application			Purpose	Testing Application		
		U	S	L		R	I	E
pH (liquid)	EPA SW-846 Method 9040			X	To determine solution pH. pH of leachate and aqueous phase of disposal environment can help estimate metal leaching tendency.	X	X	X
pH (solid)	EPA SW-846 Method 9045	X	X		Leachability of hazardous constituents (e.g., metals) may be governed by the pH. RCRA corrosivity limits are pH 2 and 12.5.	X	X	X
Oxidation/reduction potential (Eh)	ASTM D 1498-76			X	Leachate Eh can indicate potential stability of chemical species. Eh of leachate and aqueous phase of disposal environment can help estimate metal leaching tendency.			X
Major oxide components	ASTM C 114-88			X	Mineralogy of the stabilized/solidified waste may aid in interpretation of leach test results.	X	X	
Total organic carbon (TOC)	EPA SW-846 Method 9060	X	X	X	Used to approximate the nonpurgeable organic carbon in wastes and treated solids. Useful in approximating interference levels.		X	
Oil and grease - sludge	EPA SW-846 Method 9071	X	X		May be used to compare the leachable oil and grease from the treated and untreated wastes. Also a possible interference.	X		
- fluids	EPA SW-846 Method 9070			X				

TABLE 3-4. CHEMICAL TESTS (Continued)

Test Procedure	Method	<u>Material Application</u>			Purpose	<u>Testing Application</u>		
		U	S	L		R	I	E
Electrical conductivity	EPA Method 120.1 EPA SW-846 Method 9050			X	To compare ion concentrations in leachate to ion concentrations in receiving waters.			X
Acid neutralization capacity (ANC)	WTC, 1991, p. 16	X	X		To determine pH buffering capacity of S/S-treated waste.			X
Generalized Acid Neutralization Capacity (GANC)	Isenburg and Moore, 1990	X	X		To determine pH buffering capacity of S/S treated waste.			X
Alkalinity	EPA Method 403			X	To indicate the ability of a solution to neutralize acid.	X	X	
Total dissolved solids (TDS)	EPA Method 209B			X	To measure dissolve solids content of leachate.	X	X	
Reactive cyanide	EPA SW-846 Section 7.3.3.2	X	X	X	To determine potential for generation of hazardous fumes. RCRA regulatory requirement, 250 mg HCN/kg guideline.	X	X	X
Reactive sulfide	EPA SW-846 Section 7.3.4.1	X	X	X	To determine potential for generation of hazardous fumes. RCRA regulatory requirement, 500 mg/H <sub>2</sub> S/kg guideline.	X	X	X
Reactivity of silica aggregates	ASTM C 289-87	X			To evaluate potential alkali-silica reaction in aggregates.		X	X
Metals analysis	EPA SW-846 (Methods 3010, 3020, 3050, and 6010 or 7000)	X	X	X	Used to define metals content of untreated and treated wastes or TCLP leachates of such wastes for numerous specific metals.	X	X	X

TABLE 3-4. CHEMICAL TESTS (Continued)

Test Procedure	Method	<u>Material Application</u>			Purpose	<u>Testing Application</u>		
		U	S	L		R	I	E
Volatile organic compounds (VOCs)	EPA SW-846 Methods 5030 and 8240	X	X	X	Used to define VOC concentrations in S/S-treated wastes and untreated wastes or in waste extracts.	X	X	X
Base, neutral and acid (BNA) organic compounds	EPA SW-846 Methods 3510, 3520, 3540, and 8270	X	X	X	Used to define BNA concentrations of wastes or waste leachates or extracts from treated or untreated wastes.	X	X	X
Polychlorinated biphenyls (PCBs)	EPA SW-846 Methods 3540, 3520, and 8080; EPA Method 608	X	X	X	Used to define PCB concentrations of wastes or waste extracts in treated or untreated wastes.	X	X	X
Mercury	EPA SW-846 Method 7470			X	Determine Hg content in waste or waste leachates.	X	X	X
Pesticides	EPA SW-846 Methods 3510 and 8080 or 8081			X	Determine pesticide content in waste or waste leachates.	X	X	X
Herbicides	EPA SW-846 Method 8150 or 8151			X	Determine herbicide content in waste or waste leachates.	X	X	X
Ion measurements	Std. Method No. 4110			X	Used to determine leachate anionic species concentrations in aqueous solutions.			X

TABLE 3-4. CHEMICAL TESTS (Continued)

Test Procedure	Method	Material	Purpose	Testing
		Application		Application
		U S L		R I E
<u>Interferants Screen</u>			Used to screen for the presence of elements that could adversely affect S/S process or performance.	X X
Oil and Grease	EPA SW-846 Method 9071	X X X		
Potassium	EPA SW-846 Method 3050, and 6010	X X X		
Sodium	EPA SW-846 Method 3050 and 6010	X X X		
Fluoride	EPA Method 300.0	(a) (a) X		
Chloride	EPA Method 300.0	(a) (a) X		
Orthophosphate	EPA Method 365.1	(a) (a) X		
Ammonia	EPA Method 350.2	(a) (a) X		
Nitrate	EPA Method 300.0	(a) (a) X		
Sulfate	EPA Method 300.0	(a) (a) X		

(a) Extraction or digestion required.

## Material Application Guide:

U = Untreated Samples

S = S/S-treated Samples

L = Liquid Samples

## Testing Application Guide:

R = Regulatory Requirement

I = Information for S/S Process

E = Experimental Program

metals depends on their oxidation state. Therefore, Eh can indicate the stability of various chemical species in the waste's chemical environment.

### **3.3.3 Major Oxide Components**

The major oxide components can be used to characterize the mineralogy of the S/S-treated waste. Analytical techniques for determining  $\text{SiO}_2$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{MgO}$ , and loss on ignition are described in ASTM C 114. Between 10 and 30% of cementitious solids will be in the form of oxides.

### **3.3.4 Total Organic Carbon (TOC)**

The TOC analysis measures the overall level of organic compounds present in a liquid, sludge, or solid sample. TOC is measured by U.S. EPA SW-846 Method 9060. This method uses combustion with infrared, thermoconductivity, or other detection. The TOC results can be used to approximate the levels of nonpurgeable organic carbon and to estimate the potential for organic interference in the S/S process.

### **3.3.5 Oil and Grease**

Oil and grease analysis determines the total content of oil and grease in a sample. This analysis can be done by U.S. EPA SW-846 Method 9070 or 9071. The determination before and after treatment provides a method of assessing the effectiveness of the S/S process in immobilizing oil and grease in the waste. Oil and grease analysis of asphaltic solid leachates is important for determining whether the S/S process aids in stabilizing oil and grease or whether the asphalt increases oil and grease leachability. In addition, oil and grease interfere with cement or pozzolan-based S/S treatment.

### **3.3.6 Electrical Conductivity**

The electrical conductivity of a solution is a measure of its ability to carry current. Conductivity varies with the concentration and type of ions present. Solution conductivity can be measured by U.S. EPA Method 120.1 or U.S. EPA SW-846 Method 9050. Conductivity of leachates from untreated and S/S-treated wastes can be compared to find the relative ionic concentrations in the two solutions. In addition, test results from untreated and S/S-treated waste leachates can be compared with conductivities of natural



surface and subsurface waters in the vicinity of the demonstration site and/or potential disposal site. Wide differences in the conductivity of leachate and natural waters create the potential for the waste leachate to cause conductivity fluctuations in adjacent receiving waters.

### **3.3.7 Acid Neutralization Capacity (ANC and GANC)**

These buffering capacity tests indicate the capacity of the S/S-treated waste to maintain an elevated pH when exposed to acidic solutions. The ANC test involves separate extraction of S/S-treated waste samples with leaching solutions of varying levels of acidity. Ten waste samples are predried and crushed to a particle size of -100 mesh. Each sample is extracted for 24 hours in one of 10 nitric acid solutions. The acid equivalents per gram of solid increases incrementally from sample 1 to sample 10. Following the extraction, the pH of each solution is measured. The amount of decrease in pH of the leach solutions with each increase in acid concentration indicates the buffering capacity of the S/S-treated waste. Smaller decreases indicate higher buffering capacity. The higher the buffering capacity, the greater the possibility of maintaining alkaline conditions conducive to metal retention. The GANC is a similar test developed to be consistent with the TCLP test (Isenburg and Moore, 1992).

### **3.3.8 Alkalinity**

Alkalinity indicates the capacity of a solution such as a leachate to neutralize acid solutions to specific pH levels. It can be measured by U.S. EPA Method 403.

### **3.3.9 Total Dissolved Solids (TDS)**

The TDS analysis indicates the total quantity of solid material dissolved in a solution. It can be measured by U.S. EPA Method 209B. The TDS levels in leaching solutions can be used to track the degradation of S/S-treated waste solid or leaching of constituents from the sample. TDS is also a drinking water standard.

### **3.3.10 Reactive Cyanide and Sulfide**

The analyses for reactive cyanide and sulfide apply to waste containing cyanide- or sulfide-bearing material. Sulfide can be present in the waste either as a natural waste constituent or as a binder additive. If waste exposed to a pH in the range of 2 to 12.5 can generate toxic gases, vapors, or fumes in sufficient quantity to present a danger to human health or the environment, it is deemed to contain reactive cyanide or sulfide. The tests for reactive cyanide and sulfide are described in U.S. EPA SW-846 (1986c) Section 7.3. Testing for reactive cyanide and sulfide may be required for some RCRA wastes under regulation 40 CFR 261.23-(a)(5).

### **3.3.11 Reactivity of Silica Aggregates**

The test for reactivity of silica aggregates measures the propensity of silica in the waste to react with alkaline components of Portland cement/concrete mixtures or similar S/S binders. The potential for silica in suspect aggregates to react with alkaline compounds is determined by ASTM C 289. Reactive silica and alkaline compounds combine to form silicate-alkali gels that expand to cause internal stress in the S/S-treated waste. The internal stress can result in cracking or spalling.

### **3.3.12 Metal Analysis**

Metal analyses can be applied to aqueous leach solutions to determine the concentrations of metals leached from the S/S-treated waste. Metal analysis tests can also be used, following a suitable strong acid digestion step, to measure the total metal concentrations in the untreated or S/S-treated waste. Metals can be determined in accordance with U.S. EPA SW-846 Methods 6010 (analysis by inductively coupled plasma atomic emission spectroscopy [ICP]) or 7000 and associated 7000 series methods (analysis by atomic absorption spectroscopy [AA]). The material should be pretreated with the appropriate digestion procedure (U.S. EPA SW-846 Methods 3005, 3010, 3020, 3040, and 3050).

### **3.3.13 Volatile Organic Compounds**

The VOC test evaluates the types and concentrations of low-boiling-point organic materials present in a sample. U.S. EPA SW-846 Method 8240

describes the extraction and analysis of VOCs by gas chromatography/mass spectrometry (GC/MS) techniques. This method will quantify most organic compounds with a boiling point below 200° C. Concentrations of VOCs in solvent extracts of untreated and S/S-treated wastes can be used to indicate if the compounds have been stabilized during the S/S process, provided measures were taken to account for volatilization or degradation. Concentrations of VOCs in TCLP extracts can indicate the aqueous leachability of the VOCs from S/S-treated wastes. Extreme caution must be paid to the possible release of VOCs during waste sampling, handling, storage, treatability testing, or analysis. The potential for volatilization of the organic contaminants is so great that a mass balance is generally needed to demonstrate that a reduction in volatile organic content after treatment is truly due to immobilization as opposed to volatilization. Although organic leaching may be low in aqueous leaching tests, this may also be a result of low solubility of the organic in water rather than immobilization of the organic.

#### **3.3.14 Base, Neutral, and Acid (BNA) Organic Compounds**

The analyses for basic, neutral, and acidic organic compounds are performed by extraction (U.S. EPA SW-846 Method 3510, 3520, or 3540) followed by GC/MS analysis (U.S. EPA SW-846 Method 8270). Certain BNAs can be target contaminants for S/S. Measurements of BNAs in solvent extracts of untreated and treated wastes can determine the fate of organics during the S/S process, provided measures were taken to account for volatilization or degradation. Data on concentrations of BNAs in aqueous extracts can be used to assess the effectiveness of the S/S process in reducing the amount of aqueous leachable BNAs. However, like VOCs, certain BNAs have low aqueous solubility. Thus, the immobilization of such compounds should be evaluated in organic solvent extracts of appropriate polarity.

#### **3.3.15 Polychlorinated Biphenyls (PCBs)**

The PCB analysis measures the concentration of polychlorinated biphenyls. PCBs are determined by extraction (U.S. EPA SW-846 Method 3540 or 3520), followed by GC/MS analysis (U.S. EPA SW-846 Method 8080) or by U.S. EPA Method 608. Quantities of PCBs in solvent extracts of untreated and S/S-treated wastes can determine the fate of the PCBs during the S/S process,

provided that measures were taken to conduct mass balances and account for any PCB volatilization during the treatability study. Conducting aqueous leaching tests on PCB-contaminated wastes is generally fruitless because of the low aqueous solubilities of PCB compounds.

#### **3.3.16 Other Contaminant Analyses**

Several of the more common waste contaminants not specifically included in Sections 3.3.1 through 3.3.15 are mercury, pesticides, and herbicides. Analytical methods are available for measuring such constituents in either aqueous or organic solvent extracts (see Table 3-4).

#### **3.3.17 Anion Measurements**

Anions can be measured by ion chromatography, as described by Water and Wastewater Standard Method 4110 or by U.S. EPA Method 300. This analysis is used to determine the concentration of anions in leach solutions.

#### **3.3.18 Interferants Screen**

Interferants screening tests involve a series of analyses for concentrations of materials that can interfere with S/S treatment. The waste is tested for oil and grease, potassium, sodium, fluoride, chloride, ortho-phosphate, ammonia, nitrate, and sulfate.

### **3.4 BIOLOGICAL TESTS**

Biological tests applicable to S/S processes include biodegradation tests and bioassays. Table 3-5 shows some representative biological tests and presents information about the standard methods for each. Biological tests are typically conducted only in special circumstances such as testing the potential biodegradability of organic binders or the aquatic toxicity of the treated waste.

Biological testing can be used to measure either the degradation of the matrix leading to release of contaminants or the alteration of contaminant properties to increase their mobility or toxicity. Standard tests for matrix degradation exist, but none are available for biologically induced changes in the contaminants.

TABLE 3-5. BIOLOGICAL TESTS

Test Procedure	Method	Material Application			Purpose <sup>(a)</sup>
		U	S	L	
<u>Biodegradation Tests</u>					
Biodegradability of plastics	ASTM G 21-90, G 22-76		X		To determine whether biodegradation may decrease long-term stability of S/S wastes.
Biodegradability of paints	ASTM D 3273-86, D 3274-82, D 3456-86		X		To determine whether biodegradation may decrease long-term stability of S/S wastes.
Biodegradability of alkylbenzene sulfonates			X		To determine whether biodegradation may decrease long-term stability of S/S wastes.
<u>Bioassays</u>					
Assessing the hazard of a material to aquatic organisms	ASTM E 1023-84	X	X	X	To evaluate acute aquatic toxicity at a point source discharge, e.g., a leachate collection system. May be required by state or federal ARARs.

<sup>(a)</sup> Testing Application: Biological testing is normally applied as part of an experimental program.

Material Application Guide:

- U = Untreated Samples
- S = S/S-treated Sample
- L = Liquid Sample

Biodegradation tests are used to measure the biodegradability of various waste materials, almost exclusively organic binders such as asphalt or plastic. Biodegradation is one possible degradation mechanism for such binders. At present, the U.S. EPA recommends no particular methods for evaluating the biodegradation of S/S-treated wastes. In general, binders that produce an alkaline environment (e.g., Portland cement-based processes) are not favorable for microbial activity; however, this may not be true for proprietary binders and processes that are tailored to treat organic wastes.

Bioassays are performed only when the proximity of the treated waste disposal site poses a threat to an aquatic community. If a site undergoing S/S treatment has a point source discharge, such as from a leachate collection system, bioassays may be required to meet federal or state ARARs. However, note that the alkaline nature of many S/S binders may elicit a toxic response during the bioassay, which may far outweigh any acute toxic response from the contaminants in the waste.

Although the results of bioassays may provide evidence of reduced toxicity after S/S treatment, predictions of toxicity from bioassays are highly site-specific and must be combined with data on exposure pathways for a specific site. Acute bioassays may be performed rapidly and at low cost, but they do not predict the response of the test organism to chronic, low-level contamination. The bioassay techniques that most accurately predict long-term environmental effects are expensive and time-consuming.

### 3.5 MICROCHARACTERIZATION

Special methods developed for mineralogic and materials science testing are applicable to specialized, detailed characterization of materials for S/S treatment (Hannak and Liem, 1986). These nonroutine tests can be applied for detailed analysis of the structure of S/S-treated waste or to better understand the physicochemical form of the target contaminants. Table 3-6 lists a few of the many tests that can be applied to microcharacterization. However, note also that microcharacterization tests provide special research and problem-solving tools that would not be used in the vast majority of S/S treatability studies.

**TABLE 3-6. MICROCHARACTERIZATION TESTS**

Test Procedure	Method	Purpose <sup>(a)</sup>
X-Ray Powder Diffraction		To identify crystalline matrix and contaminant phases
Fourier Transform Infrared (FTIR) Spectroscopy	ASTM E 1252-88 ASTM E 168-88	To identify the presence or absence of functional groups in a molecule
Scanning Electron Microscopy (SEM) and Energy-Dispersive X-Ray Analysis (EDAX)		To examine the physical structure and chemical makeup of the surface of a material on the microscopic scale
Nuclear Magnetic Resonance (NMR) Spectroscopy		To identify and characterize molecules
Optical Microscopy Transmitted Light, Reflected Light, and Polarized Light	ASTM C 856-83 ASTM C 295-90	To study microstructure of S/S-treated waste

<sup>(a)</sup> Microcharacterization tests are typically applied to treated waste as part of an experimental program.

### **3.5.1 X-Ray Diffraction**

X-ray diffraction examines the crystal structure of a material. X-rays are scattered and diffracted by the lattice structure of crystals, yielding patterns characteristic to various crystals based on the lattice spacing. The crystalline components of a mixture, including crystalline phases of the contaminant or contaminants, in amounts of 1% or more can be identified individually by the X-ray diffraction patterns produced. However, noncrystalline components are not detected.

### **3.5.2 Fourier Transform Infrared (FTIR) Spectroscopy**

The FTIR spectroscopy analytical technique can identify the presence or absence of functional groups within a molecule. The class or type of compound can be deduced, although positive identification of the exact

composition of the unknown is not always possible. This technique can be useful in determining the physicochemical form of the contaminant in either treated or untreated waste.

### **3.5.3 Scanning Electron Microscopy (SEM) and Energy-Dispersive X-ray Analysis (EDXA)**

SEM is a technique for examining the surfaces of solid materials. The method provides a large depth of field, so it is frequently possible to observe three-dimensional structures in a sample. By adding an EDXA detector to the SEM, it is possible to obtain simultaneous, multi-element analysis. This technique can be useful in determining the physicochemical form of the contaminant in either treated or untreated waste.

### **3.5.4 Nuclear Magnetic Resonance (NMR) Spectroscopy**

NMR spectroscopy identifies and characterizes molecules. Data from NMR analysis delineate complete sequences of groups or arrangements of atoms in a molecule. This technique has been used successfully to characterize the physicochemical form of the contaminant in the treated waste and to help elucidate the mechanism of contaminant immobilization.

### **3.5.5 Optical Microscopy**

The arrangement of phase structures in a solid sample can be observed and measured by thin section transmission microscopy or reflected light microscopy. Optical properties, such as refractive index, also can be measured. Additional petrographic information can be obtained by using polarized light microscopy. This is another possible analytical tool for characterizing contaminant speciation and physicochemical form.



#### **4 STATUS OF SOLIDIFICATION/STABILIZATION TECHNOLOGY**

This chapter of the TRD reviews and summarizes existing literature on a wide variety of subjects and issues pertaining to S/S technology. A number of books and summary reports on various aspects of S/S are available. These resource documents include the following:

- ASTM (1989), STP 1033, *Environmental Aspects of Stabilization and Solidification of Hazardous and Radioactive Wastes*, American Society for Testing and Materials.
- ASTM (1992), STP 1123. *Stabilization and Solidification of Hazardous, Radioactive, and Mixed Wastes*, American Society for Testing and Materials.
- Conner, J.R. (1990), *Chemical Fixation and Solidification of Hazardous Waste*, Van Nostrand Reinhold.
- Czupyrna, G., et al. (1989), *In Situ Immobilization of Heavy-Metal-Contaminated Soils*, Noyes Data Corporation
- Pojasek, R. (1979), *Toxic and Hazardous Waste Disposal, Options for Solidification/Stabilization*, Ann Arbor Science Publishers
- U.S. EPA (1990e), *Handbook on In Situ Treatment of Hazardous Waste-Contaminated Soils*
- U.S. EPA (1989b), *Immobilization Technology Seminar, Speaker Slide Copies and Supporting Information*
- U.S. EPA (1986c), *Handbook for Solidification/Stabilization of Hazardous Wastes*
- U.S. EPA (1983), *Feasibility of In Situ Solidification/Stabilization of Landfilled Hazardous Wastes*
- U.S. EPA (1980), *Guide to the Disposal of Chemically Stabilized and Solidified Waste*.

Overview-type information on specific S/S issues can be found in Sections 4.1 through 4.10. Sections 4.1 and 4.2 describe the types of S/S binders and their binding mechanisms. Applicable waste and contaminant types are discussed in Section 2.2.3.2. Section 4.3 outlines the interferences to S/S that

arise from waste constituents. Section 4.4 deals with S/S treatment of organic contaminants. Section 4.5 discusses air emissions from organic constituents, particulates, and other emissions. Sections 4.6 and 4.7 describe leaching mechanisms and long-term stability. Sections 4.8 and 4.9 discuss reuse and disposal issues. Section 4.10 gives cost estimates for S/S testing, materials, and processes. The publications referenced in Chapter 7 provide additional technical details.

#### **4.1 S/S PROCESSES AND BINDERS**

Solidification/stabilization processes are "nondestructive" methods to immobilize the hazardous constituents in a waste. S/S processes are nondestructive in the sense that they do not remove or reduce the quantities of these constituents. Typically, S/S processes physically sorb, encapsulate, or change the physicochemical form of the pollutant in the waste, resulting in a less leachable product. Concentrations of contaminants in the treated waste are often lower than in the untreated waste, primarily because of incidental dilution by the binder rather than by destruction or removal of the contaminants.

S/S processes can generally be grouped into inorganic processes (cement and pozzolanic) and organic processes (thermoplastic and thermosetting polymers). In addition to the individual use of inorganic and organic binders, some systems combine organic with inorganic binders. For example:

- Diatomaceous earth with cement and polystyrene
- Polyurethane with cement
- Polymer gels with silicate and lime cement

The basic S/S processes are generic, and many of the basic materials are readily available. A variety of additives are used to promote the development of specific chemical or physical properties. Pretreatment may also be used to better prepare the waste for treatment by an S/S process.

S/S technology is offered commercially by a large number of vendors. The specifics of vendor technology are in most cases protected as proprietary and are not disclosed to the potential user except under agreement of confidentiality. The majority of vendors use conventional S/S technology supple-

mented by a variety of additives and know-how from previous experience in applying this technology.

#### 4.1.1 Inorganic Binders

The two principal types of inorganic binders are cement binders and pozzolanic binders (lime, kiln dust, fly ash). A pozzolan is a material containing silica or silica and alumina that has little or no cementation value itself but, under some conditions, can react with lime to produce cementitious material. Cement-based and pozzolanic processes or a combination of cement and pozzolans are the methods of choice in the S/S industry today. This probably is attributable to the low cost of the materials, their applicability to a wide variety of waste types, and the ease of operation in the field. The most common inorganic binders are:

- Portland cement
- Lime/fly ash
- Kiln dust (lime and cement)
- Portland cement/fly ash
- Portland cement/lime
- Portland cement/sodium silicate

These binders are routinely used to solidify water-based waste liquids, sludges, and filter cakes. The lime/fly ash process probably has been used most extensively in the United States, in terms of the total volume of waste treated. The treatment of flue gas desulfurization (FGD) sludges from coal-fired power plants accounts for much of the lime/fly ash process application. Specifications are available for a wide variety of cement and pozzolanic materials. ASTM standards for these materials include:

- C311: *Method for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland Cement Concrete*
- C400: *Test Methods of Testing Quicklime and Hydrated Lime for Neutralization of Waste Acid*

- C593: *Specification for Fly Ash and Other Pozzolans for Use with Lime*
- C618: *Specification for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete*
- C821: *Specification for Lime for Use with Pozzolans*
- C911: *Specification for Quicklime, Hydrated Lime, and Limestone for Chemical Uses*
- C977: *Specification for Quicklime and Hydrated Lime for Soil Stabilization*

Most concentrated industrial or Superfund wastes contain complex mixtures of contaminants, and a generic inorganic binder will frequently stabilize one contaminant to a greater extent than another. Certain constituents, such as oils and anions, can retard or prevent the setting of the binder. Chemicals that interfere with cement- and pozzolan-based processes are discussed in greater detail in Section 4.3. Complications in the stabilization of certain types of contaminants are discussed in Sections 4.2 and 4.4.

#### 4.1.1.1 Cement Processes

Of the inorganic binders, Portland cement has probably had the greatest diversity of application to a wide range of hazardous wastes, especially combined with fly ash. Because cement is a common construction material, the materials and equipment are mass-produced and generally inexpensive compared with energy-intensive treatment processes such as vitrification and incineration (McDaniel et al., 1990). Many types of cement have been used for a variety of purposes, but only those classified as Portland cement, which is primarily composed of anhydrous calcium silicate, have seen substantial use in S/S technology (Conner, 1990). Other types of cement, such as alumina or Sorel cement, have not been used extensively for S/S, primarily because of their high cost.

Advantages of cement-based processes include (McDaniel et al., 1990 and Conner, 1990):

- Availability of materials locally on a worldwide basis
- Low cost of materials and mixing equipment

- Use of naturally occurring minerals as raw materials for the matrix
- Ability to make a strong physical barrier under adverse conditions
- Flexibility of tailoring the properties for different applications
- Low variability in composition
- Well-known setting and hardening reactions and some existing data on the immobilization of metals

The disadvantages of cement-based processes include:

- Sensitivity of product quality to presence of impurities at high enough concentrations. (Specific examples of impurities are discussed in detail in Section 4.3.)
- Porosity of the S/S-treated waste.
- Waste volume typically increases due to binder addition, although not necessarily more than with other inorganic binders.
- Expertise needed for successful application, although process appears deceptively simple.

The major performance objectives of S/S treatment are to reduce the mobility of contaminants, minimize free liquids, and, occasionally, to increase the strength of the waste. Cement-based processes accomplish these objectives by forming a granular or monolithic solid that incorporates the waste materials and immobilizes contaminants. The solid matrix forms because of hydration of silicates in the cement, yielding calcium-silicate-hydrate. Sufficient free water may be present in the waste material, or additional water may be needed. In most cases, the bulk of the strength-forming ingredients are provided as an added cement binder.

ASTM provides specifications for eight types of Portland cement. Type I is the least expensive and is the most widely used for S/S treatment. Tricalcium and dicalcium silicates are the major crystalline compounds present in Portland cement, while tricalcium aluminate and a calcium aluminoferrite are present in smaller quantities. The cementation process binds free water,

increases the pH and alters other chemical properties of the mixture, reduces the surface area, and increases strength. All these mechanisms contribute to improved performance characteristics of the treated waste.

Cementation of the waste/binder mixture begins when water is added, either directly or as part of the waste. Once the cement powder contacts water, tricalcium aluminate immediately hydrates, causing the rapid setting which produces a rigid structure. In an idealized setting, the water hydrates the calcium silicates and aluminates in the cement to form calcium-silicate-hydrate. Thin, densely-packed fibrils of silicate grow out from the cement grains and interlace to harden the mixture entrapping inert materials and unreacted grains of cement. Hydration of tricalcium and dicalcium silicates results in the formation of tobermorite and crystalline calcium hydroxide. These compounds account for strength development after the initial setting. The setting rate is controlled by the amount of gypsum added to the cement. If sufficient gypsum is present, sulfates combine with tricalcium aluminate to form calcium aluminate sulfate, which coats the cement particles and retards hydration reactions.

The ratio of free water to cement (W/C) is a major factor controlling the porosity and strength of the final product. With a W/C weight ratio of about 0.48, the cement will fully hydrate, leaving some water adsorbed in the pore spaces. If the W/C ratio increases greatly above 0.48, the porosity increases rapidly and the strength declines. When estimating the required water addition, it is important to note that the total water content of the waste is not always available to hydrate the cement. Water that is held by hydration in the waste material may be unavailable or "bound" and thus not available to hydrate the cement.

In many applications, the binder is supplemented by additives to tailor the S/S process to waste-specific conditions. The additives may be used to modify the characteristics of the fresh mix to improve processing. For example, lignosulfonic or carboxylic acids can reduce the viscosity and retard the set of the mix. Low concentrations of calcium chloride accelerate setting. In other cases, additives may be needed to reduce interferences or improve the performance of the treated waste.

For cementation reagents to react, they must become wetted with water. In general, the higher the surface area of the particles, the more difficult they are to wet. Some additives may even have hydrophobic surfaces

initially. Many wastes, such as fine particulates and oils, may inhibit the setting and curing of the cement by interfering with the wetting process through coating of the reacting surfaces. Addition of surfactants to the waste may aid in the wetting of reagents, allowing thorough mixing of all components. Compounds such as alcohols, amides, and specific surfactants aid in wetting solids and dispersing fine particulates and oil. Flocculants have a similar effect by aggregating fine particles and film-formers.

The waste constituents can exhibit positive, negative, or inert contributions to the strength-forming reactions. Wastes with free calcium hydroxide can contribute to the strength-forming reactions, but excess hydroxide will increase the pH and increase the solubility of amphoteric metals. Alcohols and glycols decrease durability, while aliphatic, aromatic, and chlorinated organics increase set time and often decrease durability. Inorganic compounds such as boric acid, phosphates, iodates, sulfates, and sulfides can slow or prevent setting. Salts of some metals such as manganese, tin, zinc, copper, and lead can increase set time and reduce strength. Fine particulates such as silt, clay, or coal dust can coat cement particles and prevent the growth of calcium-silicate-hydrate crystals from the cement grain. Inerts such as soils or calcium fluoride do not directly participate in the cementation reactions but do become trapped in the solid matrix.

Cement-based solidification and stabilization processes have proven versatile and adaptable. It is possible to form waste/cement composites that have good strength and durability and that retain wastes effectively. Sorbents and/or emulsifiers can be added to reduce contaminant migration through the porous solid matrix, thus improving the leaching resistance of the treated wastes (U.S. EPA, 1986c).

#### 4.1.1.2 Pozzolanic Processes

Pozzolanic processes generally involve siliceous and aluminosilicate materials, which do not display cementing action alone but form cementitious substances when combined with lime or cement and water at ambient temperatures. The primary containment mechanisms are precipitation and physical immobilization of the contaminant in the pozzolan matrix. Common examples of pozzolans are fly ash, pumice, lime kiln dusts, and blast furnace slag. The addition of bentonite can substantially reduce the amount of fly ash required (U.S. EPA, 1986c, p. 2-11). Pozzolans contain significant amounts of sili-

cates, which distinguish them from the lime-based materials (U.S. EPA, 1989g). Typical tests of pozzolanic activity with lime and the strength of lime/pozzolan mixtures use hydrated lime-to-pozzolan ratios in the range of 1:2 to 1:6 on a weight basis (ASTM C 311 and ASTM C 593). Typically, pozzolanic reactions occur more slowly than do cement reactions.

Standard testing systems (ASTM C 311) and standard specifications (ASTM C 593 and ASTM C 618) exist for pozzolanic materials, especially for fly ash. The specifications take into account the chemical composition (percent  $\text{SiO}_2$ , percent  $\text{SO}_3$ ), moisture content, and physical properties (fineness, pozzolanic activity with lime, and specific gravity). Pozzolanic activity greater than a specified minimum can be expected if the material used meets the specification for fly ash normally produced from burning either anthracite or bituminous coal (Type F) or lignite or sub-bituminous coal (Type C). Some Type C fly ashes have enough lime to be not only pozzolanic but also self-cementing (U.S. EPA, 1986c).

Lime/fly ash treatment is relatively inexpensive and, with careful selection of materials, can reliably convert waste to a solid material. In general, lime/fly ash-solidified wastes are not considered as durable as Portland cement-treated wastes.

Common problems with lime/pozzolan reactions involve interference with the cementitious reaction that prevents bonding of materials. The bonds in pozzolan reactions depend on the formation of calcium silicate and aluminate hydrates. Therefore, the interferences are broadly the same as for cement-based processes (Sections 4.1.1.1 and 4.3).

#### 4.1.1.3 Ettringite Formation Effects

Formation of a calcium aluminate sulfate hydrate (i.e., ettringite) is typically required early in the curing process to control setting rate. However, the ettringite then dissolves and reprecipitates as calcium sulfate. Due to the high content of water of hydration, ettringite increases the volume of solids when it forms.

If the ettringite is formed while the S/S-treated waste is still plastic, the material can accommodate the expansive salt. However, if the ettringite forms after the grout has become rigid, cracking can occur and will reduce the strength of the product. The formation of this salt, with its large amount of water of crystallization and consequently large increase in



volume, can be destructive to the S/S-treated product. Figure 4-1 is an idealized representation of the progress of cementation reactions.

#### 4.1.2 Organic Binders

Application of organic binders is usually limited to special waste types. Inorganic binders are used much more frequently and are generally favored over organic binders because of cost and ease of application. The primary niche of organic S/S processes in the commercial sector is to solidify radioactive wastes or hazardous organics that cannot be destroyed thermally.

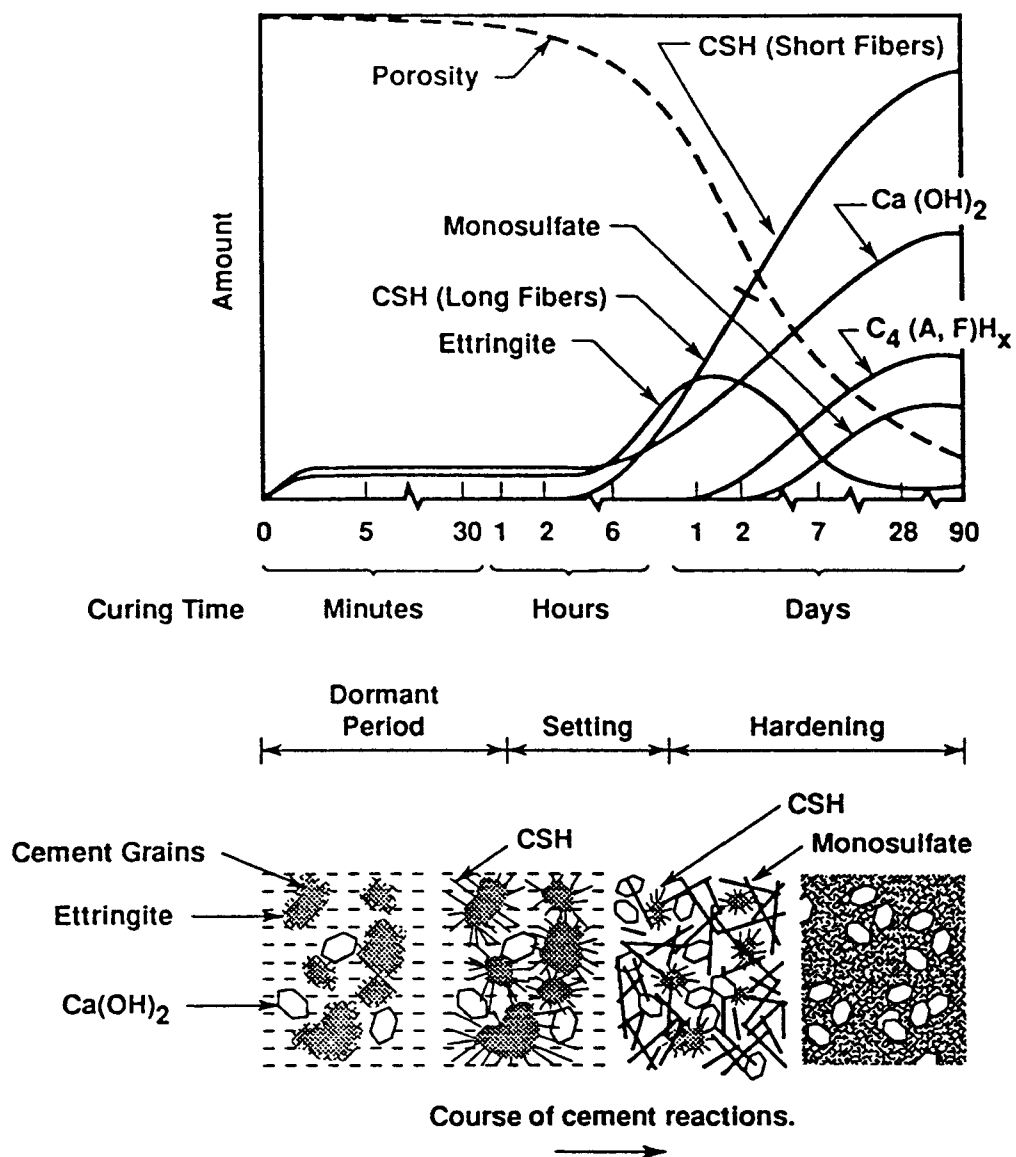
Organic binders that have been tested or used for S/S include the following:

- Asphalt (bitumen)
- Polyethylene
- Polyesters
- Polybutadiene
- Epoxide
- Urea formaldehyde
- Acrylamide gel
- Polyolefin encapsulations

The basic types of organic S/S processes are: (1) thermoplastic and (2) thermosetting with organic polymers. Thermoplastic processes involve blending waste with melted asphalt, polyethylene, or other thermoplastic binders. Liquid and volatile phases associated with the waste are driven off, and the waste is contained in a mass of cooled, hardened thermoplastic (U.S. EPA, 1986c).

Immobilization in thermosetting polymers involves mixing waste with reactive monomers, which join to form a solid incorporating the waste. Urea formaldehyde is one thermosetting resin that has been used for S/S processes.

One problem with organic processes is that many use hydrophobic binders, which are not compatible with water-based wastes unless the waste is first converted to an emulsion or a solid. Many hazardous wastes are



Legend:

CSH = Calcium Silicate Hydrate

C<sub>4</sub>(A, F)H<sub>x</sub> = Ferrite Solid Solution

Source: Dole, 1985; figure used with permission of author and symposium proceedings publisher, WM Symposia, Inc., Tuscon, Arizona.

FIGURE 4-1 PROGRESS OF CEMENTATION REACTIONS

water-based and require special pretreatment to form an emulsion prior to treatment by an organic binder.

Organic binders are also subject to deterioration from environmental factors such as biological action or exposure to ultraviolet light. Therefore, the long-term stability of organic binders for S/S processes will depend on the physicochemical characteristics of the disposal or reuse environment (as in the case of asphalt cement for roadways).

#### 4.1.2.1 Thermoplastic Processes

Thermoplastic processes are used in nuclear waste disposal and can be adapted to special industrial wastes. The thermoplastic technique for S/S treatment of waste involves drying and dispersing waste through a plastic matrix. The waste is mixed into a hot plastic mass which then cools, incorporating the waste in a rigid but deformable solid. In most cases, the hot waste/thermoplastic mix is extruded into a container, such as a fiber or metal drum, to give the final waste form a convenient shape for transport. The most common thermoplastic material used for waste incorporation is asphalt. When cost is not a limiting factor, other materials such as polyethylene, polypropylene, or wax can be employed for specific wastes to provide containment in an impermeable medium (U.S. EPA, 1986c).

One advantage of thermoplastic processes is their ability to treat soluble, toxic materials. For example, thermoplastic processing is one of the few alternatives applicable to S/S treatment of spray-dried salt (U.S. EPA, 1986c).

However, compatibility of the waste with the matrix is a limiting factor in using thermoplastic processes. Most thermoplastic S/S binders are chemically reduced materials (e.g., solid hydrocarbons) that can react (combust) when mixed with an oxidizer at elevated temperatures. The reaction can be self-sustaining or even explosive with perchlorates or nitrates (U.S. EPA, 1986c).

Other compatibility problems relate to softening or hardening of the waste/binder mix. Some solvents and greases can prevent asphalt hardening. Borate salts can initiate hardening at high temperatures, leading to stalled or clogged mixing equipment. Xylene and toluene can diffuse through asphalt (U.S. EPA, 1986c). Other interferences have been documented for salts that dehydrate at elevated temperature and for chelating and complexing agents.

Unlike inorganic S/S processes, thermoplastic processes require more complex, specialized melting and extrusion equipment. Both organic and inorganic processes require a trained operations staff to ensure safe, consistent operation. The power consumption for organic processes is higher than that for inorganic processes because of the need to dry the waste and melt the matrix material (U.S. EPA, 1986c).

#### **4.1.2.2 Thermosetting Processes**

Another type of organic S/S processes uses thermosetting resins such as urea formaldehyde. This type of process relies on polymer formation to immobilize the waste (U.S. EPA, 1989g). This technology has been evaluated for stabilizing radioactive wastes and largely abandoned due to problems with excess free water and radiolytic decomposition. Thermosetting processes have also been tested on a limited basis on hazardous wastes such as organic chlorides, phenols, paint sludges, cyanides, and arsenic as well as flue gas desulfurization sludge, electroplating sludges, nickel/cadmium battery wastes, kepone-contaminated sludge, and chlorine product wastes that have been dewatered and dried (U.S. EPA, 1989g).

Usually, there is no direct reaction between the waste constituent and the polymer. That is, thermosetting processes do not usually insolubilize, modify, or destroy the hazardous constituents. Rather, the effect of most thermosetting processes is to microencapsulate the waste, and the process is potentially applicable to a wide variety of waste types (Conner, 1990).

#### **4.1.3 Additives**

S/S processes may be used in conjunction with sorbents or other additives to improve immobilization of specific contaminants. Additives can be particularly useful for cement or pozzolan processes to decrease the mobility of contaminants in the porous, solid products. Additives to cement or pozzolan processes can also be incorporated to mitigate the effects of certain inhibitors. Some previously used additives and their applications are as follows:

- Soluble silicates, such as sodium silicate or potassium silicate. These agents will generally "flash set" Portland cement to produce a low-strength concrete.

Soluble silicates can also be beneficial in reducing interferences from metal ions.

- Selected clays to sorb liquid and bind specific anions or cations. Bentonite can reduce the amount of sorbent required in low-solids mixtures.
- Emulsifiers and surfactants to allow the incorporation of immiscible organic liquids. Waste turbine oil and grease can be mixed into cement blends if dispersing agents are used and if the proper mixing system is employed.
- Certain sorbents (e.g., carbon, silicates, zeolitic materials, and cellulosic sorbents) can help retain toxic constituents.
- Activated carbon in particular has been used primarily as a sorbent for organics, although this material will also sorb at least some metal ions and other inorganics.
- Lime ( $\text{CaO}$  or  $\text{Ca(OH)}_2$ ), soda ash (sodium carbonate,  $\text{Na}_2\text{CO}_3$ ), fly ash, sodium hydroxide ( $\text{NaOH}$ ) and, less commonly, magnesium hydroxide ( $\text{Mg(OH)}_2$ ) are added for maintaining alkaline conditions.
- Ferrous sulfate, sodium metabisulfite/bisulfite, sodium hydrosulfite, sulfides, blast furnace slag, sodium borohydride, reductive resins, and hydrazine are added as reducing agents.
- Organophilic clays have been used to increase the immobilization of certain organic contaminants within hydrophobic binders. Organophilic clays are clay minerals, such as montmorillonite, that have been modified by treatment with a quaternary ammonium compound that expands the spacing between clay layers, thus promoting the absorption of organic constituents between these layers. After treatment, the layer spacing is reduced by treatment with an alkaline substance, such as sodium chloride, to immobilize the absorbed organic constituents in the clays.
- Organosilanes have been applied to increase the binding of metals.

This list is not comprehensive but rather provides examples of additives used. Note that many additives may work for one constituent but have the opposite effect for a different constituent. An evaluation of the system performance of the additive needs to be conducted.

#### **4.1.4 Pretreatment**

Frequently, the ultimate performance of an S/S process can be improved by pretreating the waste. Improvements can sometimes be made to the physical characteristics of the waste, to alter metal speciation, to improve metal immobilization, or to remove problematic organics.

##### **4.1.4.1 Adjustment of Physical Characteristics**

Treatment by S/S involves extensive handling and mixing of the contaminated material. The presence of large pieces of debris or poor handling characteristics of the waste can interfere with sampling, analysis, and S/S processing (Barth, 1991).

Some amount of debris or large solids will be encountered in waste at almost any site. Debris such as wire, broken brick, timbers, tires, scrap metal, or scrap cloth can be encountered at many industrial or waste disposal sites. Other sites may have waste-specific debris, such as wood or bark pieces at a creosote wood preserving site. Large pieces of material present considerable obstacles to obtaining a representative sample and to characterizing the waste as well as to performing the treatment.

The preliminary site characterization should identify the presence of debris. Sample collection should be planned to allow collection of meaningful characterization data of the waste and the debris. The debris can either be removed by screening and processed separately or can be broken down with size-reduction equipment to a size compatible with the S/S processing equipment.

Mixing requires the ability to handle the waste material. Debris can damage the mixing equipment and/or prevent good mixing. Excess free liquid, high viscosity, or caking properties can all present problems in materials handling. Possible pretreatment methods to improve handling are drying, pelletizing, or adding sorbents to control liquids.

##### **4.1.4.2 Pretreatment for Inorganic Constituents**

Properly formulated inorganic binders can often incorporate metals and their inorganic salts without extensive pretreatment. In some cases, however, pretreatment can significantly improve the performance of the S/S treatment system. Examples include (Conner, 1990):

- Chemical reduction of hexavalent chromium to the less soluble and toxic trivalent state
- Elimination of problem constituents, for example, destruction of cyanide or stripping of ammonia
- Degrading soluble nickel complexes to ionic nickel
- Removing hygroscopic salts such as sodium sulfate by aqueous extraction.

#### **4.1.4.3 Pretreatment of Organic Constituents**

Organic constituents can complicate stabilization in both inorganic and organic-based S/S treatment systems. Volatile organics can make it necessary to use expensive off-gas collection and treatment systems. As described in Section 4.3, organic materials incorporated into the S/S-treated waste can prevent setting or degrade product quality.

A variety of pretreatment options are available to remove volatiles and semivolatiles or to control the effects of the organic material prior to S/S treatment:

- Soil washing
- Thermal removal
- Chemical oxidation
- Extraction
- Biodegradation
- Addition of a sorbent (such as limestone, diatomaceous earth, clays, activated carbon, or fly ash) prior to mixing

A study of RODs for Superfund sites where S/S was one component in the treatment program showed that wastes contaminated with VOCs underwent pre-treatment more often than any other wastes (U.S. EPA, 1989a).

#### **4.1.4.4 Treatment Trains Involving S/S**

In many cases, treatment of wastes containing multiple and diverse contaminants becomes so complex that S/S treatment becomes just one step in a

treatment system or a treatment train. For example, the BDAT treatment for several RCRA nonwastewater waste types includes one or more other processes followed by S/S treatment (Table 1-1). The most common BDAT treatments that prepare waste for S/S are incineration and chemical precipitation. In other cases S/S treatment can be the initial step in a treatment train. For example, it can be used to improve materials handling characteristics or to immobilize metals prior to a different type of treatment.

Pretreatment to mitigate one problem may give rise to problems of another nature. For example, oxidation of organic contaminants with permanganate leaves a permanganate residue in the waste, and permanganate will oxidize organic binders such as asphalt. Washing with solvent leaves traces of solvent that must be removed from the waste prior to S/S treatment. Incineration can leave certain metals (e.g., chromium) in the ash in their higher and more mobile oxidation states.

Selection of the appropriate combination of binders, additives, and pretreatment options for a particular waste requires careful consideration of the waste material, the contaminants, and the performance objectives of the project (Sections 2.3 and 2.4).

## **4.2 IMMOBILIZATION MECHANISMS**

Waste stabilization may involve physical mechanisms, chemical mechanisms, or a combination of the two. Physical stabilization (solidification or encapsulation) changes the physical form of the waste but does not necessarily cause chemical binding of the waste constituents. Chemical stabilization changes the chemical states of waste constituents to forms with lower aqueous solubilities. Although the mechanisms of immobilization are discussed separately for convenience, under actual S/S treatment conditions, these mechanisms usually do not work independently.

### **4.2.1 Physical Mechanisms**

Physical mechanisms of S/S operate by confining waste constituents to a certain area or zone in the waste. That is, the waste constituent may or may not occur in a soluble form, but one or more physical barriers prevent its mobilization. Containment by a barrier is a satisfactory method as long as



the barrier remains stable. Encapsulation is the most commonly used method of containment, superseding earlier use of sorbents.

Encapsulation techniques use materials that trap waste constituents in the form of stable solids, preferably as a monolith with high cohesive strength and low leachability. Waste constituents are dispersed throughout an inorganic or organic binder matrix (Section 4.1) that physically isolates them from groundwater and air. The effectiveness of isolation depends on the permeability and long-term stability of the matrix and on the degree of mixing of waste constituents throughout the matrix. In practice, mixing may be less than ideal, resulting in some of the waste material occupying cavities in the matrix. Encapsulation of inorganic wastes is generally accompanied by chemical stabilization, but encapsulation of organic wastes such as oil and grease, PCBs, pesticides, and volatile compounds usually occurs without accompanying chemical interactions (Conner, 1990). Encapsulation can be further described at three levels: microencapsulation, macroencapsulation, and embedment (Conner, 1990).

The term "microencapsulation" describes a process of adsorbing or trapping contaminants in the pore spaces of a cementitious material. The contaminants are fine waste particles that may not be visible to the naked eye. As the system ages, the waste and matrix may eventually become a homogeneous material, although this might occur in a time frame of thousands of years or more (Conner, 1990).

The term "macroencapsulation" describes a process of coating a solid or cemented waste with an impermeable layer, such as bitumen (thermoplastic) or amorphous silica (U.S. EPA, 1990e). The success of this method depends on both effective coating reactions and thorough mixing. "Macroencapsulation" may also refer to the containment of large waste solids, as in a sealed drum.

The term "embedment" describes a process of incorporating large waste masses into a solid matrix before disposal. Examples of such wastes are contaminated debris from remedial actions, laboratory protective equipment, solid medical wastes (e.g. syringes), and radioactive objects (Conner, 1990). Embedment is used in situations where it is impractical to reduce the bulk of the waste but where the waste is hazardous enough to be treated prior to disposal. In addition, special consideration may also have to be given to the strength, water permeability, and long-term stability properties of the matrix.

Finally, sorbents once were used extensively to prevent the loss of liquid wastes and to improve handling characteristics. Materials such as expanding-layer clays and vermiculite were considered attractive for liquid waste disposal because of their low cost and easy handling. However, the use of sorbents has greatly diminished since the 1985 landban on bulk liquids in landfills, although sorbents are currently permitted for certain applications (Conner, 1990). The main problem with sorbents is that they may become highly leachable under certain circumstances, for example, if oversaturation should occur and load levels become too high.

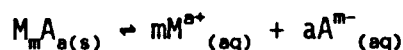
#### 4.2.2 Chemical Mechanisms

Different chemical mechanisms of S/S are operable for inorganic and organic wastes. Also, the aqueous chemistries of most inorganic and organic compounds are fundamentally different, leading to different leaching behaviors.

##### 4.2.2.1 Inorganic Wastes

**4.2.2.1.1 Basic Chemical Equilibria.** The chemistry of inorganic waste constituents is dominated by hydrolysis reactions. The term "hydrolysis" implies that a substance, usually a metal, reacts with water. Hydrolyzed products can react in the aqueous phase to form new ionic or neutral species, or they can form precipitates of hydroxides, oxides, or salts (commonly carbonates, sulfates, and sulfides).

It is useful to compare the solubilities of different metal compounds. Consider the dissolution reaction of a generic compound  $M_m A_a$ :



where  $M^{a+}$  is the cation and  $A^{m-}$  is the anion. The solubility product constant for this reaction is

$$K_{sp} = [M^{a+}]^m [A^{m-}]^a$$

where square brackets indicate concentration (activity) and  $K_{sp}$  depends only on temperature and pressure. The solubility product is therefore a constant

if temperature and pressure of the solid and solution phases remain fixed, for example, at the ambient conditions of a disposal site. Frequently, the solubility product is written as  $pK_{sp}$ , where  $pX = -\log_{10}(X)$ .

Table 4-1 lists solubility product values for the hydroxides, carbonates, sulfates, and sulfides of some regulated metals (higher  $pK_{sp}$  means lower solubility). Table 4-1 shows that the following metals salts have very low solubility products: Cr(III) hydroxide and sulfides of Cd(II), Pb(II), Hg(I), and Hg(II). The least soluble barium solid is barium sulfate (barite). This type of information can be important for deciding which form of a given hazardous metal is most stable and which metal compounds may be attainable given specific site conditions and available technology.

Actual concentrations of dissolved species depend on a number of solution parameters, such as pH, redox potential, and solution composition. The simplest and most common method of controlling speciation and precipitation is pH adjustment. To illustrate this process, consider the role of a strong acid in the solubility of a simple metal hydroxide,  $M(OH)_n$ . According to the equilibrium expression above, the solubility of  $M(OH)_n$  is described by the following reaction:

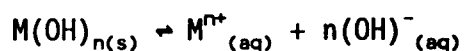


TABLE 4-1.  $pK_{sp}$  VALUES FOR SELECTED METAL PRECIPITATES<sup>(a)</sup>

Metal	Hydroxide	Carbonate	Sulfate	Sulfide
Ba	2.30	8.09	9.97	--
Cd	14.30	11.60	--	28.44
Cr(III)	30.20	--	0.50	--
Pb	19.90	13.48	7.71	27.47 <sup>(b)(c)</sup>
Hg(II)	25.52	--	1.43	48.70
Hg(I)	--	16.05	6.17	51.42

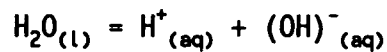
<sup>(a)</sup> Data apply to equilibria at 25°C except where otherwise noted.

<sup>(b)</sup> Equilibria at 18°C.

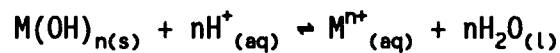
<sup>(c)</sup> Galena.

Sources: Means et al. (1991c) and Dragun (1988).

To show the dependence on pH directly, the following equation must be subtracted from the preceding one,  $n$  times:



Where  $\text{p}K_w = 14$  the result is



The solubility constant for this reaction is

$$K_a = [\text{M}^{n+}]/[\text{H}^+]^n$$

or upon rearranging terms:

$$[\text{M}^{n+}] = 10^{-n\text{pH}} K_a$$

The concentration of  $\text{M}^{n+}$  ions clearly increases as pH decreases. However,  $\text{M}^{n+}$  ions are not necessarily the dominant aqueous species of the metal M at all pH values and solution compositions. The total dissolved metal is the sum of all hydrolysis species  $[\text{M}(\text{OH})^{n-1}, \text{M}(\text{OH})_2^{n-2}, \text{etc.}]$  and complex species  $[\text{MCO}_3^{n-2}, \text{MSO}_4^{n-2}, \text{etc.}]$  that form in solution. At sufficiently high or low pH, some aqueous species can be hydroxylated or protonated. Therefore, these species are sensitive to pH and they affect the solubilities of the solid phases. The task of determining which species are present and in what concentrations is often time consuming and expensive. As an alternative, speciation calculations can be made if bulk solution compositions are known. Compilations of thermochemical data that are needed to perform these calculations are available in the chemistry literature (Means et al., 1991c).

An investigation of immobilization mechanisms for S/S of cadmium and lead (U.S. EPA, 1990f) found that, even though  $\text{Cd}(\text{OH})_2$  and  $\text{Pb}(\text{OH})_2$  have comparable and very low solubilities, the degree of leaching from cement treated wastes differed for the two metals. In leaching tests, cadmium concentrations were very low, whereas lead concentrations were considerably higher and could potentially pose a threat to groundwater. The differences were attributed to the fact that the Cd/cement system involves early formation

of  $\text{Cd}(\text{OH})_2$  which provides nucleation sites for precipitation of C-S-H and calcium hydroxide and results in Cd being in the form of an insoluble hydroxide with an impervious coating. The Pb/cement system was more complicated in terms of precipitation reactions. Mixed salts containing hydroxide, sulfate, and nitrate ions were precipitated. These salts retard the cement hydration reactions by forming an impervious coating around the cement clinker grains. Also, as pH in the cement pore water fluctuated during hydration, the Pb salts undergo solubilization and reprecipitation, resulting in Pb salts on the surface of cement minerals that are readily accessible to leach water and apparently are more soluble under basic conditions than a pure lead hydroxide.

**4.2.2.1.2 Effect of Alkaline Conditions.** Numerous compatible ionic species form solids by coprecipitation. Therefore, the application of chemical equilibria based on pure end-members may not be completely valid. Ferric iron has long been recognized for its ability to flocculate and coprecipitate toxic metals from solution (Sittig, 1973; LeGendre and Runnells, 1975; Swallow et al., 1980). Coprecipitated metals may have solubilities that are substantially lower than those of either of the pure end-member phases. For example, the Cr(III) concentrations are many times lower in solutions that are in equilibrium with coprecipitated  $\text{Cr}(\text{OH})_3$ - $\text{Fe}(\text{OH})_3$  than those that are in equilibrium with pure chromium hydroxide (Sass and Rai, 1987).

The minimum solubility of most metal hydroxides occurs within the approximate pH range of 7.5 to 11. This implies that solubility increases under extremely alkaline conditions as well as under acidic conditions (amphoteric behavior). When the waste material under consideration for S/S contains a number of different metals, it is possible that their solubility minima may not entirely overlap. In cases where pH values at these solubility minima are not too different, it may be sufficient to choose an average pH, but in cases where pH values are very different, the best recourse may be to attempt to precipitate the contaminants in a phase or phases other than a hydroxide.

As an example, suppose that Cd and Cr(III) are the predominant hazardous constituents in a waste system. Solubility minima occur at pH ~ 11 to 11.5 for  $\text{Cd}(\text{OH})_2$  (Brookins, 1988) and pH ~ 8.5 for  $\text{Cr}(\text{OH})_3$  (Baes and Mesmer, 1976). In this situation, one might elect to precipitate highly insoluble CdS by adding a soluble sulfide, such as  $\text{Na}_2\text{S}$ , and to precipitate

$\text{Cr}(\text{OH})_3$  by adjusting the pH to 8.5. Note, however, that if barium is present in the same waste, it has a high solubility in the presence of sulfide. This example illustrates the need for understanding the waste chemistry as well as the pertinent chemical equilibria in order to achieve a maximum degree of chemical stabilization.

Any alkali may be used to control pH, but the common choices are lime [either  $\text{CaO}$  or  $\text{Ca}(\text{OH})_2$ ], sodium carbonate, or sodium hydroxide. Most solidification reagents are alkaline and can substitute in part or entirely for traditional alkalies, acting both as pH controls and as binding agents. Alkaline binders include Portland cement, cement and lime kiln dusts, Type C fly ash, and sodium silicate (Conner, 1990).

Buffers provide resistance to rapid changes in pH upon exposure to acid or base. The presence of pH buffers in stabilized waste is desirable to maintain the pH at the target value for the long term, thus promoting long-term stability. Limestone (primarily  $\text{CaCO}_3$ ) is used to buffer waste acidity; Na-montmorillonite is also used for this purpose.

**4.2.2.1.3 Effect of Redox Potential.** Redox potential is another important solution parameter in S/S technology. An oxidation-reduction, or redox reaction, is one that involves the transfer of electrons between products and reactants. Experimentally, the redox potential of a half-cell reaction is measured by a quantity called "Eh." High Eh voltages indicate that the solution is oxidizing and low Eh voltages indicate that the solution is reducing. The redox potential of a waste form can be controlled to convert the valence states of hazardous metals to valence states that are more favorable for precipitation.

Among the regulated metals listed in Table 4-1, Cr and Hg have more than one common oxidation state. The table shows that trivalent chromium species precipitate as a low-solubility hydroxide. However, Cr(VI) forms mainly chromate and dichromate species, such as  $\text{CrO}_4^{2-}$  and  $\text{Cr}_2\text{O}_7^{2-}$ , which do not form precipitates with low solubility (Eary and Rai, 1987). The table also shows that mercury in both oxidation states forms very-low-solubility sulfides. Redox potential has particularly important effects on the regulated semimetals, such as As and Se, which exhibit a number of different oxidation states.

Table 4-2 lists selected stable solids of As and Se for reference. A literature review by Means et al. (1991c) shows that calcium arsenate  $\text{Ca}_3(\text{AsO}_4)_2$  is the most stable metal arsenate [As(V)] in oxidizing alkaline conditions. Under acidic conditions, calcium arsenate becomes unstable because its calcium source (calcite) is leached away. Another potentially stable phase for the immobilization of As(V) appears to be basic ferric arsenate  $\text{FeAsO}_4 \cdot x\text{Fe}(\text{OH})_3$ , which forms readily in the presence of ferric hydroxide. However, basic ferric arsenate is most stable at lower pH.

Under highly oxidizing conditions the selenate ion  $\text{SeO}_4^{2-}$  [Se(VI)] predominates in both acidic and basic solutions (Means et al., 1991c). Barium selenate appears to be the most stable solid (Elrashidi et al., 1987) but is fairly soluble (4mM activity). Other metal selenates are even more soluble. In moderately oxidizing conditions, manganese selenite,  $\text{MnSeO}_3$  [Se(IV)], is the most stable solid that persists in both neutral and acidic environments (Elrashidi et al., 1987); at pH 5 the activity of the dominant species  $\text{HSeO}_3^-$  is 2.5  $\mu\text{M}$ . According to (Elrashidi et al., 1987),  $\text{PbSeO}_3$  has a solubility minimum near pH 8. Under highly reducing conditions, selenide [Se(II)] species predominate (Elrashidi et al., 1987); lead selenide  $\text{PbSe}$  and tin selenide  $\text{SnSe}$  are the most stable solids in both neutral and alkaline conditions. Elemental Se also has a stability region, but it is more soluble than most of the metal selenides (Elrashidi et al., 1987).

The major reducing agents and their attributes are described by Conner (1990). The most common agents are ferrous sulfate, Na-metabisulfite/bisulfite, Na-hydrosulfite, sulfides, Na-borohydride, hydrazine, and reductive resins. The most widely used reducing agent in S/S technology is ferrous sulfate, which is used primarily to reduce hexavalent chromium. Its main disadvantage is that pH must be adjusted below 3 in order for the chromium reduction reaction to go to completion. The amount of acid needed can therefore be quite large, particularly if the waste material contains appreciable amounts of alkaline buffers. Na-metabisulfite/bisulfite and soluble sulfides, such as  $\text{Na}_2\text{S}$ , work similarly to ferrous sulfate but require less acid and alkali to complete a reduction/precipitation process. However, Na-metabisulfite/bisulfite is expensive, and  $\text{Na}_2\text{S}$  is unsafe to use at very low pH because of the possible evolution of toxic  $\text{H}_2\text{S}$ . Na-hydrosulfite, Na-borohydride, hydrazine, and freshly precipitated  $\text{FeS}$  (the Sulfex<sup>TM</sup> process) work under alkaline conditions, which may be more convenient for pretreated wastes.

TABLE 4-2.  $pK_{sp}$  VALUES FOR SELECTED As AND Se PRECIPITATES<sup>(a)</sup>

Element	Compound	$pK_{sp}$
As(V)	(arsenates)	
	$Ba_3(AsO_4)_2$	50.11
	$Ca_3(AsO_4)_2$	18.17
	$Cd_3(AsO_4)_2$	32.66
	$Mg_3(AsO_4)_2$	19.7
	$Pb_3(AsO_4)_2$	35.39
	$FeAsO_4 \cdot xFe(OH)_3$	20.24
As(III)	$As_2S_3$	29.6
As(II)	$AsS$	12.3
Se(VI)	(selenates)	
	$BaSeO_4$	7.46
	$CaSeO_4 \cdot 2H_2O$	3.09
	$CdSeO_4$	2.27
	$PbSeO_4$	6.84
Se(IV)	(selenites)	
	$BaSeO_3$	6.57
	$CaSeO_3 \cdot H_2O$	5.44
	$CdSeO_3$	8.84
	$HgSeO_3$	13.90
	$Hg_2SeO_3$	14.23
	$MnSeO_3$	7.27
	$PbSeO_3$	12.12
Se(-II)	(selenides)	
	$BaSe$	21.86
	$CaSe$	10.87
	$CdSe$	35.20
	$CuSe$	48.10
	$FeSe$	26.00
	$HgSe$	64.50
	$PbSe$	42.10
	$SnSe$	38.40

<sup>(a)</sup> Data apply to equilibria at 25°C.

Source: Means et al. (1991c).

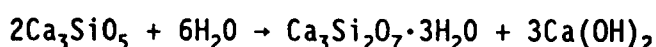


Reductive resins (e.g., Amborane™) are selective for certain metals and are used for precious metal recovery. They have potential uses for recovering hazardous metals such as silver, arsenic, mercury, and antimony (Conner, 1990). Blast furnace slag, a common binder, can serve as a reducing agent.

While adjustment of the redox-sensitive contaminant to its least soluble oxidation state is an important aspect of chemical stabilization, this objective eventually will be defeated if the treated waste is placed in a disposal or reuse environment having a very different oxidation state. Long-term stability can only be ensured if the oxidation states of the treated waste and the disposal or reuse environment are similar.

**4.2.2.1.4 Metal Silicates.** The behavior of hazardous metals in the silicate system is critical to cement-based S/S technology. However, full understanding of the chemical processes involved may be difficult to achieve because the waste constituents are often heterogeneous mixtures of solutions, suspended solids, and immiscible liquids. Reactions between metal salts in solution and soluble silicates have been studied extensively, but their insoluble products usually have not been well characterized. Metal silicates are generally nonstoichiometric and poorly crystallized. In fact, their chemical and physical properties depend considerably upon the conditions under which they are formed, for example, temperature, concentration, addition rate, and ionic speciation. The pH is also very important because it affects how readily soluble silicate (or colloidal silica) adsorbs metal ions. It has been found that adsorption occurs when the pH is 1 to 2 units below the hydroxide precipitation point (Iler, 1979).

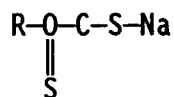
Just how the metal ions are incorporated into the cement structure is still a matter of debate. Using Portland cement as an example, the cementitious phase of calciumsilicate hydrate, or CSH, forms by a hydration reaction that takes from 28 days to 1 year to complete (Kantro et al., 1962):



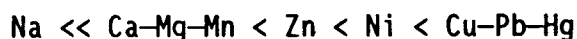
It is believed that CSH incorporates metal ions into the silicate matrix during the hydration reaction (Bhatty and Greening, 1978). The number of metal ions retained decreases as the CaO:SiO<sub>2</sub> ratio in CSH increases (Bhatty, 1987).

If metals have already been precipitated as low-solubility solids, they may gradually react with the silicate (if such a reaction is favorable) as long as free silicate is available; i.e., before it reacts with other ions in the system, such as calcium. The probable result is that the cementitious matrix will encapsulate the metal solids as hydroxides, sulfides, carbonates, etc. (see Section 4.2.1 on physical mechanisms). Continued reaction of metal ions with silicate will only occur if a continuous source of soluble silica can be created within the matrix or if the waste is pretreated to dissolve the metal hydroxides.

**4.2.2.1.5 Other Low-Solubility Phases.** Another alternative to precipitating metals as hydroxides is to bind them using insoluble substrates. Insoluble starch xanthates have been widely used for this purpose since they became commercially available in 1980 (Conner, 1990). Xanthates are produced by reacting an organic hydroxyl-containing substrate (R-OH), such as starch, cellulose, or alcohol with carbon disulfide in the presence of a strong base such as NaOH (Bricka and Hill, 1987). The structure of a Na-xanthate is represented by



Xanthates remove metals from solution by exchanging the base metal (Na) for generally heavier metals, which are bound more strongly. The selectivity for metal removal increases in the following order (Flynn et al., 1980):



Wastes stabilized by xanthates are less sensitive to pH and have better sludge dewatering properties than metal hydroxides. However, the xanthates produce large quantities of sludge that must be handled like any RCRA material (Bricka and Hill, 1987).

The effects of typical S/S techniques for binding heavy metals using cellulose and starch xanthates were investigated by Bricka (1988) and Bricka and Hill (1987), who found:

1. Xanthates of Cd, Cr, Hg, and Ni effectively immobilize these metals when bound with Portland cement.
2. Mercury precipitated by starch xanthate has lower leachability than mercury precipitated by hydroxide, even after solidification.
3. Starch xanthate binds mercury better than cellulose xanthate.
4. Cd-, Cr-, and Ni-xanthates alone do not have sufficiently low solubilities for direct disposal; thus solidification is necessary to achieve acceptable leachability levels.

#### 4.2.2.2 Organic Wastes

Aqueous wastestreams containing small amounts (10 to 1000 ppm) of organic hazardous contaminants are the most treatable organic waste forms under S/S technology (Conner, 1990). With regard to normal cement-based methods, containment will be most effective for immiscible liquids and least effective for soluble liquids (Conner, 1990). It is unclear, however, whether appreciable chemical reactions take place in the matrix. Losses may be caused by other factors, such as volatilization, which may be especially important in S/S processes that involve elevated temperatures (Weitzman et al., 1987).

As with inorganic wastes, organic constituents can undergo reactions including hydrolysis, change of oxidation state, and precipitation as some form of salt. Hydrolysis normally involves the loss of a hydroxyl group (-OH) in exchange for another functional group. Reactions must be catalyzed by a strong base to proceed at reasonable rates. Oxidation and reduction reactions can occur naturally in soils, with clays performing the role as catalysts (Warren et al., 1986). Many substituted aromatics undergo free radical oxidation. According to Dragun and Helling (1985), this group includes benzene, benzidine, ethyl benzene, naphthalene, and phenol. On the other hand, chlorinated aromatics and polynuclear organics are unlikely to be oxidized under natural conditions (Conner, 1990). Of course, oxidation can be made to occur by treating the waste with strong oxidizing agents such as potassium permanganate. However, the possible disadvantages of using such additives, such as the mobility or toxicity of the additive itself, must be weighed against the advantages. The mechanism of salt formation by organics will apply only to the ionized or ionizable organic fraction; it is not

directly applicable to nonpolar species. The formation of organic salts in S/S technology is a possible significant mechanism, but it has not been studied extensively.

One additional area of recent research is the S/S binding mechanisms of nonpolar organics in organophilic additives such as modified clays and activated carbon. Modified clays are clays that have been modified by ion exchange with selected organic compounds that have a positive charged site hence rendering the clay/organo complex hydrophilic. The binding capacity for such materials with certain types of organics has been well demonstrated. For example, Sell et al. (1992) found that sodium bentonite clay, modified with dimethyl di(hydrogenated tallow) ammonium chloride (LOCKIT®) can be used to remove phenol and chlorinated phenols from aqueous solutions. The question for organophilic additives, however, centers on whether the binding mechanism entails simple absorption or adsorption, or whether the formation of stronger covalent bonds between the additive and the contaminant is occurring. Additional evaluation is necessary.

#### **4.2.3 Concept of Surface Sealing**

The concept of "surface sealing" pertains to the situation where the surfaces of stabilized waste products are sealed, limiting the release of contaminants and the uptake of matrix-unfriendly components such as salts. Hockley and van der Sloot (1991) found that "self-sealing" may occur in some stabilized wastes. They examined the precipitation and dissolution processes in waste blocks formed from stabilization of coal combustion wastes with Portland cement and lime that had been exposed to seawater for up to 8 years. Results indicated that dissolution of calcium hydroxide, calcium sulfite, and ettringite began at the block surface and proceeded as a moving boundary toward the interior. Some calcium released by dissolution was reprecipitated as a carbonate phase, and the remainder was lost to the surrounding seawater. Magnesium ions infiltrating from the seawater were precipitated, apparently as a hydroxide phase. Concentration profiles of As, Sb, and B showed that minor elements also exhibit moving boundary effects, perhaps through association with the mineral phases. These alteration and leaching processes were confined to within 10 to 20 mm of the block surface, and many concentration profiles showed sharp discontinuities at the 10- to 20-mm region.

These discontinuities could not be explained by the simple diffusion-based models currently used to interpret leaching data. The sharp discontinuities in the concentration profiles of nonreactive sea salts led to the hypothesis that the precipitation of small crystals in pores near the block's surface restricted diffusion, a process similar to the concept of "pore refinement" previously identified in the literature on concrete durability. This process causes a slowing of all diffusion-controlled processes, including the degradation of the block matrix and the leaching of contaminants.

Similar phenomena have been observed in borosilicate glasses. Upon leaching, these glasses develop an alteration layer at the glass/water interface. The alteration layer consists of numerous growths (grouts) that precipitate and impede glass dissolution and diffusion of glass constituents into the aqueous phase (Doremus, 1979).

#### **4.3 POTENTIAL INTERFERENCES**

S/S processes can be affected by the chemical constituents present in the waste being treated and by many other factors (e.g., binder-to-waste ratio, water content, or ambient temperature). The interferences caused by the chemical constituents of the waste can affect the solidification processes and/or the chemical stabilization of the treated product as discussed in Sections 4.3.1 and 4.3.2. Waste-specific treatability studies are needed to identify and overcome such interferences. General types of interference caused by the chemical constituents include (U.S. EPA, 1990g):

- Inhibition of bonding of the waste material to the S/S material
- Retardation of setting
- Reduction of stability of the matrix resulting in increased potential for leachability of the waste
- Reduction of physical strength of the final product

The exact mechanisms for interference are not known, and because different wastes respond differently to various types of interferences, limits on

various interfering agents cannot be set. More study is needed to establish acceptable levels for interfering agents, both singly and in combination.

#### **4.3.1 Interferences with Solidification**

The contaminated materials usually treated by S/S processes are widely fluctuating, complex mixtures. Even with one waste source, the concentrations can vary by a factor of ten or more from batch to batch. Many waste constituents affect cementation chemistry by altering the setting rate or the properties of S/S-treated waste. Depending on the contaminant type and concentration, setting rate may be increased or decreased. As an example of concentration effects, mild accelerators such as chloride or nitrate anions can slow setting at higher concentrations. Treated waste properties such as porosity or flexural and compressive strength may be reduced by contaminants.

There is typically a threshold below which the contaminant has no measurable effect. Because S/S treatment performance is influenced by complex interactions of waste material and binder, it is usually not possible to quantify the threshold. Treatability studies are required to determine the feasibility of treating a specific waste.

Table 4-3 lists substances found to affect cement reactions. Many of these substances can reduce the ultimate mechanical strength of the waste form by producing cracking and spalling. Table 4-4 compiles the characteristics reported to interfere with solidification/stabilization processes and indicates their potential impacts.

#### **4.3.2 Interferences with Stabilization**

Table 4-5 summarizes some typical waste characteristics found to interfere with the stabilization processes. This table focuses on the effects of waste constituents on immobilization mechanisms, in contrast to Table 4-4, which addresses the effects on formation of a solid product. Interferences with stabilization include chemical incompatibilities and undesirable reactions. Generally, the types of effects reported in Table 4-5 are releases of noxious gases or effects resulting in the increased leaching potential of the contaminants.

**TABLE 4-3. SUBSTANCES THAT MAY AFFECT CEMENT REACTIONS:  
INHIBITION AND PROPERTY ALTERATION**

Substance or Factor	Inhibition	Property Alteration
Fine particulates	X	X
Clay	X	
Silt	X	
Ion exchange materials	X	
Metal lattice substitution	X	
Gelling agents	X	X
Organics, general	X	X
Acids, acid chlorides		X
Alcohols, glycols	X	X
Aldehydes, ketones		X
Carbonyls	X	
Carboxylates	X	
Chlorinated hydrocarbons	X	X
Grease	X	X
Heterocyclics		X
Hydrocarbons, general		X
Lignins	X	
Oil	X	X
Starches	X	
Sulfonates	X	
Sugars	X	
Tannins	X	
Organics, specific		
Ethylene glycol		X
Hexachlorobenzene		X
Phenols	X	X
Trichloroethylene		X
Inorganics, general		
Acids		X
Bases		X
Borates	X	
Chlorides	X	X
Copper compounds	X	
Lead compounds	X	
Magnesium compounds	X	
Metal salts	X	X
Phosphates	X	
Salts, general	X	X

**TABLE 4-3. SUBSTANCES THAT MAY AFFECT CEMENT REACTIONS:  
INHIBITION AND PROPERTY ALTERATION (Continued)**

Substance or Factor	Inhibition	Property Alteration
Inorganics, general (cont'd)		
Silicas	X	
Sodium compounds	X	
Sulfates	X	X
Sulfides	X	
Tin compounds	X	
Zinc compounds	X	
Inorganics, specific:		
Calcium chloride	X	
Copper hydroxide	X	
Copper nitrate		X
Gypsum, hydrate	X	
Lead hydroxide	X	
Lead nitrate	X	X
Sodium arsenate	X	
Sodium borate	X	
Sodium hydroxide		X
Sodium iodate	X	
Sodium sulfate		X
Sulfur	X	
Tin		
Zinc nitrate		X
Zinc oxide/hydroxide	X	

Adapted from: Conner, J. R. 1990. *Chemical Fixation and Solidification of Hazardous Wastes*. Van Nostrand Reinhold, New York. pp. 349-350. Used with permission of Van Nostrand Reinhold.



**TABLE 4-4. SUMMARY OF FACTORS THAT MAY INTERFERE WITH SOLIDIFICATION/STABILIZATION PROCESSES**

Possible Interfering Characteristics	Potential Interference Mechanism	Reference
Semivolatile organics or PAHs	Organics interfere with bonding of waste materials.	U.S. EPA, 1988c
Oil and grease	Weaken bonds between waste particles and cement by coating the particles. Decrease in unconfined compressive strength with increased concentrations of oil and grease.	U.S. EPA, 1988c; Cullinane and Bricka, 1989.
Phenols	Marked decrease in compressive strength.	U.S. EPA, 1988c; Cullinane and Bricka, 1989.
Nonpolar organics (oil, grease, aromatic hydrocarbons, PCBs)	May impede setting of cement, pozzolan, or organic-polymer S/S. May decrease long-term durability and allow escape of volatiles during mixing. With thermoplastic S/S, organics may vaporize from heat.	U.S. EPA, 1989g
Polar organics (alcohols, phenols, organic acids, glycols)	With cement or pozzolan S/S, phenol retards setting and may decrease short-term durability; all may decrease long-term durability. With thermoplastic S/S, organics may vaporize. Alcohols may retard setting of pozzolans.	U.S. EPA, 1989g
Solid organics (plastics, tars, resins)	Ineffective with urea formaldehyde polymers, may retard setting of other polymers.	U.S. EPA, 1989g; Wiles, 1987

TABLE 4-4. SUMMARY OF FACTORS THAT INTERFERE WITH SOLIDIFICATION/STABILIZATION PROCESSES (Continued)

Possible Interfering Characteristics	Potential Interference Mechanism	Reference
Aliphatic & aromatic hydrocarbons	Increase set time for cement.	U.S. EPA, 1989b
Chlorinated organics	Increase set time and decrease durability of cement.	U.S. EPA, 1989b
Complexing organics (hydroxycarboxylic acid, citric acid, tartaric acid, benzoic acid, EDTA)	Retard setting rate.	Dole, 1985
Presence of phenols and nitrates	Cannot be immobilized with lime/fly ash, cement, and soluble silicates; fly ash and cement; or bentonite and cement.	Stegemann et al., 1988
Metals (lead, chromium, cadmium, arsenic, mercury)	May increase setting time of cements.	U.S. EPA, 1989g
Metal salts and complexes	Increase set time and decrease durability for cement or clay/cement.	U.S. EPA, 1989b
Copper, lead, and zinc	Detrimental effect on physical properties of cement-treated waste.	U.S. Army, 1990
Halides	May retard setting, easily leached from cement and pozzolan S/S-treated waste. May dehydrate thermoplastics.	U.S. EPA, 1988c

TABLE 4-4. SUMMARY OF FACTORS THAT INTERFERE WITH SOLIDIFICATION/STABILIZATION PROCESSES (Continued)

Possible Interfering Characteristics	Potential Interference Mechanism	Reference
Soluble salts of manganese, tin, zinc, copper, and lead	Reduce physical strength of final product; cause large variations in setting time; reduce dimensional stability of the cured matrix, thereby increasing leachability potential.	U.S. EPA, 1988c
Cyanides	Cyanides interfere with bonding of waste materials.	U.S. EPA, 1988c
Arsenates, borates, phosphates, iodates, sulfides, and carbohydrates	Retard setting and curing and weaken strength of final product.	U.S. EPA, 1988c
Sulfates	Retard setting and cause swelling and spalling in cement S/S. With thermoplastic solidification may dehydrate and rehydrate causing splitting.	U.S. EPA, 1988c
Presence of coal or lignite	Coals and lignites can cause problems with setting, curing, and strength of the end product.	U.S. EPA, 1988c
Sodium borate, calcium sulfate, potassium dichromate, and carbohydrates	Interferes with pozzolanic reactions that depend on formation of calcium silicate and aluminate hydrates.	U.S. EPA, 1986c

TABLE 4-4. SUMMARY OF FACTORS THAT INTERFERE WITH SOLIDIFICATION/STABILIZATION PROCESSES (Continued)

Possible Interfering Characteristics	Potential Interference Mechanism	Reference
Oxidizers (sodium hypochlorite, potassium permanganate, nitric acid, or potassium dichromate)	May cause matrix breakdown or fire with thermoplastic or organic polymer S/S.	U.S. EPA, 1989g
Nitrates, cyanides	Increase setting time, decrease durability for cement-based.	Colonna et al., 1990
Soluble salts of magnesium, tin, zinc, copper, and lead	May cause swelling and cracking within inorganic matrix, exposing more surface area to leaching.	Colonna et al., 1990
Flocculants (e.g., ferric chloride)	Interference with setting of cements and pozzolans.	Colonna et al., 1990 p. 407
Soluble sulfates >0.1% in soil or 150 mg/L in water	Endangerment of cement products due to sulfur attack.	Jones, 1990
Soluble sulfates >0.5% in soil or 2000 mg/L in water	Serious effects on cement products from sulfur attack.	Jones, 1990
Inorganic acids	Decrease durability for cement (Portland Type I) or clay/cement.	U.S. EPA, 1989b
Inorganic bases	Decrease durability for clay/cement; KOH and NaOH decrease durability for Portland cement Type II & IV.	U.S. EPA, 1989b

TABLE 4-4. SUMMARY OF FACTORS THAT INTERFERE WITH SOLIDIFICATION/STABILIZATION PROCESSES (Continued)

Possible Interfering Characteristics	Potential Interference Mechanism	Reference
Sodium hydroxide	Increase early strength at 2 to 5% concentration but decreased early strength at 8% level.	U.S. Army, 1990
Presence of anions in acidic solutions that form soluble calcium salts (e.g., calcium chloride, acetate, and bicarbonate)	Cation exchange reactions — leach calcium from solidified/stabilized product, increases permeability of concrete; increases rate of exchange reactions.	Jones, 1990
Low-solids wastes	Large volumes of cement or other reagents required, greatly increasing the volume and weight of the end product. Waste may require reconstitution with water to prepare waste/reagent mix.	U.S. EPA, 1988c
Fine particle size	Insoluble material passing through a No. 200 mesh sieve can delay setting and curing. Small particles can also coat larger particles, weakening bonds between particles and cement or other reagents. Particle size >1/4 inch in diameter not suitable.	U.S. EPA, 1988c
Environmental/waste conditions that lower the pH of matrix	Eventual matrix deterioration.	Colonna et al., 1990 p. 407

TABLE 4-5. POTENTIAL CHEMICAL INCOMPATIBILITIES BETWEEN BINDER AND WASTE CONSTITUENTS

Characteristics Affecting Process Feasibility	Potential Incompatibilities	Reference
Volatile organics	Volatiles not effectively immobilized; driven off by heat of reaction.	U.S. EPA, 1988c
Use of acidic sorbent with metal hydroxide wastes	Solubilizes metal.	U.S. EPA, 1986c
Use of acidic sorbent with cyanide wastes	Releases hydrogen cyanide.	U.S. EPA, 1986c
Use of acidic sorbent with sulfide wastes	Releases hydrogen sulfide.	U.S. EPA, 1986c
Use of alkaline sorbent with waste-containing ammonium compounds	Releases ammonia gas.	U.S. EPA, 1986c
Use of alkaline sorbent (containing carbonates such as calcite or dolomite) with acid wastes	Releases carbon dioxide which can cause frothing.	U.S. EPA, 1986c
Use of carbonaceous sorbent (carbon, cellulose) with oily waste	May create pyrophoric waste.	U.S. EPA, 1986c
Use of siliceous sorbent (soil, fly ash) with hydrofluoric acid waste	May produce soluble fluorosilicates.	U.S. EPA, 1986c

#### **4.4 ISSUES DEALING WITH THE STABILIZATION OF ORGANIC WASTES AND OF MIXED ORGANIC AND INORGANIC WASTES**

##### **4.4.1 Introduction**

This section focuses on issues related to S/S processing of wastes in which the primary contaminants are organics or where significant quantities of organic contaminants are mixed with other types of waste, such as inorganics. Issues relating to interferences caused by low levels of organics are discussed in Section 4.3.

Threshold levels for organic interference with S/S processes exist. However, the actual level depends on the nature of the organic, the waste matrix, and the binder. Different types of interferences and some guidance on threshold levels are discussed in greater detail in Section 4.3.

Organic contaminants are more difficult to treat with inorganic S/S processes than are inorganics such as metals and metal compounds. Organics generally do not react with an inorganic matrix but instead are sorbed or encapsulated within pores. The reason organic contaminants do not react is that many of them are nonpolar and hydrophobic, whereas inorganic S/S binders are polar and hydrophilic. Therefore, additives with hydrophobic functional groups are sometimes added to binders to increase the binding affinity for the organic contaminants. Inorganics may be either entrapped or incorporated into the chemical structure, depending on the treatment process.

Wastes with very high concentrations of hazardous organic compounds are generally better suited for treatment by destructive processes such as incineration, biodegradation, chemical oxidation, and dechlorination. Another problem with organic contaminants is volatilization. Releases of volatile organics to the air during S/S treatment will occur whenever volatiles are present. Both the mixing required in treatment and the heat input (from exothermic reactions in inorganic processes or external heat input in thermoplastic processes) will contribute to the loss of volatile organics. Section 4.5 discusses air emissions in greater detail.

However, many industrial wastes and contaminated materials contain organics at low concentrations, mixed with inorganics, or in a viscous waste matrix. Application of treatment processes to destroy the organics in such wastes may be very expensive compared to the benefits and may, in some cases, be ineffective (Conner, 1990). S/S can be a very viable option. S/S processes that have been tested on or applied to various organic constituents are

listed in Table 4-6. Note, however, that an entry in this table indicates only that the binder has been successfully applied at least once in the stabilization of the indicated contaminant. The degree of stabilization and the long-term stability of the product are not indicated. Also, an entry in this table does not imply that the issue of volatilization (as opposed to immobilization) of the air emissions was properly addressed.

Mechanisms that stabilize organics are not well understood (Section 4.2.2). Some stabilization of the organics appears to occur in cementitious systems. However, it has been difficult to determine whether apparent decreased contaminant mobility is caused by sorption effects, dilution by reagent additions, sample heterogeneity, or volatilization.

Quantifying the degree of immobilization of organic contaminants in S/S-treated waste is not as straightforward as for inorganic contaminants. Aqueous leach tests may provide an estimate of the propensity for the organic contaminant to be transported in groundwater as a solute, but they do not provide a good measure of organic immobilization for nonpolar organics that have low solubility in water. For nonpolar organics, the use of nonpolar solvent extraction (e.g., the Total Waste Analysis, or TWA) has been recommended. However, this recommendation is still under consideration by EPA because it is unclear how the results of a solvent extraction relate to the environmental mobility of a contaminant in groundwater. Also, there are few if any data that demonstrate that the chemical interaction between an S/S binder and an organic contaminant is strong enough to resist leaching by an aggressive nonpolar extractant. Therefore, one of the potential pitfalls of using S/S technology to treat waste with significant nonpolar organic contaminants is the inability to adequately assess the extent of contaminant immobilization attributable to S/S treatment.

#### **4.4.2 S/S Additives Compatible with Organics**

Testing of additives to improve immobilization of organic compounds with inorganic binders has shown promising results. These additives include activated carbon, charcoal, modified clays, and condensed silica fumes (fine silica particulate prepared by condensing silica fumes).

Ontario Waste Management Corporation's Wastewater Technology Centre (WTC, 1989, p. 3) investigated the use of S/S systems with the addition of activated carbon and condensed silica fumes. The S/S process was based on



TABLE 4-6. S/S PROCESSES TESTED ON OR APPLIED TO ORGANIC-CONTAINING WASTES

Binder	Organic Contaminant	Physical Form of Waste	Reference
Bitumen	Oil and gasoline	Soil	U.S. EPA, 1989g
Chemfix <sup>a</sup>	Oil	Sludge	U.S. EPA, 1989g
Fly ash	Phenol	Sludge	Côté and Hamilton, 1984
Kiln dust	Oil	Sludge	U.S. EPA, 1989g
	Creosote	Sludge	U.S. EPA, 1989g
Lime and fly ash	Phenol	Sludge	Côté and Hamilton, 1984
	Organics	Sludge	U.S. EPA, 1989g
Lime and kiln dust	PCBs and dioxins	Sludge	U.S. EPA, 1989g
Lime and nucleophilic reagents	PCB	Soil	<i>HazTech News</i> , 1991
Organic	Kepone	Sediment	Conner, 1990
Portland cement	Latex	Waste caulk	Conner, 1990
	Phenol	Sludge	Côté and Hamilton, 1984
Portland cement and clay	Phenol	Sludge	Côté and Hamilton, 1984
	Substituted phenol	Solution	Sheriff et al., 1989

TABLE 4-6. S/S PROCESSES TESTED ON OR APPLIED TO ORGANIC-CONTAINING WASTES (Continued)

Binder	Organic Contaminant	Physical Form of Waste	Reference
Portland cement and fly ash	Phenol	Sludge	Côté and Hamilton, 1984
Portland cement, kiln dust, and a proprietary agent	Pesticides	Sludge	U.S. EPA, 1989g
Portland cement and polymer	Kepone	Soil	U.S. EPA, 1989g
Portland cement and a proprietary agent	Oil	Sludge	U.S. EPA, 1989g
	Vinyl chloride and ethylene chloride	Sludge	U.S. EPA, 1989g
	API separator sludge	Sludge	U.S. EPA, 1989g
	PCBs	Soil	U.S. EPA, 1989g
Portland cement and soluble silicate	Kepone	Sediment	Conner, 1990
	Phenol	Sludge	Côté and Hamilton, 1984
Pozzolan and proprietary agent	Oil	Soil	U.S. EPA, 1989g
Sulfur-based	Kepone	Sediment	Conner, 1990

TABLE 4-6. S/S PROCESSES TESTED ON OR APPLIED TO ORGANIC-CONTAINING WASTES (Continued)

Binder	Organic Contaminant	Physical Form of Waste	Reference
Bentonite clay modified with dimethyl di(hydrogenated tallow) ammonium chloride and mixed with Type I Portland cement	Phenol and chlorinated phenols	Soil	Sell et al., 1992

<sup>a</sup> Proprietary binder formulation.

Note: An entry in this table does not mean that the binder will work under all conditions or that it necessarily worked under the conditions of the reported study. In addition, the degree of solidification/stabilization achieved was not reported in all references, nor was the extent of contaminant volatilization uniformly addressed.

Portland cement and proprietary additives. The waste was metal-finishing sludge spiked with 500 mg/kg each of acenaphthene, aniline, bis(2-chloroethyl) ether, phenol, benzene, and trichloroethylene. WTC found both physical and chemical mechanisms to be important in containing the contaminants. Activated carbon was found to be the best additive for immobilizing organic contaminants. With the exception of phenol, none of the contaminants tested in this study were detected in the aqueous leachate. Condensed silica fumes were the best additive to entrap organic contaminants physically, and the formulation tested resulted in small increases in waste mass and volume. The physical containment factor was about ten times better than that of the other cement-based processes. Further investigation of both additives is needed to define dosages, applicability to various waste constituents, and long-term stability.

Modified clays can be added with inorganic processes to reduce the mobility of organic wastes. Investigations by Lagoutte et al. (1990) indicated that S/S processes using modified clays show promise as an effective treatment for hazardous waste containing such organic contaminants as pentachlorophenol. Some clays, such as bentonite, can be modified by introducing quaternary ammonium compounds into the spaces between the alumina and silica layers. These aqueous spaces in the clay are normally hydrophilic and polar, but they can be made more hydrophobic and less polar by introducing quaternary ammonium compounds with long-chain alkyl groups or aromatic groups attached.

One common S/S formulation combines Portland cement, treated clay, and coal fly ash. The addition of coal fly ash produces a high-strength solid, although the combination generally requires longer curing times than with Portland cement alone. The residual carbon content of the coal fly ash has been shown to have an ability comparable to that of activated carbon for adsorption of organics (Lagoutte et al., 1990). Thus, both the modified clays and the coal fly ash act to immobilize the organics.

Sheriff et al. (1989) investigated the use of activated charcoal and tetra-alkylammonium-substituted clays as prestabilization adsorbents for phenols and chlorinated phenols prior to application of cement-based S/S processes. Charcoal is a well-known adsorbent, whereas the use of the substituted clays exploits the hydrophobic properties of the alkyl groups to fix organic materials within the clay matrix. Wyoming bentonite substituted with hexadecyltrimethylammonium bromide and benzyltrimethyltetradecylammonium chloride (Chemical Abstracts Registry Number 139-08-2) was found to be very

effective in adsorbing chlorinated phenols with adsorption capacities of ~150 mg of chlorinated phenol per gram of clay. The results indicated a clear relationship between the chain length of the alkyl ammonium ion in the exchanged clay and the ability of the clay to adsorb a particular phenolic compound. Activated charcoal was found to adsorb effectively 180 mg of phenol or chlorinated phenols per gram of charcoal.

Cost is an important consideration in using additives such as modified clays and activated charcoal. Most additives are more expensive than binders such as cement. If a large quantity of additive is needed, the cost can be prohibitive. However, the additives frequently are effective in low concentrations. Costs of S/S processes are discussed in greater detail in Section 4.10.

Many of the additives used to reduce organic mobility in inorganic binders rely on sorption mechanisms. Sorption, especially with activated carbon, is at least partially reversible. The long-term performance of any S/S-treated waste is an important issue that is not fully resolved (Section 4.7). Long-term performance of binders that rely on sorption should be examined with particular attention.

#### **4.4.3 Approach to Evaluating Feasibility of S/S for Wastes Containing Organics**

Figure 4-2 presents a proposed approach in the form of a decision tree for evaluating the feasibility of S/S for treating organic-bearing wastes. This decision tree provides guidance for determining whether S/S is an acceptable alternative for treating a particular waste containing organics. At the outset of the process, the following information is needed (Wiles and Barth, 1992):

- The quantity of organic material relative to inorganic contaminants and other materials, including information on chemical and physical characteristics.
- The type and amount of inorganic compounds that would remain if all organics were destroyed or removed.
- The chemical and physical characteristics of residuals from the destroyed or removed organics.

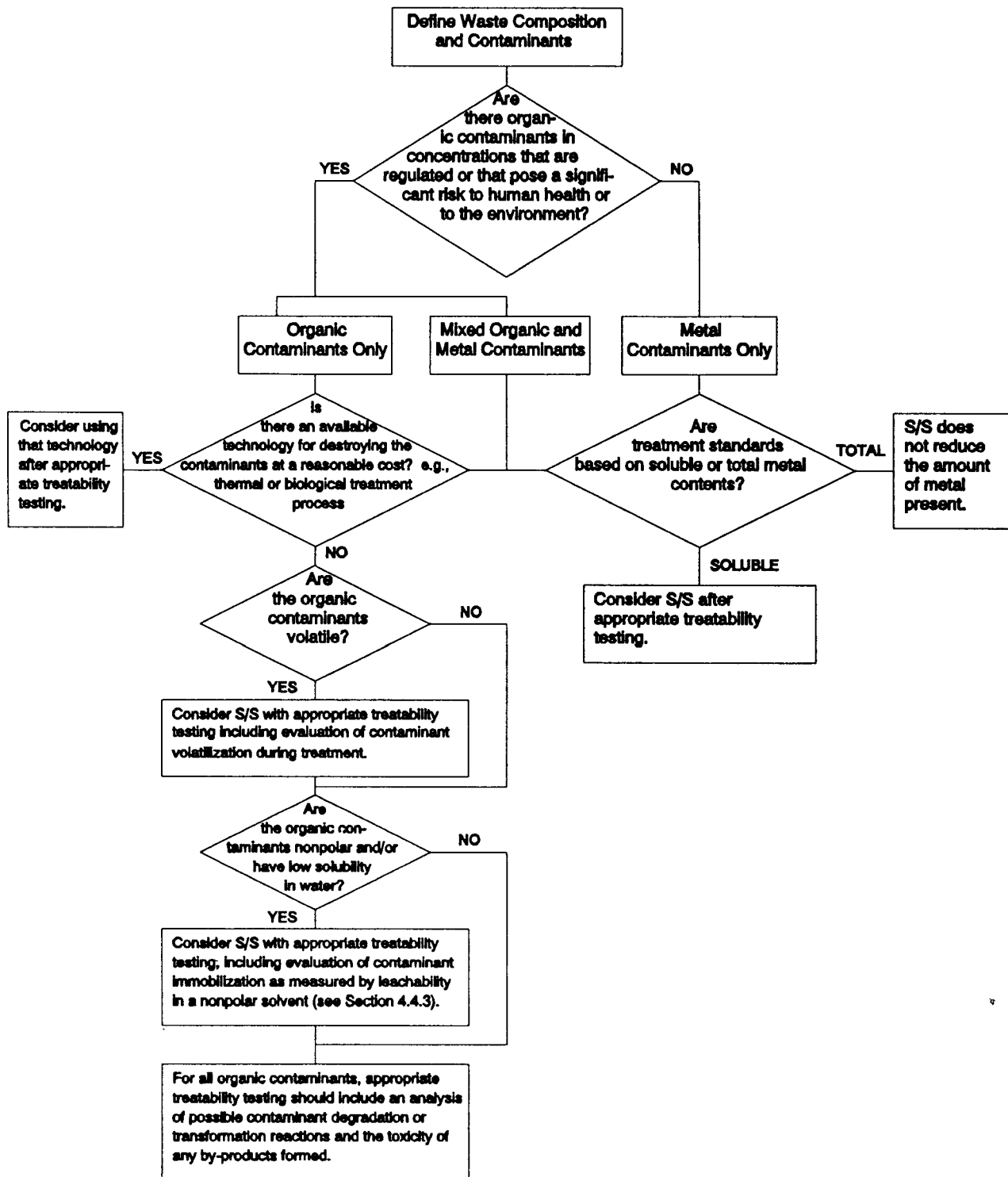


FIGURE 4-2. GENERAL DECISION TREE FOR S/S APPLIED TO ORGANIC CONTAMINANTS

The first step in the decision tree is to evaluate whether the organic contaminants present in the waste pose a significant hazard or threat to human health or the environment. This evaluation is carried out as follows:

- Determine whether the waste is either a listed or characteristic RCRA hazardous waste. If the waste is not RCRA hazardous, then the following conservative risk-based approach assuming no containment of the waste by the S/S process is proposed.
- Determine the concentration of the organic present in the waste to be treated. Determine the compound that poses the highest health or environment risk (quantity and toxicity). Then, determine the level and/or concentration of the highest risk compound that can be allowed without creating a health or environmental risk at the given site.

This conservative approach assumes that:

- The S/S process will not treat or contain the selected compound. Therefore, all of it will be released from the solidified waste; and/or
- All of the compound will be released as air emissions during the S/S process (Wiles and Barth, 1992).

If the concentration of the highest-risk compound is above the level determined to pose a health or environmental risk at the given site, then pretreatment to remove or destroy the organic(s) will be required and/or air emission controls and treatment will be needed on the S/S treatment train.

After determining that the waste contains organic contaminants that require treatment, then the decision tree in Figure 4-2 addresses the following four important issues pertaining to the feasibility and practicality of using an S/S treatment approach on the organic-bearing waste:

1. Is there an applicable technology that will either destroy or remove the organic contaminants?
2. Are the organic contaminants volatile and likely to be released as air emissions during S/S treatment?
3. Do the organic contaminants have low solubility in water? If so, the inherent potential for migration

in groundwater is low and will invalidate the meaningfulness of aqueous leach tests.

4. Will S/S treatment cause the organic contaminants to degrade or transform into toxic by-products?

These four issues are discussed further below.

#### **4.4.3.1 Destructive or Removal Technologies Versus S/S**

The concern over the use of S/S versus a destructive or removal technology for treating organic-bearing wastes relates to the hierarchy of waste treatment — that is, all other factors being equal, technologies that destroy or reduce the amount of contaminants are preferred over technologies that simply immobilize the contaminants (see Section 1.1.2). Technologies that are capable of degrading organic constituents to innocuous components such as CO<sub>2</sub> and H<sub>2</sub>O, or of separating organic contaminants from inorganic constituents based on thermal or chemical properties, are preferred over immobilization technologies. Degradation and separation-based remedies are permanent, whereas immobilization may lose its effectiveness over time (see Section 4.7 on Long-Term Performance).

Consequently, S/S treatment typically is not used at sites heavily contaminated with organic wastes (Wiles and Barth, 1992). Alternative technologies (e.g., incineration, steam stripping, vacuum extraction, bioremediation) should be considered first. However, S/S treatment is frequently appropriate for the residues remaining after the use of one of these other technologies, or for soils or sludges containing low concentrations of organics. A well-designed and controlled treatability study should be conducted to assess S/S effectiveness and to select and design a proper S/S process (see Section 2).

#### **4.4.3.2 Volatile Organic Contaminants**

A wide variety of organic constituents in hazardous waste are volatile to varying degrees. As indicated in Figure 4-2, when volatiles comprise the dominant constituents, a destructive or removal technology is usually preferred. However, there are many cases where volatiles are present as a relatively minor contaminant, but in concentrations high enough to pose a potential health or environmental risk. For example, some volatiles, mixed



with metals, salts, or semivolatile organics, may respond to S/S without pretreating to remove the organics. Pretreatment or treatment trains can add significantly to the cost and the time needed to complete the cleanup. The difficulty is that volatile contaminants are not always effectively treated using S/S.

Demonstration that the volatile organic contaminants are being immobilized during treatability studies greatly complicates the treatability study. Special precautions have to be made during treatability testing to assess volatile organic emissions. This means that proper controls must be used to perform a complete mass balance, in which all organic air emissions are collected and analyzed during the treatability study, from the point of waste compositing and mixing through the curing of the treated waste specimen. The required containment, sampling, and analysis equipment and activities can more than triple the cost of the treatability study. The testing should be structured to allow a closed mass balance to fully account for the organic materials.

Unfortunately, air emissions monitoring during treatability testing is infrequently carried out as needed, and numerous studies have reported the apparent immobilization of volatile contaminants when the post-treatment reduction in contaminant concentration was, in actuality, caused by volatilization. Nevertheless, volatile contaminants in low concentrations can be and have been successfully treated using S/S when precautions are taken to minimize volatilization. Well-designed treatability studies using technology that can be implemented in the field are needed.

#### **4.4.3.3 Nonpolar Organic Contaminants**

The third issue pertains to the low aqueous solubilities of numerous organic contaminants, both volatile and nonvolatile. Polar organic contaminants such as carboxylic acids, alcohols, and phenols are typically very soluble in water. Accordingly, the TCLP aqueous leaching test defines standards for selected organic contaminants with adequate solubility in water.

However, nonpolar organics such as polyaromatics, benzene, tetrachloroethane, and hydrocarbons are generally insoluble in water. Hence, for this latter group of compounds, an aqueous leach test is a meaningless indicator of the degree of immobilization caused by S/S. Therefore, the use of a nonpolar solvent extraction (e.g., the TWA) has been recommended.

However, this recommendation is still under consideration by EPA because it is unclear how the results of a solvent extraction relate to the environmental mobility of a contaminant in groundwater. Also, there are few if any data that demonstrate that the chemical interaction between an S/S binder and an organic contaminant is strong enough to resist leaching by an aggressive nonpolar extractant. Therefore, one of the potential pitfalls of using S/S technology to treat waste with significant nonpolar organic contaminants is the inability to adequately assess the extent of contaminant immobilization attributable to S/S treatment.

#### **4.4.3.4 Degradation and By-Product Formation**

The final issue in Figure 4-2 pertaining to the immobilization of organic contaminants applies to all types of organic contaminants — volatile and nonvolatile, polar and nonpolar — in all ranges of concentrations. Because organic constituents readily undergo chemical transformation reactions and S/S binders are associated with fairly aggressive chemical environments (such as increased temperature and alkaline pH), the potential for chemical transformation or degradation always exists; and a post-treatment reduction in the concentration of an organic contaminant may be erroneously interpreted as evidence for immobilization when it, in actuality, may be attributable to contaminant transformation. Moreover, chemical transformation or degradation may result in the formation of by-products which can be more or less toxic than the parent compound. Therefore, it is not sufficient to demonstrate the extent to which transformation is occurring. The identities of the by-products and their toxicities must also be characterized. Unfortunately, the process of detecting and analyzing by-products can be extremely expensive and can therefore be a deterrent to considering S/S as an option for the treatment of organics-bearing wastes.

### **4.5 AIR EMISSIONS AND CONTROL**

In considering S/S options, the possibility of air emissions must be taken into account. Many wastes contain VOCs that can escape into the atmosphere. Even compounds not generally considered volatile can be released by the mixing and heating operations involved in S/S. In addition to volatilization, other forms of air emissions, such as fugitive dust or particulates,

must be taken into account. The cost of installing and operating equipment to prevent air emissions can be significant. The local air board or other cognizant regulatory agency should be consulted to define air emissions issues.

#### **4.5.1 Volatile Organic Compounds**

Volatile organic compounds can escape into the atmosphere during the mixing and heating steps of the S/S process, and even during sampling, sample handling, and sample preparation prior to analysis. For example, one study of volatilization during S/S processing found that an average of 0.11% of the feed into the process was emitted to the air (Ponder and Schmitt, 1991). As a general rule, sites contaminated with only volatile contaminants should not be considered as candidates for S/S (Wiles and Barth, 1992) (see also Sections 4.4 and 2.4). However, volatile and/or semivolatile organic compounds are frequently present as secondary components in wastes that contain mostly metallic or other inorganic contaminants.

In wastes containing VOCs, significant VOC losses to the atmosphere will occur with remediation activities that involve exhumation of the waste. In situ S/S technology that produces a monolithic product is capable of reducing VOC losses but not of eliminating them (Spence et al., 1990). Also, volatiles can continue to escape from a solidified waste form, regardless of the reduction in pore space and increase in tortuosity.

The quantities of VOCs acceptable for S/S should be based on a risk assessment for the given site and/or on the result of a treatability study that includes a mass balance of the organics before, during, and after treatment. As a worst-case scenario, the risk assessment should assume that none of the highest risk compounds will be retained by the S/S process and/or that all the compounds will be lost via air emissions during S/S processing (Section 4.4).

A system for measuring the emissions of organic compounds from mixing processes such as those used in S/S activities is currently under development. This "modified headspace" sampling system, having been demonstrated at VOC emission rates ranging from less than 1 milligram per minute up to tens of grams per minute, can be used at the laboratory scale to measure organic emissions both from the S/S process and from the S/S-treated waste during curing. Such equipment can also be used in conjunction with a full-scale

remediation effort by testing samples of the treated waste from the field in the laboratory (Weitzman et al., 1990).

#### **4.5.2 Particulates and Other Emissions**

In addition to gaseous emissions from volatile organics, particulate releases to the atmosphere from operations associated with S/S treatment can also be a concern. Possible sources of air pollutants and fugitive dust in a field S/S project include excavation, the movement of trucks and other heavy equipment, and the loading and processing of waste and binder materials in the mixing device. In the study cited in Section 4.5.1 for VOC volatilization, it was also found that an average of more than 0.01% of the waste feed being processed was released as particulate emissions (Ponder and Schmitt, 1991). Care must frequently be taken to reduce the escape of both contaminated particulates and fugitive dust during treatment. Typical engineering controls include scrubbers for certain types of air pollutants and wetting the waste or ground to reduce dust.

Various guidelines exist for determining maximum air emissions of contaminants and fugitive dust during remediation projects. For example, *Toxic Air Pollutant Source Assessment Manual for California Air Pollution Control Districts and Applicants for Air Pollution Control District Permits* specifies a risk-screening methodology for evaluating air emissions and a fugitive dust concentration limit for remediation projects in California (Interagency Working Group, 1987). The risk-screening methodology is a simple, conservative estimation of the maximum possible health impacts associated with air emissions during the duration of the project. If the project fails the initial risk screening calculation, then a much more detailed risk assessment may have to be conducted prior to initiating field treatment.

#### **4.5.3 Controlling Air Emissions**

Depending on the nature of the anticipated air emissions, it may be necessary to adopt control measures to ensure that volatile and particulate emissions are within acceptable levels. Equipment such as air scrubbers or large enclosures around the treatment area may have to be employed as an adjunct to the S/S treatment process, thus increasing the complexity and costs associated with S/S. The U.S. EPA's Office of Air Quality Planning and

Standards (OAQPS) is developing guidance for controlling air emissions at RCRA treatment, storage, and disposal facilities (TSDs). This guidance will require many S/S processes to incorporate capture and control mechanisms for volatile constituents. Even those projects involving relatively low levels of volatile constituents may be affected (Wiles and Barth, 1992). However, apart from this guidance for TSD facilities, air emissions and controls are currently assessed on a project-by-project basis.

#### **4.5.4 Significance of the Amended Clean Air Act**

The purpose of the Clean Air Act (CAA) is to:

- protect and enhance the quality of the nation's air resources so as to promote the public health and welfare and the productive capacity of its population
- initiate and accelerate a national research and development program to achieve the prevention and control of air pollution
- provide technical and financial assistance to state and local governments in connection with the development and execution of their air pollution prevention and control programs
- encourage and assist the development and operation of regional air pollution control programs

Within these purposes, waste minimization or pollution prevention is encouraged but, in most cases, is not mandated. Under the CAA, regulations have been promulgated that give industry the choice of either preventing or controlling air emissions. These regulations include the National Ambient Air Quality Standards (NAAQS); National Emission Standards for Hazardous Air Pollutants (NESHAP), which control emission of specific pollutants for specific industries; and permitting requirements.

Just as the forthcoming RCRA-related guidelines for TSD facilities will affect S/S operations, the amended Clean Air Act portends increased use of capture and control mechanisms. Stricter regulations will require more careful screening of candidate sites for the application of S/S technology. This screening will be based on the potential to achieve regulatory compliance and the cost of achieving regulatory compliance.

#### 4.6 LEACHING MECHANISMS

After disposal, the S/S-treated waste may eventually come into contact with water. The S/S processes are aimed at either reducing the mobility of the contaminants or reducing access of water to the contaminant, or both. However well the S/S waste is stabilized and isolated from the hydrosphere in disposal, some transport of contamination from the S/S-treated waste into the groundwater will eventually occur. Complete immobilization of contaminants is not a realistic expectation (Bishop, 1988).

This process of slow extraction of contaminants from the S/S-treated waste by water or some other solvent is called "leaching." Leaching tests are discussed in Section 3.2. Leaching can occur when the S/S-treated waste is exposed to stagnant leachant or to a flow of leachant through or around the waste. Leaching is the general term for complex physical and chemical mechanisms. These mechanisms mobilize a contaminant and transport it away from the waste.

In a disposal scenario, the solvent will usually be groundwater. Leaching occurs when the contaminants in the S/S-treated waste come into contact with the groundwater. The leachant flow and composition are determined by the physical properties of the disposal area and any engineered barriers at the disposal site. Leaching tests may use water, aqueous solutions of acids or salts, or organic liquids to model various disposal scenarios, determine waste composition, measure diffusion coefficients, or for other specific test purposes.

There is significant experimental evidence that, when waste stabilized by cement or similar pozzolanic materials is exposed to acidic water, significant matrix dissolution occurs. Thus, the leaching rates of contaminants from stabilized waste will be a function of both the dissolution rate as well as the diffusion rate of contaminants into the leachate.

In the Netherlands, a database has been developed to collate, organize, and analyze information about the leaching of contaminants from waste and waste-containing materials (de Groot and van der Sloot, 1992). Organization of the information into a database is intended to assist identification of systematic trends in leaching behavior and mechanisms.

#### **4.6.1 Leaching Associated with Inorganic S/S Processes**

The typical S/S-treated waste resulting from use of an inorganic binder is a porous solid. The pore space contains some mixture of water and gas, so many different phases can be present. There may be several different solid phases, each containing contaminants. For example, contaminants may be present in the cement mineral phases because of substitution in the crystal structure or as a separate phase, such as a precipitated solid. There can also be one or more aqueous phases such as an adsorbed layer of fluid as well as the bulk pore fluid. The sorbed layer may have a different contaminant composition than the bulk fluid. There can also be one or more nonaqueous phases if organic contaminants are present (WTC, 1990a).

Prior to introduction of the leachant, the pore system will have approached equilibrium conditions with the surrounding solid phase. That is, the contaminants are associated with a specific phase, and there is no net transfer between the phases. The leachant changes the composition of the system and disrupts the chemical equilibrium, resulting in the mobilization of contaminants. The new system may evolve towards a new equilibrium if sufficient time passes with no leachant renewal.

The two basic mechanisms in the leaching process are mobilization and transport of the contaminant. The leachant mobilizes contaminants within the pores by dissolving the contaminant. Dissolution results from a combination of chemical and physical mechanisms. Examples include bulk dissolution of mineral phases in the S/S-treated waste, washoff of surface contaminants, changing chemical parameters such as pH or Eh dissolving a formerly insoluble phase, desorption of contaminants, or other mechanisms (deGroot and van der Sloot, 1992). Factors that affect the extent of equilibrium concentrations include the solubility of the constituent and the chemical makeup of the pore water. Under neutral conditions, the leaching rate is controlled by molecular diffusion of the solubilized species. However, if the leachant induces acid conditions, the rate will also depend on the rate of back diffusion of the hydrogen ion because the pH determines the chemical speciation within the S/S-treated waste (Cheng, et al, 1992).

As more soluble constituents are leached from a relatively insoluble solid matrix, a layer deficient in the leached constituents develops. Under low pH conditions, both  $H^+$  and the leachable constituents must diffuse through this layer in opposite directions. The leaching rate in the leached layer

should eventually be limited by diffusion of constituents, because  $H^+$  diffuses much faster than other species. However, this layer may not be rate-limiting in the overall process (Cheng and Bishop, 1990). As constituents leach, the layer may become more porous compared to the unleached solid, so that molecular diffusion in the pore water and boundary layer phenomena become the limiting factors (Conner, 1990).

Transport of the mobilized contaminants occurs by bulk advective flow or diffusion. If water flows within the S/S-treated waste, advective transport causes contaminants that have been mobilized by reactions in the pores to flow through the waste. The velocity of leachant moving through the pores will vary considerably in both magnitude and direction due to the small size and the tortuosity of the pores (WTC, 1990b). In most S/S-treated waste, the pores are small and tortuous, so the advective transport is small. However, contaminant movement still occurs by molecular diffusion (Crank, 1967).

Only a fraction of the pores within the S/S-treated waste are linked to each other and to the outside to form what is referred to as "connected porosity." The pores that are not linked to this network are referred to as "closed porosity." Also, large pore spaces may be connected by small-diameter pathways, resulting in "occluded porosity." The micromorphology of the matrix – including the number, size, and degree of connection of the pores – will determine how quickly water can permeate through an S/S waste (i.e., hydraulic conductivity) and will influence the leaching process. As might be expected, leaching occurs most quickly through the connected pores.

In most cases, cement-based monoliths have low hydraulic conductivity, which limits the amount of leaching water contacting the matrix. However, it has been shown that the hydraulic conductivity of S/S waste may vary over several orders of magnitude, from low values typical of compacted clay to higher values typical of silty soils (Côté et al., 1986). Hydraulic conductivity of the waste determines whether leaching rates will be controlled by advection or by molecular diffusion. Advection is more important than diffusion when hydraulic conductivity is larger than  $10^{-7}$  cm/s. On the other hand, slow diffusion limits transport rates when hydraulic conductivity is lower than  $10^{-7}$  cm/s. If the hydraulic conductivity of the waste is much lower than that of the surrounding material, infiltrating water such as rainwater or groundwater follows the path of least resistance and flows around



the waste. In this case, leaching is limited by molecular diffusion in the connected porosity of the S/S waste matrix because, when contaminants reach the interface of the S/S waste and surrounding material, they are carried away by the groundwater. If, on the other hand, the hydraulic conductivity of the solidified waste is on the same order of magnitude or higher than that of the surrounding material, water flows around and through the waste. In that case, the pore water solution is displaced, and leaching takes place largely by advection (Côté and Bridle, 1987, p. 60).

The surface-area-to-volume ratio (SA/V) of a waste product greatly influences the release of potentially harmful elements to the environment. A smaller SA/V results in a lower rate of release. The leaching percentage relative to the total amount of an element present in a waste form is related to the SA/V, for a given exposure time. Therefore, all measures leading to products with a smaller SA/V lead to a proportional decrease in leaching percentage but the long-term quantities released are not decreased (van der Sloot et al., 1989).

Chemical speciation also influences leaching. Van der Sloot et al. (1989) found that elements leached from cement-based waste products are mainly anionic species such as  $\text{MoO}_4^{2-}$ ,  $\text{BO}_3^{3-}$ ,  $\text{VO}_4^{3-}$ ,  $\text{F}^-$ , and  $\text{SO}_4^{2-}$ . These anions are associated with cationic species typical of cement-based waste forms such as calcium. Leaching of metals such as copper, cadmium, zinc, and lead typically is limited when the pH in the pore solution remains above 8 or 9, but can increase at very high pH (above 11.5 or 12). Van der Sloot et al. (1989) concluded that chemical speciation of potentially hazardous elements within a waste product and the interaction of these elements with matrix components within the pore system are crucial for determining the release rate to the environment. Also, they suggested that more information on different ways of contact with water is needed, particularly in relation to pH, to allow utilization of intrinsic leach parameters in a wide range of environmental conditions.

For cement-based S/S processes, sulfate can increase leaching rate. The onset of the leaching rate increase may be delayed, however, so test results immediately after setting may be misleading. Sulfate either in the cement or present in the waste causes formation of ettringite, which slowly hydrates and expands, causing an increase in porosity and possible breakdown of the waste form. Sulfites and sulfides are also a problem because they may

slowly oxidize to sulfate, increasing the solid volume and causing the waste to crack.

Poon et al. (1985) found that the microstructure of the solidified waste was important in leaching metals from the cementitious matrix. They assessed mechanisms of zinc and mercury leaching from cement/silicate stabilization processes using extended leaching tests, scanning electron microscopy, and powder X-ray diffraction. After an extended leaching period, massive breakdown of the matrix occurred with a subsequent dramatic increase in leachate concentration. Once the structural integrity of the stabilized waste was removed, massive leaching of zinc and mercury occurred.

#### **4.6.2 Leaching Associated with Organic S/S Binders**

The thermoplastic and thermosetting resin binder processes operate mainly by encapsulating the waste. The S/S-treated waste is, therefore, less porous than the material resulting from S/S processes using inorganic binders. The leaching process requires the same two fundamental mechanisms discussed in Section 4.6.1, mobilization and transport. However, the organic binder systems rely more on denying the leachant access to the contaminant than on immobilization.

#### **4.6.3 Leaching Models**

Several models of leaching mechanisms have been developed to predict the rate of release from the stabilized waste matrix. Modeling is the only existing method for predicting long-term performance because it is impractical to conduct empirical leaching tests for hundreds or thousands of years and because accelerated tests are not well developed.

##### **4.6.3.1 Dissolution/Diffusion Kinetics**

The problem of kinetics, with regard to the aqueous dissolution of a solid or to the preferential dissolution of a chemical species from a solid, has long been studied. Several factors may be involved. For example, if more than one kinetic process takes place, it must be determined which (if any) of the processes controls the overall reaction rate. The shape of the solid, the existence of any surface-connected porosity, the charge state of the dissolv-

ing species, and the chemistry of the aqueous medium into which the solid is dissolving are also considered.

The discussion that follows considers two kinetic processes from a largely qualitative point of view: the dissolution reaction itself, which consists of mass transport across the solid/liquid interface, and chemical diffusion away from this interface into the surrounding aqueous medium. It is assumed for this specific example that the rate of supply of dissolving material from the bulk of the solid to this interface occurs quickly. It is also assumed that the solution is quiescent, so that convective flow does not contribute to the mass transport. Two fundamentally different types of systems are considered within this context: a nonporous solid dissolving into an essentially infinite aqueous medium and a porous solid for which dissolution takes place principally into the interconnected solution-containing pores, coupled with diffusive transport through the pores to the solution outside the material. Idealized models of these two systems are described in Sections 4.6.3.1.1 and 4.6.3.1.2 to illustrate the concepts in leaching models.

If the dissolving chemical species is electrically charged, considerations of charge neutrality in the solution become important, as does mass transport in the solution by electromigration. The species also may react chemically with other species within the aqueous medium. Diffusing ions may also react with the matrix in the leaching zone, adsorbing or precipitating, which can slow their release. Diffusing ions may also react with the matrix in the leaching zone, adsorbing or precipitating, which can slow their release. These factors are not considered here. However, a general treatment of ionic transport within a crevice-like region, which could be applied to dissolution and diffusive transport in a porous solid, has been presented by Markworth and Kahn (1985).

**4.6.3.1.1 Nonporous Solid.** For a nonporous solid, the two kinetic processes, i.e., dissolution at the solid/liquid interface and chemical diffusion of the dissolved species away from the interface, occur sequentially. For this case, the interface may be regarded as a spatially localized "source" of the dissolving species.

At the solid/liquid interface, the flux of matter due to the dissolution reaction must be equal, point by point, to the diffusive flux in

the solution to avoid a nonphysical accumulation or depletion of matter at the interface. Consequently, the slower of the two processes is the one that dominates the overall kinetics.

Three characteristic values for the aqueous concentration of the dissolved species are important in describing the overall kinetics for this case:

1.  $C_e$ , the concentration that would exist at the interface under conditions of thermodynamic equilibrium.
2.  $C_i$ , the actual, instantaneous concentration at the interface.
3.  $C_\infty$ , the far-field concentration, i.e., the value at distances far from the interface.

The "driving force" for the dissolution reaction depends upon the difference  $C_e - C_i$ . If  $C_i < C_e$ , the net transport of matter across the interface occurs from solid to liquid as the solid dissolves, while the opposite is true for  $C_i > C_e$ . If these two concentrations are equal, there is no net transport across the interface. Likewise, the driving force for chemical diffusion is the difference  $C_i - C_\infty$ , assuming monotonic variation of the concentration from the interface to the far field. If  $C_i > C_\infty$ , matter diffuses away from the solid/liquid interface, the converse is true for  $C_i < C_\infty$ .

Two limiting or extreme cases exist for the overall kinetics; one or the other of these cases is often satisfied in nature.

1. In the dissolution-controlled case, diffusion occurs rapidly compared to the dissolution reaction so the driving force required to maintain the diffusive flow is very small and  $C_i \cong C_e$ .
2. In the diffusion-controlled case, diffusion occurs slowly compared to the dissolution rate. For this case, dissolution is rapid but a concentration gradient is needed to drive the diffusion process so  $C_i \cong C_\infty$ .

For dissolution-controlled kinetics, dependence on diffusion-related rate constants is virtually nonexistent, whereas for diffusion-controlled kinetics, dependence on dissolution-related rate constants similarly vanishes.

The intermediate case is that for which neither of the two mass transport processes controls the overall kinetics. For example, the solid is dissolving when  $C_{\infty} < C_i < C_e$ , but it is growing by supply of dissolved species from solution when  $C_e < C_i < C_{\infty}$ .

Figure 4-3 illustrates the two limiting cases and the intermediate case. The actual concentration profile of the dissolved species in the aqueous medium would generally be a complex function of position and time as well as of the geometry of the dissolving solid.

It should be noted that this view of the dissolution process, which is widely applied in practice, must be used carefully or be modified in some cases. One such case, considered by McCoy and Markworth (1987), involved the dissolution of glasses containing high-level nuclear waste. One question there concerned how these "impure" materials actually do dissolve. In their work, McCoy and Markworth assumed that the material dissolves congruently. To describe this process, they assumed first-order, dissolution-controlled kinetics, with transport of silicon across the surface/solution interface being the rate-limiting factor. As another case, consider a dissolving material which consists of two different, distinct phases, with one phase tending to dissolve into the surrounding aqueous medium much more rapidly than the other. The more soluble phase will be preferentially dissolved (i.e., leached), leaving behind a material that is enriched in the less soluble phase. Of course, the microstructure of the material left behind will depend on the morphology of the two phases prior to dissolution. If, for example, the more soluble phase exists as an interconnected network, then the leached portion of the material will consist of a porous structure that likewise contains a network of interconnected porosity, assuming that the solution can penetrate into the porous structure as it is being created. This process could be inhibited if transport of dissolved species through the solution, within the porous structure or away from the external surface, occurs slowly.

**4.6.3.1.2 Porous Solid.** The situation differs for a porous solid, with liquid penetrating and filling the porous structure, where dissolution can occur along the entire length of the pores. The distinctly sequential

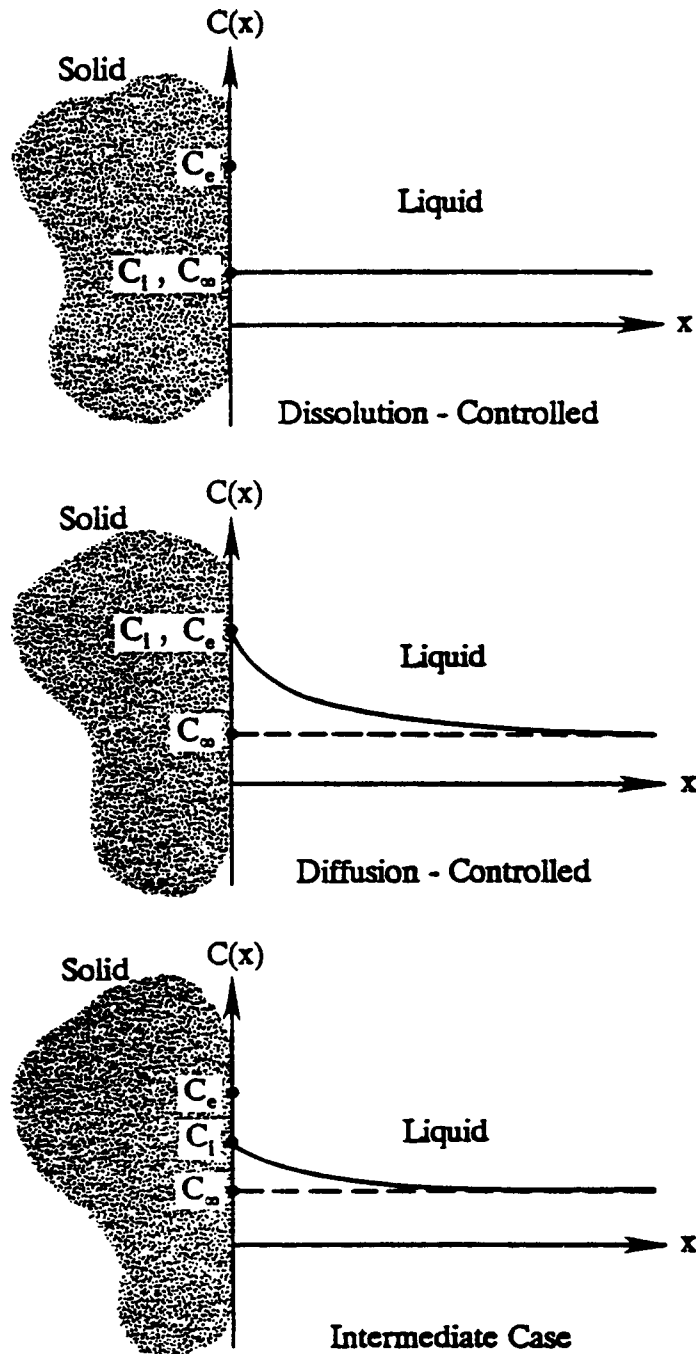


FIGURE 4-3. SCHEMATIC ILLUSTRATION OF CONCENTRATION PROFILES,  $C(x)$ , CHARACTERISTIC OF SPECIES DISSOLVING FROM A NONPOROUS SOLID INTO AN AQUEOUS MEDIUM, WITH  $x$  BEING THE DISTANCE INTO THE SOLUTION MEASURED FROM THE SOLID/LIQUID INTERFACE. THE TWO RATE-LIMITING CASES AND AN INTERMEDIATE CASE ARE SHOWN.

coupling between the two processes, characteristic of nonporous solids, does not exist. The "source" of dissolving species, considered to exist at the pore/solution interface, is *not* spatially localized as it is for a nonporous solid. Instead, dissolution can take place along the entire length of the pores within which the dissolved species is diffusing, the pore walls acting as a spatially extended "source" of this species to the solution within. Diffusion of the species takes place within the network of pores until release occurs at the intersection of the pores with the external surface. Figure 4-4 shows this type of mass-transport kinetics.

#### 4.6.3.2 Examples of Existing Models

The complex relationship between dissolution and diffusion for a porous solid means that the overall rate of release of dissolved species to the external surface depends on both dissolution-related and diffusion-related rate constants, even if one occurs faster than the other.

Godbee and Joy's widely used empirical model (Godbee et al., 1980) assumes that leaching is controlled by diffusion through the solid, and that a zero surface concentration exists (i.e., contaminant dissolves into the bulk liquid from the surface immediately). The equation takes the form:

$$\left[ \frac{\sum a_n}{A_o} \right] \left[ \frac{V}{S} \right] = 2 \left[ \frac{D_e}{\pi} \right]^{0.5} t_n^{0.5} \quad (1)$$

where

- $a_n$  = contaminant loss during leaching period  $n$  (mg)
- $A_o$  = initial amount of contaminant present in the specimen (mg)
- $V$  = volume of specimen ( $\text{cm}^3$ )
- $S$  = surface area of specimen ( $\text{cm}^2$ )
- $t_n$  = time to end of leaching period  $n$  (sec)
- $D_e$  = effective diffusion coefficient ( $\text{cm}^2/\text{sec}$ )

Models have also been developed to account for other factors and conditions in the leaching process. For example, where Godbee and Joy's model assumes leaching from an infinite depth, leaching in cementitious waste forms occurs in a narrow, but inwardly-moving, leaching zone. A model that addresses this is discussed in Section 4.6.3.3.

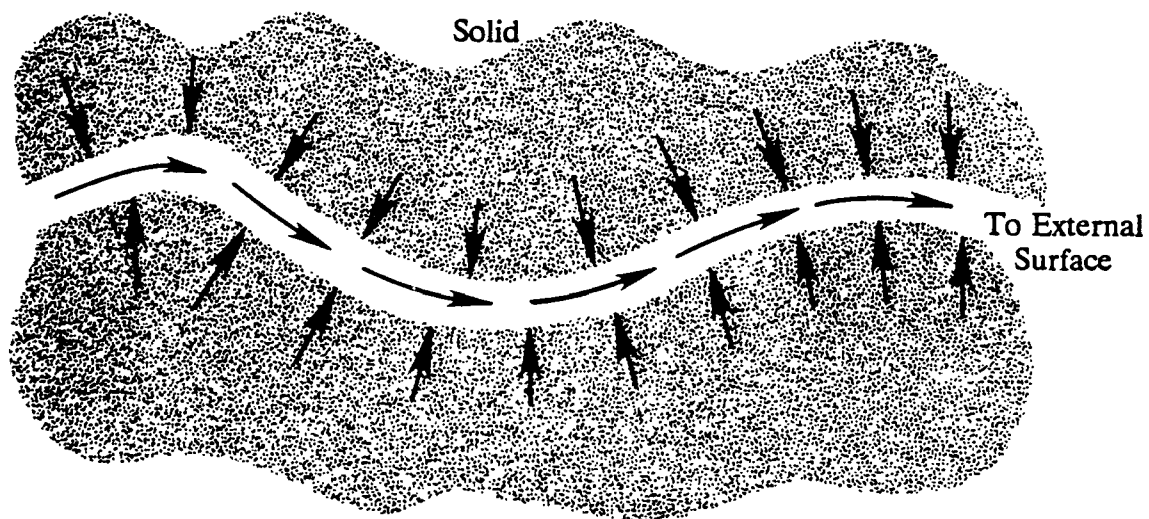


FIGURE 4-4. ILLUSTRATION OF SPECIES DISSOLUTION WITHIN A POROUS SOLID. DISSOLUTION ACROSS A PORE WALL IS SHOWN, COUPLED WITH TRANSPORT THROUGH THE SOLUTION-FILLED PORE TO THE EXTERNAL SURFACE.



Batchelor (1990) reviewed the theory and application of leaching models. His results indicate that a variety of mechanistic leaching models can be developed to describe leaching and predict the effects of process variables on the performance of solidified wastes. These models are distinguished by the assumptions made about the leaching environment and the chemical and physical mechanisms at work.

Several simple leaching models predict that the fraction of contaminant leached is proportional to the square root of leaching time. The different models assume that contaminants either do not react or react by linear sorption, by precipitation, or by an undefined mechanism that results in complete immobilization of part of the contaminant. The observed diffusivity is the parameter in these models that describes the extent of immobilization, and it can be determined by conducting a leaching test. However, these leach tests cannot themselves describe the type of immobilization occurring. Each model results in a relationship that shows that the observed diffusivity is proportional to the effective diffusivity. The effective diffusivity is the parameter that describes diffusive transport by Fick's law and therefore describes only physical immobilization. The proportionality coefficient depends on parameters that describe the particular chemical immobilization mechanisms assumed for that model.

Batchelor applied mechanistic leach models to describe performance of solidified wastes in the TCLP test by modifying a simple model to describe the effect of inward diffusion of acetic acid from the leaching solution. However, the model did not incorporate changes in the acetic acid concentration that would be observed over time as pH rises. Batchelor further notes that mechanistic leach models could also be applied to predict long-term leaching, to quantify the relative importance of chemical and physical immobilization mechanisms, to correlate and extrapolate leaching data for various contaminants and binders, and to predict ultimate performance from early characteristics of the solidified waste.

Numerous other leaching models have been developed, with a variety of intended applications. Many of these models are sophisticated and require an experienced user. For S/S remediation projects requiring application of a leaching model to evaluate long-term performance, the use of a technical expert with experience in leaching modeling is strongly recommended.

#### 4.6.3.3 The Moving Boundary or Shrinking Core Model

A qualitative model of the leaching of cement-based waste forms, in contact with an acidic leachant, has recently been developed by Cheng et al. (1992) based on experimental observations. According to this model, acids in the leachant are thermodynamically favored to be driven into the waste form. Once inside, they cause the waste form to decompose, leaving a residue (the leached layer) that is both porous and rich in silica. The unleached core of the waste form is separated from the leached portion by a very thin boundary which gradually moves into the core and thus reduces its volume. The thickness of this boundary is only about 100  $\mu\text{m}$ , but the pH varies from less than 6 on the leached side to greater than 12 within the 100  $\mu\text{m}$  distance.

What is needed is for this general physical model to be quantified, that is, to be expressed in mathematical form. Then it could be used as a predictive tool as well as an aid in the interpretation of experimental data. One way to begin would be to determine the applicability to this problem of certain mathematical models that have already been developed to describe the leaching of glasses that contain high-level nuclear waste. Although the materials, solution chemistry, distance scales, and even the associated physical processes may not be the same as for S/S wastes, the mathematical approaches may be applicable, to some extent, to the model of Cheng et al. (1992). For example, Banba et al. (1985) have developed a one-dimensional mathematical treatment of a "moving boundary" model for the leaching of nuclear-waste-containing glasses. This treatment involves a surface layer that moves into the bulk glass. Also, Harvey et al. (1984) have developed some diffusion-based mathematical models for leaching of glassy nuclear waste forms. In this latter work, they described a depleted layer in the waste-form matrix which is situated between the matrix/leachant interface and a so-called depletion front. This front is the interface between the depleted and undepleted matrix and advances into the matrix as leaching progresses. Again, the mathematical structure of these various models may have some applicability to the moving boundary model of Cheng et al. (1992).

### 4.7 LONG-TERM PERFORMANCE

A significant unresolved S/S technology issue is how well the S/S-treated waste maintains its immobilization properties over time. Although the

long-term durability of cement is well proved in conventional construction, some amount of release is virtually inevitable. S/S materials can be deposited in landfills to provide secondary barriers between natural waters and the wastes. Contaminant release begins when these secondary barriers permit natural waters to come into contact with the waste forms (Côté and Bridle, 1987). The question is not whether S/S wastes eventually release contaminants into the environment, but whether the rate of release is environmentally acceptable. S/S technologies for waste treatment have been in use for only a few decades, so the number and duration of studies on field-disposed S/S wastes are limited. Decisions about the acceptability of particular S/S products must be based on the available shorter-term field data, laboratory tests, and models of leaching behavior.

There is evidence that elements can be fixed in cementitious materials for millennia in a variety of geochemical settings (Dole, 1985). Ancient grouts from Cyprus and Greece that are 3500 to 2300 years old have held their trace metal fingerprints, allowing their constituents to be traced to nearby pits from which they had been mined. These ancient grouts are composed largely of undifferentiated, amorphous hydrosilicates, even after thousands of years. The in situ performance of these ancient grouts demonstrates the effectiveness of these metastable amorphous hydrosilicates in sequestering a wide range of elements. However, these observations are not directly applicable to S/S wastes because of differences in the physicochemical forms of the trace metals in ancient grouts versus modern waste and differences in the disposal environments in a Mediterranean climate versus the wetter climate that dominates most of the United States.

#### 4.7.1 Field Studies

There have been only a few studies of the effects of several years' duration of environmental exposure on S/S-treated waste. The Coal Waste Artificial Reef Program (CWARP) studied the environmental consequences of using stabilized coal combustion wastes as construction material for artificial fishing reefs. On September 12, 1980, some 16,000 blocks of stabilized waste were released from a hopper barge to form an artificial reef in the New York Bight. The blocks consisted of coal fly ash and flue gas desulfurization residues stabilized with lime and Portland cement additives. Blocks recovered and tested in 1988 indicated little deterioration and no decrease in compres-

sive strength. Chemical analyses and surveys of biological communities established on the reef indicated contaminants were successfully immobilized (Hockley and van der Sloot, 1991).

A Superfund Innovative Technology Evaluation (SITE) field evaluation examined the long-term performance of S/S treatment of lead and other metals, oil and grease, and mixed volatile and semivolatile organic compounds using Portland cement and a proprietary additive. Durability was tested with weathering tests, by wet-dry and freeze-thaw cycling, and by sampling S/S-treated waste after 9 and 18 months of burial. The testing showed that lead and other metals remained highly immobilized, the physical properties of the S/S-treated waste deteriorated only slightly, and the porosity decreased. The organic contaminants, however, were not effectively immobilized (de Percin and Sawyer, 1991).

The demonstrated long-term durability of concrete structures may help in the analysis of the long-term durability of S/S waste forms. Structures made with cement have lasted hundreds and even thousands of years. Long-term durability of a structure is not directly analogous to immobilization of contaminants in S/S-treated waste. However, it does indicate the ability of inorganic binders to resist gross structural degradation from exposure to the natural environment. Natural mineral deposits occurring in the environment are another possible analog to certain S/S waste forms. Metal sulfide deposits, for example, have remained stable for many millions of years in subsurface geologic formations. In general, mineral leach rates in nature do not approach those in the laboratory. The same processes that inhibit the leaching of natural substances also may apply to S/S wastes disposed in subsurface environments (Conner, 1990), provided that the chemical speciation of the materials disposed and the disposal environment are the same.

#### **4.7.2 Laboratory Studies**

At present, the environmental acceptability of a hazardous waste in the United States is based primarily upon the EPA's Extraction Procedure Toxicity Test (EP Tox) or the Toxicity Characteristic Leaching Procedure (TCLP). Neither test, however, simulates real-world, long-term conditions, although they may constitute a fairly severe set of conditions for single-exposure leaching.

Perry et al. (1992) used TCLP to examine long-term leaching performance of four types of wastes contaminated with metals or inorganics. Each waste was treated with six different commercial stabilization processes. TCLP was performed on raw waste and at 28, 90, 200, 470 and 650 days after treatment. Results indicated that the effect of time on the TCLP results was highly waste-dependent. Leachate values for some wastes remained stable over time while leachate concentrations for other wastes increased over time. In some wastes, changes in TCLP concentrations did not occur until 90-200 days after stabilizations. Similar results have been obtained by Akhter and Cartledge (1991) and Cartledge (1992), except that both increases and decreases in metals leachability as measured by the TCLP have been observed with aging. In some cases, these changes in TCLP data have been associated with changes in the chemical structure of the stabilized waste, as measured by spectroscopic analyses. These results suggest that additional evaluation of stabilization is required to ensure confidence in long-term leaching performance.

The U.S. EPA's Multiple Extraction Procedure (MEP) or other tests that expose the waste to repeated, sequential leaching can give information on leach resistance over time. Other sequential or flow-through leaching tests such as ANSI/ANS-16.1 (see Section 3.2 and Table 3-3) can give information to support prediction of long-term leach resistance.

By and large, however, attempts to correlate laboratory leaching tests with field data have not been successful. The EP Tox test, for example, can be used only to predict the potential for leaching; it cannot predict the rate of leaching over time (Bishop, 1986). Deviations between the laboratory and field are sometimes caused by testing materials under oxidized conditions (open contact with air), while the groundwater in contact with the waste may be chemically reducing. Laboratory leaching tests use continuous wetting of the waste with a leachant at controlled temperature. In situ conditions typically involve periodic contact with water and fluctuations in temperature.

#### **4.7.3 Modeling**

Numerical modeling (Section 4.6.3) is another approach to predicting the long-term performance of S/S-treated waste. Parameters based on the physical and chemical properties of a waste form can be used in conjunction with mathematical models to infer long-term leachability, based on assumptions about the leaching mechanisms and environment (Côté et al., 1986).

Mathematical models have also been combined with accelerated dynamic leaching tests to assess the long-term stability of S/S waste forms containing arsenic, cadmium, chromium, and lead.

Other recent research also indicates that metal leaching follows diffusion theory and that mathematical models combined with various leaching tests allow some predictions about metal leaching over time, with particle size, leachant velocity, and leachant acidity being key variables (Bishop, 1990). Although these models suggest good long-term stability for several S/S approaches, a test or model that simulates field conditions to a degree that would allow for confident predictions of long-term stability is lacking.

#### **4.8 USE/REUSE VERSUS DISPOSAL**

One of the principal aims of S/S processes is to produce an end product that is less environmentally threatening than the original waste. There is an added benefit if the stabilized waste can be put to some practical end use. The ability to use S/S end products eases the burden of disposing of the waste and provides obvious economic and environmental advantages over hazardous waste disposal practices. However, concerns about the long-term performance of the S/S product and the possible exposure of human or ecological receptors to contaminants released from it greatly restrict use/reuse options, and in practice relatively few S/S-treated wastes have been reused or recycled to date.

##### **4.8.1 Alternatives**

The purpose of use/reuse is to ease the burden on land disposal. Therefore, use/reuse alternatives, when deemed environmentally safe, can be a productive alternative to disposal. Possible use/reuse alternatives for stabilized/solidified waste include construction material for use in concrete, Portland cement, asphalt, road base material, landfill cover, or agricultural additives. In addition, some solidified waste may be used in direct water contact applications, such as for diking material and for forming new land from lakes, streams, marine waterways, or low-lying swamp areas. Another potential application is to help solve shoreline erosion problems by installing support structures made from incinerator ash and cement. These structures are being studied by the State University of New York not only for their

potential to reuse S/S products, but also for their ecological benefit in controlling erosion and offering a marine habitat for some species. Another potential application for S/S products is to construct artificial reefs from stabilized drilling muds from offshore drilling rigs (Kelley, 1988). In Europe, there is an emphasis on combining wastes from incineration plants with fly ash, water, and plaster to form a solid material that can be used to create sealed landfill reservoirs (Lukas and Saxer, 1990).

Until the long-term performance of S/S-treated waste in such applications is clearly demonstrated, most S/S products in the United States will still have to be disposed of in a more cautious manner, which generally means disposal in a landfill. Environment Canada (WTC, 1990b) has suggested an overall classification system for S/S waste. Classifications are based on batch extraction tests to estimate the amount of contaminant available for leaching and an evaluation of monolithic waste form leaching performance. Analysis of leaching performance uses mathematical models derived from the S/S literature with input from a database on S/S waste properties. For the purpose of this classification system, two utilization and two disposal scenarios have been selected that require different degrees of contaminant containment in a S/S waste. The scenarios are briefly described below, in order of decreasing performance requirements for S/S waste.

S/S wastes that do not qualify for utilization or disposal according to one of these scenarios would need to be disposed in a secure landfill or subjected to a more effective treatment process. In a secure landfill, containment is more a function of engineered barriers and the host geological setting than of the waste properties. Space in a secure landfill is at a premium and waste treatment that results in volume increase is usually undesirable. The performance requirements for S/S wastes disposed in secure landfills are not addressed here.

- **Unrestricted Utilization** – In an unrestricted utilization scenario, the S/S waste has a negligible leaching potential and may be used in any way that a natural material might be used, on land or in water (e.g., as a construction material). Once a given wastestream and S/S process have been approved, the resulting product becomes exempt from waste management regulations.

- **Controlled Utilization** – In this scenario, the leaching potential of the S/S waste is acceptable for a specific utilization (e.g., quarry rehabilitation, lagoon closure, road-base material). The environmental impact of S/S waste leachability measured by this classification should be assessed and utilization approved on a site-specific basis.
- **Segregated Landfill** – The S/S waste is not acceptable for utilization, or utilization is not possible or practical. The S/S waste is isolated from other wastes in a segregated landfill which does not necessarily have an engineered barrier or a leachate collection system.
- **Sanitary Landfill** – The S/S waste is not acceptable for utilization and is not acceptable for disposal in a segregated landfill without special engineered protection of the environment. Disposal with municipal garbage in a sanitary landfill is permitted (WTC, 1990b).

Use/reuse of waste materials was the subject of a recent conference on *Waste Materials in Construction, the Proceedings of the International Conference on Environmental Implications of Construction with Waste Materials* (eds., Goumans et al., 1991). The focus of this conference was on use/reuse of waste materials in general. However, several of the studies addressed wastes treated with S/S technologies. For example, the U.S. EPA Risk Reduction Engineering Laboratory (RREL) is investigating use of S/S-treated residues from combustion of municipal solid waste (fly ash, bottom ash, and combined residues). Wiles et al. (1991a and b) reported that the type of S/S treatment had little effect on the species of metals found in the municipal waste combustion residues. Instead, attenuation of metals was attributed to pH and dilution effects. In another part of this study, Holmes et al. (1991) investigated the physical properties of S/S-treated municipal waste combustion residues (bottom ash, air pollution control residue and combined ash). Results indicated that wastes treated with Portland cement only, that is, with no proprietary additives, generally produced the most durable test specimens. Of the three types of residues, the air pollution control residues produced the least durable test specimens. Kosson et al. (1991) researched the leaching properties of S/S-treated municipal waste combustion residues using a variety of leaching tests.



In addition to the U.S. EPA studies, the conference proceedings included two other investigations on the use of S/S-treated waste. Wahlstrom et al. (1991) investigated the properties of S/S-treated soils contaminated with wood preserving chemicals (As, Cr, Cu) or lead for potential use in construction of roads or storage areas in landfills. Dijkink et al. (1991) investigated the potential use of S/S-treated river sediments as building material in the Netherlands.

#### **4.8.2 Limitations**

Although there are many potential ways to use or reuse S/S-treated waste, there are many nontechnical factors to consider when evaluating any specific application. Certainly, a key question will be that of liability, which is related to political, public, and legal questions that are becoming increasingly sensitive issues of public concern.

Associated with the liability question is the lack of knowledge about the long-term performance and environmental impacts of S/S waste. The environmental consequences of the utilization of waste products or materials containing waste products on the basis of a single type of test (e.g., an extraction test) is impossible in view of the wide range of scenarios that will occur in actual use/reuse situations. Test methods to better determine the leaching mechanisms and characteristics of S/S-treated waste have been studied by van der Sloot et al. (1989), but much research remains. In addition, *Waste Materials in Construction* (eds., Goumans et al., 1991) contained numerous studies on leaching procedures for evaluating waste materials proposed for use in construction. In any event, demand will increase both for beneficial use/reuse of S/S products because of increasing constraints on land disposal and for technologies that can produce materials that are environmentally benign. However, the regulatory community is likely to be unwilling to encourage or permit reuse options unless environmental risks are clearly and confidently defined.

#### **4.8.3 Compatibility With the Disposal Environment**

In evaluating the performance of S/S technologies, the focus is often on the S/S process itself. What is often overlooked is the fact that the stabilized waste still must be evaluated in terms of its performance in

the environment into which it is placed, regardless of whether that environment is a landfill, a roadbed, or the ocean floor. Often, the interaction of the stabilized waste and its surroundings is hardly addressed, but the fact remains that both mobilization and immobilization may occur at the stabilized waste/soil or stabilized waste/water interface. The stabilized waste and the site should be evaluated together as a system to realistically assess the compatibility of the S/S product with the disposal environment. The forces and elements to which a treated waste is exposed would vary significantly, for example, depending on whether disposal occurred at the surface, in deep excavations, or in the ocean.

Environmental compatibility is a major issue at CERCLA sites, although past studies have generally not considered this factor. Compatibility with the disposal environment should have a bearing on the design and conduct of the treatability study as well as what tests are performed.

Hockley and van der Slood (1991) have modelled the interactions occurring at the waste-soil interface. They noted that the interactions between the waste and soil phases lead to phenomena that are not predicted by consideration of either phase separately, as is the case with most of the tests currently used to assess the acceptability of a waste for placement in the environment.

Another possible option to improve environmental compatibility is to codispose with the S/S waste material to modify certain physicochemical characteristics of the disposal environment. Such material could be placed between the waste and its disposal environment to improve the long-term performance of the S/S-treated waste. Environment modifiers might include bentonite or other clays to reduce groundwater infiltration; surface-reactive materials to adsorb migrating contaminants; or substances to buffer the pH or redox potential of the disposal environment. With or without the use of modifiers, however, one message clearly communicated by studies of environmental compatibility is that, to be successful, S/S process selection and design must consider the S/S product as part of a system that includes the disposal environment.

#### **4.9 COST INFORMATION**

The two major cost categories in remediation by S/S are (a) the treatability study (laboratory screening and bench-scale study), and (b) full-

scale remediation. The costs associated with these two efforts are discussed in this section. Because each project is different, it is very difficult to generalize the costs of S/S treatment. Hence the costs mentioned in this section should be regarded as estimates.

#### **4.9.1 Treatability Study Costs**

The major cost elements of a treatability study for S/S include (a) waste and site characterization (Section 2.2); and (b) bench-scale treatability screening and performance testing and associated chemical analyses (Sections 2.6 and 2.7). Since these studies are expensive, it is important to strike a balance between collection of enough data to provide statistically sound results and the available budget. Also, it is important to remember that the regulators drive the testing and that their requirements must be met before the treatability study can be accepted and full-scale remediation can proceed.

##### **4.9.1.1 Waste Characterization and Establishing Performance Objectives**

Waste sampling and characterization is conducted to determine the type, levels, and spatial distributions of the contaminants, presence of possible interferants, and for other purposes (Section 2.2). Sampling often requires the use of drill rings depending on depths to be sampled. Analyses of waste properties must be conducted in sufficient replication to permit determination of data quality by statistical methods. Refer to Section 2.2 for guidance. Some of the analytical tests conducted and their estimated costs are given in Table 4-7. Not all these analyses are necessary for every waste type.

##### **4.9.1.2 Bench-Scale Testing and Analysis**

The level of effort will depend on the number of candidate binder systems selected for testing, the number of tests performed based on the design study (or statistical design), and the types of chemical analyses to be performed, with organic analyses being significantly more expensive than inorganic analyses (Table 4-7).

**TABLE 4-7. COSTS OF TYPICAL ANALYTICAL TESTS OF UNTREATED AND TREATED WASTES**

Analysis	Method	Unit Cost <sup>(a)</sup> , \$
<b>Physical</b>		
Particle size analysis	ASTM D 422	30-160
Suspended solids	Standard Method 2092	20
Density	Various	40-240
Permeability	EPA 9100	350-450
Unconfined compressive strength (UCS) of cohesive soils	ASTM D 2166	25
Unconfined compressive strength of cylindrical cement specimens	ASTM D 1633	20-130
Cone index	ASTM D 3441	20
Flexural strength	ASTM D 1635	25
Heat of hydration	ASTM C 186	30-75
Wet/dry weathering	ASTM D 4843	530
Freeze/thaw weathering	ASTM D 4842	530
Paint filter test	EPA 9095	10-25
Atterberg limits	ASTM D 4318	40-100
Moisture	Various	10-20
<b>Chemical</b>		
pH	EPA 9045	10-20
Oxidation reduction potential	ASTM D 1498	75
Total organic carbon (TOC)	EPA 9060	50
Oil and grease	EPA 413.2	60-80
Alkalinity	EPA 403	35
Volatile organic compounds (VOCs)	EPA 5030, 8240	300-400
Semivolatile organics	EPA 3510, 8270	600-800
Base, neutral, and acidic compounds (BNA)	EPA 3540, 3520, 8270	600-1400
Polychlorinated biphenyl (PCB)	EPA 3540, 3520, 8080	150-2001
As	EPA 3050, 7060	25-30 ea <sup>(b)</sup>
Se	EPA 3050, 7740	25-30/ea <sup>(b)</sup>
Hg	EPA 7470	20-25
As, Ag, Ba, Cd, Cr, Pb, or Se	EPA 3010, 6010	10-20/ea <sup>(c)</sup>
<b>Leach Tests</b>		
- Extraction	EPA 1311 TCLP Metals	70-90
- Extraction	EPA 1311 TCLP ZHE	100-140
- Volatile organic compounds	EPA 8240	150-175
- Semivolatile organic compounds	EPA 3510, 8270	600-800
- Pesticides	EPA 3510, 8080	125-175
- Herbicides	EPA 8150	125-175
- As	EPA 3050, 7060	25-30 ea <sup>(b)</sup>
- Se	EPA 3050, 7740	25-30/ea <sup>(b)</sup>
- Hg	EPA 7470	20-25
- As, Ag, Ba, Cd, Cr, Pb, or Se	EPA 3010, 6010	10-20/ea <sup>(c)</sup>

<sup>(a)</sup> 1991 costs. May vary considerably among various laboratories. Approximate ranges are given based on quoted prices. There may be some savings of scale if a large number of samples are being analyzed.

<sup>(b)</sup> Furnace atomic absorption spectroscopy.

<sup>(c)</sup> Inductively coupled plasma atomic emission spectroscopy.

The total analytical cost will depend on the number of samples and should always include quality assurance samples. Analytical costs are the major element in treatability testing (usually  $\geq 50\%$  of the cost). Typical total costs of bench-scale treatability studies for S/S range from \$10K to \$100K, depending on process complexity, number of samples, types of analyses, and the need to capture and test air emissions. A number of different treatability laboratories are available that will conduct bench-scale treatability testing for S/S on a service basis.

#### **4.9.2 Full-Scale Remediation Costs**

The costs involved in full-scale S/S treatment fall into four major categories – planning, mobilization and demobilization, treatment, and disposal.

##### **4.9.2.1 Planning**

The planning costs are the administrative and engineering planning costs associated with the remediation. Waste and site characterization activities and the treatability study are assumed to have been completed before project planning starts. Planning costs may include permitting, engineering design (scale-up), equipment and materials procurement, and preparation of a work plan, quality assurance plan, and/or a health and safety plan. Permitting can take weeks or months, and costs can be substantial, especially for uncommon contaminants or complex sites.

Engineering costs involve designing and engineering for full-scale operation based on bench-scale (treatability testing) data. A pilot- or field-scale demonstration may be necessary, either to establish scale-up factors or to satisfy potentially responsible parties (PRPs) and/or regulators of the feasibility of the cleanup. The actual price of equipment or raw materials is not included in this category, but the labor involved in procurement is. If the remediation is to be performed through a contractor, contract procurement costs are also involved.

A site-specific work plan, quality assurance plan, and/or a health and safety plan are almost always required, and review comments from regulatory agencies and other parties must be addressed. Depending on the magnitude of the project, planning costs can range from \$25K to several hundred thousand dollars.

#### **4.9.2.2 Mobilization and Demobilization**

Mobilization costs involve transportation of personnel, equipment, and raw materials to the site, site preparation, and equipment installation and start-up. Demobilization costs include equipment shut-down and disassembly, and transportation of personnel and equipment from the site. Mobilization and demobilization (mob/demob) costs vary depending on type of equipment, facilities available at the site, decontamination requirements, and the location of the site. When large-scale equipment is necessary, mob/demob costs will range from \$25K to \$50K or more if extensive site preparation is involved.

#### **4.9.2.3 Treatment**

Treatment costs typically include costs for excavation (if treatment is ex situ), chemicals, equipment, utilities, labor, and sampling and analysis. Full-scale S/S treatment services are offered by a variety of firms, including S/S vendors, remediation companies, and construction companies certified to conduct hazardous waste remediation. Full-scale treatment should not be undertaken by anyone not fully qualified and certified, including OSHA safety certifications.

Excavation applies to sites containing contaminated materials that are to be stabilized by plant mixing. Excavation equipment consists of typical earth-moving equipment, which can be rented along with an operator at most sites. Cost for excavation ranges from about \$0.85/yd<sup>3</sup> to \$4.09/yd<sup>3</sup> (U.S. EPA, 1987b).

Chemical costs depend on the type of chemicals required for the binder system and the amounts as determined by the waste-to-binder ratio. Table 4-8 shows the costs of some typical stabilization chemicals. If chemicals are transported for large distances, the transportation costs may equal or exceed the chemical costs.

Equipment costs other than for excavation are based on the type of equipment selected for materials handling and processing. Qualified S/S vendors and remediation firms will own the necessary equipment and charge a use-rate based on the time it is used. Equipment can also be purchased (for large and long-term projects), in which case depreciation costs should be considered, or rented (for smaller sites). Customary equipment includes

**TABLE 4-8. COSTS OF TYPICAL STABILIZATION CHEMICALS**

Chemicals	Costs \$/Ton <sup>(a)</sup>
Portland cement	\$55 - 70 (bulk)
Quick lime	\$45 - 75 (bulk)
Hydrated lime	\$55 - 80 (bulk)
Kiln dust	\$10 - 40 (bulk)
Fly ash	\$ 1 - 40 (bulk)
Sodium silicate	\$160 - 239 (bulk)
Chloranan <sup>(b)</sup>	\$600
IWT-HWT 20M <sup>(c)</sup>	\$300
Concrete admixtures	\$ 2 - 12/gallon

<sup>(a)</sup> 1991 Costs obtained from suppliers. Costs may vary based on suppliers and the location of the site.

<sup>(b)</sup> Proprietary additive

<sup>(c)</sup> Proprietary modified clay binder

backhoes, front-end loaders, storage tanks, mixers, conveyors, etc. Sometimes equipment and its operators are available for an hourly, weekly, or monthly charge. The purchase costs of different types and sizes of equipment, and estimates of their rental costs are mentioned in the *Handbook for Stabilization/Solidification of Hazardous Waste* (U.S. EPA, 1986c).

Table 4-9 shows the major unit cost elements for S/S treatment with cement by typical stabilization techniques (in-drum mixing, in situ mixing, plant mixing, and area mixing as defined in Section 2.8.2). These are the unit costs for mixing only and exclude the numerous other cost elements such as mobilization and demobilization, engineering and administration, and health and safety. In addition to processing equipment, personal protective equipment may be needed, including Tyvek suits, respirators, decontamination equipment, etc.

TABLE 4-9. COMPARISON OF MAJOR COST ELEMENTS OF SOLIDIFICATION/STABILIZATION WITH CEMENT

Category	Cost, \$/Cubic Yard			
	In-Drum Mixing	In Situ	Plant Mixing	Area Mixing
Labor, overhead, and profit	216.30	1.40	1.10	3.00
Equipment and metering	65.40	1.60	0.70	3.00
Conveyance	NA	NA	1.40	
Pretreatment	NA	NA	0.50	NA
Monitoring and testing <sup>(a)</sup>	115.40	4.00	3.10	5.10
Reagents and mixing materials	31.10	31.10	31.10	31.10
Offsite disposal (nonhazardous waste)	NA	NA	3.10	NA
Supplies	84.60	0.60	0.80	1.30
TOTAL	512.80	38.70	41.80	43.50

<sup>(a)</sup> Monitoring and testing costs assume that in-drum wastes require sampling of each drum, while the other approaches require representative samples of a large, relatively uniform body of waste.

Source: Arniella and Blythe, 1990, p. 101. Excerpted by special permission from *Chemical Engineering*, February 1990, © 1990 by McGraw-Hill, Inc., New York, NY 10020.

#### NOTES:

1. NA = not applicable
2. Not included are costs for mobilization and demobilization, engineering and administration, and health and safety.
3. All mixes are based on 30% Portland cement and 2% sodium silicate.
4. Cost for delivering reagent is not included.
5. Approximate production rates assumed are: in-drum mixing, 5 drums/h; in situ, 500 yd<sup>3</sup>/d; plant mixing, 650 yd<sup>3</sup>/d; area mixing, 400 yd<sup>3</sup>/d.



Utilities normally include water and electricity. Sometimes the remediation may have to provide its own energy supply, such as diesel generators. If pretreatment is necessary, other sources of heat, such as oil, gas, or steam may be needed.

Labor costs are based on the number of equipment operators, supervisory personnel, and managers, as well as the number of hours of operation. An important factor in remediation can be the stand-by time. If operations are not scheduled appropriately or if unanticipated delays such as stop work orders are incurred, equipment or personnel will go unutilized. For example, if the operation runs short of a chemical, or if a piece of equipment breaks down, the entire operation may have to be temporarily halted. Another type of work stoppage is when sampling and analysis of treated waste show that the stabilization is ineffective. Clearly, some types of work stoppages can be avoided or minimized by effective planning. Other types of stoppages are less controllable, such as stop work orders issued by regulators so that they can review preliminary data.

Sampling and analysis are conducted during full-scale remediation to determine whether the treated process is achieving the performance goals for chemical and physical properties. A sampling and analysis and/or quality assurance plan will be prepared during planning. Implementation of the plans may be a significant part of the remediation cost. Particularly during the early stages of full-scale treatment, it may be necessary to have samples analyzed on a rush basis, in order to minimize standby time while waiting for data. Note that with rush fees, analysis costs can be 2 or 3 times higher than fees for normal turnaround-time analyses.

If a full-scale demonstration precedes full-scale cleanup, regulatory approval for the full-scale cleanup may be contingent on results of the demonstration. If the initial demonstration shows deficiencies in the process, then process modifications followed by additional demonstration runs will have to be conducted until the process is working satisfactorily. As discussed in Section 2.8, demonstration runs prior to full-scale processing are highly recommended for refining the process and verifying that process scale-up in the field has been accomplished satisfactorily. However, this step has potentially significant cost impact on the project, particularly if several demonstration runs need to be conducted prior to full-scale treatment.

#### 4.9.2.4 Final Disposal

When field treatment is completed, the S/S-treated waste has to be disposed of as planned. In some cases, depending on the characteristics of the treated waste and on regulatory approval, the S/S-treated waste can be returned to its original location. Some final steps such as compacting or capping (with the associated costs) may be required.

However in other cases, the treated waste cannot be disposed of on site. Then arrangements have to be made to transport the treated waste to a sanitary or secure landfill, again depending on waste characteristics and regulatory policy. Tipping fees at sanitary landfills typically range from approximately \$10 to \$50/ton and for secure (RCRA-permitted) landfills range from \$100 to \$300/ton. Added to this is the cost of waste transport to the landfill. The cost for transportation by covered bed dump truck or roll off box carrier typically ranges from \$0.15/yd<sup>3</sup>-mile to \$0.60/yd<sup>3</sup>-mile. Costs include the actual charge for hauling; demurrage (charge for truck waiting time); and training, licensing, and protective clothing for the truck operator (if required) (U.S. EPA, 1987b). Because there are far fewer secure landfills than sanitary landfills, the transportation distance to secure landfills will generally be much greater.

#### 4.9.3 Estimates of Stabilization Costs

Table 4-10 lists the estimated costs registered in the records of decision (RODs) for CERCLA sites. Because costs in this table are estimates, there is no indication whether or not the remediation was actually accomplished for that cost. Total costs vary according to type of contaminants and amount of wastes. Missing from this table is information on necessary pretreatment steps and other project-specific requirements that may significantly impact total cost. In general, a relatively straightforward S/S project involving more than 5,000 to 10,000 tons of waste should cost in the range of \$100 to \$150/ton of waste processed. Below this amount, unit costs can increase because of fixed costs; above 10,000 tons, unit costs can decrease because of economics of scale. Therefore, the higher unit costs in Table 4-10, some of which greatly exceed the \$100 to \$150/ton range, are almost certainly inflated by pretreatment requirements or other factors.

TABLE 4-10. ESTIMATED TREATMENT PROJECT COSTS MENTIONED IN THE RODs FOR SUPERFUND SITES WHERE STABILIZATION HAS BEEN SELECTED AS A COMPONENT OF THE REMEDIAL ACTION

Site	Media	Volume	Contaminants	Project Cost, \$	Unit Cost, \$/cy <sup>(a)</sup>
Love Canal, NY	Soil	7,500 cy	dioxin	3,675,000	490
Marathon Battery, NY	Sediment/soil	23,700 cy	Cd, Co, Ni,	16,640,000	702
Alladin Plating, PA	Soil	12,000 cy	Cr	4,461,000	372
Amnicola Dump, TN	Soil	400	As, Cd, Cr, cyanide, Pb, Hg, PAH, pesticides, VOC	256,000	640
Davie Landfill, FL	Soil/sludge	75,000 cy	As, Cd, Cr, cyanide, Pb, Hg, sulfides	3,700,000	49
Independent Nail, SC	Sediment/soil	6,200 cy	Cd, Cr, cyanide, Ni, Zn	1,132,000	183
Burrows Sanitation, MI	Sludge/soil	250 cy	Cr, Cu, Pb, Zn	237,700	951
Outboard Marine	Sediment	5,700 cy	PCB	3,150,000	553

<sup>(a)</sup> Includes planning, sampling, and pretreatment costs as well as direct S/S process costs.  
Source: Based on data contained in U.S. EPA (1989a).

#### 4.9.4 Case Study

A treatability study and field demonstration/cleanup of 1800 cubic yards of lead-contaminated soil conducted by Battelle (Means et al., 1991b) at Port Hueneme, California, demonstrates the various aspects of an S/S field project and the associated costs. To establish a baseline concentration on the amount of lead in the soil before treatment, 18 grab samples (and two blind replicates) of the untreated soil were collected and analyzed for total and soluble lead. Because the levels of lead in these samples varied greatly, seven additional samples were collected. Total lead levels averaged 178 ( $\pm 162$ ) mg/kg in the soil. The EP Tox average of 0.9 mg/L lead was lower than the U.S. EPA standard (5 mg/L lead). Previous data on the Cal WET test, however, showed that the average of 11.7 mg/L lead exceeded the STLC established by California (5 mg/L). (See Section 3.2 for further discussion of leaching tests.)

The bench-scale treatability study involved evaluating two stabilization techniques, a sulfide-based process and a silicate-based process. Eleven samples were treated with the sulfide process, which involved adding a hydrated sodium sulfide solution in water, low-alkaline Portland cement, and a small amount of detergent. Ten samples were treated with the silicate process, which involved adding sodium silicate instead of the sulfide. The sulfide process was used in this instance as an alternative to the silicate process to determine the relative attributes of the two processes. Although the sulfide process produced slightly lower soluble lead values than the silicate, the silicate process was concluded to be the preferable based on ease of application in the field.

The stabilization formulation used in the field was the same as that used during bench-scale testing; no additional testing to determine optimum ratio was done in this case. During the field demonstration, eight sets each of pre- and post-stabilization samples were collected and analyzed for pH, total lead, and Cal WET test. The average Cal WET test results were reduced from 11.7 mg/L before stabilization to 2.7 mg/L after stabilization. After a number of discussions with cognizant regulatory agencies, the treated soil was released for placement in a sanitary landfill.

Table 4-11 provides cost details for this project. A pug mill was rented for the mixing of soil, cement, silicate, and bicarbonate. Most of the

**TABLE 4-11. STABILIZATION COSTS FOR AN 1800-CUBIC-YARD SITE  
CONTAMINATED WITH LEAD**

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1.	<u>Bench-Scale Treatability Study/Planning</u>	
•	Chemist, 8 hrs @ \$50/Hr.	\$ 400
•	Chemical Analysis, 12 samples each TTLC, STLC, and pH	3,240
•	Project Manager, 16 hrs. @ \$95/hr	<u>1,520</u>
	Subtotal	\$5,160
2.	<u>Move Soil from Storage Hut to Work Area</u>	
•	End-dump trucks, 2 trucks x 1 day each x \$55/hr	\$ 880
•	Field supervision, 8 hrs @ \$78/hr	624
•	Laborers, 2 x 8 hrs each @ \$30/hr	480
•	Plastic sheeting, 10 rolls @ \$120/roll	<u>1,200</u>
	Subtotal	\$3,184
3.	<u>Steam Clean Storage Hut</u> (Subcontracted)	\$4,000
4.	<u>Power Sieving</u>	
•	Power screen @ \$4,000/wk including mobilization/demobilization	\$4,000
•	Front-end loaders, 2 loaders x 1 day each @ \$90/hr	1,440
•	Field supervision, 8 hrs @ \$78/hr	624
•	Laborers, 2 x 8 hrs each @ \$30/hr	<u>480</u>
	Subtotal	\$6,544
5.	<u>Debris Disposal</u>	
•	Front-end loader, 1 loader x 1 day @ \$90/hr	\$ 720
•	End-dump trucks, 2 trucks x 10 trips each x 1 hr/round-trip @ \$55/hr	1,100
•	Field supervision, 10 hrs @ \$78/hr	780
•	Laborers, 2 x 10 hrs each @ \$30/hr	600
•	Tippage at landfill, 300 tons @ \$18.70/ton	<u>5,610</u>
	Subtotal	\$8,810

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**TABLE 4-11. STABILIZATION COSTS FOR AN 1800-CUBIC-YARD SITE  
CONTAMINATED WITH LEAD (Continued)**

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6. <u>Stabilization</u> (approximately 4 working days and 10 hour-days, including mobilization/demobilization)		
• Cement, 150 tons @ \$0.04/lb		\$12,000
• Sodium silicate solution, 150 tons @ \$0.08/lb		24,000
• Sodium bicarbonate, 15 tons @ \$0.10/lb		3,000
• Freight for chemical deliveries		3,000
• Plastic sheeting, 5 rolls @ \$120/roll		600
• Pugmill and components, including mobilization/demobilization		29,000
• Front-end loaders, 2 loaders x 40 hrs each @ \$90/hr		7,200
• End-dump trucks, 1 truck x 40 hrs @ \$55/hr		2,200
• Baker tank, 1 month @ \$30/day		900
• Field supervision, 40 hrs @ \$78/hr		3,120
• Project Manager, 24 hrs @ \$95/hr		2,280
• Chemist, 32 hrs @ \$50/hr		1,600
• Laborers, 2 laborers x 40 hrs each @ \$30/hr		2,400
• Travel and subsistence for contractor staff, 5 persons x 7 days each @ \$100/day		3,500
• Industrial hygiene monitoring and oversight		2,000
• Analytical fees, rush basis (100% surcharge)		<u>1,080</u>
	Subtotal	\$97,880

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**TABLE 4-11. STABILIZATION COSTS FOR AN 1800-CUBIC-YARD SITE  
CONTAMINATED WITH LEAD (Continued)**

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<b>7. <u>Post-Treatment at Project Closure Activities</u></b>		
• Chemical analysis, TTLC, STLC, and pH on 12 samples, normal turnaround		<b>\$3,240</b>
• Regulator meetings concerning disposal options, Project Manager 20 hrs @ \$95/hr		<b>1,900</b>
• End-dump trucks, 4 trucks x 25 trips each x 1 hr/round trip @ \$55/hr		<b>5,500</b>
• Front-end loader, 1 truck x 25 hrs @ \$90/hr		<b>2,250</b>
• Reporting and documentation, Project Manager, 16 hrs @ \$95/hr and secretary, 16 hrs @ \$40/hr		<b><u>2,160</u></b>
	<b>Subtotal</b>	<b>\$15,050</b>
	<b>Grand Total - Expenses</b>	<b>\$140,628</b>
	<b>Contractor Fee</b>	<b><u>9,372</u></b>
	<b>Total Cost</b>	<b>\$150,000</b>

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other equipment, such as dump trucks, power screen, and front-end loaders, was also rented. A number of other cost elements are itemized to provide the reader with the variety of typical cost elements for an S/S treatment project and the stages of the project in which they were incurred. Note, however, that the unit costs associated with this project were fairly modest compared to those for other larger-scale S/S projects (e.g., Table 4-9). The total cost of the cleanup of 1800 cubic yards (approximately 2,430 tons) was \$150,000, for an average of \$83/cu yd or \$62/ton.

## **5 TECHNOLOGY SHORTCOMINGS AND LIMITATIONS**

This chapter discusses some of the shortcomings and limitations of S/S technology pertaining to S/S processes/binders, waste form and treatability/performance testing, and other issues. The topics discussed should be viewed as examples of issues rather than an exhaustive list of technology limitations.

### **5.1 PROCESS/BINDER CONSIDERATIONS**

#### **5.1.1 Hierarchy of Waste Management**

As discussed in Chapter 1, technologies that lead to the recycling, recovery, or reuse (3R) of some portion of the contaminant or waste material are preferred over treatment technologies in the waste management hierarchy. Technologies such as incineration that destroy the contaminant also are typically preferred over S/S processes. However, S/S is still an important treatment option because of its versatility and effectiveness (Section 1.1).

#### **5.1.2 Scale-Up Uncertainties**

Process scale-up from bench-scale to full-scale operation involves numerous complex issues that should not be taken for granted. These issues are no less important for S/S technology than for any other remediation technology. Variables such as ingredient flow rate control, materials mass balance, mixing, and materials handling and storage, as well as the unpredictability of the outdoor elements compared with the more controlled environment in the laboratory, all may affect the success of a field operation. These potential difficulties underline the need for a field demonstration prior to full-scale implementation (Section 2.8).

#### **5.1.3 Proprietary Binders**

The nature of the S/S business at present is such that most vendors protect their exact binder formulations as proprietary or trade secret. Relatively few formulations are covered by patent. The proprietary designation protects the formulations from being readily recognized by competitor vendors (Section 4.1). The reality is that there are several different generic binder systems that are used by the majority of S/S vendors, and each vendor has its own variations in the form of special additives.



Binder ingredients are frequently designated in the literature as, for example, "fly ash A" or "proprietary additive." As a result, the report on a treatability study lacking information on binders and additives has no technology transfer value, and the ability to evaluate the data in terms of chemical mechanisms is absent, because binder chemistry is unknown or unreported.

#### **5.1.4 Binder "Overkill"**

Too much of a particular binder ingredient can lead to unnecessary expense and even to an improperly stabilized waste form. For example, many metals are amphoteric, meaning that they are soluble under both acidic and basic conditions (Section 4.2). The metal will be at minimum solubility when a sufficient base (in the form of an S/S ingredient) is added to make the waste moderately alkaline. Too much base will cause the metal to resolubilize and/or make the waste hazardous by virtue of the RCRA corrosivity characteristic (i.e., pH >12.5).

### **5.2 WASTE FORM/CONTAMINANT ISSUES**

#### **5.2.1 Complications of Physicochemical Form of the Target Contaminants**

In a recent S/S field demonstration (Means et al., 1991b), the unsatisfactory degree of stabilization of the copper and lead was a direct result of their encapsulation in organic coatings of various types (antifouling compounds, pigments, etc.). People conducting S/S treatability tests frequently measure the type and amount of contaminant present, but, in complex waste forms such as sandblasting grit, the type and amount of contaminant do not provide sufficient information. It is important to understand the physicochemical form of the contaminant as well. However, the chemical analyses necessary to characterize the physicochemical form of the contaminant can be expensive and nonroutine (Section 3.5).

#### **5.2.2 Interferences and Incompatibilities**

As discussed in Section 4.3, numerous chemical constituents may interfere with various S/S processes. Thus, specific chemical incompatibilities should be recognized and avoided.

### 5.2.3 Volatile Organic Contaminants

Several studies have been performed that strongly indicate the inadvisability of using S/S as the principal remediation technology for organic wastes, particularly wastes containing hazardous volatile organics (Wiles and Barth, 1992). The following guidance is provided based on the current state of knowledge about using S/S for treating organics (Section 4.4):

- According to the hierarchy of waste management, treatment by a destructive technology (e.g., incineration) is preferable to contaminant immobilization (e.g., by S/S) because the former processes eliminate the contaminant and the concern over the long-term stability of the S/S process. The same is true for removal processes, such as thermal desorption, that concentrate the contaminant into a much smaller volume of material which can then be either reused as a raw material or incinerated and destroyed.
- Generally, S/S should not be used to treat a site containing only organic waste. Alternative technologies (e.g., incineration, steam stripping, vacuum extraction) should be used to remove and/or destroy the organics. If residues remain after this primary treatment, S/S treatment may be effectively used to stabilize the residue. However, a well-designed and controlled treatability study should be conducted to assess S/S effectiveness and to select and design a proper S/S process.
- There are exceptions to avoiding S/S treatment of organic wastes. For example, if the organic is generally not mobile through air, soil, and water (e.g., low levels of oil and grease), then S/S may be an acceptable, cost-effective treatment alternative for a given site. Careful attention must be paid to any existing state and federal environmental regulations concerning the particular organic contaminant (e.g., dioxins, etc.). Treatability studies must be performed incorporating appropriate test methods to evaluate the organic waste's potential for escape.
- Based on existing data, volatile organic compounds (VOCs) usually cannot be treated by current S/S technology. Whether a site containing VOCs as a minor constituent can be treated by S/S will depend on specific conditions existing at the site.
- Available data also indicate that semivolatile organic compounds generally cannot be effectively treated by

current S/S techniques. Whether a site containing low to moderate concentrations of semivolatile organics should be treated using S/S also depends upon site-specific factors.

- Notwithstanding the above factors, there are situations in which S/S can be a satisfactory treatment method for wastes containing organics. When S/S treatability tests are performed on such matrices, it is important to understand that (a) aqueous leaching tests will be a meaningless indicator of the degree of immobilization for organic compounds having low solubility in water and (b) in the aggressive chemical environments associated with certain binders, certain organic contaminants may be degraded or transformed into by-products that, in some cases, may be as toxic as or more toxic than the parent compounds.

#### **5.2.4 Multicontaminant Wastes**

Wastes containing a large number of contaminants are generally more difficult to stabilize than wastes containing one or a few contaminants, particularly when the multiple contaminants have widely varying chemistries (Section 4.2). The problem is that a given type of binder might be more compatible with an organic waste than with a primarily metallic waste. Therefore, when both organics and metals occur in the same waste form, the binder selected will not be optimal for both types of contaminants. On a more specific level, because metal chemistry varies widely, metals will respond differently to the same binder. As a general rule, a physical encapsulation process (solidification) may be the best compromise for a multicontaminant waste, whereas a chemical stabilization process may be the best approach when there is only one contaminant or when the contaminants present have similar chemical properties.

#### **5.2.5 Limitations of Cement-Based Waste Forms**

The weaknesses of cement-based waste forms are as follows:

- The fate of the waste species within the waste form is unknown.
- They are porous solid bodies.
- The total volume of material to be disposed of usually increases.

- Small changes in the waste composition or mix proportions can alter the properties, sometimes without the knowledge of those utilizing the waste form.
- Managers and operators charged with the task of waste disposal frequently do not understand the complexity of the heterogeneous material they are attempting to create.

It is of utmost importance that users of these waste forms be aware of these weaknesses and their ramifications. In most instances, problems originating from the weaknesses can be avoided or circumvented. Future research is expected to help explain and overcome these weaknesses (McDaniel et al., 1990).

#### **5.2.6 Sample Heterogeneity**

Solid wastes can be highly heterogeneous in composition, both macroscopically and microscopically. A person can analyze two different portions of the sample and obtain two very different analytical results. Therefore, sample heterogeneity should be recognized as a possible causative factor when explaining treatability data that are discrepant or difficult to interpret.

### **5.3 TREATABILITY AND PERFORMANCE TESTING ISSUES**

#### **5.3.1 Testing Limitations**

Several unresolved issues pertain to S/S processes. In particular, tests that have been developed to assess technology performance are not applicable to every disposal scenario. Testing methodologies must be tailored to the specific nature of the S/S-treated waste. Personnel involved in treatability testing should be aware of the various tests' limitations when interpreting the data (Chapter 3).

Examples of the limitations of treatability studies and S/S-treated waste testing based on actual field experience are as follows:

- Although the principal objective of the site sampling is to obtain a sample that is representative of the waste as a whole, variation from sample to sample is common and must be considered when interpreting the

analytical data. Many factors affect site sampling. If the goal is a single composite sample, site debris, such as large boulders or rocks, timbers, shingles, etc., usually should be segregated by physical screening before samples are collected from a wide range of locations, in order to produce a representative sample.

- No single leaching methodology is suitable for all waste forms or target contaminants, and none of the leaching methodologies is calibrated in terms of contaminant migration in actual groundwater. The TCLP does not provide data on long-term stability; in fact, different results are frequently obtained when the TCLP test is conducted on the same stabilized waste at different cure times. Leach tests in general are probably most useful for assessing the relative stabilization efficiencies of different binders.
- Some leaching test methods are more appropriate for metals, some are not applicable to nonvolatile organics, and others are applicable only to monolithic wastes that do not change in surface area appreciably during testing. Batch methods usually do not use sufficient acid to exhaust the acid-neutralizing capacity of most stabilized waste forms. Sequential methods accelerate leaching to assess long-term performance. The interpretation of results is difficult, however. Accelerated leaching in the laboratory may occur by different mechanisms than the longer term leaching that occurs in the field.
- At times it is appropriate to modify a standard leaching protocol to address a specific issue. Examples include the following:
  - Eliminate the leachate filtration step to address colloidal contaminant transport.
  - Use site-specific groundwater as the leachant instead of the generic leachant specified in the procedure.
  - Consider use an organic solvent (e.g., acetone) as the leachant instead of an aqueous leachant for addressing the S/S of organic contaminants (see Section 4.4.3 for discussion of pros and cons).
  - Determine when it is appropriate to create an artificial surface area prior to leaching (e.g., by crushing).
  - Deionized water can be more aggressive than acid in some cases.

- Microbes may eventually affect the long-term performance of certain waste forms, particularly organic binders (Section 3.4). However, these microbial reactions can be very slow, and accelerated tests that are generally recognized and approved and that closely simulate real-world biochemical reactions are not available.
- Bioassay data may conflict with chemical data.
- There are limitations to interpreting and applying the results of physical tests. For example:
  - The unconfined compressive strength test is not appropriate for noncohesive substrates and is not a direct indicator of constructability.
  - No correlation has been identified between the physical strength of a waste form and its leaching behavior.
  - Permeability measurements are difficult to conduct and are subject to wide variation. Also, large differences have been observed between values measured in the laboratory and in the field for the same substrates.
- With the exception of a small group of "regulatory" tests, no performance standards or acceptance criteria exist for many tests. In fact, acceptance criteria should vary, depending on waste composition, disposal or reuse site characteristics, and other factors. This leaves much to the interpretation of individual S/S project personnel.
- In general, the bench-scale treatability study should exceed the performance criteria established for the project. That is, a margin of safety should be established that allows for the greater variability of the process when implemented in the field, especially in the area of mixing. The necessary magnitude of the safety margin, however, is unknown and probably varies from project to project (Sections 2.6 and 2.7).

### 5.3.2 Long-Term Performance

The long-term performance of treated waste is not clearly understood, and no definitive test procedures exist to measure or assess this property. The TCLP is not an adequate measure of long-term leaching. Monitoring data from field disposal sites are needed to detect the premature deterioration of solidification or stabilization of previously processed

wastes. Because of the uncertainties surrounding long-term performance, wastes previously treated using S/S and disposed of may have to be retrieved and retreated in the future (Section 4.7).

### **5.3.3 Reproducibility**

The reproducibility of treatability data can be poor because of sample heterogeneity, uneven mixing, the complexity of S/S chemical reactions, and other reasons. Timing is also a critical variable. It is not unusual to see different analytical results when samples from the same treatability study are cured for different periods of time prior to leaching.

### **5.3.4 Limitations in S/S Treatability Reference Data**

S/S processes would be used more successfully if experiences were shared more effectively. However, well-documented S/S treatability data are scarce. Many of the common reporting deficiencies are as follows:

1. Proprietary binders (Section 5.1.3). Without specific information on binder characteristics the process is not reproducible, and the treatability data have no technology transfer value.
2. Incomplete treatability data and data gaps. Certain types of data that are needed to evaluate the stabilization efficiency and help understand the chemical mechanism(s) of stabilization are frequently missing, for example:
  - Baseline soluble metal concentrations in the untreated waste. This is needed as a point of comparison for the soluble metal concentrations in the treated waste so that the percent reduction attributable to treatment can be assessed.
  - Total metal concentration in the untreated waste and the treated waste. The latter is necessary to demonstrate that a low post-treatment soluble metal concentration is not attributable simply to sample heterogeneity.
  - Binder-to-waste ratio. This is needed to estimate the volume expansion of the waste during treatment and the effect of dilution on posttreatment soluble metal concentrations.

- pH of the leachate from the untreated and treated wastes. This is an important parameter for interpreting the performance data. Frequently, high soluble-metal concentrations are due to pH. The pH parameter should be routinely measured at the conclusion of leach testing.
  - Extent of dilution from binder ingredients. This can be estimated from the binder/waste ratio, where given, but should be carefully characterized in each treatability study so that the performance data can be corrected for dilution. Frequently, a significant proportion of the reduction in soluble metal concentration in the treated waste can be attributed to dilution from the binder ingredients.
3. Data reliability. Many treatability reports do not indicate whether data were collected under an appropriate quality assurance/quality control (QA/QC) program. Therefore, many existing S/S performance data have unknown validity.
  4. Treatability procedures. Similarly to data reliability, the frequent absence of detailed treatability procedural information greatly limits the technology transfer value of a treatability study. The success or failure of a treatability study may depend on small variations in the amounts of the ingredients and in the order and timing of ingredient addition.
  5. Bias of existing S/S performance data toward successful treatability studies. Treatability projects that achieved a high degree of metal stabilization are reported more frequently in the literature than projects in which the treatment systems worked poorly. Therefore, the existing S/S database is probably biased toward the most successful treatability studies.



## **6 CURRENT RESEARCH AND FUTURE DEVELOPMENT NEEDS**

### **6.1 CURRENT RESEARCH**

Solidification/stabilization is the subject of active research aimed at improving the range and efficiency of S/S process application. Some of that research is described in sections 6.1.1 through 6.1.8.

#### **6.1.1 Binders**

##### **Experimental Study of S/S Treatment of Hazardous Substances.**

Statistically designed treatability studies are being applied to identify environmentally acceptable and economically feasible methods for S/S processing of organic and inorganic wastes. The work focuses on inexpensive pozzolanic binders such as fly ash, silica fume, lime kiln dust, cement kiln dust, and ground blast furnace slag. Waste types tested include electric arc furnace dust (K061) and arsenic-contaminated soil (Fan, L.T., 1991, personal communication).

**Improvement in S/S Treatment of Hazardous Inorganic Wastes by Silica Fume (Microsilica) Concrete.** A preliminary experimental program is being conducted to assess the potential of silica fume concrete for solidification/stabilization of K061 metal arc dust from steel manufacturing. TCLP leaching tests are being used to investigate the effectiveness of the various methods of S/S processing. The study is testing S/S process performance for condensed silica fume and cement binder or fly ash, cement kiln dust, and cement binder. It was concluded that silica fume concrete can significantly enhance the stabilization of furnace arc dust as compared with the other S/S processes. The results were based on studying the concentration of metals in the leachant as specified by U.S. EPA (Fuessler and Bayasi, 1991).

**Physical and Chemical Aspects of Immobilization.** Recent studies are using sodium as an internal marker for physical retardation. Almost any product will contain some Na, K, or Cl, which can be used independently as indicators for tortuosity. The difference between the mass transfer coefficients for Na and other elements derived from leaching tests, such as the modified ANSI/ANS-16.1, reflects the contribution of chemical retention in the product matrix to the overall mass transfer coefficient for the product. The types of release mechanisms that can be distinguished are:

- dissolution
- surface wash-off
- diffusion (de Groot and van der Sloot, 1990)

#### **Evaluation of Solidification/Stabilization of RCRA/CERCLA Wastes.**

U.S. EPA Risk Reduction Engineering Laboratory is sponsoring a project to do bench-, pilot-, and field-scale evaluation of the performance of cementitious binders in S/S treatment of metal-contaminated wastes over time (Trish Erickson, 1992, personal communication). Performance will be measured in terms of lab leachability tests, solids composition and actual water quality of infiltration/runoff. Field measurements will extend over at least 5 years, while smaller tests are intended to simulate field results at a much-accelerated pace. The University of Cincinnati protocol for accelerated weathering testing described below can be tested in this project.

#### **6.1.2 Mechanisms**

**Review and Analysis of Treatability Data Involving S/S Treatment of Soils.** This project is using geochemical equilibria models to determine minimally soluble forms of the eight Toxicity Characteristic Leaching Procedure (TCLP) metals. Emphasis is on identifying physicochemical forms of these metals that are relevant to the stabilization or solidification of typical hazardous wastes and the chemical conditions needed to produce the physicochemical forms of these metals.

These data are being analyzed to identify empirical or theoretical geochemical relationships that appear to govern the success of S/S applied to metal-contaminated soils. Relationships for multiple metal systems are being quantified, where possible (Means et al., 1991c).

**Morphology and Microchemistry of S/S-Treated Waste.** Scanning electron microscopy and X-ray diffraction techniques along with solvent extractions are being used to investigate waste/binder interactions. The objectives of these investigations are to better understand S/S processes by characterizing the binder phase composition and structure and the distribution of the contaminants in the solid phases, and to determine if microstructure can be correlated to macroscale physical properties (U.S. EPA, 1990f; and

several other papers in preparation). Contaminant distribution data include analysis of the contaminant concentration, chemical forms and crystal structure, and binding mechanisms in each phase.

#### **Fate of PCBs in Soil Following Stabilization with Quicklime.**

Several researchers have reported destroying polychlorinated biphenyls (PCBs) in contaminated soil by applying quicklime. These reports are based on retrospective data from site remediation programs, anecdotal information and results of one bench-scale project. Accordingly, an investigation was conducted to verify claims that use of quicklime alone can promote decomposition of PCBs. Synthetic soil samples were spiked with three PCBs and treated with quicklime and water. Significant PCB losses (60% to 85%) were evidenced after five hours of treatment. However, evaporation and steam stripping at elevated temperature conditions, rather than PCB decomposition, accounted for most of the losses observed. Low levels of partially dechlorinated PCBs were detected in lime-treated samples, but the quantities were stoichiometrically trivial. The amounts of observed dechlorination products were not dependent on the duration of lime treatment, and no evidence of phenyl-phenyl bond cleavage was found. The use of quicklime alone as an in-situ treatment for removal of PCBs is not supported by these results (U.S. EPA, 1991c).

**S/S Treatment of Salts of As, Cd, Cr, and Pb.** The behaviors of various metal salts in cement-based S/S processes are being studied through leaching tests, conduction calorimetry, and solid-state NMR. The research is aimed at identifying the chemistry involved during cement hydration reactions in S/S processes treating metal salts (U.S. EPA, 1990f).

**The Nature of Lead, Cadmium, and Other Elements in Incineration on Residues and Their Stabilized Products.** A detailed laboratory study of metal species in raw and S/S-treated wastes is being conducted to test how the chemical nature and binding state affect leachability. Focus will be on the application of sophisticated surface analysis techniques to characterize poorly crystalline inhomogeneous metal forms. Existing geochemical models will be applied to test if they can predict the formation of solubility-controlling solid phases as determined analytically (Eighmy et al., 1992).

### **6.1.3 Interferences**

**Factors Affecting the S/S Treatment of Toxic Waste.** Research on interfering agents is being done to quantify the physical and performance characteristics of S/S-treated waste containing interfering chemicals. The data are being analyzed to determine whether physical properties can be correlated with durability and leach resistance. Interferences from inorganics such as Pb, Cd, and Zn and from sulfates and organics such as oil, grease, hexachlorobenzene, trichloroethylene, and phenol are being studied (Jones et al., 1992).

**Effects of Selected Waste Constituents on S/S-Treated Waste Leachability.** The effects of 10 common waste constituents on the strength and contaminant immobilization of S/S-treated waste were studied. The contaminants were cadmium, chromium, mercury, and nickel. The potential interferences were nitrate salts, sodium hydroxide, sodium sulfate, and five organic substances. The S/S binders tested were Portland cement, cement plus fly ash, and lime/fly ash (Jones et al., 1992).

### **6.1.4 Organics and Air Emissions**

**Roles of Organic Compounds in Solidification/Stabilization of Contaminated Soils.** Organic compounds pose problems for solidification/stabilization processes in three ways:

1. Nontarget organics can interfere with the immobilization of target metals.
2. Target organics are more difficult to stabilize than metals.
3. Some organics can volatilize during mixing with treatment agents, leading to unacceptable air emissions.

The University of Cincinnati, on behalf of the U.S. EPA, is evaluating the effectiveness of S/S processing for organic/metal wastes, in terms of organic immobilization and organic-induced effects on metal immobilization. Organic emissions during S/S processing are being measured. Polyaromatic hydrocarbons (PAHs) will be used in this project to represent a common class of organic compounds of concern in waste remediation.

**Measurement of Volatile Emissions from S/S-Treated Waste.** Although the mechanical strength and leaching characteristics of S/S-treated wastes have been investigated, few data are available on the emissions of organics from the S/S process and from the treated waste. Acurex Corporation at Research Triangle Park, North Carolina, is developing organic measurement methods and using them to test S/S-treated waste to address this data gap. A "Wind Tunnel" system, a "Modified Headspace" sampling system, and a "Sample Venting" system have been developed and are being used to measure organic releases from S/S-treated waste (Weitzman et al., 1990).

**Field Assessment of Air Emissions From Hazardous Waste S/S Processing.** The U.S. EPA is collecting information to develop standards necessary to control air emissions from hazardous waste treatment, storage, and disposal facilities. Field tests have been conducted to quantify emissions of volatile, semivolatile, and particulate emissions from S/S treatment processes (Ponder and Schmitt, 1991).

**S/S Treatment of Metal Wastes Contaminated with Volatile Organics.** S/S-treatment of sludge contaminated with about 1% metal ions and about 0.04 % VOCs was tested. Waste sludge containing 11 metal contaminants was spiked with 8 VOCs. Four different cement based S/S processes were applied to treat sludge samples (Spence et al., 1990).

**Immobilization of Organics in S/S Waste Forms.** U.S. EPA RREL is sponsoring a laboratory study to investigate (1) the immobilization of target organics by selected S/S formulations and (2) the effects of nontarget organics on the immobilization of target metals. Initial studies will be performed on spiked soils to systematically vary relative contaminant concentrations (Trish Erickson, U.S. EPA, personal communication, 1992).

#### **6.1.5 Test Methods**

**Method Development.** Laboratory and field test methods are needed to support optimum binder selection, assess short-term and long-term performance of S/S-treated waste, and allow better correlation of laboratory and field tests. A project is being conducted to study these three areas (U.S. EPA, 1991a):

- Evaluate the effect of sample size and configuration on results from leaching tests.
- Assess durability tests such as ANSI/ANS-16.1 and the accelerated aging/weathering protocol being developed through cooperative agreement between the U.S. EPA and the University of Cincinnati.
- Evaluate methods to monitor S/S-treated waste in situ.

**Investigation of Test Methods for Solidified Waste.** An effort was conducted with Environment Canada to evaluate several leaching and physical property measurement methods. This research is leading toward development of a protocol for evaluating S/S-treated waste. The protocol is based on the measurement of several physical, engineering, and chemical properties of S/S-treated wastes to allow different use and disposal scenarios to be evaluated. Several of the testing methods in the protocol have been evaluated in a cooperative project with industry initiated by Environment Canada. Others are methods recommended by standards organizations in the fields of hazardous and radioactive wastes. Finally, some properties of S/S wastes were measured using methods in the developmental stage (Stegemann and Côté, 1991).

**Critical Characteristics of Hazardous S/S-Treated Waste.** The physical and chemical characteristics of the waste affect performance, as do the climatic (temperature and humidity) conditions during curing and after placement in the final disposal or reuse environment. This research is being conducted to determine the critical characteristics affecting waste performance and how to measure them. The work is leading to quality control procedures for use in the field to better assure performance of S/S-treated waste (Wiles and Howard, 1988).

**Advanced Test Methods.** A program evaluating test methods for construction materials and stabilized waste is ongoing at Energieonderzoek Centrum Nederland (ECN). Aspects being dealt with are changes within the product with time, problems in determining the proper geometrical surface area, boundary conditions for modeling the release from products, development of a three-dimensional leaching model, and chemical speciation within a waste form. Testing involves radionuclide tracers in specific chemical forms in the S/S-treated waste (van der Sloot, ECN, personal communication, 1991).

**Assessment of Long-Term Durability of Solidified/Stabilized Hazardous Waste Forms — Lab Component and Field Component.** U.S. EPA RREL is sponsoring a laboratory study of synthetic and real hazardous wastes to develop a protocol for accelerated weathering testing of cementitious waste forms. Durability testing is focused on the use of elevated temperature or acid to speed degradation reactions.

A field project is also being conducted to develop and utilize sampling and analysis methods that allow assessment of waste form durability after various periods of exposure to field conditions. Early efforts are concentrating on detection of the interface between buried waste forms and adjacent fill material. Subsequent work will focus on sampling to obtain surficial (<1 cm) weathered material for analysis as well as bulk sampling. The observed weathering patterns will be compared with those induced under laboratory or lysimeter conditions. (Trish Erickson, U.S. EPA, personal communication, 1992).

#### **6.1.6 Leaching and Transport Models**

**Contaminant Profile Analysis.** Chemical and X-ray diffraction analysis methods are being used to determine the composition profiles in blocks of S/S-treated waste that have experienced long-term leaching. These analyses evaluate the actual release from S/S-treated waste and provide insight into the processes occurring within the waste during leaching (Hockley and van der Sloot, 1991).

**The Binding Chemistry and Chemical Leaching Mechanism of Hazardous Substances in Cementitious S/S Binders.** Type I Portland cement samples containing the soluble nitrates of the priority pollutant metals chromium, lead, barium, mercury, cadmium, and zinc have been investigated using thermogravimetric and Fourier-transform infrared techniques, including diffuse reflectance. The major vibrational bands and thermal stability of the carbonate, sulfate, silicate, water, and nitrate species have been tabulated in comparison to uncontaminated Portland cement. Immobilization mechanisms and their effect on contaminant leaching are being studied (Ortego et al., 1989; Ortego, 1990; and Ortego et al., 1991).

**Development of a Numerical Three-Dimensional Leaching Model.** The overall goal of this research effort is to improve the fundamental understanding of binding chemistry and leaching mechanisms in S/S-treated waste and to apply this understanding to development of improved S/S technology and of improved methods for predicting the environmental impacts of disposing of S/S-treated waste. This work is taking the approach of developing mechanistic leach models and developing characterization methods that can be used with the leach models. An underlying theme throughout this research is the need to separately describe the physical and chemical immobilization mechanisms. A set of simple leach models has been developed based on various simple reaction systems and rectangular geometry. Irreversible immobilization, reversible linear sorption, reversible precipitation, and reaction between a precipitate and inwardly diffusing reactant are the mechanisms considered in the simple leach model. A general numerical three-dimensional leaching model is being developed based on the Crank-Nicholson finite difference algorithm (Batchelor, 1991, personal communication).

**Acid Leaching Rate and Advancement of Acid Front in S/S-Treated Waste.** This program is studying the behavior of leaching of a cement-based waste form. The investigations indicate that acids in the bulk solution diffuse through the pores of the waste form leading to a reduction in pH and dissolution of metals.

The dissolved metals leach out of the solid matrix into the bulk solution, leaving a leached layer on the surface of the waste form. A sharp leaching boundary was identified in every leached sample, using pH indicators. The movement of the leaching boundary was found to be a single diffusion-controlled process. Studies were conducted using both static and semidynamic leaching procedures (Cheng and Bishop, 1992).

**Leaching Test Methods and Models.** Several leaching mechanisms, including dissolution of the matrix, washoff of surface contaminants, and diffusion-controlled release, were studied. A variety of leach testing methods were described and the capabilities compared. A diffusion model for leaching was developed (de Groot and van der Sloot, 1992).



**Review and Analysis of Treatability Data Involving Solidification/Stabilization of Soils.** A paper study of existing treatability data for S/S of 18 metals and application of geochemical models is being conducted to identify factors controlling metal solubility. The data base contains approximately 2600 records representing approximately 80 studies. Despite the volume of data, inconsistent data collection and procedural uncertainties limit interpretation. No statistically significant correlations could be found when post-treatment parameters were tested against measured waste characteristics. However, subsets of the data base will continue to be tested to identify chemical controls as the modeling work proceeds (Means et al., 1991a).

#### **6.1.7 Compatibility with Disposal or Reuse**

**Assessment of Long-Term Durability of S/S-Treated Waste.** The mechanisms governing the durability of S/S-treated waste are not well understood. Studies are needed to examine how the disposal environment interacts to modify the physical and chemical performance of the waste. In one study, S/S-treated waste is being tested to quantify waste form performance and examine degradation mechanisms. Testing involves accelerated freeze/thaw and wet/dry cycles and various environments, such as high or low pH, high pressure, high- or low-redox potential. Conventional and advanced large-scale leaching tests are being performed. The S/S-treated waste is being characterized by sophisticated techniques such as laser holography, acoustic stress wave testing, and dye injection (Bishop et al., 1990a).

**Effect of Curing Time on Leaching.** The effect of curing time on metal leaching, as measured by the TCLP test, is being studied in synthetic wastes for a variety of metal contaminants. Initial results indicate a significant effect of curing time, both on TCLP results and on the chemical structure of the stabilized waste as evidenced by spectroscopic analyses (Akhter and Cartledge, 1991; Cartledge, 1992). Both increased and decreased leaching is being observed, depending on the metal contaminant, binder, and other factors. These observations underline the limitations of the TCLP test as an indicator of the long-term leaching of stabilized waste and emphasize the need for other types of leaching data.

**Field Performance of S/S-Treated Waste.** Solidification/stabilization is used at CERCLA sites and in other waste treatment applications. However, durability of S/S-treated waste remains unclear due, in part, to the relative newness of the technology and the lack of information from sites currently applying S/S processes. A three-phase project is under way (U.S. EPA, 1991b):

- Identify sites using S/S processes.
- Core sample and test S/S-treated waste from several sites.
- Design and implement a program to solidify representative wastes by various S/S processes and monitor the wastes over an extended period.

**Utilization and Disposal.** The performance of S/S-treated waste depends on the environment the material is exposed to as well as the treated waste and contaminant properties. The Waste Technology Centre in Canada is developing an evaluation protocol as a decision-making tool for management of S/S-treated waste. One factor in the protocol is identification and definition of use and disposal scenarios. Scenarios include unrestricted use, approved use, sanitary landfill, segregated landfill, and secure landfill (WTC, 1990b).

#### **6.1.8 Treatability Tests and S/S Process Application**

**Superfund Innovative Technology Evaluation (SITE) Program.** The SITE Program was established to accelerate the development and use of innovative cleanup technologies at hazardous waste sites across the country. The Demonstration Program of SITE focuses on field demonstration of emerging site remediation technologies. The Demonstration Program has 37 active tests, including the eight low-temperature S/S technologies summarized in Table 6-1.

**Municipal Waste Combustion Residue S/S Program.** Vendors of S/S processes are cooperating with the U.S. EPA Office of Research and Development Risk Reduction Engineering Laboratory to demonstrate and evaluate the performance of S/S processes for treating residues from the combustion of municipal solid waste (MSW). The program includes four S/S processes: cement, silicate, cement kiln dust, and a phosphate process. The aim of the project is to

TABLE 6-1. SUPERFUND INNOVATIVE TECHNOLOGY EVALUATION PROGRAM:  
SOLIDIFICATION/STABILIZATION TECHNOLOGIES

Developer	Solidification/ Stabilization Technology	Applicable Waste Media	Applicable Waste	
			Inorganic	Organic
Chemfix Technologies, Inc. Metairie, LA	Soluble silicates and silicate setting agents	Soil, sludge, other solids	Metals	High-molecular- weight organics
HAZCON, Inc. Brookshire, TX	Cement and proprietary additive	Soil, sludge	Metals	Not an inhibitor
International Waste Technologies/Geo-Con, Inc. Wichita, KS	In situ — silicate and proprietary additives	Soil, sediment	Nonspecific	PCBs, other nonspecific organic compounds
S.M.W. Seiko, Inc. Redwood City, CA	In situ — proprietary binder	Soil	Metals	Semivolatile organic compounds
Separation and Recovery Systems, Inc. (SRS) Irvine, CA	Lime and proprietary additives	Liquid/solid	Low-level metals	Specific for acidic sludges with at least 5% hydrocarbons
Silicate Technology Corp. Scottsdale, AZ	Silicate, cementitious material, and proprietary additives	Groundwater, sludge, soil	Metals, cyanide, ammonia	High-molecular- weight organics
Soliditech, Inc. Houston, TX	Pozzolan or cement and proprietary liquid additives	Soil, sludge	Metals	Nonspecific
Wastech, Inc. Oak Ridge, TN	Proprietary	Soil, sludge, liquid waste	Nonspecific, radioactive	Nonspecific

Nonspecific = Technology is generally applicable to that waste type.

Sources: U.S. EPA, 1988d and Barth, 1991)

enhance the environmental performance of S/S-treated MSW combustion residue in a range of final environments. The final environment may be disposal in the land or use as roadbed aggregate, building blocks, or artificial reefs for shore erosion control (Wiles et al., 1991a and b).

**Leaching Mechanisms and Performance of S/S-Treated Hazardous Waste Substances in Modified Cementitious and Polymeric Matrices.** In this study, a latex polymer additive is being used with Portland cement to treat inorganic- and organic-contaminated waste. The latex polymer is used to reduce the porosity of the S/S-treated waste in order to improve immobilization (Daniali, 1990).

**Stabilization Potential of Lime Injection Multistage Burner (LIMB) Product Ash Used With Hazardous Distillation Residues.** A study is under way to investigate the trace metal binding mechanisms in S/S high-sulfur coal fly ash and flue gas desulfurization (FGD) sludges. Fly ash and sludge from a typical wet FGD process and dry flue gas desulfurization by-product from a demonstration LIMB process are being evaluated. The latter material contains substantial portions of available lime and may prove amenable as a solidifying agent with the fly ash. This work is being done to characterize the waste, determine the solidified/stabilized waste formulation, and measure the influence of liquid/solid ratio on metal leaching from the waste forms (Bishop et al., 1992; Dusing et al., 1991).

**Stabilized Incinerator Residue in a Shore Protection Device.** The goals of this research are to stabilize potentially toxic incineration residues and to use the stabilized material to construct energy-deflecting or absorbing structures to reduce shore erosion. The initial phases of the project will deal with developing the proper mix design for stabilized materials in high-wave energy environments and with determining their engineering properties, leachate characteristics, and potential toxicity to organisms. Permits will be secured to construct a model wave deflector/absorber in a marine system. The actual construction will occur in the next phase (Swanson, 1990).

## **6.2 FUTURE DEVELOPMENT**

For more than 20 years, S/S processes have been used to treat industrial and radioactive waste. More recently, the technology has been used to treat contaminated soils at CERCLA sites, fly ash, incinerator ash, and metal-contaminated sludges.

Despite extensive application and considerable research, there still are areas that could profit from additional effort. An increased understanding of S/S mechanisms, interferences, leaching behavior, and long-term performance would all help to improve process efficiency and increase confidence in the technology. Some areas to consider for future research are summarized in sections 6.2.1 through 6.2.8.

### **6.2.1 Binders**

- Increase immobilization performance by modifying existing binders.
- Develop advanced binders to minimize volume increase inherent in most existing S/S processes.
- Develop advanced binders with better tolerance to organic contaminants and interferences.
- Determine factors affecting optimum binder addition rate. Too much of a particular binder ingredient can lead to an improperly stabilized waste form. For example, many metals are amphoteric, meaning that they are soluble under both acidic and alkaline conditions. The metal will be at minimum solubility when a sufficient base (S/S ingredient) is added to make the waste moderately alkaline. Too much base will cause the metal to resolubilize and/or make the waste hazardous by virtue of the RCRA corrosivity characteristic (i.e., pH >12.5).

### **6.2.2 Mechanisms**

- Develop an understanding of chemical speciation and how it affects immobilization.
- Gain understanding of S/S process bonding mechanisms with presently used binders and additives.
- Gain understanding of microstructure and chemistry of the complex interactions among binder phases and contaminants (McDaniel et al., 1990).

### **6.2.3 Interferences**

- Organic matter in the waste can prevent setting of the S/S-treated waste or reduce the strength or immobilization performance of the final product. Research is needed to determine threshold levels for interfering organic compounds with inorganic and organic S/S binders.
- Interfering agents should be classified into groups based on similarity of interference mechanisms. Once the mechanisms are defined and interferences grouped, control parameters could be set for interfering chemicals such as volatile organics, insoluble organics, soluble organics, soluble salts, sulfates, and ammonia.

### **6.2.4 Organics and Air Emissions**

- Develop methods to efficiently remove organic contaminants from sludge, soil, and soil-like wastes (Barth, 1990).
- Develop methods to determine whether bonding occurs between binder and organic waste. Increased understanding of the mechanisms for organic immobilization will speed development of better binders for organic contaminants.

### **6.2.5 Test Methods**

- Characterize the chemical interaction within the S/S-treated waste and at the waste/soil interface by diffusion tube measurements with radiotracers.
- Develop methods to more accurately predict and measure the performance of S/S processes and products in the laboratory and to improve the correlation of laboratory results with performance in the field (McDaniel et al., 1990).
- Develop and evaluate simple methods for determination of metal speciation for use in binder evaluation and selection.
- Develop and evaluate methods for inexpensive determination of metal speciation.
- Develop better test methods for detailed research of S/S-treated waste performance (e.g., X-ray fluorescence, computer imaging, laser holography).

- Identify factors affecting scale-up of treatability test results to determine the safety margin needed in performance measures. Scale-up from bench-scale to field-scale involves a number of variables that cannot be exactly replicated in the bench-scale experiments, e.g., field-curing conditions, degree of mixing, and ingredient control, among others. Therefore, the results of the bench-scale tests should exceed the performance measures for the field project by a wide enough margin to allow for unknown contingencies. As a general rule, if a bench-scale test meets the field performance measures by only a slim margin, then one may expect problems with full-scale implementation.
- Quantify the effect of the small-scale treatability test environment on S/S-treated waste performance. The jar environment promotes good contact between the binder and waste form and can enhance the degree of stabilization.

#### **6.2.6 Leaching and Transport Models**

- Develop approaches to better predict field performance from laboratory results.
- Quantify containment release rates by diffusion and advection over long-term exposure to environmental conditions. Use the transport data to evaluate the acceptability of the release rates.
- The TCLP does not fully address the main leaching mechanisms for many organics. In many cases, the organics in leachates are associated with particulate matter. Methods need to be developed to assess the fraction of organics mobilized by mechanisms not directly related to diffusion or dissolution such as sorption on particulates.
- Develop better, more economical, and more rapid leaching tests that allow reliable prediction of long-term performance of S/S-treated waste.

#### **6.2.7 Compatibility with Disposal or Reuse**

- Identify and validate methods to produce S/S-treated waste that can be reused or recycled (Barth, 1990).
- Determine the long-term physical durability and contaminant retention properties of S/S products by the following means:

- Define the physical and chemical environments for various end uses.
  - Develop accelerated weathering tests.
  - Define biodegradation potential.
  - Determine the relative merits of granular versus monolithic materials.
- Analyze the conditions needed for long-term environmental protection for S/S-treated waste placed in a disposal or use environment. Analysis will include determination and evaluation of the ultimate release pathways.
  - Evaluate and develop criteria for reuse of S/S-treated waste (e.g., bricks or subgrade fill).

#### **6.2.8 Treatability Tests and S/S Application**

- Determine the effectiveness of S/S processes and equipment for treating contaminated soil and impounded liquid.
- Determine effectiveness of mixing methods (including in situ methods).
- Evaluate effectiveness of slag addition or other pretreatment options to alter the valence states of metal contaminants prior to S/S processing.
- Establish a database recording important characteristics of S/S processing, such as binders, waste characteristics, interferences, and performance.
- Develop expert systems to aid in planning and evaluating treatability studies, S/S processes, and pretreatment options. The expert systems can be used to screen potential S/S processes for specific waste types and contaminated site conditions.
- Develop real-time QA/QC methods for S/S process control.
- Evaluate uses, based on experience with S/S treatment of industrial sludge, for similar wastes such as dredged materials from harbors and waterways or ashes and residues from combustion of coal and municipal solid waste.
- Develop strategies to optimize sample collection and analysis to increase efficiency and reduce cost.



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## **APPENDIX A**

### **SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS**

#### **INTRODUCTION**

This section summarizes the steps in the technology screening process for S/S technology. It provides a checklist of the material described in detail in Chapter 2. The organization of the checklist parallels the organization of Chapter 2, integrating the issues covered in that section into a user-friendly format. The checklist worksheets help the uninitiated user to follow orderly and comprehensive screening procedures. The screening could be repeated at several stages throughout a project, as appropriate. For the first use, the checklist would serve as a tool to guide preparation of test plans. The checklist would then be applied at major milestones, such as selection of an S/S process or completion of bench-scale screening, to review progress, identify weaknesses in the project, and develop methods to improve the testing. Later in the testing the checklist would be applied to review and evaluate the project.

#### **INSTRUCTIONS**

Each major subheading in the checklist is followed by 1) a brief statement or question that clarifies the scope and aspect of S/S technology covered in that section and 2) a series of questions to guide evaluation of the S/S project with respect to that aspect. The question can be evaluated as "favorable," "neutral," "unfavorable," "not known," or "not applicable." "Favorable" means lower complexity or a higher probability of success for the S/S project. "Neutral" means that the issue has a known effect but the effect is not significant to the outcome of the project. "Unfavorable" means greater challenges to S/S technology. "Not known" means there is high probability of an effect but the magnitude and/or direction are not known. "Not applicable" means a low probability of any effect. The questions are typically clarified or elaborated with notes in the "Issues" column. In most cases the evaluations are qualitative, but in a few cases quantitative performance criteria are given as guidance. Typically, an answer of "yes" to the question equals a favorable condition. Cases where the reverse is true are noted.

A summary sheet for tallying the responses for each subheading is provided at the conclusion of this chapter. The purpose of the summary sheet



is to assist in identifying trends or possible weaknesses in the treatability study.

Not every issue listed in the checklist is applicable to every treatability study. Irrelevant issues should be ignored. It is hoped that, through consideration of the issues contained herein, future S/S treatment projects can be improved in terms of both planning and conduct.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS

Information Requirements*		Indicator					Issues
		Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<b>1 SITE-SPECIFIC BASELINE INFORMATION REQUIREMENTS</b>							
1.1	Waste Sampling - Do the waste samples accurately reflect the chemical and physical characteristics of the entire volume of the waste?						
1.	Are preliminary field surveys available?						Planning for sampling
2.	Are waste sampling procedures documented and consistent with guidance in SW-846 (U.S. EPA, 1986a) and/or other agency guidance?						Representativeness, holding times, chain-of-custody, etc.
3.	Are sampling locations statistically randomized?						Representativeness
4.	Is sample variability addressed by statistical analysis?						Representativeness
5.	Were samples composited prior to analysis?						Composites preferred for comparative treatability testing but do not define extremes in waste composition. Variation is particularly important for testing of continuous processes, e.g., pug mill mixing.
6.	Were debris, large rock fragments, vegetative material, etc. removed prior to analysis?						Representativeness
7.	Is material available sufficient for pilot-scale testing?						Need to support waste characterization and bench- and pilot-scale tests.
8.	Is some material being archived for possible later tests?						QA/QC

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*		Indicator					Issues
		Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
1.2	Waste Acceptance - Is the waste material toxicity low enough to allow contact handling needed for S/S testing and application?						
	1. Was a representative sample analyzed prior to shipping?						Identification of chemical hazards
	2. Is waste composition in compliance with shipping regulations?						Toxicity and U.S. DOT shipping regulations
	3. Is the hazard to S/S workers acceptably low?						Worker safety
1.3	Waste Characterization - Is there an adequate, statistically valid database to support selection of binding agents?						
	1. Is historical information available?						Optimize data collection
	2. Does characterization include a "total waste analysis"?						Identify target contaminants
	3. Were TCLP data generated on the untreated waste?						Baseline leaching data; RCRA toxicity characteristic
	4. Have other hazard characteristic tests been performed or are they known to be unnecessary?						Ignitability, corrosivity, reactivity, toxicity, infectivity
	5. Have other chemical analyses been performed to establish baselines and possible S/S interferences?						pH, redox potential, acid neutralization capacity, etc.; Interferants screen, e.g., oil and grease, salt content, nitrate, sulfate, etc.
	6. Have baseline physical characteristics of the untreated waste been measured?						UCS, specific gravity, Paint Filter Test, permeability, etc.

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*		Indicator					Issues
		Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
7.	Are any other data available on the physico-chemical form of the target contaminants?						X-ray diffraction, SEM-EDXA, microscopy, spectroscopy, etc.
1.4	Site Characterization - Are fundamental site characteristics established to give baseline data for the design of the treatment system?						
1.	Does the site support the setup and operation of S/S equipment?						Available space, topography, excavation difficulty, climate
2.	Are necessary resources close to the site?						Design flexibility
	<ul style="list-style-type: none"> <li>• Water, gas, electricity</li> <li>• Supplies and chemicals</li> <li>• Equipment</li> <li>• Access routes</li> <li>• Disposal facilities</li> </ul>						
3.	What proportion of the waste occurs above the groundwater table (or uppermost aquifer)?						Excess water can make excavation difficult and require dewatering of waste material.
	100% = favorable						
4.	Has the total waste volume been estimated, measured, or calculated?						Smaller volumes, more limited treatability study; larger volumes, more extensive treatability study
5.	Does the waste contain debris that may interfere with field treatment?						Pretreatment and handling requirements; interferences may be process-specific.
	no = favorable						

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

**SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)**

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>6. What are the textural characteristics of the waste?</p> <p>dry, granular = favorable clayey, sludge, or liquid = neutral hard, blocky = unfavorable</p> <p>7. How heterogeneous is the distribution of the target contaminant(s) within the waste?</p> <p>fairly homogeneous = favorable</p>						<p>Pretreatment and handling requirements</p> <p>More analytical data needed to compensate for higher variability.</p>
<p>1.5 Quality Assurance/Quality Control - Is QA/QC sufficient to determine and document data quality?</p> <p>1. Does the analytical laboratory performing the analyses on the untreated waste possess appropriate qualifications/certifications?</p> <p>2. Are the characterization data collected under an appropriate QA/QC program, or is there some other indication of the quality of the analytical measurements?</p> <p>3. Are there a sufficient number of replicates analyzed to permit a statistical analysis of the results?</p> <p>4. Is a second analytical laboratory available for interlaboratory verification on a portion of the more critical measurements?</p>						<p>CLP, other qualifications/certifications</p> <p>Blind replicates, duplicates, bracketed calibration, standard additions, blanks, etc.</p> <p>Mean, standard deviation, confidence intervals, etc.</p> <p>Data accuracy, interlaboratory verification</p>

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<b>2 PERFORMANCE OBJECTIVES</b>						
<b>2.1 Regulatory Requirements - Have CERCLA and RCRA regulatory-driven requirements been considered in developing performance requirements?</b>						
1. Is the site close to possible receptors of noise, fugitive dust, volatiles, or odors?						Possible source of location-specific ARAR
2. Is the site close to sensitive environmental areas such as floodplains, wetlands, or the breeding grounds of protected species?						Possible source of location-specific ARAR
3. Are the primary contaminants metals or organics, or both?  metals = favorable metals and organics = neutral organics only = unfavorable						S/S BDAT for many metals; some types of organics may require pretreatment unless present in low concentrations.
4. If mostly metals, how many metals are present in regulated concentrations?  1 = favorable 2-3 = neutral 4 or more = unfavorable						Potential for incompatible chemistries; complex wastes are more difficult to satisfactorily stabilize
5. If arsenic and chromium are among the target contaminants, have their valence states been determined?						Toxicity issues; may affect binder selection; data may also be inferred from waste origin in some cases.

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>6. If mercury, nickel, tin, arsenic or lead is among the target contaminants, are analyses planned for organic (e.g., tetraethyl lead, tributyl tin, organoarsenic) or other unusual and toxic forms (e.g., nickel carbonyl)?</p>						Toxicity issues; may affect binder selection; data may also be inferred from waste origin in some cases.
<p>7. Does the waste contain volatile organic contaminants and, if so, in what concentrations?</p> <p>no or &lt; 50 ppb = favorable</p>						Levels of concern will vary with the contaminant; S/S not demonstrated for volatiles; probable release during mixing and curing; pretreatment probably necessary.
<p>8. Does the waste contain other high hazard or special contaminants, such as PCBs, dioxins, pesticides, chlorophenols, radionuclides, or cyanide?</p> <p>no = favorable</p>						Levels of concern vary with the contaminant; pretreatment will likely be necessary; S/S may not be preferred approach, unless a strong rationale is provided.
<p>2.2 Technical and Institutional Requirements - Have technical and institutional factors been considered in developing performance requirements?</p>						
<p>1. Will testing determine the leaching (e.g., TCLP) or physical properties (e.g., compressive strength) of treated waste?</p>						Demonstrate basic feasibility.
<p>2. Are reagent costs consistent with project economics?</p>						Calculate binder cost per volume of stabilized waste.
<p>3. Does the waste contain compounds that may decompose or volatilize to produce off-gas?</p> <p>no = favorable</p>						Off-gas treatment increases processing costs.

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
4. Will the waste mix well with the binder?						Good mixing and wetting is needed to ensure a strong, uniform product.
5. Does the waste interfere with setting or cause unfavorable reactions with the binder?  no = favorable						Interferences should be identified.
6. Is the waste/binder mixture fluid and amenable to material handling and mixing?						Pumpable waste/binder mix makes handling easier.
7. Does S/S increase waste volume significantly?  no = favorable						Large volume increase raises costs and increases disposal problems.
8. Is the S/S-treated waste amenable to placement?						Need long-term structural integrity and ability to support heavy equipment soon after placement.
9. Is the binder material subject to possible biodegradation?  no = favorable						Long-term stability
10. Are longer-term leaching tests on the treated waste planned?						TCLP is not a good indicator of long-term stability.
<b>3 INITIAL TECHNOLOGY SCREENING</b>						
3.1 Technology Screening/Feasibility Study - Has S/S been compared to other treatment alternatives and been found to be the most appropriate technology?						

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.



# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<b>3.1.1 CERCLA Technology Screening</b>  1. Do the selected methods protect human health and the environment?  2. Do the selected methods meet ARARs?  3. Do the selected methods reduce toxicity, mobility, or volume?  4. Do the selected methods minimize impact to human health and the environment?  5. Do the selected methods reliably maintain low residual risk to human health and the environment?  6. Do the selected methods allow efficient, cost-effective application at the site?  7. Are the selected methods likely to receive state acceptance?  8. Are the selected methods likely to receive community acceptance?						Methods should attain threshold criteria.  Methods should attain threshold criteria.  Methods should provide good trade-off of primary balancing criteria.  Methods should provide good trade-off of primary balancing criteria.  Methods should provide good trade-off of primary balancing criteria.  Methods should provide a good trade-off of primary balancing criteria.  Modifying criteria are evaluated after the public comment period.  Modifying criteria are evaluated after the public comment period.
<b>3.1.2 Technology Screening at RCRA TSD Facilities</b>  1. Is the waste banned under another regulatory system such as TSCA?  yes = not suitable for S/S						Review waste for suitability of S/S treatment.

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>2. Is the waste classified as "not suitable" for S/S or land disposal under the landbans, or is a technology other than S/S recommended as BDAT?</p> <p>yes = not suitable for S/S</p>						Adherence to RCRA landban and BDAT recommendations
<p>3. Is the waste not yet covered or extended under landbans?</p> <p>yes = S/S not required</p>						Landban requirements
<p>4. Does the generator certify that the waste meets landban requirements?</p> <p>yes = S/S not required</p>						Landban requirements
<p>5. Is the waste restricted or banned under site permit conditions or otherwise unacceptable to a TSD facility?</p> <p>yes = not suitable for S/S</p>						Permit compliance
<p>6. Is treatment required to prepare waste for a TSD facility's S/S system?</p> <p>yes = less favorable</p>						Treatment process complexity
<p>3.2 General Criteria for Not Using S/S - Is the waste compatible with S/S technology?</p>						
<p>1. Is the waste amenable to recycling, reuse, or recovery technology, all other factors being equal?</p> <p>no = favorable for S/S</p>						Recycling, reuse, and recovery are preferred over treatment or disposal.

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>2. Is the waste treatable by a destruction technology, all other factors being equal?</p> <p>no = favorable for S/S</p> <p>3. Are there ARARs that cannot be satisfied by existing S/S technology?</p> <p>no = favorable for S/S</p> <p>4. Is S/S waste treatment inefficient or expensive when compared to another remedy?</p> <p>no = favorable for S/S</p> <p>5. Does the waste exhibit poor mixing, incompatibility, or other unacceptable characteristics?</p> <p>no = favorable for S/S</p> <p>6. Does the waste contain volatile organics or a large fraction of total organics?</p> <p>no = favorable for S/S</p>						<p>Contaminant destruction is preferred over disposal.</p> <p>Can S/S meet regulatory requirements?</p> <p>Cost effectiveness</p> <p>Amenability to S/S</p> <p>Organics can be difficult to stabilize.</p>
<p>4 WASTE/BINDER COMPATIBILITY LITERATURE SCREENING - Has a comprehensive review and selection process found a group of test S/S binder formulations that have a high probability of providing good stabilization?</p>						

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>1. Are interferences and chemical incompatibilities considered as part of the binder selection?</p> <p>2. Has metal chemistry been considered in the binder formulation?</p> <p>3. Is S/S-treated waste compatible with the planned end use?</p> <p>4. Are the S/S costs known, and are they competitive with other treatment and disposal methods?</p> <p>5. Does the S/S process have a proven track record on similar wastes?</p>						<p>Pozzolanic binders are incompatible with high concentrations of oil, grease, organics, chlorides, and other soluble salts. Sodium sulfite binder is incompatible with acids.</p> <p>Formation of metal hydroxides is an important stabilization mechanism with alkaline binders; however, high pH can increase the solubility of some metals (e.g., As and Cr).</p> <p>Possible end use includes disposal such as landfill, monofill, or burial or reuse as fill, road base, or construction material.</p> <p>Cost is a consideration but should be secondary to performance.</p> <p>While proven performance is desirable, innovative methods should not be discouraged.</p>
<p><b>5 LABORATORY BENCH-SCALE SCREENING OF THE WASTE/BINDER MIXTURES</b> - Although laboratory screening can be conducted in a variety of ways, it is typically an interactive process involving two sequential steps. A wide range of formulations are given simple tests. Then a more refined group are tested against more complex or demanding criteria. Test criteria and issues are discussed below.</p>						

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
1. Has an appropriate pretreatment step been devised, if necessary?						Highly toxic constituents; contaminants that do not respond well to S/S; interferants; debris
2. Have at least 3 to 4 different binders been selected for bench-scale testing?						Maximize potential for successful treatability study.
3. Are several different binder-to-waste ratios used in the testing?						Cost/benefit; excess binder may hinder S/S process.
4. Have waste/binder compatibility issues been considered in selecting a binder?						Target contaminants; interferants; compatibility with disposal environment
5. Is laboratory testing being based on composite or "worst-case" samples, or both?						Composite best for process comparison; may be necessary to design for worst case.
issue was considered = favorable						
6. Are any chemical additives to the binder carefully monitored and controlled?						Reproducibility, interpretability, sensitivity analysis
7. Are several rounds of bench-scale testing performed, i.e., have the most successful processes been adapted to the site-specific waste form?						Process optimization is an iterative process; ability to "engineer" solutions to treatability problems
8. Are the chemical compositions of the binder and of any other chemicals added during S/S (e.g., fatty dust) known?						Hazardous properties
9. Are all of the additives mentioned in item 8 above nontoxic and nonhazardous?						Corrosivity (pH), reactivity (free sulfide or lime), etc.

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

**SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)**

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>10. Are there any new ARARs that result from the binder additives?</p> <p>no = favorable</p> <p>11. Is there provision for a third party or regulatory agency to observe the treatability study?</p> <p>12. Were anticipated field conditions simulated during waste curing?</p> <p>13. Were the samples allowed to cure for an appropriate time period prior to analysis?</p> <p>14. Does the test program cover critical ARARs?</p> <p>15. Does the test plan provide for split samples to be sent to a second laboratory?</p> <p>16. Does the test include good statistical design, replication, blind controls, laboratory QA/QC, etc?</p> <p>17. Is the waste volume increase resulting from binder additions determinable from the test?</p>						<p>Toxicity and hazard characteristics, e.g., pH, reactive sulfide, metal leach criteria, volatile emissions, dust, etc.</p> <p>Objectivity</p> <p>"Jar effect" enhances performance</p> <p>28 days recommended before UCS testing for most pozzolans</p> <p>Leaching and critical chemical/physical properties</p> <p>Interlaboratory comparison to increase confidence in results</p> <p>Data accuracy and reliability</p> <p>End use compatibility, economical feasibility</p>

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<b>6 BENCH-SCALE PERFORMANCE TESTING/PROCESS OPTIMIZATION</b> - Does the bench-scale performance test demonstrate that the S/S-treated waste meets predetermined performance standards?						
1. Are the guidelines applied in the bench-scale screening also considered in the bench-scale performance testing?						Completeness and consistency
2a. If subsurface disposal is anticipated, are appropriate physical tests being conducted?						e.g., UCS, permeability etc.
2b. If surface or near-surface disposal is anticipated, are the appropriate physical tests being conducted?						e.g., wet/dry, freeze/thaw, etc.
2c. Is the longer-term stability of the waste toward leaching being evaluated?						e.g., multiple extraction procedure, ANSI/ANS/16.1, etc.
2d. For wastes containing organic contaminants with low aqueous solubilities, are leaching tests in an organic solvent being conducted?						Aqueous leachate is a meaningless indicator of process effectiveness because of low solubility of contaminant.
2e. Are there any technical reasons to suspect that colloidal contaminant transport may be important at this site?  no = favorable						Assess in leach test by modifying or eliminating filtration step.
2f. Is there any technical reason for conducting leach tests with site-specific groundwater as leachant?  no = favorable						e.g., humic-rich groundwater, or groundwater with other complexing ligands (e.g., carbonate, fluoride, high chloride, etc.)

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>2g. If the binder is biodegradable, is a biodegradation performance test being conducted?</p> <p>2h. If the disposal site could potentially leak into an aquatic system, are leachate bioassays being performed?</p> <p>2i. Are specific binding agent properties considered in the test plan?</p> <p>3. Is a total metal analysis being performed on the same subsample as the leach test?</p> <p>4. Have the leaching performance data been corrected for dilution by binder additives?</p> <p>5. Is there a safety margin in the performance data compared to the performance criteria?</p> <p>6. Is the process implementable in the field?</p> <p>7. Is the bulking factor (volumetric expansion of the waste due to binder additives and water) compatible with disposal constraints?</p> <p>&lt; 25 % expansion = favorable</p>						<p>Waste form stability</p> <p>Leachate toxicity to aquatic ecosystem</p> <p>Examples include:</p> <ul style="list-style-type: none"> <li>pH and reactive sulfide analyses for sulfide-containing treatment chemicals</li> <li>biodegradation tests for thermoplastic or other organic binders</li> </ul> <p>Eliminate false negatives.</p> <p>Subtract out effect of dilution.</p> <p>Mixing, ingredient control, and curing environments are not as well controlled in the field.</p> <p>Materials handling issues; process complexity; mixing, throughput, and storage requirements</p> <p>Criteria will vary depending on the site.</p>

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.



# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>8. Is the estimated cost of field treatment reasonable?</p> <p>&lt; \$100/ton = favorable \$100 - \$150/ton = neutral</p>						<p>Will vary depending on several factors, such as waste volume, binder type, and process complexity. Includes both operating and capital costs.</p>
<p>9. Does the process or binder selected have a successful track record for this type of waste?</p>						<p>Innovative processes may require slower implementation, e.g., mandatory pilot-scale test, more extensive field performance data.</p>
<p>10. Does the test plan provide for split samples to be sent to a second laboratory?</p>						<p>Interlaboratory comparison to increase confidence in results</p>
<p>11. Is there provision for a third party or regulatory agency to observe the bench-scale performance study?</p>						<p>Objectivity</p>
<p>12. Does the study simulate field conditions as closely as possible during curing?</p>						<ul style="list-style-type: none"> <li>• Representative of field conditions</li> <li>• Improve use of data for scale-up</li> </ul>
<p>13. Is the S/S-treated waste allowed to cure for the appropriate period of time?</p>						<p>Test reliability</p>
<p>14. Is the amount of performance testing consistent with the guidance provided in Section 2.7.2 regarding project risk?</p>						<p>The greater the risk, the more performance testing is needed.</p>
<p>15. Does the analytical laboratory performing the analyses on the treated waste possess appropriate qualifications/certifications?</p>						<p>CLP, other qualifications/certifications</p>

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
16. Were the performance data collected under an appropriate QA/QC program, or is there some other indication of the quality of the analytical measurements?						Binder replicates, duplicates, bracketed calibration, standard additions, blanks, interlaboratory verification, etc.
17. Have a sufficient number of replicates been analyzed to permit a statistical analysis of the results?						Mean, standard deviation, confidence intervals, etc.
<b>7 PILOT-SCALE AND FIELD DEMONSTRATIONS</b>						
7.1 The Need for Process Scale-Up - Is technical, regulatory, and institutional confidence in the S/S binder and binder/waste ratio high enough to obviate the need for bench-scale testing?						
1. Has the binder been used successfully in field applications?						Field application increases confidence.
2. Does the waste to be treated have physical and chemical characteristics similar to waste successfully treated in a prior field application?						<ul style="list-style-type: none"> <li>• Similar wastes' characteristics imply similar binder performance.</li> <li>• Particular attention should be given to complex mixtures and possible interferences.</li> </ul>
3. Are site surroundings similar?						Review site-specific performance and institutional issues.
4. Are regulatory requirements similar?						Site-specific regulatory issues and ARARs
5. Are process scale-up issues well understood?						<ul style="list-style-type: none"> <li>• Material handling</li> <li>• Mixing</li> <li>• Vapor evolution</li> </ul>

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
6. Are process costs known?						<ul style="list-style-type: none"> <li>Pilot plant test will improve accuracy of cost estimate.</li> </ul>
7. Is waste reasonably homogeneous and well characterized?						Waste composition variations can affect S/S binder performance.
7.2 Scale-Up Issues - Do your pilot-scale tests address the major remediation steps?						
1. Is the performance of earth-moving or other waste removal equipment known?						<ul style="list-style-type: none"> <li>Throughput</li> <li>Free liquid handling</li> <li>Operator safety</li> </ul>
2. Is the performance of material-handling equipment known?						<ul style="list-style-type: none"> <li>Throughput</li> <li>Caking/Plugging</li> <li>Spillage</li> </ul>
3. Is the storage and handling system for the S/S binder known?						<ul style="list-style-type: none"> <li>Inventory needs</li> <li>Throughput</li> <li>Space</li> </ul>
4. Is waste pretreatment needed to improve material handling?						<ul style="list-style-type: none"> <li>Size adjustment by crushing and/or screening</li> <li>Moisture adjustment</li> </ul>
5. Is waste pretreatment needed to improve binder compatibility or efficiency?						Blending, homogenization, pH adjustment, volatile organic removal
6. Are the mixing system for the S/S binder and the waste disposal approach known?						In situ, batch, continuous
7. Is the S/S-treated waste disposal approach known?						Handling, placement, compaction, moisture content, final closure and capping

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

# SOLIDIFICATION/STABILIZATION TECHNOLOGY SCREENING WORKSHEETS (cont'd)

Information Requirements*	Indicator					Issues
	Favorable	Neutral	Unfavorable	Not Known	Not Applicable	
<p>7.3 Analytical Testing of the Treated Waste - Is sampling and analysis of pilot plant S/S-treated waste sufficient to determine performance?</p> <p>1. Is basic testing included?</p> <p>2. Are additional tests required?</p>						<p>Leaching and physical strength</p> <p>Permeability, moisture content, chemistry</p>

\* An answer of "yes" to a question indicates a favorable condition unless otherwise indicated.

## SUMMARY SHEET

I. Site:

II. Reviewer:

III. Date:

IV. Review Summary:

	Favorable	Neutral	Unfavorable	Not Known	Not Applicable
1.1 WASTE SAMPLING					
1.2 WASTE ACCEPTANCE					
1.3 WASTE CHARACTERIZATION					
1.4 SITE CHARACTERIZATION					
1.5 QUALITY ASSURANCE/QUALITY CONTROL					
Subtotal, Waste and Site Characterization					
2.1 REGULATORY REQUIREMENTS					
2.2 TECHNICAL AND INSTITUTIONAL REQUIREMENTS					
2.3 APPROACH TO SETTING PERFORMANCE CRITERIA					
Subtotal, Performance Objectives					
3.1 TECHNOLOGY SCREENING/FEASIBILITY STUDY					
3.2 GENERAL CRITERIA FOR NOT USING S/S					
Subtotal, Initial Technology Screening					

# SUMMARY SHEET (Continued)

	Favorable	Neutral	Unfavorable	Not Known	Not Applicable
4. WASTE/BINDER COMPATIBILITY SCREENING					
Subtotal, Waste/Binder Compatibility Screening					
5. BENCH-SCALE LABORATORY SCREENING					
Subtotal, Bench-Scale Laboratory Screening					
6. BENCH-SCALE PERFORMANCE OBJECTIVES					
Subtotal, Bench-Scale Performance Testing/Process Optimization					
7.1 THE NEED FOR PROCESS SCALE UP					
7.2 SCALE UP ISSUES					
7.3 ANALYTICAL TESTING OF THE TREATED WASTE					
Subtotal, Pilot-Scale and Field Demonstration					

## **APPENDIX B**

### **DRAFT REPORT: SAMPLING AND ANALYTICAL PROCEDURES**

**Note: The sampling and analytical procedures document presented in this appendix was developed for sampling piles of waste material contaminated with copper and lead. The document is included here only as an example and has been modified to protect client confidentiality.**

**DRAFT REPORT**

**SAMPLING AND ANALYTICAL PROCEDURES**

**February 25, 1992**

**by**

**Andrea Leeson  
Jeffrey Means  
Gregory Headington  
Bruce Buxton**

**BATTELLE  
Columbus Division  
505 King Avenue  
Columbus, Ohio 43201-2693**

**(B-2)**



## TABLE OF CONTENTS

1.0 INTRODUCTION .....	1
2.0 PROJECT SCOPE .....	2
3.0 SAMPLING PROGRAM .....	7
4.0 ANALYSIS PROGRAM .....	8
5.0 STATISTICAL DESIGN .....	10
5.1 Overview .....	10
5.2 Approach .....	10
5.2.1 NUMBER OF SAMPLES PER WASTE PILE .....	10
5.2.2 GRID SIZE .....	14
5.2.3 SELECTION OF GRIDS .....	14
5.2.4 SAMPLING METHOD WITHIN A GRID .....	16
6.0 SAMPLING EQUIPMENT AND OPERATION .....	17
6.1 Dipper .....	17
6.2 Stainless Steel Spoon or Scoop .....	19
6.3 Glass Tube Thief .....	19
6.4 Auger and Thin-Wall Tube Sampler .....	21
7.0 SAMPLE COLLECTION AND PRESERVATION .....	25
7.1 Sample Collection .....	25
7.2 Sample Preservation .....	26
8.0 PERSONAL PROTECTIVE EQUIPMENT AND DECONTAMINATION .....	27
8.1 Personal Protective Equipment .....	27
8.1.1 SAMPLING .....	27
8.1.2 CLEANING OPERATIONS (DECONTAMINATION) .....	27
8.2 Decontamination .....	27
9.0 SAMPLE CUSTODY, LABELING, PACKAGING, AND TRANSPORTATION .....	30
9.1 Sample Custody .....	30
9.2 Sample Labeling .....	30
9.3 Sample Packaging .....	33
9.4 Sample Transportation .....	34

**TABLE OF CONTENTS**  
(Continued)

10.0	SAMPLE QUALITY ASSURANCE AND QUALITY CONTROL . . . . .	35
10.1	Rinsate Blanks (Equipment Washes) . . . . .	35
10.2	Laboratory Quality Control and Certification . . . . .	35
10.2.1	MATRIX SPIKE ANALYSIS . . . . .	35
10.2.2	MATRIX SPIKE DUPLICATES . . . . .	36
10.2.3	METHOD BLANK TESTS . . . . .	36

**LIST OF TABLES**

TABLE 2-1.	SUMMARY OF COPPER AND LEAD LEVELS IN WASTE BOXES . . . . .	4
TABLE 2-2.	SUMMARY OF COPPER LEVELS IN WASTE PILES . . . . .	5
TABLE 2-3.	SUMMARY OF LEAD LEVELS IN WASTE PILES . . . . .	6
TABLE 5-2.	SAMPLE SIZE REQUIRED TO DEMONSTRATE COMPLIANCE WITH REGULATORY THRESHOLD (RT) AS A FUNCTION OF ANTICIPATED AVERAGE CONTAMINATION LEVEL (X) AND COEFFICIENT OF VARIATION . . . . .	11
TABLE 5-2.	RANDOM NUMBERS TABLE . . . . .	15

**LIST OF FIGURES**

FIGURE 2-1.	SCHEMATIC DIAGRAM OF WASTE PILES . . . . .	3
FIGURE 6-1.	SCHEMATIC DIAGRAM OF DIPPER . . . . .	18
FIGURE 6-2.	SCHEMATIC DIAGRAM OF GLASS TUBE THIEF . . . . .	20
FIGURE 6-3.	SCHEMATIC DIAGRAM OF AUGERS AND THIN-WALL TUBE SAMPLER . . . . .	22
FIGURE 9-1.	CHAIN-OF-CUSTODY SHEET . . . . .	31
FIGURE 9-2.	SAMPLE LABEL . . . . .	32

**DRAFT REPORT**  
**FOR**  
**SAMPLING AND ANALYTICAL PROCEDURES**

**1.0 INTRODUCTION**

[The introduction is specific to each project and should briefly describe the project background and objectives.]

## 2.0 PROJECT SCOPE

The existing waste consists of three accumulated piles of material situated on pavement in an approximately rectangular shape (Figure 2-1). Approximate estimates of the dimensions of the piles are: Pile 1: 43 ft by 27 ft and 2 ft deep; Pile 2: 53 ft by 38 ft and 2 to 2.5 ft deep; and Pile 3: 53 ft by 20 ft and 3 ft deep.

A preliminary sampling of the waste was conducted by Battelle to obtain an estimate of the number of contaminants of concern as well as the concentrations. In addition, previous sampling of other similar waste which had been collected in rolloff boxes and stored in the parking area was analyzed in order to obtain a better estimate of the contaminants likely to be found in the piles. Copper and lead were the primary contaminants from both sampling surveys. Average concentrations of copper and lead from the rolloff boxes and piles are shown in Tables 2-1, 2-2, and 2-3. These preliminary measurements of the metal concentrations were used to design the sampling program.

The waste tends to be fairly uniform in consistency throughout, but possible variations in metal concentrations require that samples be collected at varying locations, both spatially and as a function of depth. Specific details of the sampling design are discussed in the following section.

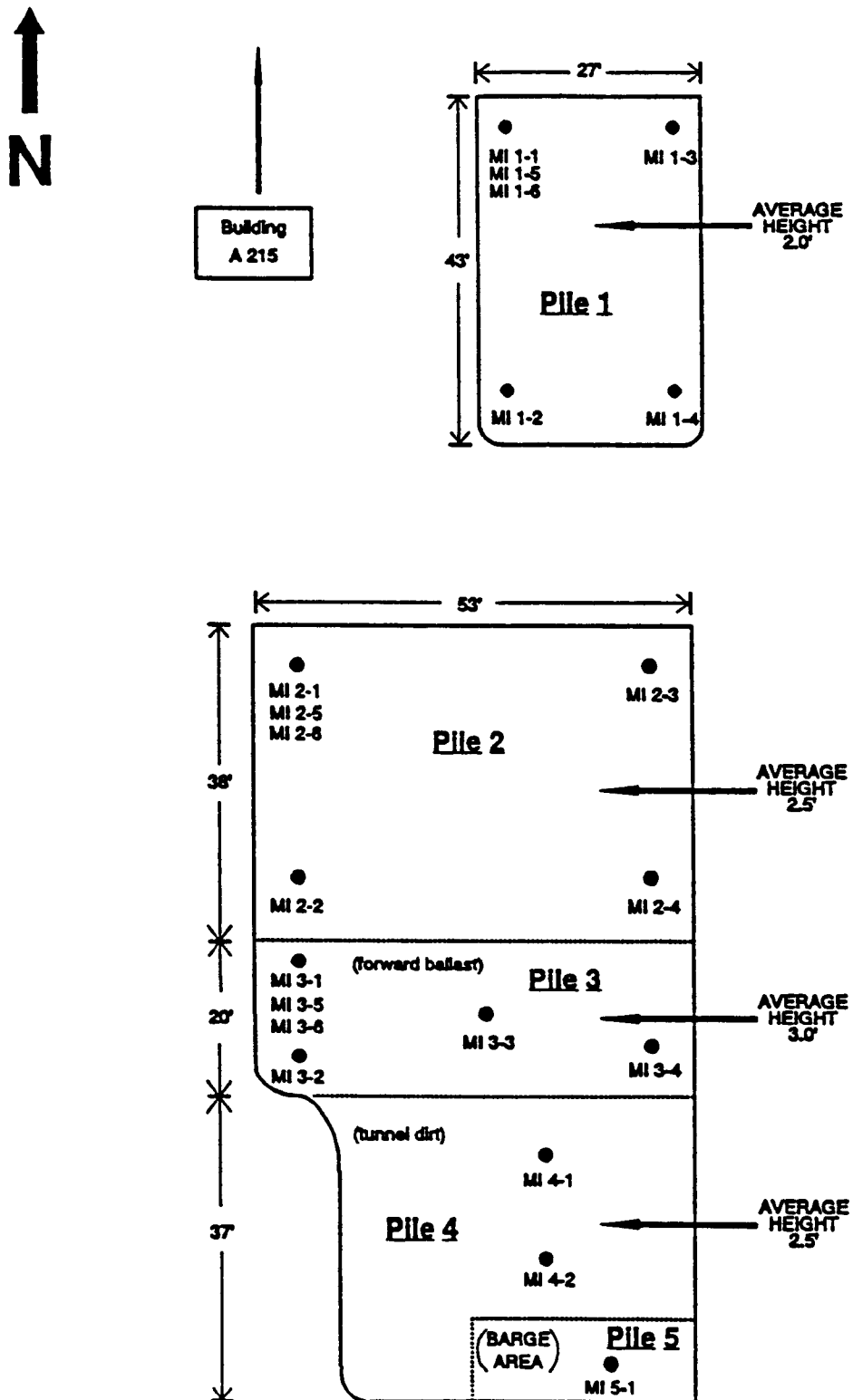


FIGURE 2-1. SCHEMATIC DIAGRAM OF SPENT WASTE PILES

TABLE 2-1. SUMMARY OF COPPER AND LEAD LEVELS IN WASTE BOXES

Results by Analytical Methods	Copper	Lead
<b>STLC</b>		
Regulatory Threshold (mg/L)	25	5.0
Mean (mg/L) <sup>1</sup>	35	2.2
Coefficient of Variation	0.97	0.43
<b>TTLC</b>		
Regulatory Threshold (mg/kg)	2500	1000
Mean (mg/kg) <sup>1</sup>	3240	28
Coefficient of Variation	0.33	0.40

<sup>1</sup> Samples which contained nondetectable concentrations were used in calculations as the mean between 0 and the detection limit.

TABLE 2-2. SUMMARY OF COPPER LEVELS IN WASTE PILES

Results by Analytical Methods	Pile #		
	1	2	3
<b>STLC</b>			
Regulatory Threshold (mg/L)	25		
Mean (mg/L) <sup>1</sup>	45	79	31
Coefficient of Variation	0.33	0.91	0.85
<b>TTLIC</b>			
Regulatory Threshold (mg/kg)	2500		
Mean (mg/kg) <sup>1</sup>	2550	3080	2600
Coefficient of Variation	0.14	0.27	0.11

<sup>1</sup> Average of four samples.

TABLE 2-3. SUMMARY OF LEAD LEVELS IN WASTE PILES

Results by Analytical Methods	Pile #		
	1	2	3
<b>STLC</b>			
Regulatory Threshold (mg/L)	5.0		
Mean (mg/L) <sup>1</sup>	3.0	2.0	2.4
Coefficient of Variation	0.23	0.26	0.33
<b>TTLIC</b>			
Regulatory Threshold (mg/kg)	1000		
Mean (mg/kg) <sup>1</sup>	66	58	64
Coefficient of Variation	0.21	0.11	0.05

<sup>1</sup> Average of four samples.



### 3.0 SAMPLING PROGRAM

The sampling design will be of a random grid layout. Piles 1 and 3 will be gridded into equal surface areas by marking a coordinate every 3 ft, both horizontally and laterally. Due to the variation in size between piles, this will result in approximately 130 grids for Pile 1 and approximately 120 grids for Pile 3. Each grid will have a surface area of 9 square ft. Pile 2 will be gridded into equal surface areas by marking a coordinate every 4 ft, both horizontally and laterally. This will result in approximately 125 grids. Each grid will have a surface area of 16 square ft. The grid areas will be numbered consecutively so that sample locations may be referenced.

Six different samples will be collected along with two blind replicates for each pile. Location of the sampling points will be selected for each of the sampling locations from a random number table (see Section 5.2.3).

Trained technicians will be required to collect samples of waste from the piles using the techniques described in Sections 5.0 through 10.0. Composite samples will be collected from each randomly selected grid. Composite sampling will consist of collecting five subsamples from each of two different depths in the randomly selected grid for a total of ten subsamples. The depths will be 0.5 ft from the surface of the pile and approximately 0.5 ft from the pavement. Subsamples will be collected from four corners of the grid in addition to one subsample from the center. The subsamples will then be composited in a tray and mixed using a stainless steel or Teflon spoon. The composited sample will be split and placed into two or three precleaned polyethylene bottles for analysis as follows:

- 500 cc from all sampling points. These samples will be sent to the primary analytical laboratory.
- 1000 cc from all sampling points. These samples will be archived in the event that additional analyses are required.
- 500 cc from 1 out of 10 sampling points. These samples will be sent to a separate analytical laboratory to verify results from the primary laboratory.

This type of sampling versus a single grab sample will provide a better estimate of the mean concentration of the contaminants within the sampling grid and, correspondingly, a better estimate of the mean concentration of the contaminants in the waste pile.

#### 4.0 ANALYSIS PROGRAM

One sample from each pile will be analyzed for the seventeen California Assessment Manual (CAM) metals plus Cr (VI). Total metal concentration is to be compared to California Total Threshold Limit Concentrations (TTLCs) for the seventeen metals plus Cr (VI) using appropriate methods as found in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, 3rd Edition. The remaining samples need be analyzed for only total copper and lead since previous testing has shown these to be the major metals. The waste will be analyzed for soluble metals using the following two methods:

- The Toxicity Characteristic Leaching Procedure (TCLP) will be carried out on 1 out of 5 samples in future sampling programs to ensure the waste is not a RCRA waste. The waste piles which are now undergoing analyses have already been tested by this method.
- Soluble metal concentrations using the California Title 22 Waste Extraction Test (WET), to be compared with the California Soluble Threshold Limit Concentrations (STLCs) standards for these metals.

The total metal analyses (all 17 metals plus Cr(VI)) are conducted first and are conducted to determine major metals for subsequent analysis. A major metal is one whose total concentration is ten times above the STLC for that metal. Then all the remaining samples are analyzed for total metals content for just the major metals. Finally, any sample whose total metal concentration is  $\geq$  ten times its STLC should be analyzed by the WET to determine any STLC exceedances. The approach to analysis described in this paragraph is relatively simple, quick, and cost-effective.

It is important to inform the analytical laboratory to use as large a sample volume as possible for analyses in order to obtain an accurate representation of the metal concentrations in each sample. A minimum of 100 g of sample should be used for the WET and a minimum of 5 g of sample should be used for acid digestion.

The analytical laboratory must meet the following quality control and quality assurance standards:

- The minimum acceptable detection limit is 100 times lower than TTLCs and 10 times lower than STLCs for WET analysis.

- Results from spike analyses must be provided to demonstrate the accuracy and reproducibility of laboratory methods. An error of  $\pm 20\%$  is acceptable.

Also, in future sampling programs we recommend that approximately one out of ten samples be analyzed for total metal concentrations of all 17 CAM metals plus CR(VI). It is not necessary or cost-effective to analyze every sample for all 17 metals. However, a representative fraction of the samples used needs to be completely characterized in order to determine the major metals present.

Additional details on the statistical design of the sampling program are provided in Section 5.0. Sampling equipment and operation, sample collection and preservation, personal protective equipment and decontamination, and quality assurance and quality control are discussed in Sections 6.0 through 10.0.

## **5.0 STATISTICAL DESIGN**

An overview of the sampling effort will be discussed first followed by details on each aspect of the sampling design. The overview is intended to provide a general understanding of how the waste will be sampled. The details which follow the overview will include information on how the number of samples and grid sizes were selected, as well as detailing the method for selection of the grids and the sampling method within a grid.

### **5.1 Overview**

Each waste pile will first be subdivided into either 3 ft by 3 ft grids (Piles 1 and 3) or 4 ft by 4 ft grids (Pile 2). Random sampling will then be used to select six grids for sampling. Within each of these grids, ten samples will be taken and composited, five samples from each of two levels.

The key elements which must be defined for this type of sampling design include: 1) the number of samples; 2) the grids (spatial area) to be sampled; 3) the selection of the grids; 4) the sampling method within a grid; and 5) the estimators used to characterize the population.

### **5.2 Approach**

#### **5.2.1 NUMBER OF SAMPLES PER WASTE PILE**

Factors affecting the number of samples which should be collected are the homogeneity of the contaminant in the waste, the desired confidence interval, and the cost per sample. Based on previous sampling at the site (Tables 2-1 - 2-3), an estimate of the number of samples which would provide statistical confidence in the results may be determined.

In order to provide a basis for the determination of the number of samples to acquire per pile, a table was generated which compares the coefficient of variation of a sample set (standard deviation/mean) versus K, which is a ratio of the mean of the sample set to the regulatory threshold (Table 5-1). In order to generate this table, the sample mean, standard deviation, and sample size are related to determine an upper bound,  $T_U$ , which represents the highest value for the

TABLE 5-1. SAMPLE SIZE<sup>1</sup> REQUIRED TO DEMONSTRATE COMPLIANCE WITH REGULATORY THRESHOLD (RT) AS A FUNCTION OF ANTICIPATED AVERAGE CONTAMINATION LEVEL (X) AND COEFFICIENT OF VARIATION

CV	K = 100X/RT				
	10	30	50	70	90
80% CONFIDENCE LEVEL					
0.1	1	1	1	1	1
0.5	1	1	1	2	15
0.9	1	1	1	4	38
1.3	1	1	2	6	63
1.7	1	1	2	8	87
2.0	1	1	3	9	103
90% CONFIDENCE LEVEL					
0.1	1	1	1	1	2
0.5	1	1	1	3	34
0.9	1	1	3	8	108
1.3	1	2	4	13	147
1.7	1	2	5	18	202
2.0	1	2	6	21	239
95% CONFIDENCE LEVEL					
0.1	1	1	1	1	3
0.5	1	1	2	5	55
0.9	1	2	4	13	145
1.3	1	3	6	22	242
1.7	1	3	8	29	332
2.0	1	4	10	35	393

<sup>1</sup> These sample sizes correspond to a statistical power of 50% at a contamination level  $x$ , and were calculated assuming a lognormal probability distribution for the metal concentrations, along with assumptions that the standard deviation of the measurements is known, and that spatial correlation effects are not important.

concentration that is plausible based on the samples taken. If  $T_U$  is found to be below the regulatory threshold, then it is decided that the true average concentration is also below that threshold. From an environmental point of view, the use of  $T_U$  is probably most defensible because it requires that an area be demonstrated free of contaminants at the regulated levels.

$T_U$  is calculated from the statistical formula shown below:

$$\ln(T_U) = m + g_{1-\alpha} - \frac{\sigma}{\sqrt{n}} + \frac{\sigma^2}{2} \quad (5.1)$$

where  $m$  is the mean of the log-transformed metal concentrations:

$$m = \frac{\sum_{i=1}^n t(x_i)}{n} \quad (5.2)$$

where:  $t(x_i)$  = the log-transformed metal concentrations  
 $n$  = sample number  
 $g_{1-\alpha}$  = the  $(1-\alpha)$  percentile point of the standard normal distribution  
 $\sigma$  = the standard deviation of the log-transformed metal concentrations

The sample sizes shown in Table 5-1 have been generated by assuming an average metal concentration ( $\bar{x}$ ), a standard deviation ( $\sigma$ ), and a desired  $T_U$  to give a range of CVs ( $\sigma/\bar{x}$ ) and Ks ( $100\bar{x}/RT$ ). In order to use Table 5-1, it is necessary to either assume an expected  $\bar{x}$  and CV or a small preliminary sample should be taken to provide an estimate of  $\bar{x}$  and the CV. These values can then be used to select an appropriate sample size. The mean and standard deviation of the sample set may be calculated in the standard method as shown. The mean of a sample set may be calculated as follows:

$$\bar{x} = \frac{\sum_{i=1}^n x_i}{n} \quad (5.3)$$

The standard deviation of the sample set may be calculated as follows:

$$s = \sqrt{\frac{\sum_{i=1}^n (y_i - \bar{y})^2}{n - 1}} \quad (5.4)$$

The coefficient of variation (CV) is simply the ratio of the sample standard deviation to the sample mean:

$$CV = \frac{s}{\bar{x}} \quad (5.5)$$

From Table 5-1, one can see that as the K value increases or the coefficient of variation increases, a greater number of samples are required to demonstrate compliance. In other words, as the expected sample mean approaches the regulatory threshold, it will require many more samples to demonstrate that the actual metal concentration in the waste is below the threshold.

As shown by the preliminary sampling (Tables 2-1 - 2-3), the results demonstrated that most of the waste in the piles contain copper concentrations above the regulatory thresholds for both soluble and total metals content, although a high coefficient of variance was often found with these results. Theoretically, additional sampling of any pile of waste might result in finding the metal concentrations to be below the regulatory limits (although this is not recommended for these particular piles because the soluble copper content is too high); however, one must balance the cost of sampling with the likelihood of being able to dispose of the waste as nonhazardous.

Although the calculations in Table 5-1 show that in some cases one sample would be sufficient to demonstrate compliance, this would be difficult to justify from a regulatory perspective. From a statistical standpoint, a minimum of six samples per waste pile (where a waste pile is equal to 300 yd<sup>3</sup> or less) would provide relatively good confidence in the calculated average metal concentration. The number of samples required if, for example, the average metal concentration is expected to be close to the regulatory threshold and the coefficient of variation is high, can be as high as 390 samples, which would clearly be economically unfeasible. Therefore, it is recommended that six samples per pile be taken to determine the average metal concentration. If waste piles generated in the future are significantly larger than those now in question, sample size should increase proportionally.

### **5.2.2 GRID SIZE**

The grid size selected was based upon the area required to collect the samples and a "rule of thumb" that for a sample of size  $n$ , there should be  $20 \times n$  grids. There are six samples to be taken from each waste pile, therefore, 120 grids would be adequate. This number of grids indicates a grid size of 3 ft by 3 ft would be appropriate for Piles 1 and 3 (generating approximately 130 and 120 grids, respectively), while a grid size of 4 ft by 4 ft would be appropriate for Pile 2 (generating approximately 125 grids).

For sampling of other piles, the following steps may be followed:

- 1) Determine the number of samples to be taken as discussed in the previous section.
- 2) Multiply the number of samples,  $n$ , by 20 to determine the number of grids required per strata.
- 3) Based upon the dimensions of the pile, determine the size of the grids required. For example, to take 5 samples from a waste pile with dimensions of 40 ft by 50 ft would require 100 grids. Selecting a grid size of 4.5 ft by 4.5 ft would yield approximately 100 grids.

### **5.2.3 SELECTION OF GRIDS**

Grid areas should be numbered consecutively. Selection of the grids for sampling will be done randomly. In order to select the grids, use the set of random numbers shown in Table 5-2. Select the first, middle, or last three digits from each five-digit number, but decide which digits will be selected prior to beginning. Choose any number randomly in the table as a starting point. From this number, go down the column, then to the top of the next column on the right, until six numbers have been selected with no repetitions. If a number is selected for which there is no grid, select the next consecutive random number. For example, if we choose to select the middle three digits from the five-digit number and we begin in the seventh column, proceeding down column 7 would give us the numbers 46, 119, 75, 22, 95, and 130. The grids corresponding to these numbers would then be selected for sampling.



TABLE 5-2. RANDOM NUMBERS TABLE<sup>1</sup>

Line/Col.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
1	10480	15011	01536	02011	81647	91646	69179	14194	62590	36207	20969	99570	91291	90700
2	22368	46573	25595	85393	30995	89198	27982	53402	93965	34095	52666	19174	39615	99505
3	24130	48360	22527	97265	76393	64809	15179	24830	49340	32081	30680	19655	83348	58629
4	42167	93093	06243	61680	07856	16376	39440	53537	71341	57004	00849	74917	97758	16379
5	37570	39975	81837	16656	06121	91782	60468	81305	49684	60672	14110	06927	01263	54613
6	77921	06907	11008	42751	27756	53498	18602	70659	90655	15053	21916	81825	44394	42880
7	99562	72905	56420	69994	98872	31016	71194	18738	44013	48840	63213	21069	10634	12952
8	96301	91977	05463	07972	18876	20922	94595	56869	69014	60045	18425	84903	42508	32307
9	89579	14342	63661	10281	17453	18103	57740	84378	25331	12566	58678	44947	05585	56941
10	85475	36857	53342	53988	53060	59533	38867	62300	08158	17983	16439	11458	18593	64952
11	28918	69578	88231	33276	70997	79936	56865	05859	90106	31595	01547	85590	91610	78188
12	63553	40961	48235	03427	49626	69445	18663	72695	52180	20847	12234	90511	33703	90322
13	09429	93969	52636	92737	88974	33488	36320	17617	30015	08272	84115	27156	30613	74952
14	10365	61129	87529	85689	48237	52267	67689	93394	01511	26358	85104	20285	29975	89868
15	07119	97336	71048	08178	77233	13916	47564	81056	97735	85977	29372	74461	28551	90707
16	51085	12765	51821	51259	77452	16308	60756	92144	49442	53900	70960	63990	75801	40719
17	02368	21382	52404	60268	89368	19885	55322	44819	01188	65255	64835	44919	05944	55157
18	01011	54092	33362	94904	31273	04146	18594	29852	71585	85030	51132	01915	92747	64951
19	52162	53916	46369	58586	23216	14513	83149	96736	23495	64350	94738	17752	35156	35749
20	07056	97628	33787	09998	42698	06691	76988	13802	51851	48104	88916	19509	25625	58104
21	48663	91245	85828	14346	09172	30168	90229	04734	59193	22178	30421	61666	99904	32812
22	54164	58492	22421	74103	47070	25306	76468	26384	58151	06646	21524	15227	96909	44592
23	32639	32363	05597	24200	13363	38005	94342	28728	35806	06912	17012	64161	18296	22851
24	29334	27001	87637	87308	58731	00256	45834	15398	46557	41135	10367	07684	36188	18510
25	02488	33062	26834	07351	19731	92420	60952	61280	50001	67658	32586	86679	50720	94953

<sup>1</sup> Ott, L. 1984 An Introduction to Statistical Methods and Data Analysis, Second Edition, Duxbury Press, Boston

#### **5.2.4 SAMPLING METHOD WITHIN A GRID**

Spatial composite sampling will be used to characterize the waste within a grid. Five subsamples will be taken within each grid from the corners of the grid and the center at a depth of 0.5 ft from the surface. An additional five subsamples will be taken in the same manner from a depth of 0.5 ft from the pavement. These ten subsamples will then be composited via mixing in a lined container into a homogenous sample for the various analyses.

## **6.0 SAMPLING EQUIPMENT AND OPERATION**

The following pieces of equipment will be used to perform sampling of the waste placed in roll-off bins, grit piles, and the rinsate water. The two main requirements for the sampling equipment are:

- The tool must not contribute any chemical contamination to the sample, and
- The tool must be capable of collecting a representative sample.

Stainless steel equipment is generally the most durable and is often used for sampling sludge, sediments, and soils. The following paragraphs below discuss the pieces of sampling equipment which are recommended for use in sampling the waste and the rinsate water resulting from decontamination.

### **6.1 Dipper**

A dipper consists of stainless steel, glass, or Teflon beaker constructed with or clamped to the end of a handle (Figure 6-1). Dippers are used for sampling tanks, bins, outfalls, and discharge. The following precautions should be observed:

- A stainless steel dipper should have a riveted handle not a soldered handle, because metals from the solder could leach into and contaminate the sample.
- Use only Teflon, stainless steel, or glass to sample wastes containing organic materials.
- When using a beaker clamped to a pole, the handle and clamp should be painted with a 2-part epoxy or other chemically-inert paint when sampling either alkaline or acidic materials.

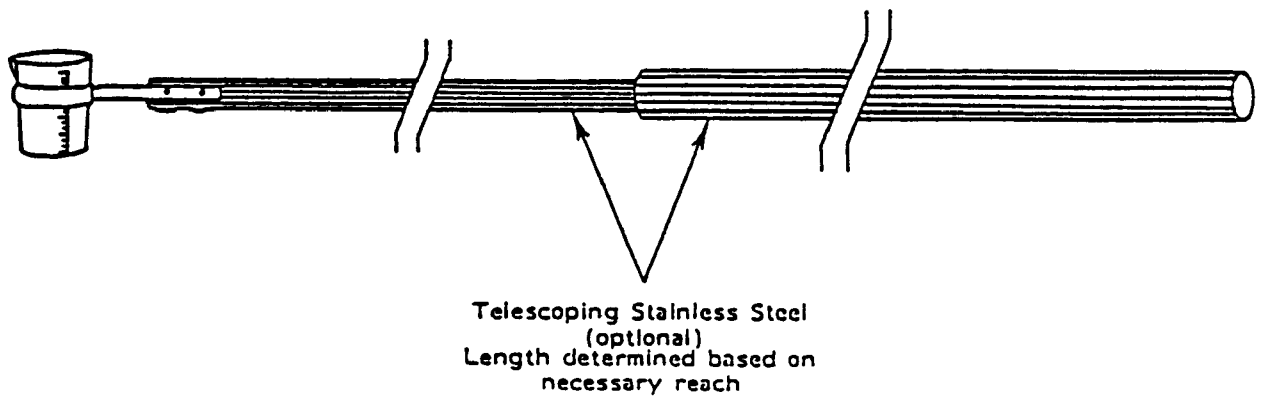


FIGURE 6-1. SCHEMATIC DIAGRAM OF DIPPER

(B-22)

**Procedures for Use:**

1. Decontaminate the dipper, clamp, and handle (see Section 6.2).
2. In tanks, turn the dipper so the mouth of the dipper faces down and insert it into the waste material. Turn dipper right side up when dipper is at desired depth. Allow dipper to fill completely as shown by the cessation of air bubbles. When sampling drums, submerge the dipper to the desired depth, allow the beaker to fill.
3. Raise dipper and pour the sample material into an appropriate container.
4. Decontaminate the dipper.

**6.2 Stainless Steel Spoon or Scoop**

A stainless steel spoon or scoop is the simplest, most direct method for collecting soil samples. In general, the procedure is used to sample the first three inches of surface soil. However, samples from greater depths and samples of sludges, sediments and bulk samples may also employ this technique in some situations.

**Procedures for use:**

1. Collect and composite samples from the top three inches of soil.
2. Mix the samples in a lined container, then deposit in the appropriate container.
3. Wipe sample containers clean of surface contamination.
4. Place in individual plastic bags in an insulated ice chest with freezer packs if refrigeration is necessary.

**6.3 Glass Tube Thief**

A hollow glass tube is a simple tool which is used to sample liquids from drums (Figure 6-2). The advantages of using a glass tube thief include inexpensive cost, ease of disposal, its availability in variable lengths, and capability to sample a vertical column of waste. The tool

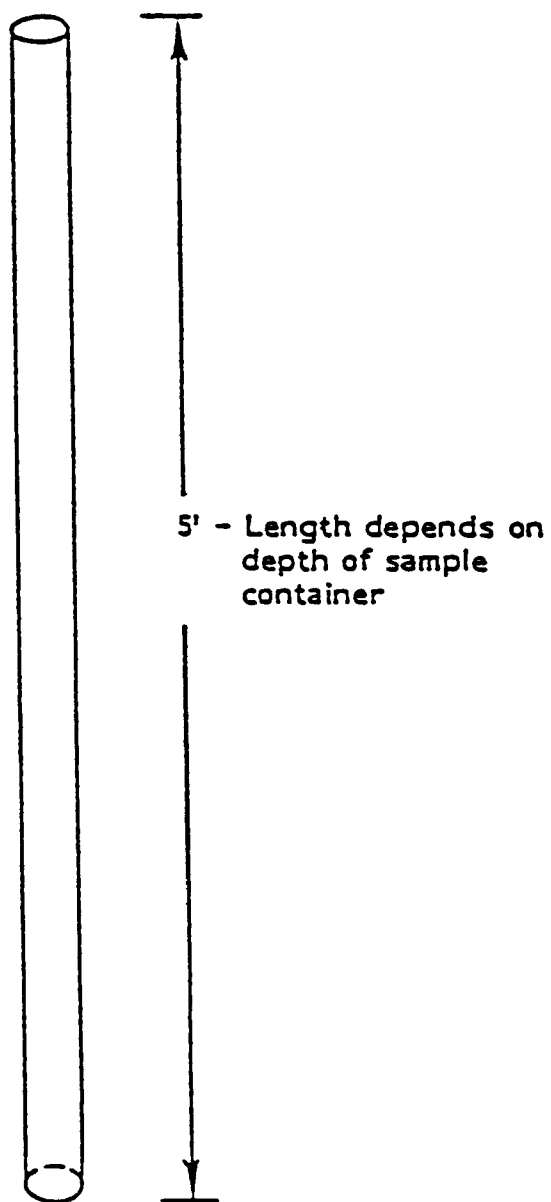


FIGURE 6-2. SCHEMATIC DIAGRAM OF GLASS TUBE THIEF

(B-24)

consists of a glass tube, typically between 8 and 16 mm in diameter. This device will be used to sample the drums containing rinsate from the decontamination of the dipper.

**Procedures for use:**

1. Decontaminate the glass tube (see Section 6.2)
2. Slowly insert the tube into the waste container. This should be done at a rate which permits the level of the liquid inside and outside the sampler to remain the same. If the level of waste in the sampler tube is lower inside than outside, the sampling rate is too fast and may yield a non-representative sample.
3. When the tube contacts the bottom of the waste container, place a rubber stopper or attach a squeeze bulb over the exposed end of the sampling tube. The use of a squeeze bulb improves the ability of a glass tube to retain very viscous fluids during sampling. It is important that none of the fluid comes in contact with the rubber squeeze bulb. If using your thumb, ensure your hands are protected by gloves which are resistant to the chemicals sampled. With the end of the tube plugged, slowly draw the tube from the waste container. In order to enable the sampler to retain the fluid in the glass tube, the glass tube may be withdrawn at an angle such that the thumb may be kept over the end of the glass tube.
4. Place the end of the glass tube in the sample container and remove plug from the end of the tube.
5. Repeat steps 2 through 5 until the required amount of sample has been collected.
6. Place the contaminated glass tube in a plastic storage tube for subsequent cleaning, as described in Section 6.2. If used to sample a drum of waste, the glass tube may be disposed in the drum prior to resealing the bung. Notch the glass with a steel file to avoid shattering the glass when breaking long pieces.

#### **6.4 Auger and Thin-Wall Tube Sampler**

The system consists of an auger bit, a series of drill rods, a "T" handle, and a thin-wall corer (Figure 6-3). The auger bit is used to bore a hole to the desired sampling depth and is then withdrawn. The auger tip is replaced with the tube corer, lowered down the borehole, and forced into the soil at the completion depth. The corer is then withdrawn and the sample collected.

Alternatively, the sample may be recovered directly from the auger. This technique however, does not provide an "undisturbed" sample as would be collected with a thin-tube

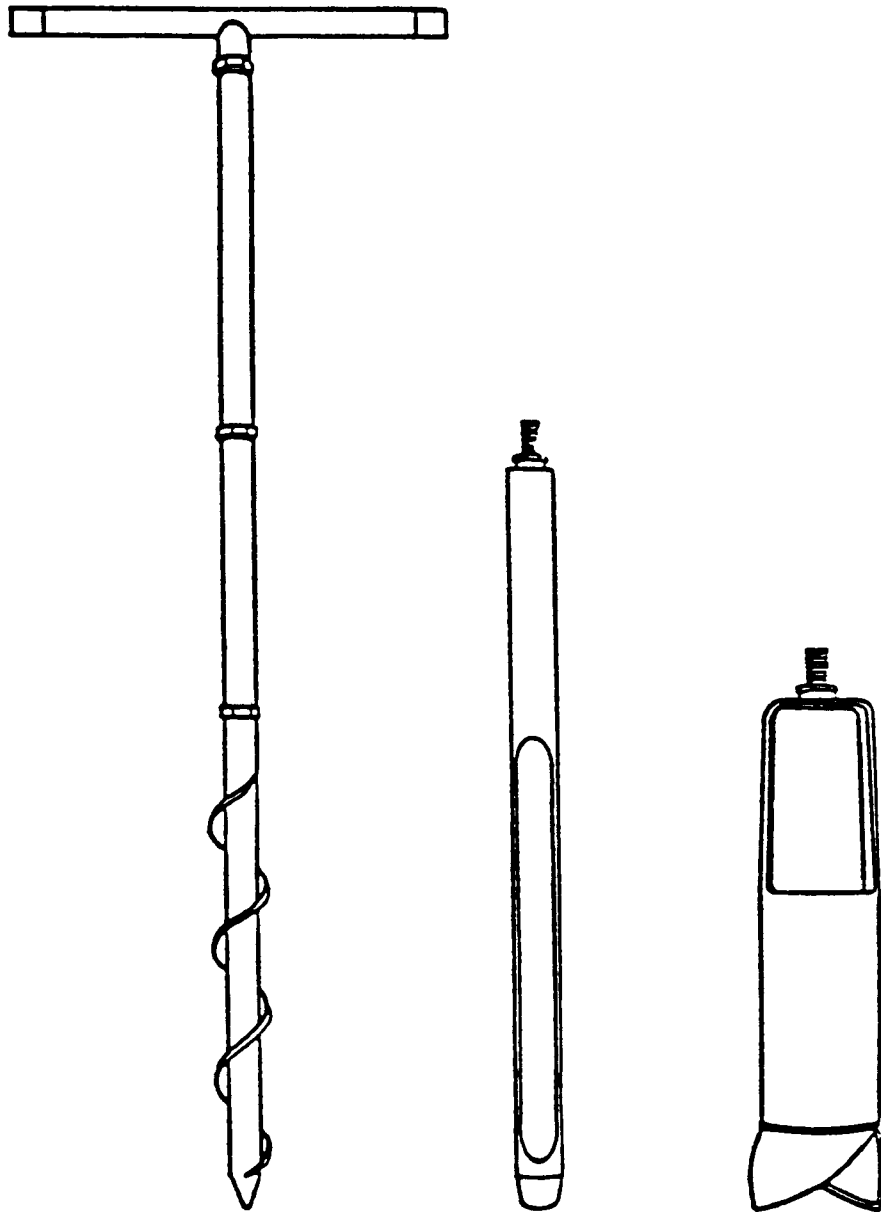


FIGURE 6-3. SCHEMATIC DIAGRAM OF AUGERS AND THIN-WALL TUBE SAMPLER

(B-26)



sampler. When the soil is rocky, it may not be possible to force a thin-tube sampler through the soil or sample recovery may be poor. Sampling directly from the auger may be the only viable method. Several auger types are available: bucket type, continuous-flight (screw), and posthole augers. Bucket types are good for direct sample recovery, are fast, and provide a large volume of sample. When continuous flight (screw) augers are used, the sample may be collected directly off the flights, however, this technique will provide a somewhat unrepresentative sample as the exact sample depth will not be known. The continuous-flight augers are satisfactory for use when a composite of the entire soil column is desired. Posthole augers have limited utility for sample acquisition as they are designed more for their ability to cut through fibrous, heavily rooted, swampy areas. In soils where the borehole will not remain open when the tool is removed, a temporary casing may be used until the desired sampling depth is reached.

**Procedures for use:**

1. Attach the auger bit to a drill rod extension and attach the "T" handle to the drill rod.
2. Clear the area to be sampled of any surface debris (twigs, rocks, litter). It may be advisable to remove the first 8 to 15 cm of surface soil from a 30-cm diameter area around the drilling location.
3. Begin drilling, periodically removing accumulated soils. This prevents accidentally brushing loose material back down the borehole when removing the auger or adding drill rods.
4. After reaching desired depth, slowly and carefully remove auger from boring. (Note: When sampling directly from auger, collect sample after auger is removed from boring and proceed to Step 10).
5. Remove auger top from drill rods and replace with a precleaned thin-wall tube sampler. Install proper cutting tip.
6. Carefully lower corer down borehole. Gradually force corer into soil. Take care to avoid scraping the borehole sides. Do not hammer the drill rods to facilitate coring as the vibrations may cause the boring walls to collapse.
7. Remove corer and unscrew drill rods.
8. Remove cutting tip and remove core from device.

9. Discard top of core (approximately 2.5 cm) which represents material collected by the core before penetrating the layer in question. Place remaining core into sample container.
10. Verify that a Teflon liner is in the cap if required. Secure the cap tightly.
11. Label the sample bottle with the appropriate sample tag. Label the tag carefully and clearly, addressing all the categories or parameters. Complete all chain-of-custody documents and record in the field logbook.

## **7.0 SAMPLE COLLECTION AND PRESERVATION**

### **7.1 Sample Collection**

The following procedures will be followed for sampling waste from waste piles:

1. Identify the specific pile which will be sampled.
2. Construct the sampling grid as described in Section 5.2.3.
3. Go to the random numbers table (Table 5-2) and select six numbers. Each number represents the grid unit which will be sampled.
4. Use the appropriate instrument to obtain the sample. Follow the recommended procedures for use as stated in Section 6.0.
5. Collect a composite sample from each randomly selected grid. Composite sampling will consist of collecting five subsamples at two different depths (0.5 ft from the surface and 0.5 ft from the pavement) from each corner of the randomly selected grid in addition to one sample from the center for a total of 10 subsamples. The samples will then be composited in a tray and mixed using a stainless steel or Teflon spoon. The composited sample will be placed in precleaned polyethylene bottles for analysis.
6. From each sampling point, split the composite sample into a 500 cc subsample for the analytical laboratory and a 1000 cc subsample to archive. From 1 out of 10 sampling points, reserve 500 cc of the composite sample to send to a separate analytical laboratory. No preservation is required for samples. Rinsate blanks must be preserved with a solution of nitric acid. This can be provided in the sample jar by the analytical laboratory. Holding time for the samples is 6 months, unless sampling for mercury which has a holding time of 28 days.
7. The collection of the sample does not require filling the sample jar in any special manner.
8. Discard the outer latex gloves after each sample into an appropriate container and then replace them for the next sampling event.
9. For the rinsate blank (which will be required once for every twenty samples), simply run deionized water over the sampling instrument after it has been decontaminated.

10. The sampler must pay attention while filling the sample bottle for the rinsate blank due to the fact that the sample bottle will have a preservative already in it. If the bottle were to be overfilled during collection, some of the preservative would be lost resulting in insufficient preservative remaining in the bottle and an inaccurate analysis.

## **7.2 Sample Preservation**

No preservatives will be required for the sampling of the waste itself. Only the rinsate blank (equipment washing) will require a preservative of nitric acid in order to lower the pH of the sample below 2. The analytical laboratory can provide the sample containers containing the appropriate quantities of preservative for this. Caution should be exercised when these samples are collected to prevent accidental exposure by splashing.

## **8.0 PERSONAL PROTECTIVE EQUIPMENT AND DECONTAMINATION**

### **8.1 Personal Protective Equipment**

#### **8.1.1 SAMPLING**

The following personal protective equipment shall be worn during the sampling of the waste:

- Tyvek suit
- Latex gloves (two pairs)
- Dust protector
- Safety glasses with splash shields (only necessary for when rinsate blanks (equipment washes) are collected).

#### **8.1.2 CLEANING OPERATIONS (DECONTAMINATION)**

The following personal protective equipment shall be worn during all cleaning operations for sampling equipment:

- Safety glasses with splash shields
- Latex gloves (water decontamination)
- Neoprene or nitrile gloves (when using solvents)
- Tyvek or cloth coveralls

### **8.2 Decontamination**

Decontamination (cleaning) of sampling devices prior to and after use is required. Decontamination is important so that material from a previous sampling event does not contaminate subsequent samples. Decontamination should be performed as follows:

- Scrub the sampling tool with a brush in a laboratory-grade detergent (Alconox, Liquinox, or the equivalent) and tap water solution
- Rinse with water
- Rinse again with deionized water or the equivalent
- If sampling for organic contamination, rinse a final time with pesticide-grade isopropanol or pesticide-grade acetone or methanol (a minimal amount is necessary for rinsing and this should be conducted under a fume hood or in the open, but never in a closed room without adequate ventilation)
- Allow sampling tool to air dry
- Wrap in aluminum foil or other similar protective covering to avoid contamination before the next use
- No eating, smoking, drinking, chewing, or any hand to mouth contact will be permitted during cleaning operations.

The following are cleaning procedures for the glass tube thief:

- Wash thoroughly with laboratory detergent and hot water using a brush to remove any particulate matter or surface film
- Rinse thoroughly with hot tap water
- Rinse with at least a 10 percent nitric acid solution
- Rinse thoroughly with tap water
- Rinse thoroughly with deionized water
- Rinse twice with solvent and allow to air dry for at least 24 hours
- Wrap completely with aluminum foil to prevent contamination during storage

The following are cleaning procedures for stainless steel sampling equipment:

- Wash thoroughly with laboratory detergent and water with a brush
- Rinse thoroughly with tap water
- Rinse thoroughly with deionized water
- If sampling for organic contamination, rinse twice with solvent and allow to air dry

- Wrap completely with aluminum foil
- Rinse with tap water after use in the field

Decontamination wash waters should be collected and containerized separately from solvent rinses in a 55-gallon drum. Since potentially hazardous wastes are being rinsed from sampling equipment, the collected rinse waters should be handled and sampled for hazardous constituents using a glass tube thief prior to disposal. The storage area should have a drum staged for the disposal of rinse waters and one for disposal of solvents. Upon filling the rinse water drum, it should be sampled for metals to determine if it must be disposed of as a hazardous waste or down the industrial drain. The contents of the solvent drum may be recycled.

## **9.0 SAMPLE CUSTODY, LABELING, PACKAGING, AND TRANSPORTATION**

### **9.1 Sample Custody**

The purpose of a sample chain-of-custody is to document the possession of a sample from the time of collection, through all transfers of custody, until it is delivered to the analytical laboratory. This requires that a form (Figure 9-1) be filled out in permanent ink and sent along with the samples to the storage area. This form will contain the following minimum information:

- Sample number
- Date and time of collection
- Shipyard location
- Waste type
- Signature of collector
- Preservation
- Container type
- Analysis request
- Appropriate notations relative to sample integrity and handling practices
- Signature of all persons involved in the chain of possession
- Inclusive dates and times of possession

### **9.2 Sample Labeling**

A sample label is applied to a sample container before a sample of waste is collected (Figure 9-2). The label will be completely filled out with permanent ink. It will contain the following information:





# Battelle

### Columbus Laboratories

### CHAIN OF CUSTODY RECORD

Form No. \_\_\_\_\_

[illegible]

FIGURE 9-1. CHAIN-OF-CUSTODY SHEET

( B-35 )

SAMPLE NO.	_____
SAMPLE MATRIX	_____
SAMPLE PRESERVATIVE	_____
SAMPLING LOCATION	_____
ANALYSIS REQUIRED	_____
INITIALS OF SAMPLER & TIME AND DATE OF COLLECTION	_____

FIGURE 9-2. SAMPLE LABEL

(B-36)

- **Sample number**
- **Date and hour the sample was taken**
- **Sampler's initials**
- **Sampling site**
- **Tests required, if known**
- **Preservative used, if any**

### **9.3 Sample Packaging**

The laboratory will typically provide all sample containers, preservatives, and packaging for transportation of samples. Proper preparation of sample containers for transport to the laboratory is essential to prevent breakage of containers and spillage of potentially hazardous material. The following steps will be taken during sample packaging:

- **Ensure sample container is labeled correctly**
- **After sampling activities are complete, clean the outer surface of all sample containers**
- **Wrap each glass container with plastic insulating material (bubble wrap) and enclose in a plastic bag to prevent contact with other sample containers. Plastic containers also should be placed into bags, however, bubble wrap is not needed.**
- **Place sample containers in thermally-insulated, rigid ice chests which contain ice or reusable ice packs if the temperature must be held at 4°C. If the sample does not need to be held at 4°C, an ice chest is not required. However, an ice chest is a lightweight, rigid, and easily secured container in addition to being thermally efficient.**
- **Ensure the chain-of-custody forms are filled out and secure the inside the sample chests. Packers should retain one copy.**

#### **9.4 Sample Transportation**

Transport samples to the laboratory as soon as possible after collection.

## **10.0 SAMPLE QUALITY ASSURANCE AND QUALITY CONTROL**

### **10.1 Rinsate Blanks (Equipment Washes)**

Equipment washes serve as checks of field decontamination procedures. They are obtained after final wash and decontamination of equipment by pouring reagent-grade water into/through/over a decontaminated piece of sampling equipment. The water is collected in appropriate sample containers and transported to the laboratory with other samples. The equipment blanks are analyzed in the same manner as the field samples. Equipment blanks should be collected prior to each sampling event at each sampling site. However, once good equipment decontamination technique (equipment blanks are "clean") has been demonstrated, the frequency of equipment wash samples may be reduced to an occasional basis. Initially, one rinsate blank (equipment wash) will be collected for every twenty samples taken.

### **10.2 Laboratory Quality Control and Certification**

Laboratory quality control procedures are instituted to ensure the reliability of analytical data obtained throughout the sampling effort. Procedures include the analysis of laboratory samples to measure the accuracy and precision of laboratory procedures. A laboratory duplicate should typically be analyzed one time in twenty samples. Any analytical laboratory used should have current certification from the state of California for performing all the necessary chemical analyses.

#### **10.2.1 MATRIX SPIKE ANALYSIS**

Matrix spike analyses are performed to assist the accuracy of laboratory methods. Spiked samples are used to determine if chemical interferences are occurring. One spike analysis per sample set is generally adequate.

### **10.2.2 MATRIX SPIKE DUPLICATES**

Matrix spike duplicates are used to evaluate the reproducibility of the analytical procedures. A field sample is analyzed and the results are compared to the original matrix spike sample test results. In general, this is only necessary for large numbers of samples (> 30).

### **10.2.3 METHOD BLANK TESTS**

Method blank tests are performed in the laboratory by analyzing distilled, deionized water for all analytical methods employed by the laboratory. Method blanks are analyzed for each matrix to verify that laboratory-induced contaminants are identified and distinguished from environmental contaminants of concern.

**APPENDIX C**  
**GLOSSARY OF SOLIDIFICATION/STABILIZATION TERMS AND ABBREVIATIONS\***

**AA** – atomic absorption spectroscopy, a microcharacterization method.

**ANC** – Acid Neutralization Capacity, a chemical test.

**ANS** – American Nuclear Society.

**ANSI** – American National Standards Institute.

**ANSI/ANS/16.1** – American Nuclear Society test 16.1, a leaching test.

**AOC** – area of contamination.

**APC** – air pollution control.

**API** – American Petroleum Institute.

**ARAR** – applicable or relevant and appropriate requirement. These are cleanup standards, standards of control, and other substantive requirements, criteria, or limitations promulgated under federal, state, or local environmental laws or facility siting laws that: (1. applicable) specifically address a hazardous substance, pollutant, contaminant, remedial action, location, or other circumstance found at a CERCLA site or (2. relevant and appropriate) address problems or situations similar to those encountered at a CERCLA site (40 CFR 300.5, pp. 7 and 12).

**ASTM** – American Society for Testing and Materials.

**absorption** – assimilation of fluids into interstices (ASTM D 653, p. 129).

**acidity** – the quantitative capacity of materials to react with hydroxyl ions.

**additives** – materials included in the binder to improve the S/S process. Examples of some types of additives are: (1) silicates or other materials that alter the rate of hardening, (2) clays or other sorbents to improve retention of water or contaminants, or (3) emulsifiers and surfactants that improve the incorporation of organic compounds.

**adsorption** – attraction of solid, liquid, or gas molecules, ions, or atoms to particle surfaces by physiochemical forces. The adsorbed material may have different properties from those of the material in the pore space at the same temperature and pressure due to altered molecular arrangement (after ASTM D 653 and Parker, 1989, p. 37).

**advection** – unidirectional, progressive bulk movement, such as water under the influence of a hydraulic gradient.

**alkalinity** – the quantitative capacity of aqueous media to react with hydrogen ions.

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\*Acronyms and abbreviations are listed at the beginning of each letter of the alphabet.

**anion** — an ion that is negatively charged.

**asphalt** — a brown, black, hard, brittle, or plastic bituminous material composed principally of hydrocarbons. It is found in nature or can be prepared by pyrolysis of coal tar, certain petroleums, and lignite tar. It melts on heating and is insoluble in water but soluble in gasoline.

**BDAT** — Best Demonstrated Available Technology.

**BNA** — base, neutral, and acid (organic) compounds, a chemical analysis identification.

**bentonite** — a clay formed from volcanic ash decomposition and largely composed of montmorillonite and beidellite. Usually characterized by high swelling on wetting.

**binder** — a cement, cementlike material, or resin (possibly in conjunction with water, extender, or other additives) used to hold particles together.

**bitumen** — naturally occurring or pyrolytically obtained dark or black colored, tarry hydrocarbons consisting almost entirely of carbon and hydrogen, with very little oxygen, nitrogen, or sulfur.

**buffer** — a solution selected or prepared to minimize changes in pH (hydrogen ion concentration). Also known as buffer solution.

**CAA** — Clean Air Act.

**Cal WET** — California Waste Extraction Test, a leaching test.

**CERCLA** — Comprehensive Environmental Response, Compensation, and Liability Act.

**CERCLA hazardous substance** — any substance, pollutant, or contaminant as defined in CERCLA sections 101(14) and 101(33), except where otherwise noted in the Hazard Ranking System (see 40 CFR 302.4).

**CERCLA hazardous wastestream** — any material containing CERCLA hazardous substances that was deposited, stored, disposed, or placed in or that migrated to a site being evaluated by the HRS; any material listed in the NPL.

**CERCLA waste** — a term with no regulatory meaning that is often used as a shortened form of CERCLA hazardous wastestream.

**CFR** — Code of Federal Regulations.

**CLP** — Contract Laboratory Procedures.

**COE** — U.S. Army Corps of Engineers.

**CRN** — Core Research Needs for Containment Systems.

**CSH** — Calcium Silicate Hydrate.

**CWA** — Clean Water Act.



**CWARP** — Coal Waste Artificial Reef Program.

**cation** — a positively charged atom or group of atoms.

**cement** — a mixture of calcium aluminates and silicates made by combining lime and clay while heating.

**characteristic waste** — see *RCRA characteristic waste*

**clay** — fine-grained soil or the fine-grained portion of soil that can be made to exhibit plasticity (putty-like properties) within a range of water contents and that exhibits considerable strength when air-dry.

**colloid** — the phase of a colloidal system made up of particles having dimensions of 1 to 1000 nanometers and which is dispersed in a different phase.

**colloidal system** — an intimate mixture of two substances, one of which, called the dispersed phase (or colloid), is uniformly distributed in a finely divided state through the second substance, called the dispersion medium.

**compressive strength** (unconfined or uniaxial compressive strength) — the load per unit area at which an unconfined cylindrical specimen of soil or rock will fail in a simple compression test. Commonly the failure load is the maximum that the specimen can withstand in the test.

**contaminant** — typically undesirable minor constituent that renders another substance impure.

**corrosiveness characteristic** — exhibiting the hazardous characteristic of corrosivity due to extreme pH or failing under the test conditions defined in 40 CFR 261.22.

**DLT** — Dynamic Leach Test, a leaching test where the specimen is exposed to an actual or simulated flow of the leachant.

**DQO** — Data Quality Objective, a planned quantitative measure of precision, accuracy, and completeness of data.

**DRE** — destruction-removal efficiency. The combined efficiencies of one or more processes intended to reduce the target contaminant(s). The DRE may be expressed as a ratio or percentage.

**density, apparent** (of solids and liquids) — the mass of a unit volume of a material at a specified temperature. Only the volume that is impermeable is considered.

**density, bulk** (of solids) — the mass of a unit volume of the material at a specified temperature.

**diffusion** — movement of molecules towards an equilibrium driven by heat or concentration gradients (mass transfer without bulk fluid flow).

**diffusivity** — diffusion coefficient, the weight of material, in grams, diffusing across an area of 1 square centimeter in 1 second due to a unit concentration gradient.

**dimensional stability** — the ability of the solidified/stabilized waste to retain its shape.

**disposal facility** — a facility or part of a facility at which waste is intentionally placed into or on any land or water, and at which waste will remain after closure.

**durability** — the ability of solidified/stabilized wastes to resist physical wear and chemical attack over time.

**ECN** — Energieonderzoek Centrum Nederland (Netherlands Energy Research Foundation).

**EDXA** — energy dispersive X-ray analysis, a microcharacterization method.

**EE/CA** — Economic Evaluation/Cost Analysis, CERCLA technology screening process for a removal action 40 CFR 300.415.

**ELT** — Equilibrium Leach Test, a leaching test where, under the conditions of the test, an equilibrium between the specimen and the leachant is attained.

**EP Tox** — Extraction Procedure Toxicity Test, a regulatory leaching test used since 1980 to determine if a waste is toxic (40 CFR 261, Appendix II).

**embedment** — the incorporation of waste masses into a solid matrix before disposal.

**emulsifier** — a substance used to produce an emulsion of two liquids which do not naturally mix.

**emulsion** — a colloidal mixture of two immiscible fluids, one being dispersed in the other in the form of fine droplets.

**ettringite** — a mineral composed of hydrous basic calcium and aluminum sulfate. The formula for ettringite is  $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})12 \cdot 26 \text{H}_2\text{O}$ .

**extender** — an additive whose primary function is to increase the total bulk of the S/S-treated waste.

**FGD** — flue gas desulfurization, a pollution abatement process.

**FR** — *Federal Register*.

**FS** — Feasibility Study, a study undertaken to develop and evaluate options for a treatment process.

**FTIR** — Fourier transform infrared spectroscopy, a microcharacterization method.

**FY** — fiscal year.

**fly ash** — the finely divided residue from the combustion of ground or powdered coal and which is transported from the firebox through the boiler by flue gas.

**free water** — water that is free to move through a soil or rock mass under the influence of gravity.

**freeze/thaw cycle** — alternation of a sample temperature to allow determination of weight loss and visual observation of sample disintegration resulting from phase change from water to ice.

**GC/MS** — gas chromatography/mass spectrometry.

**grout** — as used in soil and rock grouting, a material injected into a soil or rock formation to change the physical characteristics of the formation. The term "grout" is not used in this document but is frequently encountered in the S/S industry as a synonym for the term "binder."

**HCB** — hexachlorobenzene.

**HRS** — Hazard Ranking System, the primary mechanism for considering sites for inclusion on the NPL.

**HSL** — Hazardous Substance List, a list of designated CERCLA hazardous substances as presented in 40 CFR 302.4.

**HSWA** — Hazardous and Solid Waste Amendments of 1984.

**hazardous characteristics** — ignitable, corrosive, reactive, and toxic as defined in 40 CFR Part 261.10.

**hazardous waste** — see *RCRA hazardous waste*, *CERCLA hazardous substance*, and *CERCLA hazardous wastestream*.

**heat of hydration** (in S/S reactions) — the heat generated due to the reaction of cementitious or pozzolanic materials with water.

**hydrate** — a compound containing structural water.

**ICP** — inductively coupled plasma atomic emission spectroscopy.

**ignitability characteristic** — exhibiting the hazardous characteristic of ignitability as defined in 40 CFR 261.21.

**immobilization** — the reduction in the ability of contaminants to move through or escape from S/S-treated waste.

**incineration** — a treatment technology involving destruction of waste by controlled burning at high temperatures.

**inhibitor** — a material that stops or slows a chemical reaction from occurring. Used in this document to apply to stopping or slowing of the setting of S/S-treated material.

**interference (S/S)** — an undesirable change in the setting of the S/S material resulting in lower strength, poorer leach resistance, or evolution of noxious or hazardous gases, or other degradation of the S/S-treated material.

**ion** — an atom or molecule which by loss or gain of one or more electrons has acquired a net electric charge.

**interstitial** — see *pore water*.

**kaolin** — a variety of clay containing a high percentage of kaolinite.

**kaolinite** — a common clay mineral having the general formula  $\text{Al}_2(\text{Si}_2\text{O}_5)(\text{OH})_4$ .

**kiln** — a heated and usually rotating enclosure used for drying, burning, or firing materials such as ore or ceramics. In this document "kiln" typically refers to a kiln used for production of lime or cement.

**kiln dust** — fine particulate by-product of cement production or lime calcination.

**LDR** — Land Disposal Restriction.

**LIMB** — Lime Injection Multistage Burner.

**LRT** — Liquid Release Test.

**leachability** — a measure of release of constituents from a waste or solidified/stabilized waste. Leachability is one measure of the mobility of a constituent. High leachability means high constituent mobility.

**leachant** — liquid that comes in contact with a material either from natural exposure (e.g., water in a disposal site) or in a planned test of leachability. The typically used leachants are pure distilled water or water containing salts, acids, or both.

**leachate** — any liquid, including any suspended components in the liquid, that has soaked, percolated through, or drained from material during leaching.

**leaching** — the release of constituents from a solid through contact with the leachant. The leaching may occur by either natural mechanisms at waste sites or as part of a laboratory leaching test.

**leaching agent** — leachant.

**leaching rate** — the amount of a constituent of a specimen or solid waste form which is leached during a unit of time (usually normalized by sample volume, area, or weight).

**leaching resistance** — the inverse of leachability. High leach resistance means low contaminant mobility.

**leaching test** — exposure of a representative sample of contaminated waste, S/S-treated waste, or other material to a leachant under controlled conditions to measure the release of constituents.

**lime** — specifically, calcium oxide (CaO); also loosely, a general term for the various chemical and physical forms of quicklime, hydrated lime, and hydraulic hydrated lime.

**listed waste** — see *RCRA listed waste*.

**long-term stability** — the ability of solidified/stabilized wastes to maintain their properties over time while exposed to the environment.

**MCL** — maximum concentration limit.

**MEP** — Multiple Extraction Procedure, a leaching test in which the sample is repeatedly leached with fresh batches of leachant.

**MSDS** — Material Safety Data Sheet.

**MSW** — municipal solid waste.

**MWEP** — Monofilled Waste Extraction Procedure, a leaching test.

**macroencapsulation** — a process of encasing a mass of solid or S/S-treated waste in a protective layer, such as bitumen (thermoplastic).

**meq** — milliequivalent.

**microencapsulation** — containment of the contaminants on a microscopic or molecular scale.

**microstructure** — the structure of an object or material as revealed by a microscope at a magnification over 10 times.

**mixer** — machine employed for blending the constituents of grout, mortar, or other mixtures.

**modified clays** — clays (such as bentonite) that have been modified by ion exchange with selected organic compounds that have a positive charged site (often a quarternary amine), hence rendering the clay/organo complex hydrophobic.

**monolith** — a free standing solid consisting of one piece.

**monomer** — a simple molecule which is capable of combining with a number of like or unlike molecules to form a polymer.

**montmorillonite** — a group of clay minerals characterized by a weakly bonded sheet-like internal molecular structure; consisting of extremely finely divided hydrous aluminum or magnesium silicates that swell on wetting, shrink on drying, and have ion exchange capacity.

**multimedia** — air, land, and water.

**NAAQS** — National Ambient Air Quality Standards.

**NCP** — National Oil and Hazardous Substances Contingency Plan, provides the organizational structure and procedures for preparing and responding to

discharges of oil and releases of hazardous substances, pollutants, and contaminants (40 CFR 300.1).

**NESHAP** — National Emission Standards for Hazardous Air Pollutants.

**NMR** — nuclear magnetic resonance spectroscopy, a microcharacterization method.

**NPL** — National Priorities List, list of CERCLA sites (40 CFR Part 300 Appendix B).

**NRC** — U.S. Nuclear Regulatory Commission.

**NYSC-HWM** — New York State Center for Hazardous Waste Management.

**OAQPS** — Office of Air Quality Planning and Standards (of the U.S. EPA).

**OSHA** — Occupational Safety and Health Act; Occupational Safety and Health Administration.

**PAH** — polynuclear aromatic hydrocarbon.

**PCB** — polychlorinated biphenyl.

**PFT** — Paint Filter Test, a physical characterization test.

**ppb** — part per billion.

**ppm** — part per million.

**PRP** — potentially responsible party, potentially liable for the contamination and cleanup of CERCLA sites.

**percolation** — movement of water under hydrostatic pressure or gravity through the smaller interstices of rock, soil, wastes, or S/S-treated wastes.

**performance criterion** — a measurable performance standard set for an individual property or parameter.

**performance indicator** — an easy-to-measure property or parameter selected to characterize the S/S process or S/S-treated waste.

**permeability** — a measure of flow of a fluid through the tortuous pore structure of the waste or S/S-treated waste. It is expressed as the proportionality constant between flow velocity and the hydraulic gradient. It is a function of both media. If the permeating fluid is water, the permeability is termed as hydraulic conductivity.

**phase (of a material)** — a region of a material that is physically distinct and is homogeneous in composition and morphology.

**polymer** — a chemical with repetitive structure formed by the chemical linking of single molecules (monomers).

**pore** — a small cavity or void in a solid.

**pore size distribution** — variations in pore sizes in solids; each material has its own typical pore size distribution and related permeability.

**pore water** — water contained in voids in the solid material.

**porosity** — the ratio of the aggregate volume of voids or interstices to the total volume of the medium.

**Portland cement** — a hydraulic cement produced by pulverizing clinker consisting essentially of hydraulic calcium silicates, usually containing one or more of the forms of calcium sulfate.

**pozzolan** — a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds with cementitious properties. The term is derived from an early source of natural pozzolanic material, Pozzuoli, Italy.

**QA/QC** — Quality Assurance/Quality Control.

**QAPjP** — Quality Assurance Project Plan.

**QAPP** — Quality Assurance Program Plan.

**3Rs** — recovery, reuse, and recycle.

**RCRA** — Resource Conservation and Recovery Act.

**RCRA characteristic waste** — any solid waste exhibiting a characteristic of ignitability, corrosivity, reactivity or toxicity, as defined in 40 CFR 261, Subpart C.

**RCRA hazardous waste** — any RCRA solid waste, as defined by 40 CFR 261.3, that is not excluded from regulation under 40 CFR 261.4 and that meets any one of the characteristic or listing criteria (including mixtures) described in 40 CFR 261.3(a)(2). For more detail, see 40 CFR 260, Appendix I.

**RCRA listed waste** — any solid waste listed in 40 CFR 261, Subpart D; or a mixture that contains a solid waste listed in 40 CFR 261, Subpart D that has not been excluded under the provisions of 40 CFR 261.3 in accordance with 40 CFR 260.20 or 40 CFR 260.22.

**RCRA solid waste** — any garbage, refuse, or sludge; or any solid, liquid, semi-solid or contained gaseous material that is: (1) discarded, (2) no longer to be used for its original purpose, or (3) a manufacturing or mining by-product and is not excluded by the provisions of 40 CFR 261.4(a). For more detail, see 40 CFR 260, Appendix I. Also note that the definition of solid waste includes materials that are not "solids" in the normal sense of the word.

**RI** — Remedial Investigation, a process undertaken by the lead agency to determine the nature and extent of the problem presented by a CERCLA site (40 CFR 300.430(d)).

**RI/FS** — Remedial Investigation/Feasibility Study, see RI or FS.

**ROD** — Record of Decision, a document prepared to explain and define the final remedy selected for a CERCLA site (40 CFR 300.430 (f)(4)(i)).

**RP** — Responsible Party, persons or corporate entities found to be responsible for contamination and cleanup at a CERCLA site.

**RPM** — Remedial Project Manager, the official designated by the lead agency to coordinate, monitor, or direct remedial or other response actions under subpart E of the NCP (40 CFR 300.5).

**RREL** — Risk Reduction Engineering Laboratory (of the U.S. EPA).

**reactivity characteristic** — exhibiting the hazardous characteristic of reactivity as defined in 40 CFR 261.23.

**redox** — abbreviation for oxidation-reduction, now accepted as a word.

**residual liquid** — free liquid remaining in the S/S-treated waste after treatment.

**SARA** — Superfund Amendments and Reauthorization Act.

**SDWA** — Safe Drinking Water Act.

**SCE** — sequential chemical extraction, a leaching test with a variety of aqueous chemicals used sequentially to characterize the contaminant bonding.

**SEM** — scanning electron microscopy, a microcharacterization method.

**SET** — Sequential Extraction Test, a leaching test with a series of sequential acid extractions used to determine the sample buffering capacity.

**SITE** — Superfund Innovative Technology Evaluation.

**SRS** — Separation and Recovery Systems, Inc.

**S/S** — solidification/stabilization, used in this document to encompass the variety of processes that may contribute to increased physical strength and/or contaminant immobilization.

**S/S-treated waste** — a waste liquid, solution, slurry, sludge, or powder that has been converted to a stable solid (granular or monolithic) by an S/S treatment process.

**STLC** — Soluble Threshold Limit Concentration, limit applied to Cal WET leaching results (Ca 22 California Code of Regulations 66699).

**silica fume** — very fine silica dust produced by condensation of silica fumes.

**sludge** — in this document, sludge means a viscous semi-solid or fluid containing contaminants requiring treatment. The regulatory definition is any solid, semi-solid, or liquid waste generated from a municipal, commercial, or industrial wastewater treatment plant, water supply treatment plant, or air pollution control facility with the exception of specific exclusions such as the treated effluent from a wastewater treatment plant (40 CFR 260.10).



**solid waste** — see *RCRA solid waste*.

**solidification** — a process in which materials are added to the waste to convert it to a solid or to simply improve its handling and physical properties. The process may or may not involve a chemical bonding between the waste, its contaminants, and the binder. In solidification, the mechanical binding of contaminants can be on the microscale (microencapsulation, absorption, or adsorption) or the macroscale (macroencapsulation).

**solubility** — the maximum concentration of a substance dissolved in a solvent at a given temperature.

**solubility product** — a type of simplified equilibrium constant defined for and useful for equilibria between solids and their respective ions in solution.

**solution** — a single, homogeneous phase of liquid, solid, or gas in which a solute is uniformly distributed.

**sorption** — a general term used to encompass the processes of adsorption, absorption, desorption, ion exchange, ion exclusion, ion retardation, chemisorption, and dialysis.

**stability** — the stabilization and solidification provided by an S/S process.

**stabilization** — a process by which a waste is converted to a more chemically stable form. The term may include solidification, but also includes chemical changes to reduce contaminant mobility.

**storage** — the holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere (40 CFR 260.10).

**surfactant** — surface-active agent, a soluble compound that reduces the surface tension of liquids, or reduces interfacial tension between two liquids or a liquid and a solid.

**TCE** — trichloroethylene.

**TCLP** — Toxicity Characteristic Leaching Procedure, the primary leach testing procedure required by 40 CFR 261.24 and the most commonly used test for degree of immobilization offered by an S/S process.

**TDS** — total dissolved solids.

**TOC** — total organic carbon, a chemical analysis.

**TRD** — Technical Resources Document.

**TSCA** — Toxic Substances Control Act.

**TSD** — treatment, storage, and disposal facility (RCRA).

**TTLC** — Total Threshold Limit Concentration, limit applied to Cal WET leaching results (Ca 22 California Code of Regulations 66699).

**TWA** — Total Waste Analysis, total concentration of priority pollutants, organics, and metals in the waste

**technology screening** — the logistic of technology selection, evaluation, and optimization. A treatment technology properly screened prior to full-scale implementation has the highest probability of success in the field.

**thermoplastic resin** — an organic polymer with a linear macromolecular structure that will repeatedly soften when heated and harden when cooled; for example styrenes, acrylics, cellulose, polyethylenes, vinyls, nylons, and fluorocarbons.

**thermosetting resin** — an organic polymer that solidifies when first heated under pressure, and which cannot be remelted or remolded without destroying its original characteristics; for example epoxies, melamines, phenolics, and ureas.

**tortuosity** — the ratio of the length of a sinuous pathway between two points and the length of a straight line between the points.

**toxicity characteristic** — exhibiting the hazardous characteristic of toxicity as defined in 40 CFR 261.24.

**transportation** — the movement of hazardous waste by air, rail, highway, or water (40 CFR 260.10).

**treatability study** — a study in which hazardous waste is subjected to a treatment process to determine: (1) whether the waste is amenable to the treatment process, (2) what pretreatment (if any) is required, (3) the optimal process conditions needed to achieve the desired treatment, (4) the efficiency of a treatment process for a specific waste or wastes, or (5) the characteristics and volumes of residuals from a particular treatment process (40 CFR 260.10).

**treatment** — any method, technique, or process, including neutralization, designed to change the physical, chemical, or biological character or composition of any hazardous waste so as to neutralize such waste, or so as to recover energy or material resources from the waste, or so as to render such waste non-hazardous, or less hazardous; safer to transport, store, or dispose of; or amenable for recovery, amenable for storage, or reduced in volume (40 CFR 260.10).

**triaxial compression** — compression caused by the application of normal stress in lateral directions (ASTM D 653, p. 152).

**triaxial shear test (triaxial compression test)** — a test in which a cylindrical specimen encased in an impervious membrane is subjected to a confining pressure and then loaded axially to failure.

**UCS** — unconfined compressive strength, the load per unit area at which an unconfined cube or cylindrical specimen of material will fail in a simple compression test without lateral support.

**U.S. DOE** — United States Department of Energy.

**J.S. DOT** — United States Department of Transportation.

**U.S. EPA** — United States Environmental Protection Agency.

**VOC** — volatile organic compound, an organic compound with a low boiling point.

**WDW** — wet/dry weathering.

**WET** — see Cal WET, a leaching test.

**WTC** — Wastewater Technology Centre, formerly of Environment Canada.

**wet/dry cycle** — alternation of soaking and drying a sample to allow determination of material loss and visual observation of sample disintegration resulting from repeated soaking and drying cycles.