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Feasibility Study of Sorbent Treatment for Hazardous Wastes

Final Technical Report Phase I and Phase II

June 6, 1996

Task No. N.063
CDRL No. A006
Contract No. DAAA21-93-C-0046

NDCEE

*National Defense Center
for Environmental Excellence*

*Prepared by
National Defense Center for Environmental Excellence*

Operated by Concurrent Technologies Corporation

**Feasibility Study of Sorbent Treatment
For Hazardous Wastes**

Final Technical Report - Phase I and Phase II

June 6, 1996

Requests for this document shall be referred to:

Commander, U.S. Army Armament Research
Development & Engineering Center
ATTN: AMSTA-AR-ET (Mr. M. Wrazen)
Building 172
Picatinny Arsenal, NJ 07806-5000

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Submitted by

Concurrent Technologies Corporation
1450 Scalp Avenue
Johnstown, PA 15904

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1.0 EXECUTIVE SUMMARY

In 1995, Concurrent Technologies Corporation (*CTC*), under the National Defense Center for Environmental Excellence (NDCEE) Program, was tasked by the Defense Logistics Agency (DLA) to evaluate the potential use of sorbent materials for treating hazardous wastes. The results of *CTC*'s bench scale testing of several classes of nonbiodegradable sorbents were presented in three reports: Phase II Technical Report (August 29, 1995); Phase II Follow-up Report (December 15, 1995); and Phase II Report: Addendum, Cellulosic Sorbent Testing (January 15, 1996). These reports, as well as a number of related reports and correspondence prepared by *CTC* as part of the task, also discussed the selection of the candidate sorbents and the rationale for the testing procedures. This report summarizes all of this prior information into a brief, concise document.

To be permitted for land disposal, the sorbed wastes tested in this study would have to meet three requirements:

- Pass the Paint Filter Test
- Meet the Land Disposal Restrictions (LDR) standards for the chemical constituents
- Eliminate flammability or corrosivity of the waste, if applicable.

At the given test conditions and hazardous constituent concentrations, the test results showed that only one waste/sorbent combination — aqueous heavy metals and clay sorbent — passed all three tests for all chemical constituents (Table 1). This test combination met the LDR standards with a significant margin of safety, eliminated the corrosivity, and allowed no free liquids to pass.

Another waste/sorbent combination, aqueous heavy metals waste treated with zeolitic sorbent, also passed two of the screening criteria and partially passed the third, i.e., it was able to stabilize one of the five heavy metals (lead).

For the paint related and degreasing solvent wastes, none of the sorbed wastes would be able to be land disposed. None of the LDR standards were met and none of the associated hazardous characteristics of the wastes were eliminated.

Based on the positive results for the clay and zeolitic sorbents, it is recommended that an economic analysis (Phase III task) be performed for the use of clay and zeolitic sorbents in treating aqueous heavy metals waste. The results of the economic analysis will be used to justify whether or not a full-scale treatment study should be conducted for either the clay or the zeolitic sorbent.

Table 1. Summary of Sorbent Test Results

Sorbent Class	Waste Stream											
	Paint Related Waste					Aqueous Heavy Metal Waste ¹					Degreasing Solvent Waste	
	Free Liquids	LDR Stds.	Haz. Prop ²	Free Liquids	LDR Stds.	Haz. Prop ³	Free Liquids	LDR Stds.	Haz. Prop ³	Free Liquids	LDR Stds.	Haz. Prop ³
Peat (non-biodegradable)	Pass	Fail	Fail	Pass	1 Pass (Pb)	Fail	Pass	1 Pass (Pb)	Fail	Pass	Fail	N/A
Polymeric (non-encapsulating)	Fail	NT	NT	Pass	5 Fail	Fail	Pass	5 Fail	Fail	Fail	NT	N/A
Polymeric (encapsulating)	Fail	NT	NT	Pass	3 Pass (Cd, Cr, Pb)	Fail	Pass	3 Pass (Cd, Cr, Pb)	Fail	Pass	Fail	N/A
Zeolitic (Original Test)	Pass	Fail	Fail	Pass	1 Pass (Pb)	Pass	Pass	1 Pass (Pb)	Pass	Pass	Fail	N/A
(Re-Testing)	Pass	Fail	Fail	Pass	1 Pass (Pb)	Pass	Pass	1 Pass (Pb)	Pass	Pass	Fail	N/A
Clay	Pass	Fail	Fail	Pass	5 Pass	Pass	Pass	5 Pass	Pass	Pass	Fail	N/A
Cellulosic	Pass	Fail	Fail	Pass	5 Fail	Fail	Pass	5 Fail	Fail	Pass	Fail	N/A

Recommended for Phase III, Economic Analysis

¹ Five metals tested were Cd, Cr, Pb, Ni, and Zn.

² Flammability test

³ Corrosivity test

NT = not tested because failed Paint Filter Test

N/A = no hazardous properties associated with this waste

2.0 BACKGROUND

Sorbents, for the purpose of this task, are defined as solid materials that take up and hold liquid by means of absorption, adsorption, or both. Sorbents have been extensively studied and used to absorb free liquids from spills. As such, sorbents are commonplace at industrial facilities, laboratories, and other operations where oil, fuel, and chemical spills may occur. Occasionally, sorbents are also used for waste treatment before land disposal. In the latter case, however, only a limited number of studies have been conducted to determine the feasibility of using sorbents for nonspill applications.

In 1995, Concurrent Technologies Corporation (*CTC*), under the NDCEE Program, was tasked by the Defense Logistics Agency (*DLA*) to evaluate the potential use of sorbent materials for treating hazardous wastes. The Statement of Work for the task was comprised of four distinct phases. Phase I consisted of identifying waste streams and classes of sorbents for testing, and developing a test plan for the bench scale studies. Five deliverables were prepared by *CTC* as part of the Phase I investigation: a Candidate Waste Streams Report, Technical Assessment Report, Safety Plan, Test Plan, and QA/QC Plan. Phase II was the bench-scale laboratory investigation of waste/sorbent combinations. Table 2 briefly describes the Phase I and Phase II deliverables. Planned activities are the Phase III economic analysis and the Phase IV on-site demonstration testing.

During Phase I of this sorbent task, performance criteria were identified to assist in evaluating and comparing candidate sorbents. These criteria are listed in Table 3. Of the thirteen performance criteria identified, eight criteria were fully addressed by data obtained during the technology survey. The remaining five criteria were to be addressed wholly or in part by data generated during the Phase II laboratory bench-scale testing at *CTC*. These five criteria were:

- Suitability for immobilizing free liquids
- Suitability for eliminating characteristically hazardous properties or constituents
- Sorbent/sorbate material compatibility
- Waste volume reduction
- Sorbent economics.

Table 2. Sorbent Task Deliverables

	Deliverable	Date	Focus of Report
Phase I	Candidate Waste Streams Report	May 23, 1995	Select waste streams for Phase II bench-scale testing
	Technical Assessment Report (included regulatory assessment)	May 24, 1995	Select sorbent materials for testing using information from literature searches and surveys
	Safety Plan	June 19, 1995	Provide protection for <i>CTC</i> employees from potential hazards during bench-scale testing
	Test Plan	June 27, 1995	Provide methods and procedures for bench-scale testing
	QA/QC Plan	June 28, 1995	Provide an approach for implementing the quality requirements of the bench-scale testing
Phase II	Phase II Technical Report	August 29, 1995	Present results of <i>CTC</i> 's bench-scale testing of 5 sorbent classes and 3 waste streams
	Phase II Follow-up Report	December 15, 1995	Present results of <i>CTC</i> 's re-testing of a zeolitic sorbent and 3 waste streams
	Phase II Report: Addendum, Cellulosic Sorbent Testing	January 15, 1996	Present results of <i>CTC</i> 's bench-scale testing of a cellulosic sorbent and 3 waste streams

1 2 3 4 5 6 7 8 9 10 11 12

Table 3. Performance Criteria and Means of Evaluation

Performance Criteria *	Means of Evaluation
I. SUITABILITY FOR LANDFILLING	
A. Long-term stability/ nonbiodegradability	Vendor data
B. Immobilization of free liquids	CTC laboratory data
C. Immobilization of hazardous constituents	CTC laboratory data
II. PRODUCT QUALITY, PERFORMANCE, AND ECONOMICS	
A. Sorbent/sorbate compatibility	CTC laboratory data
B. Development risk	Technology survey information
C. Vendor quality assurance	Vendor data
D. Product availability	Technology survey information
E. Waste volume reduction	CTC laboratory data
F. Sorption selectivity and efficiency	Vendor data
G. Sorbent economics	Vendor data CTC laboratory data Economic analysis
III. OPERATIONAL ISSUES	
A. Human health data	Vendor data Technology survey information
B. Worker safety impact	Vendor data
C. Ease of handling	Vendor data

* Boldface items comprise the data objectives for the CTC bench-scale testing.

Phase II Testing

The general objective of Phase II was to conduct bench-scale testing of sorbent materials and waste streams to quantify the effectiveness of sorbent materials for treating hazardous wastes. A technology report discussing the results of the waste/sorbent testing was issued August 29, 1995.

A specific objective of the Phase II testing was to generate laboratory data to address the five criteria highlighted in Table 3 for the candidate waste streams and sorbents. The Phase II laboratory study was designed also to provide data to support the Phase III economic assessment.

Zeolitic Sorbent Re-Testing

The Phase II Technical Report issued on August 29, 1995 presented the results of the lab scale tests which were performed on the five sorbents and the three waste streams. After reviewing this document, the DLA asked *CTC* to make this report available to the six sorbent suppliers for their comments on the test plan and the reported results.

On August 31, 1995, *CTC* sent the Phase II Technical Report to the six sorbent vendors whose product(s) were tested. Follow-up telephone calls to the vendors indicated that five of the vendors were satisfied with the reported results and had no concerns with the testing program. However, one of the vendors offered some suggestions for improving the effectiveness of the zeolitic sorbent and asked to have their zeolitic sorbent re-tested. The vendor suggested three changes to the testing protocol: (1) different waste/sorbent mixing ratios, (2) the addition of water following the sorbent addition, (3) a seven day cure time before subsequent testing.

Acting on instructions from the DLA, *CTC* agreed to re-test the zeolitic sorbent under the new directions provided by the vendor and asked that their principal investigator be present to monitor this re-test. These arrangements were made and the re-test occurred in November 1995. Results were reported in the "Phase II Follow-up Report" dated December 15, 1995.

Cellulosic Sorbent Testing

When *CTC* was completing its first round of sorbent bench-scale testing, the U.S. Environmental Protection Agency published a notice in the Federal Register (July 11, 1995) modifying the definition of "biodegradable sorbent." Under this new definition, some sorbents that were previously "biodegradable" and, therefore, not tested earlier by *CTC*, might be considered "nonbiodegradable." In response to this change, the DLA published a notice in the Commerce Business Daily (CBD) on October 12, 1995 asking for manufacturers of sorbents that meet the new definition to submit information on their products. *CTC* was to select the best candidate(s) for bench-scale testing using the same criteria as before. *CTC* received 16 responses to the CBD announcement. Of these 16 responses, only 8 were relevant to the DLA request and, of these, only one sorbent was recommended for testing at the bench-scale. The others, which were not considered relevant to this requirement, included liquid products, foam products, flue gas treatment products, material storage cabinets, workstations, drums, containers, pallets, and containment systems. The one sorbent that was recommended for testing at the bench scale was a cellulose based material. Two other products met the primary screening requirements, but were not recommended for testing because the two classes of sorbents (peat moss and polymers) had already been tested. Testing of the cellulosic sorbent occurred in December 1995, and the results were reported in the "Phase II Report: Addendum, Cellulosic Sorbent Testing" (January 15, 1996).

3.0 SUMMARY OF PHASE I AND PHASE II ACTIVITIES

This section summarizes the objectives, significant results, and conclusions from the key subtasks performed by CTC from May 1995, through January 1996. In addition, the evaluation criteria and rationale used to select sorbent materials and waste streams are also summarized. The five activities were:

1. Selection of candidate waste streams
2. Selection of sorbent materials
3. Regulatory assessment
4. Bench-scale test planning
5. Waste stream/sorbent testing.

3.1 Selection of Candidate Waste Streams

Investigation of those DOD hazardous waste streams indicated by DLA to be of highest priority for waste disposal cost reduction, and potentially suited to sorbent treatment was conducted to complete the first task deliverable, the "Candidate Waste Stream Report" (issued May 23, 1995). The objective was to identify a manageable number of waste streams for laboratory testing against various sorbents, considering regulatory issues and covering a broad range of possible waste types.

The process of selecting the candidate wastes was comprised of two steps: screening of EPA waste codes; and selection of actual waste streams representative of those waste codes. The screening process started with the initial consideration of the entire group of EPA listed wastes, i.e., those wastes designated as U, P, F, K, and D. The wastes in this list were then screened to eliminate those wastes that appeared to be ill-suited to the bench-scale sorbent testing, and to maximize the amount of information obtained from a single test. The following six criteria were used in the screening process:

1. Wastes were omitted from consideration if they represented a potential health or safety danger to CTC laboratory personnel. For example, because many of the P- and F-listed wastes are comprised of highly dangerous materials such as cyanides and dioxins, the entire P-list, as well as all F-listed wastes above F019, were dropped from consideration. D003, F007, F008, F009, F010, F011, F012, K044-047, and D018 wastes were eliminated for the same health and safety reasons.

2. Wastes produced in very low quantities were dropped from consideration. To determine the quantity of waste produced, annual waste generation data from the Defense Reutilization and Marketing Service (DRMS) were reviewed and analyzed. The U-listed wastes, which are primarily virgin or off-specification materials, represented a very low waste volume category, and therefore were dropped from further consideration. In addition, DRMS data showed relatively low volumes of D-listed waste above D008, so all but D001 through D008 were immediately dropped from further consideration. D004 (arsenic) was also a low volume waste, and thus excluded.
3. Wastes specific to certain industries and not common to the DOD were dropped from consideration. For example, because the K-listed wastes are industry-specific and not very common to the DOD, all K waste were dropped from further consideration except the K waste associated with steel manufacturing: K062. The F004 waste code was dropped for the same reason.
4. Sludge-type wastes, namely F006 and F019, were dropped from consideration based on the engineering judgment that sorbent treatment of such high-solids waste would be impractical. Although treatment would successfully sorb any free liquids in such waste, it was believed the sorbent material would not adequately stabilize the heavy metals contained within the solids portion of the waste.
5. Wastes for which a particular non-sorbent technology was the preferred treatment, or the only allowable treatment, were dropped from consideration. For example, the EPA has ruled that, under the Land Disposal Restrictions, "recovery of organics" is the only allowable treatment technology for D001 wastes having greater than 10 percent Total Organic Carbon (TOC). Because the DRMS data showed that almost all of their D001 waste was greater than 10 percent TOC, the flammable liquid category of D001 waste was dropped from further consideration. D001 oxidizers were also dropped because the Land Disposal Restrictions require specific treatment technologies.
6. Wastes for which the constituent chemicals were listed as hazardous under more than one waste code were screened to avoid potential duplication during testing. For example,

trichloroethylene is listed as a D040 waste and also as a listed solvent in F001 and F002. There was no need to test all three types of wastes. In addition, the F001 and F002 wastes represent a broader mixture of chemical constituents than D040. Therefore, the D040 waste was excluded. Because the F001 and F002 waste codes represent exactly the same chemicals, the F002 was dropped and only F001 was carried through for further evaluation.

This same criterion also was applied in the exclusion of other D listed chemicals similar in property to solvents listed in the F001-F005 codes.

The result of the six-step screening process was a list of ten waste codes for consideration: F001, F003, F005, F006, F019, K062, D002, D006, D007, and D008.

Because the purpose of the Phase II testing was to evaluate the potential applicability of sorbents in treating "real" waste streams (i.e., waste characteristic of a particular manufacturing or maintenance process), CTC and DLA agreed that two additional factors should be considered in selecting the waste streams:

1. The number and type of waste streams should be optimized to obtain the most data from a given series of laboratory tests. This would be accomplished by combining waste codes of like character into one group. For example, F003 and F005 waste codes are very similar in character; therefore, these two waste codes were grouped together.
2. The selected waste streams should be as representative as possible of actual waste streams observed in DOD industrial operations, i.e. either collected from actual production lines or made in the laboratory to simulate real wastes as closely as possible. The heavy metals wastes (D006-08) and acids (D002) are commonly found together in pickling wastes (K062) and plating wastes (F006, F019). Therefore, all of these waste codes were grouped together.

The result of incorporating these final two decision factors was the selection of three wastes for laboratory testing: paint-related waste; degreasing solvent waste; and aqueous, heavy metals waste (Table 4). The primary constituents of the three wastes, as shown in the table, were monitored throughout the testing. Waste codes directly applicable to the waste are also shown. One advantage of using the selected wastes is that a number of waste codes also indirectly apply. Therefore, by using these three waste streams, the performance of the various sorbents can be

reasonably extrapolated to dozens of waste codes that are indirectly related to the wastes.

Table 4. Wastes Selected for Phase II Bench-Scale Testing

Waste Stream			
Parameter	Paint-Related	Aqueous Heavy Metals	Degreasing Solvent
Primary Constituents	methyl ethyl ketone 4-methyl-2-pentanone toluene xylenes ethylbenzene	cadmium chromium lead nickel zinc	1,1,1-trichloroethane trichloroethylene tetrachloroethylene
Directly Related Waste Codes	F003 and F005 (non-halogenated solvents)	D002 (corrosives) D006 (cadmium) D007 (chromium) D008 (lead)	F001 (degreasing solvents)
Indirectly Related Wastes	non-halogenated solvents listed under U and D waste codes. flammable waste	K062 some F006 some F019 D004-11 acidic/alkaline waste	halogenated solvents listed under F, U, and D waste codes.

The paint-related waste offered the advantage of exhibiting two hazardous characteristics in the same waste, i.e., containing non-halogenated solvents and being flammable. Therefore, the performance of the sorbents in reducing or eliminating both of these hazardous properties were obtained from one sample. The paint-related waste sample was obtained from the paint spray gun cleaning operation at CTC's Environmental Technology Facility (ETF) in Johnstown, Pennsylvania. The degreasing solvent waste and heavy metals waste were both prepared in the laboratory at the same facility. The primary regulated constituent in the paint waste was methyl ethyl ketone.

The degreasing solvent was prepared by mixing together 30 percent by volume each of trichloroethylene, 1,1,1-trichloroethane, and tetrachloroethylene, and 10 percent by volume of SAE 30 motor oil. This solvent mixture was representative of the types of degreasing solvents and oil/grease waste products found in industrial operations.

The aqueous, heavy metals waste was simulated by preparing an acidic solution of 10 percent by volume nitric acid and adding the five metals. The metals were added at levels exactly 100 times their respective Land Disposal Restriction standards: chromium, lead, nickel, and zinc were

dosed at 500 mg/l and cadmium was added at 100 mg/l. One reason for choosing the 100-fold concentration factor was that the resulting baseline metal concentrations would be typical of major industrial wastes. The other reason was that the overall effect of sorbent dilution, which can account for 50 percent or more of the reduction in chemical constituents, would be minimized, and thus the data would more accurately represent actual treatments. Nickel and zinc were added to the aqueous heavy metals waste because they are commonly found together with the other metal constituents.

3.2 Selection of Sorbent Materials

The identification, screening, and selection of candidate sorbent materials was the principal objective of the technology assessment, which is described in detail in the "Technology Assessment Report" dated May 24, 1995. The approach to performing the technology assessment involved six steps: (1) organizing and reviewing vendor responses to the DLA's sources sought notification in the CBD; (2) conducting a literature search; (3) conducting a telephone/facsimile survey; (4) performing an assessment of relevant regulations; (5) identifying performance criteria; and (6) using the performance criteria to screen the sorbents and recommend those best suited for the bench-scale test program.

The vendor responses to the CBD announcement were used as a foundation for developing an initial list of sorbent classes. The category of clay sorbents was added as a control standard, to compare the performance of the other candidate sorbents, because clay sorbents are very common among DOD installations.

Some of the sorbent materials identified by the vendor's responses did not fit the required definition of sorbents as outlined in the CBD announcement and were immediately removed from consideration. These sorbent materials included ion exchange resins, molecular sieves, and known biodegradable sorbents.

Each of the sorbent classes represented by the CBD responses, and clay, were evaluated against each other based on six screening criteria. These criteria, in order of importance, were:

1. Is the sorbent listed or certified as non-biodegradable, thus suitable for landfill disposal?
2. How well do the selected classes of sorbents, as a whole, address the range of DOD candidate wastes selected for laboratory testing at *CTC*? Are all three categories of sorbents (organic, synthetic, inorganic) represented by the chosen sorbent classes?
3. Is the sorbent class well-established in the marketplace? To what extent has research been done on the sorbent class been commercialized? To what extent is the sorbent class already used at DOD facilities for spill cleanup?
4. Is the sorbent readily available in loose particulate form and a variety of easy-to-use package volumes?
5. Has the sorbent class been shown to adequately immobilize free liquid and hazardous constituents in similar waste streams?
6. How well does the sorbent class meet other product quality and operational performance criteria?

The result of the screening analysis was the selection of four categories of sorbents for the first round of bench-scale testing, plus clay as a control:

- Peat, certified non-biodegradable (per testing performed and documented by the sorbent vendor)
- Polymeric, non-encapsulating
- Polymeric, encapsulating
- Zeolitic
- Clay.

Two other rounds of testing were conducted during the study period. First, at the request of DLA and one of the sorbent vendors, the zeolitic sorbent used in the first round of testing was re-tested under conditions that the vendor felt were more favorable to the sorbent product. Second, a cellulosic sorbent was later tested using the same procedures used during the first round of bench-scale testing. The cellulosic sorbent was added because of a change in the regulatory definition of “biodegradable sorbent” that qualified at least one vendor’s cellulosic sorbent as non-biodegradable sorbent, and thus suitable for testing consideration.

3.3 Regulatory Assessment

The regulatory assessment was conducted concurrently with the technology assessment. This was done to provide focus and direction to the sorbent screening process. The three waste streams identified in the "Candidate Waste Streams Report" were used, in part, as the foundation for the regulatory analysis. From this report, CTC examined and compiled a list of relevant regulations that affect the use of sorbent materials for treating the three DOD candidate waste streams.

The regulatory assessment found that, if a sorbed liquid is properly treated according to RCRA Land Disposal Restrictions (LDR) and exhibits none of the characteristics that originally made it hazardous, the waste may be land disposed. If a waste is sent for reuse or recycling, LDRs do not apply. Facilities must bear in mind, however, that the states in which they operate may impose more stringent regulations on the management and disposal of hazardous wastes than the federal program.

3.4 Bench-Scale Test Planning

The "Test Plan," dated June 6, 1995, was developed with the objective to outline the test procedures, methods, and rationale for evaluating the performance, material compatibility, and waste volume reduction of the candidate sorbents on the three waste streams. The specific tests outlined in the Test Plan were designed to evaluate several aspects of sorbent performance, most importantly the sorbent's ability to: immobilize free liquids, stabilize hazardous constituents to meet LDRs, and eliminate any associated hazardous properties, such as flammability or corrosivity. As part of the bench-scale testing, the sorbent materials, representing the six sorbent classes of peat, encapsulating polymeric, non-encapsulating polymeric, zeolitic, clay, and cellulosic, were tested against all three waste streams.

3.5 Waste Stream/Sorbent Testing

Bench-scale testing of the sorbents and waste streams was conducted in three phases. The test results were reported in the: "Phase II Technical Report" (August 29, 1995); "Phase II Follow-up Report" (December 15, 1995); and "Phase II Report: Addendum, Cellulosic Sorbent Testing" (January 15, 1996), respectively. All tests were carried out as specified in

the Test Plan. Details of testing procedures, including the waste/sorbent mixing ratios, can be found in the reports.

4.0 SUMMARY OF BENCH-SCALE TEST RESULTS

Tests were conducted to provide data to help determine whether or not the sorbed wastes would meet the Land Disposal Restrictions. Prior to the performance testing of the sorbent, laboratory screening tests were run to confirm vendor recommended mixing ratios, evaluate compatibility with the waste, and establish correct mixing ratios of waste to sorbent for subsequent testing. This section of the report summarizes the results from all three rounds of Phase II testing for the three most important tests: immobilization of free liquids; stabilization of hazardous constituents to meet LDR standards; and elimination of associated hazardous properties, such as flammability or corrosivity.

4.1 Immobilization of Free Liquids

The Paint Filter Test (PFT) was used to determine whether or not the sorbed wastes released any liquids (Table 5). If a particular waste/sorbent mixture yielded free liquid during the PFT, the sample failed, and no follow-on tests were conducted on that waste/sorbent mixture.

Table 5. PFT Results

Sorbent Category	Waste Stream		
	Paint-Related	Aqueous Heavy Metals	Degreasing Solvents
Peat (non-biodegradable) ¹	Pass	Pass	Pass
Polymeric (non-encapsulating) ¹	Fail	Pass	Fail
Polymeric (encapsulating) ¹	Fail	Pass	Pass
Zeolitic (Original Test) ¹	Pass	Pass	Pass
(Re-Testing) ²	Pass	Pass	Pass
Clay ¹	Pass	Pass	Pass
Cellulosic ³	Pass	Pass	Pass

¹ Phase II Technical Report (8/95), Tables 14, 21, and 27.

² Phase II Follow-up Report (12/95), Tables 5, 8, and 11.

³ Phase II Report: Addendum, Cellulosic Sorbent Testing (1/96), Tables 11 and 12.

As shown in Table 5, many of the sorbents were able to pass the PFT, indicating that liquids could be immobilized.

4.2 Stabilization of Hazardous Constituents to Meet LDRs

This post-treatment testing of the sorbed waste involved determining the extent to which the hazardous constituents had been stabilized by the sorbent, and whether or not LDR standards were met. For the paint-related waste and degreasing solvent waste, this testing involved measuring VOC concentrations of the sorbed waste. For the aqueous, heavy metals waste, measurements were made of the metals concentration in the TCLP (Toxic Characteristic Leaching Procedure) extract of the sorbed waste. These tests were performed only on wastes that first passed the Paint Filter Test.

Table 6. Hazardous Constituent Stabilization Test Results

Sorbent Category	Waste Stream		
	Paint-Related	Aqueous Heavy Metals	Degreasing Solvents
Peat (non-biodegradable) ¹	Fail	Pass (Pb only)	Fail
Polymeric (non-encapsulating) ¹	NT	Fail	NT
Polymeric (encapsulating) ¹	NT	Pass (Cd, Cr, Pb only)	Fail
Zeolitic (Original Test) ¹	Fail	Pass (Pb only)	Fail
(Re-Testing) ²	Fail	Pass (Pb only)	Fail
Clay ¹	Fail	Pass (Cd, Cr, Pb, Ni, Zn)	Fail
Cellulosic ³	Fail	Fail	Fail

¹ Phase II Technical Report (8/95), Tables 16, 23, and 28.

² Phase II Follow-up Report (12/95), Tables 7, 10, and 12.

³ Phase II Report: Addendum, Cellulosic Sorbent Testing (1/96), Tables 14, 16 and 17.
NT = not tested because failed PFT.

As shown in Table 6, many of the sorbents failed to meet LDR standards. None of the sorbents were able to meet the LDR standards for the paint-related and degreasing solvent wastes.

The best-performing sorbent for stabilizing the metals in the waste was clay. The clay sorbent resulted in leachable metal concentrations that were below detection limits (<0.02 mg/l) for all metals; the LDR standards are 1.0 mg/l for cadmium and 5.0 mg/l each for chromium, lead, nickel, and zinc. The two zeolitic test samples resulted in leachable lead

concentrations of 1.2 and 1.4 mg/l, which were below the LDR of 5.0 mg/l for lead. The peat and encapsulating polymeric sorbents also appeared to stabilize some metals. However, these two sorbents were later eliminated from further consideration because of their inability to neutralize the waste's acidity, as discussed below.

4.3 Elimination of Flammability and Corrosivity

This post-treatment testing involved determining whether or not the characteristic flammability (ignitability) of the paint-related waste or the corrosivity of the aqueous heavy metals waste was eliminated by the sorbent. A waste sample passed the ignitability test if the sample failed to ignite when directly exposed to a flame. A sorbed waste passed the corrosivity test if the final pH was between 2 and 12. The degreasing solvent did not exhibit any associated characteristics, so no ignitability/corrosivity tests were run on that sample. Ignitability/corrosivity tests were only performed on samples in which the waste/sorbent combination passed the preceding Paint Filter Test.

Table 7. Ignitability and Corrosivity Test Results

Sorbent Category	Waste Stream and Test	
	Paint-Related, Ignitability	Aqueous Heavy Metals, Corrosivity
Peat (non-biodegradable) ¹	Fail	Fail
Polymeric (non-encapsulating) ¹	NT	Fail
Polymeric (encapsulating) ¹	NT	Fail
Zeolitic (Original Test) ¹	Fail	Pass
(Re-Testing) ²	Fail	Pass
Clay ¹	Fail	Pass
Cellulosic ³	Fail	Fail

¹ Phase II Technical Report (8/95), Tables 15 and 22.

² Phase II Follow-up Report (12/95), Tables 6 and 9.

³ Phase II Report: Addendum, Cellulosic Sorbent Testing (1/96), Tables 13 and 15.

NT = not tested because failed Paint Filter test.

As shown in Table 7, all of the sorbents failed to eliminate the flammability of the paint related waste. With regard to eliminating the corrosivity of the aqueous heavy metals waste, only the zeolitic and clay sorbents passed this test. The other sorbed wastes had pH values less than 1.0, similar to the pH of the original waste.

5.0 SUMMARY AND CONCLUSION

To be permitted for land disposal, the sorbed wastes tested in this study would have to meet of three requirements:

- Pass the PFT
- Meet the LDR standards for the chemical constituents
- Eliminate the flammability or corrosivity of the waste, if applicable.

As shown in Table 1 (see Page 2), only one waste/sorbent combination — aqueous, heavy metals treated with clay — passed all three tests for all chemical constituents. The peat, encapsulating polymeric, and zeolitic sorbents also passed all three tests, but only for selected metals, and in most cases, only met the LDR standards by a slim margin.

The zeolitic sorbent offered one important benefit over the peat, polymeric, and cellulosic sorbents; it was the only sorbent other than clay to eliminate the corrosivity of the aqueous, heavy metals waste. Because the zeolitic sorbent also passed the PFT and met the LDR standards for one metal (lead), this waste/sorbent combination also is worthy of further consideration.

For the paint-related and degreasing solvent wastes, none of the sorbed wastes would be able to be land disposed. None of the LDR standards were met and none of the associated hazardous characteristics were eliminated for these two wastes.

6.0 RECOMMENDATIONS

Based on the results of the three rounds of waste/sorbent testing, two waste/sorbent combinations deserve further consideration:

- aqueous, heavy metals waste with clay sorbent (for removing cadmium, chromium, lead, nickel, and zinc), and
- aqueous, heavy metals waste with zeolitic sorbent (for removing lead only).

Both of these test combinations met the LDR standards, eliminated the corrosivity, and allowed no free liquids to pass.

CTC recommends that an economic analysis (Phase III task) be performed for both of these waste/sorbent combinations. The results of the economic analysis will be used to justify whether or not a full-scale treatment study should be conducted.

APPENDIX A

**Feasibility Study of Sorbent Treatments for Hazardous Wastes
Phase II Technical Report**

Feasibility Study of Sorbent Treatments for Hazardous Wastes

Phase II Technical Report

August 29, 1995

Task No. N.063
CDRL No. A005
Contract No. DAAA21-93-C-0046

*Prepared by
National Defense Center for Environmental Excellence
(NDCEE)*

Operated by Concurrent Technologies Corporation

**Feasibility Study of Sorbent Treatments
for Hazardous Wastes**

Phase II Technical Report

Draft

August 29, 1995

Contract No. DAAA21-93-C-0046

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CDRL No. A005

Submitted by

Concurrent Technologies Corporation

1450 Scalp Avenue

Johnstown, PA 15904

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1.0 SUMMARY OF PHASE II TESTING

1.1 Introduction

Sorbents, for the purpose of this task, are defined as solid materials that take up and hold liquid by means of absorption, adsorption, or both. Sorbents have been extensively studied and used to absorb free liquids from spills. As such, sorbents are commonplace at industrial facilities, laboratories, and other operations where oil, fuel, and chemical spills may occur. Occasionally, sorbents also are used for waste treatment before land disposal. In the latter case, however, only a limited number of studies have been conducted to determine the feasibility of using sorbents for nonspill applications and whether or not they meet regulatory requirements for land disposal. A sorbed waste may be land disposed if it meets RCRA Phase II Land Disposal Restrictions (LDR) and no longer exhibits any of the characteristics that originally made it hazardous.

The Defense Logistics Agency (DLA) tasked Concurrent Technologies Corporation (CTC), through the National Defense Center for Environmental Excellence (NDCEE), to perform a comprehensive feasibility study of sorbent treatments for hazardous waste. The objective of this task is to identify, demonstrate, and validate a simple, efficient, and cost effective technology, based on sorbent materials, for treating Department of Defense (DOD) generated hazardous waste streams, including contaminated media, and thereby reduce disposal costs.

The proposed feasibility study of using sorbent materials for the treatment of hazardous waste supports the Environmental Excellence Program of the DLA for Fiscal Year 1995. One of the principle requirements of this program is for the DLA and its primary field-level activities to establish specific goals, objectives, and measures to reduce DOD generated hazardous waste disposal costs.

This task, which started in April 1995, is comprised of four distinct phases. Phase I consisted of identifying waste streams and classes of sorbents for testing and developing a test plan for the bench-scale studies. As part of the Phase I investigation, the following five deliverables were prepared by CTC and completed by June 1995: a Candidate Waste Streams Report, Technical Assessment Report, Safety Plan, Test Plan, and QA/QC Plan. Phase II is the bench-scale laboratory investigation of waste/sorbent combinations and forms the basis for this report. Planned activities are the Phase III economic analysis and the Phase IV on-site demonstration testing.

1.2 Objective of the Phase II Testing

The objective of Phase II was to conduct bench-scale testing of sorbent materials and waste streams to quantify the effectiveness of sorbent materials for treating hazardous wastes. This technology report discusses the results, and presents conclusions from the waste/sorbent treatability testing.

During Phase I of this sorbent task, performance criteria were identified to assist in evaluating and comparing candidate sorbents. These criteria are shown in Table 1. Of the thirteen performance criteria identified, eight criteria were fully addressed by data obtained during the technology survey. The remaining five criteria were to be addressed, wholly or in part, by data generated during the Phase II laboratory bench-scale testing at CTC. These five criteria, highlighted in Table 1, were:

- Suitability for immobilizing free liquids,
- Elimination of characteristic hazardous properties or constituents,
- Sorbent/sorbate material compatibility,
- Waste volume reduction, and
- Sorbent use economics.

Table 1. Performance Criteria and Means of Evaluation

Performance Criteria	Means of Evaluation
I. Suitability for Landfilling	
A. Long-term stability/ nonbiodegradability	vendor data
B. Immobilization of free liquids	CTC laboratory data
C. Immobilization of hazardous constituents	CTC laboratory data
II. Product Quality, Performance, and Economics	
A. Sorbent/sorbate compatibility	CTC laboratory data
B. Development risk	technology survey
C. Vendor quality assurance	vendor data
D. Product availability	technology survey
E. Waste volume reduction	CTC laboratory data
F. Sorption selectivity and efficiency	vendor data
G. Sorbent economics	vendor data, CTC laboratory data, economic analysis
III. Operational Issues	
A. Human health data	vendor data, technology survey
B. Worker Safety Impact	vendor data
C. Ease of Handling	vendor data

As a result, the key objective for the Phase II task was to generate laboratory data for the designated waste streams and sorbents to support these five criteria and to provide recommendations for the economic assessment.

1.3 Summary

This report discusses the findings from the Phase II laboratory bench-scale investigation to determine the technical feasibility of five classes of sorbents for treating three surrogate waste streams: paint related waste, aqueous heavy metals waste, and degreasing solvent waste. The tasks involved in the bench-scale investigation fell into three broad areas:

- Baseline chemical and physical characterization of the waste streams,
- Characterization of the sorbents' capacity, ability to immobilize free liquids and hazardous constituents, and material compatibility, and
- Post-treatment testing and characterization of the sorbed waste for residual chemical and physical hazardous constituents.

1.4 Major Conclusions

Zeolitic, peat, and clay sorbents passed the Paint Filter Test (PFT) for the paint related waste. However, these sorbents failed to reduce the flammability, or the concentration of organic constituents below the Land Disposal Restriction (LDR) Phase II standards required for landfill disposal of such waste.

All five sorbents, i.e., zeolitic, polymeric encapsulating, polymeric non-encapsulating, peat, and clay, passed the PFT for the aqueous heavy metals waste. However, not all of the sorbents performed adequately in reducing all of the heavy metals concentrations below LDR standards. Clay and zeolitic were the top ranked sorbents for this waste.

Similar to the paint waste samples, some of the sorbents passed the PFT for degreasing solvent waste, but each of these failed in reducing organic constituents below LDR standards.

In conclusion, only the clay and zeolitic sorption of aqueous heavy metals waste resulted in wastes that would be suitable for landfill disposal, and then only for wastes containing selected metals. These two waste/sorbent combinations are recommended for Phase II economic assessment, as shown in Table 2.

Table 2. Waste/Sorbent Recommendations for Economic Analysis

Sorbent Classes	Waste Streams		
	Paint Related Waste	Aqueous Heavy Metal Waste	Degreasing Solvent Waste
Peat (non-biodegradable)			
Polymeric (non-encapsulating)			
Polymeric (encapsulating)			
Zeolitic		√	
Clay		√	

√ = Recommended

1.5 Major Recommendations for Future Work

Several factors are recommended for further examination to optimize the sorbent treatment of the three DOD candidate waste streams:

Waste-to-sorbent mixing ratio—Waste-to-sorbent mixing ratios were predetermined and not varied in the bench-scale study. In instances where samples failed either the PFT or the post-treatment test for hazardous characteristics, it is possible that lower waste-to-sorbent ratios would have yielded samples that passed these tests.

Time—The effects of time on sorbent performance was not investigated as part of this study. It is possible that allowing the waste matrix to set for several days might help to stabilize hazardous constituents.

pH—The pH of the sorbed waste increased significantly in the case of zeolitic and clay sorbents. It is possible that the pH had a significant effect in reducing metals concentrations, but this must be investigated.

Additives—The effect of additives, such as water or Portland cement, to enhance sorbent performance also was not investigated. It is also possible that the addition of small amounts of these or other materials might enhance sorbency as well as “fix” the hazardous constituents.

Alternative disposal—The bench-scale test program was designed to test waste-sorbent combinations for meeting landfill disposal requirements. However, there are other post-sorbent treatment options besides landfilling that may deserve further study, such as aggregation, bioremediation, and vitrification.

2.0 SUMMARY OF PHASE I TASKS AND DELIVERABLES

The Phase I activities called for five deliverables or reports, as indicated in Table 3. The content of these reports is summarized below to provide the background for the Phase II activities.

Table 3. Summary of Phase I Deliverables

Report Title	Date	Focus of Report
Candidate Waste Streams Report	May 23, 1995	Selection of waste streams for Phase II bench-scale testing
Technical Assessment Report (included regulatory assessment)	May 24, 1995	Use information from literature searches and surveys to select sorbent materials for testing
Safety Plan	June 19, 1995	Measures for protecting CTC employees from potential hazards during bench-scale testing
Test Plan	June 27, 1995	Methods and procedures for bench-scale testing
QA/QC Plan	June 28, 1995	Approach for implementing the quality requirements of the bench-scale testing

2.1 Candidate Waste Streams Report

The first task deliverable, "Candidate Waste Stream Report", investigated those DOD hazardous waste streams indicated by DLA to be of highest priority for waste disposal cost reduction, and potentially suited to sorbent treatment. The objective was to identify a manageable number of waste streams for laboratory testing with various sorbents, considering regulatory issues and a broad range of possible waste types. The procedure involved using the EPA listed wastes as an initial list and then narrowing the list based on specified screening criteria. These screening criteria were: consideration as a DOD high priority waste stream, as defined by the total cost of disposal to DLA; regulations that ruled out treatment of some wastes by sorbents, or that made treatment not worthwhile; wide applicability; and environmental considerations. Applications of this screening

procedure resulted in the recommendation of the following three categories of DOD candidate wastes:

- Paint related waste,
- Aqueous heavy metal waste, and
- Degreasing solvent waste.

These three waste streams were selected for use in the Phase II bench-scale testing.

2.2 Technology Assessment Report

The principal objective of the technology assessment was to obtain and analyze information on various sorbent materials, and recommend several sorbent materials for Phase II bench-scale testing with the three DOD candidate waste streams. The approach to performing the technology assessment involved six steps:

(1) organizing and reviewing vendor responses to the DLA's sources sought notification in the Commerce Business Daily (CBD); (2) conducting a literature search; (3) conducting a telephone/facsimile survey; (4) performing an assessment of relevant regulations; (5) identifying performance criteria; and (6) using the performance criteria along with the DLA/CBD requirements to screen the sorbents and recommend those best suited for the bench-scale test program.

The vendor responses to the CBD announcement were used as a foundation for developing an initial list of sorbent classes. At least one sorbent class, clay, was not represented in the CBD responses. Because of the potential importance of clay as an analytical control, clay was added to the initial list of sorbent classes. At the same time, some of the sorbent materials identified by the vendors responses did not fit the required DLA/CBD definition of sorbents. The types of sorbent materials that were removed from consideration included known biodegradable sorbents, ion exchange resins, and molecular sieves.

Each of the sorbent classes on the initial list were evaluated relative to the other sorbent on the list based on six screening criteria. These criteria, in order of importance, were:

1. Is the sorbent listed or certified as non-biodegradable and thus suitable for landfill disposal?
2. How well do the selected classes of sorbents, as a whole, address the range of DOD candidate wastes selected for laboratory testing at CTC? Are all three categories of sorbents (organic, synthetic, inorganic) represented by the chosen sorbent classes?
3. Is the sorbent class well-established in the marketplace? To what extent has research been done, or has the sorbent class been

commercialized? To what extent is the sorbent class already used at DOD facilities for spill cleanup?

4. Is the sorbent readily available in loose, particulate form and a variety of easy-to-use packaged volumes?
5. Has the sorbent class been shown to adequately immobilize free liquid and hazardous constituents in similar waste streams?
6. How well does the sorbent class meet other product quality and operational performance criteria?

The result of the screening analysis was the selection of four distinct categories of sorbents for further evaluation, along with selection of clay sorbent as a control. The final five candidates selected for evaluation were:

- Peat, non-biodegradable,
- Polymeric, non-encapsulating,
- Polymeric, encapsulating,
- Zeolitic, and
- Clay.

2.3 Regulatory Assessment

The regulatory assessment was conducted concurrently with the technology assessment. This was done to provide focus and direction to the sorbent screening process. The three waste streams identified in the "Candidate Waste Streams Report" were used, in part, as the foundation for the regulatory analysis. From this report, *CTC* examined and compiled a list of relevant regulations that affect the use of sorbent materials for treating the three DOD candidate waste streams.

The regulatory assessment found that, if a sorbed liquid is properly treated according to RCRA Land Disposal Restrictions (LDR) and exhibits none of the characteristics that originally made it hazardous, the waste may be land disposed. If a waste is sent for reuse or recycling, LDRs do not apply. Keep in mind, however, that the states in which facilities are located may impose more stringent regulations on the management and disposal of hazardous wastes than the federal program.

2.4 Safety Plan

The objective of the "Safety Plan" was to detail safe-laboratory practices and other measures to protect *CTC* employees from potential hazards during the bench-scale testing. The Safety Plan provided guidelines for anticipating, recognizing, evaluating, and controlling health and physical hazards throughout the workplace. The Safety Plan provided employees with the training, materials, and equipment

necessary to protect themselves from hazards created by the sorbents, wastes and other hazardous chemicals that might be used.

2.5 Test Plan

The principal objective of the "Test Plan" was to outline the test procedures, methods, and rationale for evaluating the performance, material compatibility, and waste volume reduction of the candidate sorbents on the three waste streams. The specific tests outlined in the Test Plan were designed to determine the sorbents' capacity for the three waste streams, ability to immobilize free liquid and hazardous constituents in the waste, and the material compatibility of the waste/sorbent mixtures.

The Test Plan called for two of the three types of DOD waste samples, aqueous heavy metal waste and degreasing solvent waste, to be simulated in the laboratory using reagent and standard grade chemicals. This offered cost and personal safety advantages over the alternative of shipping the waste to CTC from an actual DOD facility. For convenience as well as representativeness, the paint related waste sample was taken from a painting operation at CTC.

The selection of three waste streams and five sorbent classes resulted in a test matrix consisting of fifteen test combinations (Table 4). All fifteen experimental combinations were tested during Phase II.

Table 4. Candidate Waste Streams and Sorbent Combination Matrix

Sorbent Classes	Candidate Waste Streams		
	Paint Related Waste	Aqueous Heavy Metals Waste	Degreasing Solvent Waste
Peat (non-biodegradable)	√	√	√
Polymeric (non-encapsulating)	√	√	√
Polymeric (encapsulating)	√	√	√
Zeolitic	√	√	√
Clay	√	√	√

2.6 Quality Assurance/Quality Control Plan

The principal objective of the "QA/QC Plan" was to describe the approach for managing and implementing the quality requirements of the laboratory bench-scale

test program. A key component of the QA/QC Plan is the identification of standard protocols for laboratory activities, such as sample handling, test equipment calibration, data reduction and reporting, and quality audits.

3.0 SCOPE OF PHASE II TESTING

The test plan for bench-scale testing in the laboratory was designed to test each selected sorbent material with the three waste streams. According to the Statement of Work requirements and the CBD announcement, these tests were designed to provide data to help the DLA establish the feasibility for the broader use of non-biodegradable, spill-type sorbents for treating hazardous wastes. The Phase II testing was not intended, nor should it be construed, to be a comprehensive investigation, including optimizing waste/sorbent mixing ratios and procedures.

Three major parameters were observed during this testing to establish the effectiveness of each sorbent. These three factors were:

1. the compatibility of each sorbent with each waste stream;
2. the ability of the sorbents to pass the EPA's PFT after sorbing the waste;
and
3. the ability of the sorbents to retain, or bind, the constituents that are listed in the Land Disposal Restrictions Phase II standards for that specific waste.

In addition to these three parameters, other testing was performed to determine the efficiency of each sorbent, namely:

- L-Test (Sorbency Testing) - used to determine the maximum amount of waste each sorbent was capable of sorbing.
- Volume, Mass, and Temperature Change - used to determine how the volume and mass of the waste was affected by the addition of sorbents.

These tests are described in detail along with the results in Section 6.0. The results were used to form final conclusions and recommendations for this task. The recommendations will be used in Phase III of this task to formulate a cost comparison to existing treatment methods of DOD hazardous waste. No recommendations will be made regarding any Phase IV on-site demonstration testing until Phase III is completed.

4.0 TEST MATERIALS AND METHODS

The materials used in the laboratory tests and the methods used to perform the bench-scale tests are discussed below. Details were first presented in the Test Plan, Safety Plan, and QA/QC Plan.

4.1 Hazardous Waste Streams

The "Candidate Waste Streams Report" describes the process used to select those hazardous waste streams showing high potential for treatment with a sorbent material. The three waste streams selected represent a large portion of the waste produced by the DOD.

4.1.1 Paint Related Waste

The paint related waste stream was chosen for testing for two reasons: 1) to determine if sorbents can remove waste ignitability, and 2) to determine if sorbents can retain or bind the organic solvents responsible for making this waste hazardous, in order to meet the LDR Phase II standards. The paint related waste sample was obtained from the paint spray gun cleaning operation at CTC's Environmental Technology Facility (ETF) in Johnstown, Pennsylvania. While the major component of this waste is paint, the primary regulated constituent causing this waste to be hazardous is methyl ethyl ketone (MEK). The latter is one of several solvents the EPA has classified as hazardous if used in certain processes. Using MEK as a cleaning solvent for paint guns is one such process that makes the resultant waste hazardous by "definition."

Toluene and xylene are two other constituents of the paint waste. As with MEK, these two solvents are listed by the EPA as hazardous by the same definition. Toluene is primarily used for paint gun cleaning, while xylene is used for thinning. The remaining two solvents identified in the paint waste, 4-methyl-2-pentanone and ethylbenzene, are listed on the Material Safety Data Sheets (MSDSs) for some of the paints used in CTC's painting operation and, therefore, are part of those paint formulations.

The paint related waste sample was tested to its characteristics, or baseline. The results of the baseline testing are provided below in Table 5. The methods used to test this material are discussed further in Section 4.4.

Table 5. Baseline Analysis of Paint Related Waste

Test	Parameter	Result ***	
Ignitability* (ASTM D-93-90)	Flash Point	< -5.0°C (< 23°F)	
		mg/l	mg/kg
Solvent Content** (EPA Method 8260A)	Methyl ethyl ketone	271,000	330,000
	4-Methyl-2-pentanone	4,630	5,700
	Toluene	7,580	9,300
	Total xylenes	16,300	20,000
	Ethylbenzene	3,290	4,000

* Ignitability test performed using a Pensky-Martens Closed Cup Tester.

** Solvent content measured using a Gas Chromatograph/Mass Spectrometer.

*** Measured values converted from mg/l to mg/kg using the density of the paint waste sample, 0.81 g/ml. Units of mg/kg are consistent with Land Disposal Restriction Phase II standards.

4.1.2 Aqueous Heavy Metals Waste

The aqueous heavy metal waste stream was chosen for two reasons: (1) to determine if the sorbents can neutralize an acidic liquid, and (2) to determine if the sorbents can retain or bind the metals in order to meet the LDR Phase II standards.

The aqueous heavy metals waste sample is a surrogate prepared in the CTC laboratory. The aqueous heavy metal waste sample consisted of five metals: cadmium (Cd), chromium (Cr), lead (Pb), nickel (Ni), and zinc (Zn) in a 10% by volume nitric acid (HNO₃) solution. Cadmium, chromium, and lead are commonly found in several DOD and commercial process wastes. Nickel and zinc are common underlying constituents found in several metal waste streams.

The surrogate sample of aqueous heavy metals waste was prepared using metal concentration standards normally used for calibrating analytical equipment. The metals were added to water to obtain final solution concentrations approximately 100 times the EPA's LDR Phase II standard for each metal. A concentration factor of 100 was selected to simulate heavy metal concentrations found in many industrial wastes.

A baseline analysis was performed on this sample to verify the actual metal concentrations in the solution. The results of the baseline analysis are shown in Table 6.

Table 6. Baseline Analysis of Aqueous Heavy Metals Waste

Test	Parameter	Result ***	LDR Standard
Acidity/ Alkalinity* (EPA Method 9040A)	pH	BDL (0.0)	N/A
		mg/l	mg/l
Heavy Metals Content** (EPA Methods 1311 and 200.7)	Cd	102	1.0
	Cr	508	5.0
	Pb	540	5.0
	Ni	519	5.0
	Zn	507	5.3

BDL = Below detection level of instrument

* pH measured with electrometric measurement device

** Metals analysis performed by Inductively Coupled Plasma-Atomic Emission Spectroscopy

*** The density of the aqueous heavy metals waste sample was 1.07 g/ml

These metal concentrations are within the range of concentrations seen in waste streams of major metal-waste-producing industries. A study by IT Corporation for USEPA (1991) of nine major industries (e.g., metal coatings, smelting and refining, paint and ink) showed the following average metals concentrations for cadmium, chromium, lead, and nickel for several waste categories (Table 7). The study mentioned that zinc is also found in many of these waste categories, particularly F006.

Table 7. Average Metal-Waste Stream Composition Data (USEPA, 1991) for Nine Major Industries

Concentration (mg/l)				
EPA Hazardous Waste No.	Cd	Cr	Pb	Ni
F006	1,320	39,730 24.5*	408	14,760
F009	21.6	525.9	25.39	2.954
F019	0.62	11,361 2.2* 11,358.6**	<17	1,275

* Chromium as Cr (VI)

** Chromium as Cr (III)

4.1.3 Degreasing Solvent Waste

Degreasing solvent waste was chosen for one main reason -- to determine if sorbents can retain or bind degreasing solvents effectively enough to meet the LDR Phase II standards. This stream was not flammable and contained no heavy metals.

The degreasing solvent waste sample was a surrogate prepared in the CTC laboratory. The sample was prepared by using laboratory grade standard reagents. The waste sample consisted of 30% trichloroethylene, 30% 1,1,1-trichloroethane, 30% tetrachloroethylene and 10% SAE 30 motor oil by volume. These three solvents represent a majority of degreasing solvents used in DOD and commercial operations.

A baseline analysis was performed on the sample with the results found in Table 8.

Table 8. Baseline Analysis of Degreasing Solvent Waste

Test	Parameter	Result*	
		mg/l	mg/kg
Solvent content** (EPA Method 8260A)	1,1,1-Trichloroethane	372,600	273,000
	Trichloroethylene	406,000	297,000
	Tetrachloroethylene	460,100	337,000

* Measured values converted from mg/l to mg/kg using the density of the degreasing solvent waste sample, 1.37 g/ml. Units of mg/kg are consistent with Phase II standards.

** Solvent measurement made with Gas Chromatography/Mass Spectroscopy.

4.2 Sorbent Materials

The "Technology Assessment Report" describes the process used to select sorbent classes showing high potential for treatment of hazardous waste. Five classes of sorbents were chosen for the laboratory scale testing: non-biodegradable peat, non-encapsulating polymeric, encapsulating polymeric, zeolitic, and clay. The following 3x5 matrix illustrates the test plan matrix.

As shown by the alphanumeric abbreviations in Table 9, the non-biodegradable peat and non-encapsulating polymeric sorbent classes were represented by a single sorbent product for their respective class. The encapsulating polymeric, zeolitic, and clay sorbent classes were represented by two commercial sorbents for their respective class. Different sorbents were used for these three cases because no all-purpose sorbent was identified for that class that could effectively sorb both organic and aqueous waste streams. As a result, a total of eight commercial sorbent products were tested.

Table 9. Candidate Waste Streams and Sorbent Combination Matrix

Sorbent Classes	Candidate Waste Streams		
	Paint Related Waste	Aqueous Heavy Metal Waste	Degreasing Solvent Waste
Peat (non-biodegradable)	P	P	P
Polymeric (non-encapsulating)	N	N	N
Polymeric (encapsulating)	E-1	E-2	E-1
Zeolitic	Z-1	Z-2	Z-1
Clay	C-1	C-2	C-1

The manufacturer or brand names of the eight sorbent materials were not provided to the CTC laboratory technicians during testing, nor are such names referenced in this report. Rather, all sorbents were referred to by their respective sorbent class name.

4.3 Physical Testing Methods

The following sections discuss the physical tests performed on the sorbents and waste/sorbent mixtures prior to and after mixing. These tests were used to determine the sorbency of the sorbents, the effect the sorbent had on the waste, and the characteristic of the resultant mixture.

4.3.1 L-Test

The L-Test, per Canadian General Standards Board (CGSB) Method CAN/CGSB-183.2-94 section 9.5.2, was used to determine the sorption capacity of each sorbent material. The method requires the use of a mesh container filled with a specific mass of sorbent. The container is then submersed in the waste for 15 minutes and then removed. The container is allowed to drain for 30 seconds then it is again weighed. The standard L-Test Ratio is obtained by recording the mass of waste absorbed per gram of sorbent.

4.3.2 Screening Paint Filter Test for Sorbent Dose Determination

In preparation for the bench-scale Paint Filter Test (PFT), it was necessary to develop and verify a uniform and rational procedure to determine the appropriate amount of sorbent to add to each waste sample. This was necessary for two reasons. First, in a few instances, vendors did not

provide waste/sorbent mixing ratios that would ensure no free liquids. Second, in most instances when vendors did provide dosing recommendations, the recommendations were based on results from similar, but not identical, waste streams. Without a procedure for verifying the vendors' recommended mixing ratio for a given waste, there was no way of determining whether or not the recommendation was appropriate. Therefore, it was necessary to conduct a "screening" PFT for each waste/sorbent combination where a mixing ratio was provided by the vendor.

The L-Test Ratio shows the upper mass limit of waste that can be absorbed by each gram of sorbent. However, using the exact L-Test Ratio to set the sorbent dose to produce no free liquids does not provide any safety factor to account for variations in mixing, temperature, sorbent quality, and other uncontrolled factors. Therefore, a conservative value of one-half the L-Test Ratio was used in the bench-scale PFT. Halving the L-Test Ratio allows twice as much sorbent material to be added to the waste sample and, in essence, provides a safety factor of 100 percent. The idea of adding additional sorbent beyond the point at which the waste appears to produce no free liquids is also supported by statements in product literature from a zeolitic and polymeric sorbent vendor. The zeolitic vendor recommended adding 25 percent additional sorbent to the waste, while the polymeric vendor did not provide a specific excess quantity of sorbent to add.

Since it was believed that this approach should provide for a suitable sorbent dose to allow the sorbed wastes to pass the PFT, it was necessary to compare these waste/sorbent mixing ratios with mixing ratios recommended by vendors. This mixing ratio verification testing was done before performing any bench-scale PFT. For each specific sorbent and waste combination, the sorbent vendor was asked to recommend an appropriate mixing ratio to ensure no free liquids. Of the 15 waste/sorbent mixtures from the 3x5 test matrix, vendors provided mixing ratios for 11 of the combinations. Vendors were unable to provide mixing ratios for four test combinations because of lack of knowledge about how well their product would perform on that particular waste stream.

For three of the test combinations, paint waste/clay, aqueous heavy metals/zeolitic, and aqueous heavy metals/clay, the one-half L-Test procedure resulted in very low ratios: 0.49:1, 0.27:1, and 0.28:1 respectively. If mixtures were made using these ratios, one would see a 3-fold to 4.5-fold increase in the mass of waste to be handled. The 4.5-fold increase, in particular, seemed impractical in terms of achieving pollution prevention/waste minimization goals. The 3-fold increase also appeared undesirable, but potentially acceptable in lieu of any economic analyses. A further assumption was made that, for mixing ratios less than 0.5:1,

thorough mixing of the sorbent and waste would be very difficult, if not impractical. As a result, a mixing ratio of 0.5:1 was set as the lower practical limit for this task. Consequently, for the paint waste/clay and aqueous heavy metals/clay tests, a ratio of 0.5:1 was used as the appropriate mixing ratio for the PFT.

4.3.3 Paint Filter Test

The PFT was used to determine whether or not sorbed wastes release any liquids. The PFT was critical to this task because it helped determine regulatory compliance of the waste.

The procedure for the PFT is outlined in EPA Method 9095. The Method calls for a specific mass of sample to be placed in a funnel lined with a paint filter. The sample is left standing for 5 minutes. If any free liquid is collected in a container below the funnel, the sample has failed the test. If no free liquid is visible, the sample passes the test.

Because two different sorbents were used to represent the zeolitic, encapsulating polymeric, and clay sorbent classes, a PFT was conducted on the particular sorbent intended to be used for the given waste stream.

4.3.4 Waste/Sorbent Compatibility

A compatibility test was performed on the waste/sorbent samples to observe any obvious signs of incompatibility in the form of (1) visual physical degradation and/or (2) chemical reaction. In the compatibility test, the sorbent and waste were mixed, and observations were made at pre-established time intervals. For these tests, waste/sorbent mixing ratios were set at twice the L-Test Ratio to intentionally overexpose the sorbent to approximately twice as much waste as it could absorb. This volume of waste ensured that all the sorbent was in contact with the waste and the maximum sorption occurred during the observation period.

This procedure required a specific mass of sorbent to be added to a graduated cylinder. The mass of waste from the L-Test Ratio was doubled and mixed with the sorbent. The mixture was then covered. Observations were made at the initial mixing and at 5 minute intervals for 15 minutes. After a 24 hour standing period, the mixture again was observed.

4.3.5 Volume/Mass/Temperature Change

In conjunction with the compatibility test, observations were also made to determine if there was a change in the total volume, mass, and temperature

of the resulting waste. A volume change illustrates whether or not the mixture will have a larger or smaller volume than the original waste stream. A temperature change typically indicates either an exothermic or endothermic chemical reaction has occurred. Exothermic reactions can pose a threat of spontaneous combustion, runaway chain reactions, or explosion. Changes in the mass of waste can impact waste disposal decisions when disposal cost is on a weight basis.

4.4 Analytical Methods

The baseline testing and final testing involved both physical and instrumental analyses. This section discusses the instrumental testing that was performed during the laboratory bench-scale testing.

4.4.1 Ignitability Testing

The ignitability test was performed in accordance with the EPA regulations found in 40 CFR Part 261, Identification and Listing of Hazardous Waste. This regulation identifies the use of an ASTM test method D093-90 to determine ignitability for liquids. The instrument used was a Pensky-Martens closed cup tester.

Once the waste/sorbent mixture was created, the waste was no longer a liquid. The same test as above was performed in addition to a flame test. The definition of ignitability in the above referenced regulation states:

“(2) It is not a liquid and is capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture or spontaneous chemical changes and, when ignited, burns so vigorously and persistently that it creates a hazard.”

The flame test was performed by moving a flame slowly toward the sample. Observations were made to determine if the flame “jumped” to the sample before it contacts the sample. Observations were also made if and when the sample caught fire. If the flame jumped to the sample, it could potentially be considered ignitable. Once the sample is ignited, subjective observations must be made to determine if the sample burns vigorously and persistently enough to create a hazard.

4.4.2 Corrosivity Testing

Corrosivity testing also was performed in accordance with the same regulation as above. EPA Method 9040A was used to determine the pH of

a liquid sample. A pH meter properly calibrated with pH buffer solutions was used to perform this test. A pH reading less than or equal to 2 or greater than or equal to 12.5 is considered to be hazardous.

4.4.3 Organic Solvent Testing

Volatile organic compound (VOC) analysis was performed in accordance with EPA Method 8260A. A Gas Chromatograph/Mass Spectrometer configured to analyze samples by purge and trap was used to perform these tests.

4.4.4 Heavy Metals Testing

Metal concentrations were determined in accordance with EPA Methods 1311 (Toxicity Characteristic Leaching Procedure) and 200.7 (Inductively Coupled Plasma Spectroscopy).

4.5 Quality Assurance and Quality Control

The QA objectives for this task were met by using standard accepted protocols to generate the waste/sorbent stream comparison data. All samples were identified with unique numbers and split out samples were traceable to parent samples. Instruments used in the bench-scale testing were calibrated with traceable standards. Duplicate samples were analyzed to calculate data precision. If the same standardized protocols are used in testing by others, the results should be comparable and reproducible.

4.5.1 Sampling and Traceability

All samples were prepared, labeled, and handled according to written procedures. Samples were properly logged into the laboratory's sample tracking logbook. Each sample of waste was given a unique laboratory identification number and the split out samples for chemical analysis were traceable to the parent sample.

4.5.2 Accuracy

Instruments used for testing were calibrated at prescribed intervals using traceable standards and documented calibration procedures. All calibrations were performed as described in the "QA/QC Plan" and according to written procedures. Calibration results were properly logged and there were no discrepancies that required corrective action.

4.5.3 Precision

Duplicate samples were analyzed in all quantitative tests and also for the pass/fail PFT. Relative Percent Differences (RPD) evaluations were calculated for all duplicate quantitative results. For the pass/fail PFT, if the duplicate did not agree with the initial test, a third referee replicate was run and the final result was based on the two results in closest agreement. This occurred with duplicate sample number 95-1837-C, which failed and the triplicate also failed; and duplicate sample number 95-1842, which failed and the triplicate also failed. A nonconformance report was not deemed necessary.

All quantitative test RPDs were within specified control limits. RPD is the absolute difference between the initial test and the duplicate test result divided by the average of the two readings times 100.

4.5.4 Comparability

Comparability was insured by using standard test protocols. Standard U.S. EPA and ASTM test protocols were used for all tests except the L-Test, which is a Canadian General Standards Board (CGSB) Method (CAN/CGSB-183.2-94 section 9.5.2). The basket used for the Type II (loose sorbent) test was a mesh stocking because other materials did not retain the finer sorbent materials. A value of one-half the L-Test ratio was used for all but three of the waste/sorbent mixing ratios. Exceptions to this rule were applied in three instances when one-half the L-Test ratio was calculated to be less than 0.5 gram of waste to 1.0 gram of sorbent. This 0.50:1 ratio was deemed to be the practical cut-off ratio. The three exceptions were: (1) Paint Related Waste and clay sorbent (2) Aqueous Heavy Metals Waste and zeolitic sorbent (3) Aqueous Heavy Metals Waste and clay sorbent. For these, one half the L-Test ratio was less than 0.5 gram of waste to 1.0 gram of sorbent, so in these cases 0.50 grams of waste to 1.0 grams of sorbent was used as the mixing ratio. All three of these combinations passed the PFT and were further tested. Based on the positive test results, the 0.5:1 limit did not appear to adversely affect the bench-scale testing.

5.0 EFFICACY TRIAL TEST DISCUSSION

The objectives of the efficacy trial testing were to: (1) verify laboratory QA/QC, material handling and mixing procedures, and other laboratory protocols, and (2) troubleshoot any problems, such as analytical interferences, prior to conducting bench-scale testing. The approach to the efficacy trial involved selecting representative waste samples and targeting the same suite of laboratory tests to be used in the bench-scale study. Two waste streams were selected and used during the efficacy trials; a kerosene waste stream and an aqueous heavy metals waste stream. These two wastes represented the broad waste categories of organics and aqueous waste. Both samples were surrogates made in the laboratory at CTC. All laboratory testing procedures were performed under ambient laboratory conditions. All procedures and observations were documented as they were conducted.

Some bench-scale procedures were added or modified based on findings during the efficacy trial testing. Briefly, these modifications were:

- the use of nitric acid instead of sulfuric acid to overcome interferences,
- the addition of the L-Test for sorbency testing, and
- the use of compatibility testing.

The following is a discussion of the major results and conclusions from the efficacy trial testing.

5.1 Kerosene Waste Stream

The kerosene waste stream consisted of 98% kerosene by volume and 2% deionized water by volume. The primary hazardous constituents of interest in this waste stream were VOCs and ignitability. Baseline testing consisted of a VOC Scan by Gas Chromatograph/ Mass Spectroscopy (GC/MS) and Flash Point by Pensky-Martens Closed Cup Tester. Because the kerosene contained only low levels of VOCs, the VOC levels detected were too low to be practical for analytical purposes. In addition, the flash point of the waste was 75°C and therefore not flammable by regulatory definition (<60°C). The waste stream was useful to observe the interactions of sorbent materials with organic phase materials and to troubleshoot analytical problems that may arise in the analysis of sorbed liquids by the GC/MS and flash point procedures. Efficacy in the measurement of VOCs by the heated purge and trap technique was demonstrated with laboratory standards.

5.2 Acid Waste Stream

For the efficacy testing, the aqueous heavy metals waste stream consisted of five metals; cadmium (Cd), chromium (Cr), lead (Pb), nickel (Ni), and zinc (Zn) in a 10% by volume concentrated Sulfuric Acid (H₂SO₄) matrix. The aqueous heavy

metals baseline testing consisted of quantification of the metals by Inductively Coupled Plasma (ICP) spectroscopy and pH determination by Electrometric Measurement. Metals were spiked at levels that should have resulted in baseline concentrations of 2 ppm Cd and 10 ppm Cr, Pb, Ni, and Zn. Instead, the observed metals concentrations were 1.53 ppm Cd, 8.46 ppm Cr, 3.85 ppm Pb, 7.87 ppm Ni, and 7.87 ppm Zn. In the case with Pb in H₂SO₄, much of Pb apparently precipitated out of solution as PbSO₄, thus the measured low dissolved concentration of Pb. Another problem appeared to be the fact that the very low pH of the H₂SO₄ (pH = 0.35) caused a shift in the standard curve of the instrument, resulting in erroneously low results. Therefore, based on observations from the efficacy trial and other experience, it was concluded that nitric acid (HNO₃) would be more compatible with metals in solution and with the ICP measurement techniques.

5.3 Waste/Sorbent Mixing Ratios

During this phase of testing it became evident that laboratory testing would be needed to experimentally determine reasonable waste/sorbent mixing ratios. This arose because of two reasons; mixing instructions were either not available or, if available, were not verified for all waste streams. For example, the zeolitic sorbents had no vendor information on mixing and the polymeric vendor instructions were for one specific type of waste stream. The polymeric sorbent would not stabilize the liquid constituent. As discussed below, sorbent ratios should be determined for each waste/sorbent combination prior to on-site application, unless the goal is to use an obvious excess of material. In addition, test mixing with small portions of waste should occur to determine compatibility and handling precautions.

In the treatment of the kerosene waste with the non-encapsulating polymeric sorbent, it was evident that free liquid remained after treatment using the generic vendor-supplied ratio of 25 grams waste to 1 gram sorbent. The test then was carried further with new ratios of 16.7 grams waste to 1 gram sorbent and 12.5 grams waste to 1 gram sorbent. Free liquid was evident in each case despite lowering the relative amount of waste. The waste/sorbent mixtures were allowed to stand for 24 hours and free liquid was still evident. The findings clearly indicated that a waste-specific sorbency test, such as the "L-Test", should be introduced into the test plan so that a proper mixing ratio is established for each combination. Another conclusion was that, when vendor mixing information is available, the recommended ratios should be verified by the user.

5.4 L-Test for Sorbency

Sorbency, for the purpose of this study, can be defined as the mass of waste absorbed per gram of sorbent applied to that waste. The Canadian General

Standards Board L-Test calls for the use of a mesh basket. Various metallic mesh screen materials were considered; however, the fine grain size of some sorbents precluded the use of these baskets. To sufficiently retain all of the sorbent material, the "basket" material required a very fine mesh size. The material ultimately chosen was nylon mesh from commercially available hosiery. The nylon did not degrade in the presence of the wastes, and held even the finest sorbent material. For fine material, such as the zeolitic sorbent, three overlapping stockings were required to sufficiently contain all of the sorbent. The sorbency ratio found for the polymeric type was 7.85 grams/1g of sorbent. The sorbency ratio found for the zeolitic type was 1.27 grams/1g of sorbent.

6.0 BENCH-SCALE TEST RESULTS AND DISCUSSION

This section provides detailed analytical discussions concerning the laboratory bench-scale test plan. Each waste stream is discussed separately. Section 6.1 discusses the results of the verification testing of vendor-recommended mixing ratios, Section 6.2 reviews test data for the Paint Related Waste test data, Section 6.3 reviews the Aqueous Heavy Metals Waste, and Section 6.4 reviews the Degreasing Solvent Waste test data.

6.1 Sorbent Dose Determination Testing

The mixing ratios recommended by the sorbent vendors were used to prepare the corresponding 1 l waste/sorbent mixtures used in the screening PFT. As shown in Table 10, of the 11 waste/sorbent combinations, only three passed the screening PFT at the vendor recommended mixing ratios. These three test results are highlighted in bold in the table. For comparison, the vendor ratios are also shown with the one-half L-Test Ratios determined in the laboratory.

Table 10. Screening Paint Filter Test Results Using Vendor-Recommended Mixing Ratios

Sorbents	Waste Streams					
	Paint Related		Aqueous Heavy Metals		Degreasing Solvents	
	Waste/Sorbent Mix Ratio (g waste/ 1 g sorbent)					
	Vendor	½ L-Test	Vendor	½ L-Test	Vendor	½ L-Test
Zeolitic	Failed		Failed		Passed	
	0.9	0.67	0.9	0.27	0.9	1.00
Polymeric (encapsulating)	Failed				Failed	
	3	1.98	NR	5.46	5	2.01
Polymeric (non-encapsulating)						
	NR	7.86	NR	7.77	NR	11.96
Peat (non-biodegradable)	Failed		Passed		Failed	
	4.52	3.10	5.86	4.64	7.60	4.09
Clay	Failed		Failed		Passed	
	0.9	0.49	2.12	0.28	0.9	0.65

NR = No recommendation provided by sorbent vendor.

Italicized numbers represent very low ratios that were not used during actual testing.

In examining the mixing ratio data, it is interesting to note that for the samples that passed the screening PFT, the difference between the vendor-recommended mixing ratio and the laboratory-calculated mixing ratio was small compared to samples that failed the screening PFT. Clearly, performing an L-Test and halving the

resulting ratio appears to be a valid procedure for verifying vendor-recommended mixing ratios. In addition, the use of one-half the L-Test Ratio may provide a good first approximation of the appropriate amount of sorbent to add to a given waste, particularly if the vendor cannot provide a recommendation.

As previously discussed, problems may occur when using waste/sorbent ratios less than 0.5:1. For the three test combinations where this occurred (i.e., paint waste/clay, aqueous heavy metals/zeolite, and aqueous heavy metals/clay), a mixing ratio of 0.5:1 was used as the appropriate mixing ratio.

6.2 Paint Related Waste Testing

The sample used for this waste material was collected from paint waste generated by the organic finishing line at CTC's Environmental Technology Facility. As stated earlier, the waste consists primarily of various paints, methyl ethyl ketone, toluene, and mixed xylenes.

6.2.1 Paint Related Waste L-Test

The L-Test (sorbency test) results for the five sorbent classes are shown below in Table 11. These results are reported as a ratio of waste to sorbent, and represent the mass of waste that can potentially be absorbed by one gram of sorbent. Also included in this table are the results of taking one half the L-Test ratio and doubling the L-Test ratio. The one-half L-Test Ratios were used in the PFT, and the two-times L-Test Ratios were used in the compatibility tests.

As shown in Table 11, the non-encapsulating polymeric sorbent has the highest sorbency ratio for this waste. All things being equal, this ratio suggests that the non-encapsulating polymeric sorbent should provide the least volume of waste, though may not necessarily represent the least-cost option.

Table 11. Paint Related Waste L-Test Results

Sorbent Class	Waste/Sorbent Ratios (g waste/ g sorbent)		
	L-Test Ratio	½ L-Test Ratio	2 L-Test Ratio
Zeolitic	1.33 :1	0.67 :1	2.66 :1
Polymeric (encapsulating)	3.96 :1	1.98 :1	7.92 :1
Polymeric (non-encapsulating)	15.7 :1	7.86 :1	31.4 :1
Peat (non-biodegradable)	6.20 :1	3.10 :1	12.4 :1
Clay	0.97 :1	0.49 :1	1.94 :1

6.2.2 Paint Related Waste Compatibility Test

The last column of the Table 11 shows the ratio for twice the L-Test. This ratio was used to test the compatibility of the five sorbent classes with the paint related waste. Table 12 illustrates the results of the compatibility test performed on the waste/sorbent mixture.

Table 12. Paint Related Waste Compatibility Test Results

Waste Type and Description	Sorbent Class	Sorbent Description	Waste/Sorbent Post Mix Description	Result
Paint Related Waste Greenish-brown and cloudy	Zeolitic	Dusty, very fine, dense, yellow in color	Saturated and darker in color	Compatible
	Polymeric (non-encapsulating)	Fluffy, medium texture, granular, white	Saturated and green in color	Compatible
	Polymeric (encapsulating)	Fluffy, soft very coarse texture, gray in color	Saturated and dark green in color	Compatible
	Peat (non-biodegradable)	Fluffy, soft varying texture, dark brown color	Saturated	Compatible
	Clay	Hard, coarse texture, beige color	Saturated and dark in color	Compatible

A color change in the sorbents was noted upon mixing with the waste. However, this was simply due to sorption of the colored paint waste, and there were no signs of waste/sorbent incompatibility.

6.2.3 Paint Related Waste Volume/Mass/Temperature Change

Table 13 shows the change in volume, mass, and temperature of the paint waste when mixed with the sorbents. In some cases, the sorbents added significant volume to the original waste. The polymeric non-encapsulating sorbent showed the lowest volume increase after sorbent addition, 10 to 18 percent.

A reduction in total volume after setting for 24 hours was noted in four out of the five samples. A possible explanation for this effect may be initial sorbent swelling followed by compaction. Most sorbents will immediately begin to swell when wetted. After time, the sorbents may start to compact from their own weight. Therefore, an increase in volume initially occurs, followed by varying degrees of volume decrease with time.

The results for the mass increase in waste are also presented in Table 13. The mass increase data was directly related to the values for one-half L-Test Ratio for each waste/sorbent combination. The non-encapsulating polymeric, which had the most favorable one-half L-Test Ratio, resulted in the lowest weight increase of waste. Clay, which had the least favorable one-half L-Test Ratio, resulted in the highest weight increase. Weight increase can be an important factor in waste transportation costs and waste disposal costs.

6.2.4 Paint Related Waste Liquid Stabilization

The five waste/sorbent combinations were tested for free liquids using the PFT. Table 14 shows the results of these tests. Two paint waste/sorbent combinations, with encapsulating and non-encapsulating polymers, did not pass the PFT; therefore, no further testing (i.e., post-treatment characteristic testing) was performed on these two combinations.

Table 13. Paint Related Waste Volume/Mass/Temperature Changes

Sorbent Category	Time	1/2 L-Test Ratio	Initial Waste Amount		Sorbent Added	Volume Mixing Ratio*		Final Waste Amount		Waste Volume Increase		Waste Mass Increase		Max Temp Increase (°C)
			ml (g)	ml (g)		ml waste/ml sorbent	ml waste/ml sorbent	ml (g)	ml (g)	ml waste/ml waste	Rank	g waste/g waste	Rank	
Zeolitic	0	0.67:1	25.5 (20.0)	50.0 (29.9)	0.51:1	49.0	1.92:1	-	-	-	-	-	8.8	
	24					43.0 (49.0)	1.69:1	3	2.45:1	4	-	-		
Polymeric (encapsulating)	0	1.98:1	26.0 (20.0)	42.0 (10.1)	0.62:1	44.0	1.69:1	-	-	-	-	-	4.8	
	24					40.0 (29.4)	1.54:1	2	1.47:1	3	-	-		
Polymeric (non-encapsulating)	0	7.86:1	25.5 (20.0)	50.0 (2.5)	0.51:1	30.0	1.18:1	-	-	-	-	-	4.3	
	24					28.0 (22.2)	1.10:1	1	1.11:1	1	-	-		
Peat (non biodegradable)	0	3.10:1	25.0 (20.0)	42.0 (6.5)	0.60:1	50.0	2.00:1	-	-	-	-	-	6.6	
	24					47.0 (25.7)	1.88:1	4	1.28:1	2	-	-		
Clay	0	0.50:1 **	25.0 (20.0)	70.0 (40.0)	0.36:1	72.5	2.90:1	-	-	-	-	-	10.6	
	24					73.0 (59.6)	2.92:1	5	2.98:1	5	-	-		

* For the clay sorbent, a mixing ratio of 0.50:1 was used because the 1/2 L-Test Ratio was less than 0.50:1.

** Volume Mixing Ratio = ml initial waste / ml sorbent added.

Table 14. Paint Related Waste Paint Filter Test Results

Sorbent Class	Paint Related Waste	
	Paint Filter Test Results	Waste/Sorbent Mix Ratio
Zeolitic	Passed	0.67 :1
Polymeric (encapsulating)	Failed	1.98 :1
Polymeric (non-encapsulating)	Failed	7.80 :1
Peat (non-biodegradable)	Passed	3.10 :1
Clay	Passed	0.50 :1

6.2.5 Paint Related Hazardous Constituent Stabilization

The final (post-treatment characteristic) testing involved determining if any or all of the hazardous constituents had been stabilized by the sorbent material. For the paint related waste, the testing was designed to measure flammability and VOC concentrations. The polymeric encapsulating and non-encapsulating sorbents were excluded from the post-treatment testing due to failure in the PFT. Table 15 shows the post-treatment ignitability test results with the baseline results and EPA LDR Phase II standards.

Table 15. Paint Related Waste Ignitability Test Results

Test	Baseline Results	Zeolitic	Peat	Clay	EPA LDR Phase II Stds.
Ignitability	< -5.0°C	< 6.0°C	< -2.0°C	< -5.0°C	≥ 60°C

Table 16 shows the post-treatment VOC test results with the baseline results and EPA LDR Phase II standards.

Table 16. Paint Related Waste Hazardous Constituents Stabilization

A	B	C	D	E	F	G	H
Sorbent	VOC	Baseline Waste Conc. (mg VOC/kg soln)	Bulk Mix (Waste+ Sorbent) Conc. (mg VOC/kg mixture)	VOC Reduction Due To Sorbent Addition	Observed Final VOC Conc. (mg VOC/kg mixture)	Apparent VOC Retention by Sorbent	EPA LDR Phase II Sids. (mg VOC/kg mixture)
Zeolitic	Methyl ethyl ketone	333,000	134,000	60%	142,000	< 0%	36
	4-Methyl-2-pentanone	5,690	2,280		< 0%	NA	
	Toluene	9,320	3,740		10%	10	
	Total xylenes	20,000	8,040		4%	30	
	Ethylbenzene	4,040	1,620		4%	10	
	Total VOC	372,000	150,000		< 0%	157,000	
Peat	Methyl ethyl ketone	333,000	252,000	24%	210,000	17%	36
	4-Methyl-2-pentanone	5,690	4,300		< 0%	NA	
	Toluene	9,320	7,050		15%	10	
	Total xylenes	20,000	15,100		4%	30	
	Ethylbenzene	4,040	3,060		5%	10	
	Total VOC	372,000	282,000		16%	238,000	
Clay	Methyl ethyl ketone	333,000	111,000	67%	116,000	< 0%	36
	4-Methyl-2-pentanone	5,690	1,900		< 0%	NA	
	Toluene	9,320	3,110		9%	10	
	Total xylenes	20,000	6,680		0%	30	
	Ethylbenzene	4,040	1,350		4%	10	
	Total VOC	372,000	124,000		< 0%	129,000	

Column D = Baseline Mass Conc [mg/kg soln] X Mass Soln + Mass Waste/Sorbent mixture

Example: For methyl ethyl ketone and zeolitic sorbent

Column D = 333,000 mg methyl ethyl ketone/kg soln X (240.72 g soln + 600 g mixture)

Column D = 134,000 mg methyl ethyl ketone/kg mixture

Column E = (Column C - Column D) / Column C x 100

Column E = (333,000 - 134,000) / 333,000 x 100 = 60%

Column G = (Column D - Column F) / Column D x 100

Column G = (134,000 - 142,000) / 134,000 x 100 = < 0%

Table 16 clearly illustrates that the sorbents did not reduce the concentrations of organics to levels below the LDR Phase II standards (Column H). As a result, the solid mixture would not be landfillable. Moreover, within possible experimental error, most or all of the VOC reduction that was observed was due to dilution by the sorbent, rather than stabilization. This is evidenced by the fairly large differences between baseline waste concentrations (column C) and the calculated bulk mixture concentrations (column D) and the small difference between the calculated bulk mixture concentrations (column D) and the observed concentrations (column F). Among the three samples, sorbent addition alone accounted for anywhere from a 24 to 67 percent reduction in the original VOC concentrations.

Column G, Apparent VOC Retention By Sorbent, is the percent difference between the calculated bulk mixture concentration (column D) and the observed concentration (column F). This numerical difference represents the amount of VOCs that could not be accounted for in the final analysis. In the case of peat, approximately 16 percent of the available total VOCs in the bulk mixture were not detected in the final analysis. Thus, it is possible that some amount of VOC retention on the peat may be occurring. However, such sorption onto peat was not directly measured, and the observed difference could be due to VOC losses during handling. Regardless of where the VOCs ended up, peat, like the zeolitic and clay sorbents, was unable to reduce the VOC concentrations anywhere close to the Phase II LDR standards.

Flammability test results are shown in Table 17. None of the tested sorbents removed the characteristic of ignitability when tested on the Pinsky-Martens closed cup tester. However, because the final mixtures are solid, this test will not suffice to declare the final mixture as hazardous for ignitability. The flame test is implemented at this point to determine if the sample, when ignited, burns with such vigor and persistence that it can create a hazard.

Clearly, all mixtures would be considered hazardous for ignitability. Each mixture ignited before the flame physically touched the sample. Once lit, each mixture burned vigorously and could pose potential hazards.

Table 17. Results of Flame Test with Paint Related Waste

Sorbent in Paint Booth Related Waste	Observation	Result
Zeolitic	Sample ignited while flame was at a distance of 2.5 cm from the sample. Burned more vigorously than paper. Flame was about 50% blue and 50% yellow.	Failed
Peat (non-biodegradable)	Sample ignited while flame was at a distance of 2.5 cm from the sample. Burned more vigorously than paper (popped and cracked). Flame was about 75% blue and 25% yellow.	Failed
Clay	Sample ignited while flame was at a distance of 2.5 cm from the sample. Burned slightly more vigorously than paper. Granular particles would become very hot and pop and crack. Flame was about 75% blue and 25% yellow.	Failed

6.3 Aqueous Heavy Metals Waste

The sample used for this waste was prepared in CTC's laboratory using deionized water, nitric acid, and laboratory metal standards. The metal standards used were cadmium, chromium, lead, nickel, and zinc.

6.3.1 Aqueous Heavy Metal Waste L-Test

The L-Test (sorbency test) results for the five sorbent classes are shown below in Table 18. These results are reported as a ratio of waste to sorbent, and represent the mass of waste that can potentially be sorbed by one gram of sorbent. Also included in this table are the results of taking one half the L-Test ratio and doubling the L-Test ratio. The one-half L-Test Ratios were used in the PFT, with the exception of the zeolitic and clay sorbents, where a 0.50:1 mixing ratio was used for practical reasons. The two-times L-Test Ratios were used in the compatibility tests.

Table 18. Aqueous Heavy Metal Waste L-Test Results

Sorbent Class	Waste/Sorbent Ratios (g waste/ g sorbent)		
	Aqueous Heavy Metals Waste		
	L-Test Ratio	½ L-Test Ratio	2 L-Test Ratio
Zeolitic	0.54: 1	0.27: 1	1.08: 1
Polymeric (encapsulating)	10.9: 1	5.46: 1	21.8: 1
Polymeric (non-encapsulating)	15.5: 1	7.77: 1	31.1: 1
Peat (non-biodegradable)	9.27: 1	4.64: 1	18.5: 1
Clay	0.56: 1	0.28: 1	1.12: 1

As shown in Table 18, the non-encapsulating polymeric sorbent has the highest sorbency ratio for this waste. All things being equal, this ratio suggests that the non-encapsulating polymeric sorbent should provide the smallest volume of waste, though may not necessarily represent the smallest-cost option.

6.3.2 Aqueous Heavy Metals Waste Compatibility Test

The last column in Table 18 shows the ratio for twice the L-Test. This ratio was used to test the compatibility of the five sorbent classes with the aqueous heavy metal waste. Table 19 shows the results of the compatibility test performed on the waste/sorbent mixture.

As the table shows, changes in color and physical appearance of the sorbents were noted after waste addition; however, there were no signs of waste/sorbent incompatibility.

6.3.3 Aqueous Heavy Metals Waste Volume/Mass/Temperature Change

Table 20 shows the change in volume, mass and temperature of the aqueous heavy metal waste when mixed with the sorbents. The sorbents added significant volumes to the original waste. As with the paint waste, the polymeric non-encapsulating sorbent showed the smallest volume increase.

Table 19. Aqueous Heavy Metals Waste Compatibility Test Results

Waste Type and Description	Sorbent Class	Sorbent Description	Waste/Sorbent Post Mix Description	Result
Aqueous Heavy Metals	Zeolitic	Dusty, very fine, dense, pale yellow in color	Saturated and darker in color. Some bubbling during first 15 min.	Compatible
Transparent but bluish-green due to the metals present	Polymeric (non-encapsulating)	Fluffy, medium (0.8 to 3mm dia.), granular, white in color	Became gel-like and translucent.	Compatible
	Polymeric (encapsulating)	Fluffy, soft, very coarse, gray in color	Saturated and darker in color.	Compatible
	Peat (non-biodegradable)	Fluffy, soft, fine to coarse, dark brown in color	Saturated.	Compatible
	Clay	Dusty, fine (up to 2 mm dia.), dense, sandy or granular, pink in color with white specs.	Same but became hard like cement. Bubbled and fumed during first 15 min.	Compatible

An additional finding was the generation of heat when zeolitic and clay sorbents were added to the waste. Upon closer investigation, these two sorbents were found to have high pH levels. These sorbents were mixed with deionized water to form a paste, then the pH level was measured. The pH of the zeolitic paste was 12.4 and the clay paste 13.0. The high pH of the sorbents could explain the heat emitted during the mixing process. One possible explanation is that the mixing of high pH sorbent with a very low pH sample caused a neutralization reaction that generated heat.

6.3.4 Aqueous Heavy Metals Waste Liquid Stabilization

The five waste/sorbent combinations were tested for free liquids using the PFT. Table 21 summarizes the results of these tests. All waste/sorbent combinations passed the test.

Table 20. Aqueous Heavy Metal Waste Volume/Mass/Temperature Changes

Sorbent Category	Time hours	1/2 L-Test Ratio* g waste/ g sorbent	Initial Waste Amount ml (g)	Sorbent Added ml (g)	Volume Mixing Ratio* ml waste/ ml sorbent	Final Waste Amount ml (g)	Waste Volume Increase		Waste Mass Increase		Max Temp Increase (°C)
							ml waste/ ml waste	Rank	g waste/ g waste	Rank	
Zeolitic	0	0.50:1 **	24.0 (25.0)	56.0 (50.0)	0.43:1	60.0	2.50:1	-	-	23.1	
	24						2.21:1	5	2.98:1	5	-
Polymeric (encapsulating)	0	5.46:1	24.0 (25.0)	11.0 (4.6)	2.2:1	43.0	1.79:1	-	-	11.1	
	24						1.42:1	1	1.18:1	2	-
Polymeric (non-encapsulating)	0	7.77:1	24.0 (25.0)	57.0 (3.2)	0.42:1	40.0	1.67:1	-	-	5.1	
	24						1.67:1	2	1.13:1	1	-
Peat (non biodegradable)	0	4.64:1	24.0 (25.0)	36.0 (5.4)	0.67:1	50.0	2.08:1	-	-	6.1	
	24						2.08:1	4	1.21:1	3	-
Clay	0	0.50:1 **	24.0 (25.0)	42.0 (50.1)	0.57:1	58.0	2.42:1	-	-	24.1	
	24						1.88:1	3	2.94:1	4	-

* For the zeolitic and clay sorbents, a mixing ratio of 0.50:1 was used because the 1/2 L-Test Ratio was less than 0.50:1.

** Volume Mixing Ratio = ml initial waste / ml sorbent added.

Table 21. Aqueous Heavy Metals Waste Paint Filter Test Results

Sorbent Class	Aqueous Heavy Metals Waste	
	Paint Filter Test Results	Waste/Sorbent Mix Ratio
Zeolitic	Passed	0.50: 1
Polymeric (encapsulating)	Passed	5.46: 1
Polymeric (non-encapsulating)	Passed	7.77: 1
Peat (non-biodegradable)	Passed	4.64: 1
Clay	Passed	0.50: 1

6.3.5 Aqueous Heavy Metal Hazardous Constituent Stabilization

The post-treatment testing of the aqueous heavy metals waste involved determining whether or not hazardous constituents had been stabilized by the sorbent material. The same tests performed in the baseline analysis, i.e., pH and VOC content, were again performed on the mixtures. These results were compared to the baseline results.

Table 22 compares the of final pH of the waste/sorbent mixtures with the baseline pH of the aqueous heavy metals solution. In the case of zeolitic and clay sorbents, the pH increased significantly. The pH of these two waste streams were 11.7 and 11.8, respectively. As noted before, these same two sorbents also resulted in a significant temperature change when added to the aqueous heavy metals waste. The other three sorbents failed to raise the acidity of the waste above pH 2.

Table 22. Aqueous Heavy Metal Waste pH Test Results

Aqueous Heavy Metals Waste	Measured Value
Baseline pH	BDL (0.00)
Post-Treatment Sorbent/Waste pH	
Sorbent Class	Measured Value
Zeolitic	11.7
Polymeric (encapsulating)	0.99
Polymeric (non-encapsulating)	0.31
Peat (non-biodegradable)	0.41
Clay	11.8

BDL = Below detection limit of instrument

Table 23, Column I, shows the observed metal concentrations in the TCLP extract of the bulk mixture, i.e., aqueous heavy metals waste and sorbent.

To make a meaningful comparison between the final metal testing results and the baseline data, baseline metal concentrations in solution should be recalculated to take into account dilution due to sorbent addition. The initial metals concentration in solution, as presented in column C and D, was calculated from the initial volume of water and the known amount of metal standard spiked into the water. Columns E and F show the metal concentrations in the combined waste/sorbent mixture. Column G illustrates the effect of sorbent addition alone in reducing metals concentrations. Column H shows the theoretical maximum metals concentration if all metals were to leach from the waste/sorbent mixture. Column I shows the observed metals concentration in the acidic test extract, as required by EPA Method 1311, Toxicity Characteristic Leaching Procedure. Column J shows the amount of metals that could not be accounted for and, therefore, assumed to be held by the sorbent. Finally, for most metals there are two different LDR standards that apply, as shown by columns K and L in Table 23. Column K lists the LDR Phase II standard applicable to "characteristically" hazardous waste (D-waste). Column L shows the Universal Treatment Standards that may apply to a metals waste in certain circumstances, as discussed below.

If a metal concentration is high enough to make the waste characteristically hazardous for that metal, then that metal must be lower than the LDR Phase II standard in column K before being landfilled. If the concentration of a metal does not make the waste hazardous but it exists in a hazardous waste, then the metal is an underlying constituent. The number in column L represents the treatment standard applicable to underlying metal contaminants in any hazardous waste. These standards are known as the LDR Universal Treatment Standards. Essentially, the Universal Treatment Standards apply to all heavy metal constituents whose concentrations are not high enough to render the waste hazardous but are nonetheless present.

All LDR Phase II standards listed are based on non-wastewater waste. Non-wastewater wastes are defined as having $\geq 1\%$ total organic carbon and $\geq 1\%$ total suspended solids. The surrogate aqueous heavy metals waste used in this testing would not qualify as a non-wastewater waste, and would not be subject to the standards in column K and L. However, because many aqueous heavy metals waste streams are non-wastewaters, and they represent the most stringent standards, the use of non-wastewater LDRs is a valid baseline comparison.

Table 23. Aqueous Heavy Metal Waste Metals Test Results

A	B	C	D	E	F	G	H	I	J	K	L
Sorbent Category	Metal	Baseline Metal Conc.		Bulk Mix (Waste + Sorbent) Metal Conc.		Metals Reduction Due To Sorbent Addition	Theoretical Maximum Conc., TCLP (mg/l extract)	Observed Conc., TCLP (mg/l extract)	Apparent Metals Retention by Sorbent	LDR Phase II Std. (mg/l extract)	Univ. Treatment Std. (mg/l extract)
		(mg/l soln.)	(mg/g soln.)	(mg/400g mixture)	(mg/g mixture)						
Zeolitic	Cd	102	0.0953	12.7	0.032	67%	1.6	1.2	25%	1.0	0.19
		508	0.475	63.3	0.158		7.9	6.8	14%	5.0	0.86
		540	0.505	67.3	0.168		8.4	1.4	83%	5.0	0.37
		519	0.485	64.7	0.162		8.1	5.7	30%	5.0	5.0
		508	0.475	63.3	0.158		7.9	5.5	30%	5.3	5.3
Polymetric (encapsulating)	Cd	102	0.0953	32.2	0.081	15%	4.0	0.9	78%	1.0	0.19
		508	0.475	160.5	0.401		20.1	3.2	84%	5.0	0.86
		540	0.505	170.6	0.427		21.3	3.0	86%	5.0	0.37
		519	0.485	164.0	0.410		20.5	7.8	62%	5.0	5.0
		508	0.475	160.5	0.401		20.1	8.0	60%	5.3	5.3
Polymetric (non-encapsulating)	Cd	102	0.0953	33.8	0.084	11%	4.2	4.1	2%	1.0	0.19
		508	0.475	168.3	0.421		21	21.1	0%	5.0	0.86
		540	0.505	178.9	0.447		22.4	19.5	13%	5.0	0.37
		519	0.485	171.9	0.430		21.5	20.5	5%	5.0	5.0
		508	0.475	168.3	0.421		21.0	22.1	0%	5.3	5.3
Peat (non-biodegradable)	Cd	102	0.0953	31.4	0.078	18%	3.9	3.4	13%	1.0	0.19
		508	0.475	156.2	0.391		19.5	12.3	37%	5.0	0.86
		540	0.505	166.1	0.415		20.8	4.1	80%	5.0	0.37
		519	0.485	159.6	0.399		20.0	15.8	21%	5.0	5.0
		508	0.475	156.2	0.391		19.5	18.9	3%	5.3	5.3
Clay	Cd	102	0.0953	12.7	0.032	67%	1.6	<0.02	>98.8%	1.0	0.19
		508	0.475	63.3	0.158		7.9	<0.02	>99.7%	5.0	0.86
		540	0.505	67.3	0.168		8.4	<0.02	>99.8%	5.0	0.37
		519	0.485	64.7	0.162		8.1	<0.02	>99.8%	5.0	5.0
		508	0.475	63.3	0.158		7.9	<0.02	>99.7%	5.3	5.3

Example (refer to Table 23): For cadmium with zeolitic sorbent,

Column D = Column C + 1.07 g/ml + 1000 ml/l

= 102mg Cd/l soln + 1.07 g/ml + 1000 ml/l = 0.0953 mg Cd/g soln.

Column E = Column C [mg/liter soln] X liter soln/ 400g waste/sorbent mixture

= 102mg Cd/l soln X (133.33g soln + 1.07 g/ml + 1000 ml/l) +400 g mixture

= 12.7 mg Cd/400 g mixture

Column F = 12.7 mg/Cd/400 g mixture = 0.032 mg Cd/g mixture

Column G = (Column D - Column F) / Column D x 100

= (0.0953 - 0.032) / 0.0953 x 100 = 67%

Column H = Column F (mg/g mixture) X (100g mixture + 2 liter extraction fluid)

= 0.032 mg Cd/g mixture X (100 g mixture + 2 liter extraction fluid)

= 1.6 mg Cd/liter extraction fluid

Column J = (Column H - Column I) / Column H x 100

= (1.6 - 1.2) / 1.6 x 100 = 25%

Of the 25 final metals/sorbent test combinations, ten of them met the LDR Phase II treatment standards. These ten samples, highlighted in boldface in column I, were: Pb/zeolitic; Cd, Cr, and Pb/ polymeric encapsulating; Pb/peat; and Cd, Cr, Pb, Ni, and Zn/ clay. The polymeric non-encapsulating sorbent failed the test for all five metals.

The only samples to meet the Universal Treatment Standards for underlying metal constituents (column L) were from the clay mixture. All five metals met the Standards when clay sorbent was used.

6.4 Degreasing Solvent Waste

The sample used for this waste was prepared in CTC's laboratory using a mixture of 1,1,1-trichloroethane, trichloroethylene, tetrachloroethylene, and motor oil.

6.4.1 Degreasing Solvent Waste L-Test

The L-Test (sorbency test) results for the five sorbent classes are shown in Table 24. These results are reported in a ratio of waste to sorbent, and represent the mass of waste that can potentially be sorbed by one gram of sorbent. Also included in this table are the results of taking one half the L-Test ratio and doubling the L-Test ratio. The modified L-Test ratios are used in later testing.

Table 24. Degreasing Solvent Waste L-Test Results

Sorbent Class	Waste/Sorbent Ratios (g waste/ g sorbent)		
	Degreasing solvent Waste		
	L-Test Ratio	½ L-Test Ratio	2 L-Test Ratio
Zeolitic	2.00 :1	1.0 :1	4.0 :1
Polymeric (encapsulating)	4.01 :1	2.01 :1	8.02 :1
Polymeric (non-encapsulating)	23.9 :1	12.0 :1	47.8 :1
Peat (non-biodegradable)	8.18 :1	4.09 :1	16.4 :1
Clay	1.30:1	0.65 :1	2.60 :1

As shown in Table 24, the non-encapsulating polymeric sorbent has the best sorbency for this waste when compared to the other sorbents tested. This ratio would suggest that this sorbent would lead to the least volume increase in the original waste but may not produce the best cost evaluation.

6.4.2 Degreasing Solvent Waste Compatibility Test

The last column of the above table shows the ratio for twice the L-Test. This ratio was used to test the compatibility of the five sorbent classes with the degreasing solvent. Table 25 illustrates the results of the compatibility test performed on the waste/sorbent mixture.

The importance of a waste/sorbent combination passing the PFT is that it represents one of the first steps in determining whether or not treated hazardous waste can be landfilled. The first step is usually to determine whether or not the sorbent is non-biodegradable. If the resultant mixture did not pass the PFT, the sorbent, in general, failed to meet regulatory compliance. If a waste passed the PFT, it became a candidate for further consideration. At that point, two other criteria must be used to evaluate the sorbed waste for suitability in landfilling: (1) Does the sorbent eliminate the hazardous constituents or characteristics of the original waste?, and (2) Does the sorbent process represent "treatment," in terms of stabilization and simply not dilution? In the use of corrosive wastes, a third criteria must be evaluated, i.e., does the sorbent neutralize the liquid?

Table 30 summarizes the results of the post-treatment hazardous characteristics testing. There were two characteristics by which the wastes/sorbents could be judged, flammability and chemical concentrations (in terms of meeting the LDR standards). If a candidate waste were to fail a test for hazardous constituent stabilization, it could not be disposed in a landfill.

As shown in Table 30, four of the five aqueous waste/sorbent combinations passed the hazardous constituent test for selected metals, but only one combination passed for all five metals. The aqueous waste/clay combination was the only sample where the TCLP extract resulted in all five metals concentrations being below the LDR standards. The non-encapsulating polymeric was the one sorbent that failed for all five metals. Only the clay and zeolitic sorbents neutralized the acidic waste.

Because of the wide variation in positive and negative test results among the different wastes, sorbents were evaluated and ranked relative to other sorbents for each of the three candidate waste streams. For example, sorbents for paint waste treatment were not compared with sorbents for aqueous heavy metals or degreasing solvent waste treatment. As called for in the Statement of Work for this task, the sorbent technologies were ranked in order of decreasing likelihood of being successfully developed and optimized for treating DOD generated hazardous wastes or contaminated media. The objective of the ranking is to specify those highest performing sorbent materials for which an economic analysis will be conducted (Phase III).

Table 26. Degreasing Solvent Waste Volume/Mass/Temperature Changes

Sorbent Category	Time	1/2 L-Test Ratio	Initial Waste Amount		Sorbent Added	Volume Mixing Ratio*		Final Waste Amount	Waste Volume Increase		Waste Mass Increase		Max Temp Increase (°C)
			ml (g)	ml (g)		ml waste/ml sorbent	ml waste/ml sorbent		ml waste/ml waste	Rank	g waste/g waste	Rank	
Zeolitic	0	1.0 : 1	18.0 (25.0)	18.0 (25.0)	39.0 (25.0)	0.46 : 1	0.46 : 1	37.0	2.06 : 1	-	-	5.0	
	24				-			35.0 (48.4)	1.94 : 1	1.93 : 1	4	-	
Polymeric (encapsulating)	0	2.01 : 1	18.0 (25.0)	18.0 (25.0)	57.0 (12.4)	0.32 : 1	0.32 : 1	44.0	2.44 : 1	-	-	4.5	
	24				-			40.0 (36.2)	2.22 : 1	1.45 : 1	3	-	
Polymeric (non-encapsulating)	0	11.96 : 1	18.0 (25.0)	18.0 (25.0)	36.5 (2.1)	0.49 : 1	0.49 : 1	26.0	1.44 : 1	-	-	4.0	
	24				-			23.0 (26.4)	1.28 : 1	1.06 : 1	1	-	
Peat (non biodegradable)	0	4.09 : 1	18.0 (25.0)	18.0 (25.0)	40.5 (6.1)	0.44 : 1	0.44 : 1	44.0	2.44 : 1	-	-	5.0	
	24				-			41.5 (30.2)	2.31 : 1	1.21 : 1	2	-	
Clay	0	0.65 : 1	18.0 (25.0)	18.0 (25.0)	64.0 (38.5)	0.28 : 1	0.28 : 1	70.5	3.92 : 1	-	-	8.0	
	24				-			64.0 (62.6)	3.56 : 1	2.50 : 1	5	-	

* Volume Mixing Ratio = ml initial waste / ml sorbent added

6.4.4 Degreasing Solvent Waste Liquid Stabilization

The five degreasing solvent waste/sorbent combinations were tested for free liquids by use of the PFT. Table 27 shows the results of these tests. Four samples, zeolitic, polymeric encapsulating, peat, and clay, passed the PFT; the polymeric non-encapsulating sorbent sample failed. This one mixture will be excluded from further testing.

Table 27. Degreasing Solvent Waste Paint Filter Test Results

Sorbent Class	Degreasing Solvent Waste	
	Paint Filter Test Results	Waste/Sorbent Mix Ratio
Zeolitic	Passed	1.0 :1
Polymeric (encapsulating)	Passed	2.01 :1
Polymeric (non-encapsulating)	Failed	12.0 :1
Peat (non-biodegradable)	Passed	4.09 :1
Clay	Passed	0.65 :1

6.4.5 Degreasing Solvent Hazardous Constituent Stabilization

The final (post-treatment characteristic) testing involved determining if all or any hazardous constituents were stabilized by the sorbent material. For the degreasing solvent waste, the testing was designed to measure final VOC concentrations. Table 28 shows the comparison of these post-treatment test results with the baseline results and LDR Phase II standards.

As with the other waste/sorbent mixtures, the baseline value is not a valid comparison due to the change in volume from the initial sample. When the sorbent is mixed with the waste, the total volume changes, which changes the concentration of each solvent. Therefore, column E in Table 28 compares the bulk mix concentration with the baseline concentration. In addition, column G compares the final VOC content of the mixture, as measured by GC/MS, with the adjusted baseline analysis. Column G shows the amount of solvent unaccounted for, and therefore possibly retained by the sorbent. However, some reduction may be attributed to evaporative losses.

Table 28 clearly illustrates that the sorbents did not reduce the concentration of organics to levels below the LDR Phase II standards. Although the sorbents did reduce VOC concentrations, the resulting solid mixture would not be landfillable. Moreover, a measureable portion of the VOC reduction was due to dilution by sorbent addition.

Depending on the sorbent, 22 to 31 percent of the total VOCs could not be accounted for in the final analysis. This observation may indicate an ability by these sorbents to hold some organics. However, VOC sorption was not directly measured, and the observed difference could be due to VOC losses during handling.

6.5 Critical Review and Ranking of Sorbents for Treating DOD Candidate Wastes

Table 29 shows the PFT results for all 15 test combinations, as well as, the 11 screening PFTs conducted at the vendor-recommended ratios. Very few, if any, similarities or trends can be drawn based on this information. This suggests two possibilities. First, the three waste streams and the five sorbents selected for testing were so different in characteristics that few comparisons can be made between them in terms of sorbent capacity or ability to retain free liquids. The other possibility is that factors other than waste type or sorbent type must be used to determine whether or not a sample will retain free liquids.

One overriding conclusion that can be drawn from the data in Table 29 is that many more samples passed the PFT when a one-half L-Test Ratio was used rather than a vendor-recommended mixing ratio. Only 3 of 11 samples at vendor-recommended ratios passed the PFT, while 12 of 15 samples passed the PFT at one-half the L-Test Ratio. The three laboratory samples that failed were: paint waste/encapsulating polymeric, paint waste/non-encapsulating polymeric, and degreasing solvent/non-encapsulating polymeric. This suggests that the procedure of performing an L-Test and halving the resulting ratio is a good first approximation for an appropriate waste/sorbent mixing ratio.

Table 29. Vendor-Recommended and Laboratory-Recommended Mixing Ratios and Paint Filter Test Results

Sorbents	Waste Streams					
	Paint Related		Aqueous Heavy Metals		Degreasing Solvents	
	Waste/Sorbent Mix Ratio (g waste/ 1 g sorbent)					
	Vendor	½ L-Test	Vendor	½ L-Test	Vendor	½ L-Test
Zeolitic	Failed 0.90	Passed 0.67	Failed 0.90	Passed 0.50	Passed 0.90	Passed 1.00
Polymeric (encapsulating)	Failed 3.00	Failed 1.98	NR	Passed 5.46	Failed 5.00	Passed 2.01
Polymeric (non- encapsulating)	NR	Failed 7.86	NR	Passed 7.77	NR	Failed 11.96
Peat (non- biodegradable)	Failed 4.52	Passed 3.10	Passed 5.86	Passed 4.64	Failed 7.60	Passed 4.09
Clay	Failed 0.90	Passed 0.50	Failed 2.12	Passed 0.50	Passed 0.90	Passed 0.65

Note: NR = No recommendation provided by sorbent vendor

One possible explanation for the inconsistent performance of the polymeric sorbents in the PFT may be the physical nature of the sorbents and/or a limitation of the test program. In the laboratory, it was noted that the encapsulating and non-encapsulating polymeric sorbents were very light, "fluffy" materials. One possibility is that, while these sorbents may be good at wicking up liquids, they may not have the internal mechanical strength to retain the liquids under the pressure of their own weight over a long period of time, as would be seen in the PFT. Furthermore, the PFT called for observations for free liquids to be made upon 5 minutes of sample sitting. The L-Test, on the other hand, called for measurements of capacity to be made after only 30 seconds of sample sitting/draining. Consequently, liquids held in such mechanically unstable sorbents would be gradually released, causing the sample to fail the PFT. Unfortunately, this theory does not explain why the polymeric sorbents passed the PFT for three other waste/sorbent combinations. One possibility is that the one-half L-Test ratio represents a "borderline" mixing ratio for polymers, where some samples will pass the PFT and other samples will not pass. The current task did not allow for examination of whether or not a modified L-Test, i.e., 5 minutes of sample drain time, or a more conservative mixing ratio favoring the sorbents would have resulted in these three waste/polymeric combinations passing the PFT.

Table 30. Results of Hazardous Constituent Stabilization Testing

Sorbents	Waste Streams					
	Paint Related		Aqueous Heavy Metals*		Degreasing Solvents	
	Test Results					
	Flame	Chemical Conc.	pH	Chemical Conc.	Flame	Chemical Conc.
Zeolitic	Failed	Failed	Passed	1 Passed (Pb)	NA	Failed
Polymeric (encapsulating)	NT	NT	Failed	3 Passed (Cd, Cr, Pb)	NA	Failed
Polymeric (non-encapsulating)	NT	NT	Failed	5 Failed	NA	NT
Peat (non-biodegradable)	Failed	Failed	Failed	1 Passed (Pb)	NA	Failed
Clay	Failed	Failed	Passed	5 Passed	NA	Failed

NA = Not applicable for this waste.

NT = Not tested because failed previous PFT.

* Five metals tested were Cd, Cr, Pb, Ni, and Zn.

Tables 31, 32, and 33 summarize the evaluation of sorbents for each waste stream. In general, sorbents that passed the PFT and/or hazardous constituents stabilization test were favored most highly. In addition, the non-biodegradable peat was given higher preference because of signs that it may absorb and bind organics. The final rankings for each sorbent are shown in the bottom row of each of the three tables. A "1" ranking is the highest, a "5" ranking is the lowest.

Rankings were based on pass or fail. A "pass" on the PFT received 50 points for the organic wastes and 33.3 points for the aqueous waste; a "fail" received 0 points. Points for stabilization of hazardous constituents were equal to 50 points (or 33.3 points for the aqueous waste) divided by the total number of constituent chemicals for each "pass"; "fail" received 0 points per constituent chemical. Aqueous waste samples that were noncorrosive passed and received a full 33.3 points; corrosive aqueous wastes received 0 points. Samples with scores of 50 or less for organic wastes and 67 or less for aqueous waste were deemed unsuitable for ranking (NR).

Table 31. Sorbent Evaluation Matrix for Paint Related Waste

Evaluation Criterion	Weight Percent	Zeolitic	Encapsulating Polymers	Non-Encapsulating Polymers	Peat, Non-biodegradable	Clay
Suitability for landfilling						
A. Immobilization of free liquids	50%	50 (Pass)	0 (Fail)	0 (Fail)	50 (Pass)	50 (Pass)
B. Immobilization of hazardous constituents	50%	0 (Fail)	(NT)	(NT)	0 (Fail)	0 (Fail)
C. Deactivate corrosivity	0%	NA	NA	NA	NA	NA
Score (maximum = 100)		50	0	0	50	50
Relative Rank		NR	NR	NR	NR	NR

NR = Not recommended because failed either the PFT or a hazardous constituents test.

NA = Not applicable, waste not corrosive.

Table 32. Sorbent Evaluation Matrix for Aqueous Heavy Metals Waste

Evaluation Criterion	Weight Percent	Zeolitic	Encapsulating Polymers	Non-Encapsulating Polymers	Peat, Non-biodegradable	Clay
Suitability for landfilling						
A. Immobilization of free liquids	33.3%	33.3 (Pass)	33.3 (Pass)	33.3 (Pass)	33.3 (Pass)	33.3 (Pass)
B. Immobilization of hazardous constituents	33.3%	6.7 (1 Pass)	20 (3 Pass)	0 (0 Pass)	6.7 (1 Pass)	33.3 (5 Pass)
C. Deactivate corrosivity	33.3%	33.3 (Pass)	0 (Fail)	0 (Fail)	0 (Fail)	33.3 (Pass)
Score (maximum = 100)		73	53	33	40	100
Relative Rank		2	NR	NR	NR	1

Table 33. Sorbent Evaluation Matrix for Degreasing Solvents Waste

Evaluation Criterion	Weight Percent	Zeolitic	Encapsulating Polymers	Non-Encapsulating Polymers	Peat, Non-biodegradable	Clay
Suitability for landfilling						
A. Immobilization of free liquids	50%	50 (Pass)	50 (Pass)	0 (Fail)	50 (Pass)	50 (Pass)
B. Immobilization of hazardous constituents	50%	0 (Fail)	0 (Fail)	(NT)	0 (Fail)	0 (Fail)
C. Deactivate corrosivity	0%	NA	NA	NA	NA	NA
Score (maximum = 100)		50	50	0	50	50
Relative Rank		NR	NR	NR	NR	NR

NR = Not recommended because failed either the PFT or a hazardous constituents test.

A summary of the sorbent rankings is shown in Table 34.

Table 34. Summary of Waste/Sorbent Rankings

Waste Stream	Sorbent Ranking				
	Zeolitic	Encapsulating Polymers	Non-Encapsulating Polymers	Peat, Non-biodegradable	Clay
Paint Related Waste	NR	NR	NR	NR	NR
Aqueous Heavy Metals Waste	2	NR	NR	NR	1
Degreasing Solvent Waste	NR	NR	NR	NR	NR

Note: NR = Not recommended because failed PFT.

Based on the Phase II findings in this report, the treatment of aqueous heavy metals waste with zeolitic and clay sorbents appear to be the only potentially feasible candidates for further investigation for landfill disposal. These two waste/sorbent combinations are recommended for the Phase III economic assessment, as noted by check-marks in Table 35 below.

Table 35. Waste/Sorbent Recommendations for Economic Analysis

Sorbent Classes	Waste Streams		
	Paint Related Waste	Aqueous Heavy Metal Waste	Degreasing Solvent Waste
Peat (non-biodegradable)			
Polymeric (non-encapsulating)			
Polymeric (encapsulating)			
Zeolitic		√	
Clay		√	

√ = Recommended

7.0 SUMMARY AND CONCLUSIONS

The objective of Phase II was to conduct bench-scale testing of sorbent materials and waste streams to quantify the effectiveness of sorbent materials for treating hazardous wastes. Five criteria were to be addressed wholly or in part by data generated during the Phase II laboratory bench-scale testing: (1) suitability for immobilizing free liquids, (2) suitability for eliminating characteristically hazardous properties or constituents, (3) sorbent/sorbate material compatibility, (4) waste volume reduction, and (5) sorbent economics.

Prior to conducting the bench-scale testing, efficacy trial testing was performed to verify laboratory procedures and troubleshoot any problems, such as analytical interferences. Testing on a kerosene waste stream and an aqueous heavy metal waste stream led to implementation of the following modifications to the original test plan: (1) the use of nitric acid instead of sulfuric acid for preparing the aqueous heavy metals waste stream, (2) the addition of the L-Test for sorbency testing, and (3) the use of compatibility testing.

Zeolitic, peat, and clay sorbents passed the PFT for the paint related waste. However, these sorbents failed to reduce the flammability or the concentration of organic constituents below the LDR Phase II standards required for landfill disposal of such waste.

All five sorbents, i.e., zeolitic, polymeric encapsulating, polymeric non-encapsulating, peat, and clay, passed the PFT for the aqueous heavy metals waste. However, not all of the sorbents performed adequately in reducing all of the heavy metals concentrations below Phase II standards. Clay and zeolitic were the top ranked sorbents for this waste stream.

Similarly to the paint waste samples, some of the sorbents passed the PFT for degreasing solvent waste, but each of these failed in reducing organic constituents below Phase II standards.

In preparation for the bench-scale PFT mentioned above, it was necessary to develop and verify a procedure to determine the appropriate amount of sorbent to add to each waste sample. The L-Test was subsequently chosen to serve as a basis for making this determination. Because using the exact L-Test Ratio would not provide any safety factor by which sorbents would produce no free liquids, a conservative value of one-half the L-Test Ratio was evaluated against vendor-recommended mixing ratios. Only 3 of the 11 waste/sorbent samples passed the PFT at the vendor-recommended ratios. However, 12 of the 15 samples passed when the waste/sorbent ratio was one-half the L-Test. Therefore, it appears that taking one-half the L-Test ratio may provide a valid means of determining the appropriate mixing ratio.

Based on an analysis of the test results, the following waste/sorbent combinations are recommended for the Phase III economic analysis:

8.0 RECOMMENDATIONS FOR FURTHER WORK

The results and conclusions from this Phase II work effort provide a basis for the Phase III economic assessment. Phase IV on-site demonstration testing is contingent upon the findings of Phase III and various site-specific factors. In addition, several issues discussed in this report are recommended for further study to gain a better understanding of sorbent mechanisms prior to on-site demonstration.

Only two factors, waste type and sorbent type, were varied in this task. Waste-to-sorbent mixing ratio was fixed by the L-Test results and was not varied. In instances where samples failed either the PFT or the post-treatment test for hazardous characteristics, it is possible that lower waste-to-sorbent ratios would have yielded samples that passed these tests.

The effects of time on sorbent performance was not investigated as part of this task. A recommendation contained in literature from one zeolitic sorbent manufacturer stated that the waste matrix after mixing should set for hours to several days to allow the waste matrix to stabilize the hazardous constituents. If samples in this laboratory study were allowed to set undisturbed for a longer period of time, it is possible that more waste/sorbent combinations would have passed the post-treatment characteristic tests.

In the aqueous heavy metals waste testing, the zeolitic and clay sorbents were found to have high pH levels, and the resulting post-treatment pH levels of the sorbed aqueous waste were also very high. One possibility is that, when these two sorbents were added to the aqueous heavy metals waste stream, the high pH caused the metals to precipitate out of solution. The precipitated metals would not be present in the bulk solution analyzed for heavy metals, which would reduce the measured concentrations of dissolved metals. Therefore, for these two sorbents, it may be that the heavy metals reduction is due to a combination of stabilization and precipitation. Further testing and data evaluation incorporating material balances would be required to verify this assumption.

With regard to removal of organic constituents and/or flammability, none of the paint waste/sorbent or degreasing solvent waste/sorbent combinations passed the respective hazardous constituent stabilization tests. This clearly indicates that the tested sorbents do not sufficiently bind organic chemicals under these laboratory test conditions or render them nonhazardous, as is sometimes claimed in sorbent vendor literature. At this time, the only possible exception to this statement might be non-biodegradable peat sorption of organics. For the paint waste/peat and degreasing solvent/peat samples, it appeared that a measurable portion of the total VOC reduction was not due to sorbent dilution and might be due to adsorption/absorption of organics by the peat. This possibility may represent an area for further study, such as whether or not peat could be used on samples with lower organic concentrations, such as cleaning or paint stripping rinsewaters.

Although meeting the requirements for free liquids and hazardous constituent stabilization is of major importance in determining suitability for landfilling, one should not read too

much into the test results for those waste/sorbent samples that failed in this Phase II testing. For example, for those samples that failed the PFT, additional sorbent may have resulted in a strong chance of the mixture passing the PFT. The option is always available in the field to simply add more sorbent to a waste until it passes the PFT. Similarly, there appeared to be a few "borderline" cases in the sorption of aqueous heavy metals where the metals concentrations were only slightly greater than the LDR standards. One possibility is that a slightly higher dose of sorbent would lower the metals' concentrations enough to meet LDR standards. However, the EPA may view this procedure as dilution instead of physical removal of the metals. The role of the PFT and hazardous constituent tests in this task was one of evaluating whether or not a reasonably appropriate waste-to-sorbent ratio would result in a waste suitable for landfilling. More conservative or less conservative mixing ratios could have been used in the PFT to determine the optimum mixing ratio, but this was out of the scope of this task. Therefore, it should be understood that testing of these wastes and sorbents under more favorable conditions might result in a greater percentage of samples passing.

The effect of additives to enhance sorbent performance was not investigated. For example, one zeolitic sorbent manufacturer recommends that water be added to their sorbent to initiate reactions that stabilize the waste matrix. Lack of water was not an issue with sorption of the aqueous heavy metals waste because more than enough water was already present in the matrix. However, no water was present in the paint waste and degreasing solvent waste streams. One possibility is that the addition of water to these two waste/zeolitic combinations would have caused the matrices to stabilize hazardous constituents. Another possibility is that the addition of small amounts of other solidification-type materials to the selected waste/sorbent combinations might enhance sorbency as well as "fix" the hazardous constituents. Such additives might include Portland cement, flyash, kiln dust, and bentonite.

The Phase II laboratory test program was designed to test waste-sorbent combinations for meeting landfill disposal requirements, of which only four combinations were shown to be potentially feasible. Other alternative applications of sorbents may be to add them prior to certain types of on-site or off-site waste treatment processes, such as aggregation, thermal treatment, or bioremediation. Testing different from the type performed in this bench-scale study would be required to assess the technical feasibility of any of these alternatives.

APPENDIX B

**Feasibility Study of Sorbent Treatments for Hazardous Wastes
Phase II Follow-up Report**

1.0 INTRODUCTION

The Phase II Technical Report, which was issued on August 29, 1995, presented the results of the laboratory scale tests which were performed on the five sorbents and the three waste streams. After reviewing this document, the Defense Logistics Agency asked CTC to make this report available to the six sorbent suppliers for their comments and recommendations on the test plan and the reported results.

CTC sent the report to the six vendors on August 31. Follow-up phone calls were made to the vendors on September 7 and September 29. Five of the vendors were satisfied with the reported results and had no concerns. One of the vendors, offered some comments and asked for a re-test of their zeolitic sorbent. The vendor suggested three changes to the testing protocol: (1) different waste/sorbent mixing ratios, (2) the addition of water following the sorbent addition, (3) a seven day cure time.

CTC and the DLA agreed to re-test the zeolitic sorbent under the new directions provided by the vendor and asked that their principal investigator be present to monitor this re-test. These arrangement were made and the re-test occurred on November 20. This report summarizes the testing done and compares the results to the original tests done during August, 1995.

2.0 TEST MATERIALS AND METHODS

2.1 Hazardous Waste Streams

The same three waste streams used in the earlier sorbent testing (Phase II Technical Report, August 29, 1995) were used in this study: paint related waste, aqueous heavy metals waste, and degreasing solvent waste.

2.1.1 Paint Related Waste

A baseline analysis performed of the paint related waste sample used in the zeolitic re-test (Table 1) shows the waste was very comparable in chemical concentration to the paint related waste sample used in the earlier testing. The difference between the re-test and earlier baseline concentrations is representative of the variations in the painting line at CTC's Environmental Technology Facility where the samples were collected.

Table 1 Baseline Analysis of Paint Related Waste

Test	Parameter	Baseline Waste Conc. ***			
		Zeolitic Re-test		8/95 Testing	
*Ignitability (ASTM D-93-90)	Flash Point	< -5.0°C (< 23°F)		< -5.0°C (< 23°F)	
		mg VOC /L soln.	mg VOC /kg soln.	mg VOC /L soln.	mg VOC /kg soln.
**Solvent content (EPA Method 8260A)	Methyl ethyl ketone	482,000	565,000	271,000	330,000
"	4-methyl-2- pentanone	6,340	7,430	4,630	5,700
"	Toluene	6,180	7,250	7,580	9,300
"	Total xylenes	14,400	16,900	16,300	20,000
"	Ethylbenze ne	3,140	3,670	3,290	4,000

* Ignitability test performed using a Pensky-Martens Closed Cup Tester.

** Solvent content performed using a Gas Chromatograph/Mass Spectrometer.

*** Measured values converted from mg/L to mg/kg using the density of the paint waste sample. Units of mg/kg are consistent with Land Disposal Restriction Phase II standards.

2.1.2 Aqueous Heavy Metals Waste

A baseline analysis of the aqueous heavy metals waste used in the re-test shows the waste was very comparable in chemical concentration to the waste used in the earlier testing (See Table 2).

Table 2 Baseline Analysis of Aqueous Heavy Metals Waste

Test	Parameter	Baseline Waste Conc. ***		LDR Standard
		Zeolitic Re- test	8/95 Testing	
*Acidity/ Alkalinity (EPA Method 9040A)	pH	<0.0	<0.0	N/A
		mg/L soln	mg/L soln	mg/L soln
**Heavy Metals Content (EPA Methods 1311 and 200.7)	Cd	99	102	1.0
"	Cr	530	508	5.0
"	Pb	541	540	5.0
"	Ni	527	519	5.0
"	Zn	524	507	5.3

* pH measured with electrometric measurement device

** Metals analysis performed by Inductively Coupled Plasma-Atomic Emission Spectroscopy

***The density of the aqueous heavy metals waste sample was used to convert measured values, in mg/L, to mg/kg.

2.1.3 Degreasing Solvent Waste

A baseline analysis performed of the degreasing solvent waste used in the re-test shows the waste was very comparable in chemical concentration to the waste used in the earlier testing (See Table 3).

Table 3 Baseline Analysis of Degreasing Solvent Waste

Test	Parameter	Baseline Waste Conc.*			
		Zeolitic Re-test		8/95 Testing	
		mg VOC /L soln	mg VOC /kg soln	mg VOC /L soln	mg VOC /kg soln
**Solvent content (EPA Meth. 8260A)	Trichloroethylene	404,000	295,000	406,000	297,440
"	1,1,1-Trichloroethane	410,000	300,000	460,100	336,576
"	Tetrachloroethylene	458,000	335,000	372,600	272,568

* Measured values converted from mg/L to mg/kg using the density of the degreasing solvent waste sample. Units of mg/kg are consistent with Phase II standards.

** Solvent measurement made with Gas Chromatography/Mass Spectroscopy.

2.2 Sorbent Materials

The sorbents used in this testing were two types of zeolitic sorbents from the same sorbent manufacturer. One of the zeolitic sorbents, marketed as better for sorbing organic liquids, was used on the testing for the paint related waste and the degreasing solvent waste. The other sorbent, a zeolitic mixture applicable to sorbing aqueous wastes, was used on the aqueous heavy metals waste.

2.3 Physical Testing Methods

The same physical testing methods used in the earlier testing was used to re-test the zeolitic sorbents. These physical tests were:

- L-Test, per Canadian General Standards Board (CGSB) Method CAN/CGSB-183.2-94 section 9.5.2 — to determine the sorption capacity of each sorbent material.
- The Paint Filter Test (PFT), EPA Method 9095 — to determine whether or not sorbed wastes release any liquids. The PFT was critical to this task because it helped determine regulatory acceptance of the waste.

- **Waste/Sorbent Compatibility** — to observe any obvious signs of incompatibility in the form of visual physical degradation and/or chemical reaction.
- **Volume/Mass/Temperature Change** — to observe changes in the total volume, mass and temperature of the resulting waste.

2.4 Analytical Methods

The same analytical methods used in the earlier testing was used to re-test the zeolitic sorbents. These analytical methods were:

- **Ignitability Testing, ASTM test method D093-90** — To perform the ignitability test, a portion of the sample was taken and exposed to a flame. If the sample ignites before the flame comes into contact with the sample or if the sample burns more vigorously than paper, the sample is considered hazardous. This test was carried out twice, once immediately after mixing and also after the seven day curing time.
- **Corrosivity Testing, EPA Method 9040A**
- **Organic Solvent Testing, EPA Method 8260A**
- **Heavy Metal Testing, EPA Methods 1311 (Toxicity Characteristic Leaching Procedure)**

3.0 BENCH SCALE TEST RESULTS AND DISCUSSION FOR ZEOLITIC SORBENT

The sections below have been organized to discuss the major results of the zeolitic sorbent re-testing. Section 3.1 discusses the waste stream/ sorbent mixing procedures, section 3.2 reviews test data for the paint related waste, section 3.3 reviews the aqueous heavy metal waste test results, and section 3.4 reviews the degreasing solvent waste test results.

3.1 Sorbent Dose Determination Testing

To generate the samples for subsequent free liquids testing and post-treatment chemical analysis, all wastes were treated with the appropriate sorbent at the vendors suggested mix ratio. The paint related waste as well as the degreasing solvents waste had some additional water mixed with the combination. These samples were then allowed to "cure" for a duration of seven days. Table 4 shows the mix ratios for the three waste streams and sorbents. In each column, the first bullet item indicates the actual mass of waste plus the mass of sorbent that were used, plus any additional water. The second bullet is the calculated mixing ratio of waste to sorbent. The third bullet item is the calculated mixing ratio of waste to the combined mass of the sorbent and added water. Finally, the last two rows in Table 4 show the final mass of the sorbed waste mixture and the corresponding mix ratio for the earlier tests. Note that for the re-test, two to three times more sorbent was used than in the earlier tests.

Table 4 Waste Stream/ Sorbent Mixing Ratios

Sorbent	Waste Streams		
	Paint Related	Aqueous Heavy Metals	Degreasing Solvents
Zeolitic re-test	<ul style="list-style-type: none"> • 85 g waste : 276 g sorbent + 137 g H₂O • 0.31 g waste : 1 g sorbent • 0.21 g waste : 1 g sorbent and water 	<ul style="list-style-type: none"> • 107 g waste : 210 g sorbent • 0.51 g waste : 1 g sorbent 	<ul style="list-style-type: none"> • 137 g waste : 200 g sorbent + 45 g H₂O • 0.69 g waste : 1 g sorbent • 0.56 g waste : 1 g sorbent and water
Total Mass	498 g	317 g	382 g
Zeolitic 8/95 testing	0.67 g waste: 1 g sorbent	0.50 g waste: 1 g sorbent	1.0 g waste: 1 g sorbent

3.2 Paint Related Waste Testing

The sample used for this waste was collected from paint waste generated by the organic finishing line at CTC's Environmental Technology Facility. The waste consists primarily of various paints, methyl ethyl ketone, toluene and mixed xylenes.

3.2.1 Paint Related Waste Free Liquid Stabilization

The paint related waste/zeolitic sorbent combination was tested for free liquids by use of the Paint Filter Test. Table 5 shows the results of this test, as well as the test results for the same zeolitic sorbent from the August 1995 report.

Table 5 Paint Related Waste Paint Filter Test Results

Mix Ratio (g waste/ g sorbent)		
Sorbent Class	Zeolitic Re-test*	8/95 Testing
Zeolitic	Passed (0.21 :1)	Passed (0.67 :1)

* Includes amount of water added after addition of sorbent to the waste.

3.2.2 Paint Related Hazardous Constituent Stabilization

The final (post-treatment characteristic) testing involved determining if all or any hazardous constituents were stabilized by the sorbent material. The same tests performed in the baseline analysis were again performed on the mixtures. These results were compared to the baseline results. Table 6 shows the test results for ignitability. The zeolitic sorbent failed to successfully remove the characteristic of ignitability.

Table 6 Paint Related Waste Post-Mix Ignitability Test Results

Sorbent	Observation
Zeolitic re-test	Sample ignited when flame was in contact with sample. Flame was blue in color and would only burn for approximately one second and extinguish. Upon reintroduction of the flame it would behave in a consistent manner for about three times , then no ignition would occur.
Zeolitic 8/95 testing	Sample ignited while flame was at a distance of one inch from the sample. Burned more vigorous than paper. Flame was about 50% blue and 50% yellow.

Table 7 shows the results for chemical stabilization. The zeolitic sorbent did not reduce the concentrations of organics to levels below the LDR Phase II standards. As a result, the solid mixture would not be landfillable.

Table 7 Paint Related Waste Hazardous Constituents Stabilization

Test	Compound	Re-test		8/95 Testing		EPA LDR Phase II Stds.
		Baseline Waste Conc.	Observed Final VOC Conc.	Baseline Waste Conc.	Observed Final VOC Conc.	
		(mg VOC/ kg soln)	(mg VOC/ kg mix)	(mg VOC/ kg soln)	(mg VOC/ kg mix)	
VOCs by GC/MS	Methyl ethyl ketone	565,000	55,200	333,000	142,000	36
"	4-Methyl-2-pentanone	7,430	930	5,690	2,300	NA
"	Toluene	7,250	720	9,320	3,350	10
"	Total xylenes	16,900	2,220	20,000	7,740	30
"	Ethylbenzene	3,670	460	4,040	1,550	10
	Total VOCs	600,000	59,500	372,000	157,000	

3.3 Aqueous Heavy Metal Waste

The sample used for this waste was prepared in CTC's laboratory using deionized water, nitric acid and laboratory metal standards. The metal standards used were cadmium, chromium, lead, nickel and zinc.

3.3.1 Aqueous Heavy Metal Waste Free Liquid Stabilization

The aqueous heavy metals waste/zeolitic sorbent combination was tested for free liquids by use of the Paint Filter Test. Table 8 shows the results of this test, as well as the test results for the same zeolitic sorbent from the August 1995 report. It can be seen that the aqueous waste/zeolite combination successfully passed the test.

Table 8 Aqueous Heavy Metal Waste Paint Filter Test Results

Mix Ratio* (g waste/ g sorbent)		
Sorbent Class	Zeolitic Re-test	8/95 Testing
Zeolitic	Passed (0.51 :1)	Passed (0.50 :1)

* No water was added after addition of sorbent to the waste.

3.3.2 Aqueous Heavy Metal Hazardous Constituent Stabilization

The sorbed aqueous waste stream was first tested for pH. The result is shown below in Table 9. It can be seen that the pH became fairly neutral after treatment (pH 6.01). While this pH value differed from the earlier test sample, both samples would still be considered neutralized by the EPA definition for landfills which is in the range of pH 2.0 to pH 12.0.

Table 9 Aqueous Heavy Metal Waste pH Test Results

Aqueous Heavy Metals Waste	Measured Value
Baseline pH	BDL (0.00)
Post-Treatment Sorbent/Waste pH	
Sorbent Class	Measured Value
Zeolitic re-test	6.01
Zeolitic 8/95 testing	11.67

BDL = Below detection limit of instrument

The aqueous heavy metals sorbent treated waste was tested for Cd, Cr, Pb, Ni, and Zn by ICP-AES from a Toxicity Characteristic Leaching Procedure (TCLP) extract. To make a proper comparison of the final metal testing results with the baseline results, initial concentrations were recalculated due

to the change in mass of the sample analyzed. Most of the reduction in metal concentration is the result of the increased volume from mixing the waste stream with the sorbent prior to the analysis. As part of the TCLP test, additional sample dilution also occurs which appears to reduce the final metal concentrations. These calculations are shown in Table 10. The results are comparable to the earlier test results. Only the lead (Pb) concentration was reduced to a level that meets the LDR requirements for landfill disposal.

Table 10 Aqueous Heavy Metal Waste Metals Test Results

A	B	C	D	E	F	G	H	I	J	K	L
Sorbent Category	Metal	Baseline Metal Conc.		Bulk Mix (Waste + Sorbent) Metal Conc.	Metals Reduction Due to Sorbent Addition	Theoretical Maximum Conc., TCLP	Observed Conc., TCLP	Apparent Metals Retention by Sorbent	LDR Phase II Std.	Univ. Treatment Std.	
		(mg/l soln)	(mg/g soln)	(mg/317g mixture)	(mg/g mixture)	(mg/l extract)	(mg/l extract)	(mg/l extract)	(mg/l extract)	(mg/l)	
Zeolitic Re-test	Cd	99	0.0925	9.9	0.031		1.6	1.1	31%	1.0	0.19
	Cr	530	0.495	53.0	0.167	66%	8.4	7.2	14%	5.0	0.86
	Pb	541	0.506	54.1	0.171		8.5	1.2	86%	5.0	0.37
	Ni	527	0.492	52.7	0.166		8.3	7.0	16%	5.0	5.0
	Zn	524	0.490	52.4	0.165		8.3	5.6	33%	5.3	5.3
Zeolitic 8/95 Testing	Cd	102	0.0953	12.7	0.032	67%	1.6	1.2	25%	1.0	0.19
	Cr	508	0.475	63.3	0.158		7.9	6.8	14%	5.0	0.86
	Pb	540	0.505	67.3	0.168		8.4	1.4	83%	5.0	0.37
	Ni	519	0.485	64.7	0.162		8.1	5.7	30%	5.0	5.0
	Zn	508	0.475	63.3	0.158		7.9	5.5	30%	5.3	5.3

Column D = Column C + 1.07 g/ml + 1000 ml/l
 Column E = Column C x 1 liter soln + 317 g waste-sorbent mixture
 Column F = Column E + 317 g mixture
 Column G = (Column D - Column F) + Column D x 100
 Column H = Column F x (100g mixture + 2 liters extraction fluid)
 Column J = (Column H - Column I) + Column H x 100

3.4 Degreasing Solvent Waste

The sample used for this waste was prepared in CTC's laboratory using a mixture of 1,1,1-trichloroethane, trichloroethylene, tetrachloroethylene, and motor oil.

3.4.1 Degreasing Solvent Waste Free Liquid Stabilization

The degreasing solvent waste/zeolitic sorbent combination was tested for free liquids by use of the Paint Filter Test. Table 11 shows the results of these tests.

Table 11 Degreasing Solvent Waste Paint Filter Test Results

Mix Ratio (g waste/ g sorbent)		
Sorbent Class	Zeolitic Re-test*	8/95 Testing
Zeolitic	Passed (0.56 :1)	Passed (1.0 :1)

* Includes amount of water added after addition of sorbent to the waste.

3.4.2 Degreasing Solvent Hazardous Constituent Stabilization

The final testing involved determining if all or any hazardous constituents have been stabilized by the sorbent material. The same tests performed in the baseline analysis are again performed on the mixtures. The mixture was sampled immediately after mixing and then after the seven day cure time. Table 12 shows the final test results, along with data from the August 1995 report.

Table 12 Degreasing Solvent Waste Hazardous Constituents Stabilization

Test	Compound	Re-test		8/95 Testing		EPA LDR Phase II Stds.
		Baseline Waste Conc.	Observed Final VOC Conc.	Baseline Waste Conc.	Observed Final VOC Conc.	
		(mg VOC/ kg soln)	(mg VOC/ kg mix)	(mg VOC/ kg soln)	(mg VOC/ kg mix)	
VOCs by GC/MS	1,1,1-Trichloroethane	295,000	61,500	273,000	79,700	6.0
"	Trichloroethylene	300,000	58,200	297,000	106,000	6.0
"	Tetrachloroethylene	335,000	93,400	337,000	153,000	6.0
	Total VOCs	930,000	213,000	907,000	339,000	

Table 12 clearly illustrates that the zeolitic sorbent did not successfully reduce the concentrations of organics to levels below the LDR Phase II standards. As a result, the solid mixture would not be landfillable.

4.0 SUMMARY

This re-testing of the zeolitic sorbent was performed to validate the earlier waste/sorbent treatment tests reported in August, 1995. During this re-test, three test conditions were changed from the earlier testing: (1) vendor-recommended mixing ratios were used, (2) water was added to the sorbed organic wastes, and (3) a seven-day cure time was instituted.

Although the vendor was present during this round of testing and the the conditions were potentially more favorable, the results were essentially the same as in the earlier testing. In all cases, the zeolitic sorbent did not sufficiently stabilize the hazardous constituents in any of the waste streams to allow landfill disposal. The results indicated that the three factors — additional sorbent, the addition of water, and a seven-day cure time — did not significantly change the treatment effectiveness of the zeolitic sorbents.

APPENDIX C

**Feasibility Study of Sorbent Treatments for Hazardous Wastes
Phase II Report: Addendum Cellulosic Sorbent Testing**

EXECUTIVE SUMMARY

In 1995, Concurrent Technologies Corporation (*CTC*) was tasked by the Defense Logistics Agency (*DLA*) to evaluate the potential use of sorbent materials for treating hazardous wastes. The results of *CTC*'s bench scale testing of several classes of nonbiodegradable sorbents were presented in the Phase II Technical Report, Feasibility Study of Sorbent Treatment for Hazardous Wastes (August 29, 1995). After this work was completed, the U.S. Environmental Protection Agency modified the definition of "biodegradable sorbent." Under this new definition, some sorbents that were previously "biodegradable" and, therefore, not tested in the earlier round, might be considered "nonbiodegradable." In response to this change, the *DLA* directed *CTC* to determine the best sorbent(s) that now met this new definition and perform bench scale testing on the material(s). One sorbent product — a cellulosic sorbent — was selected for bench scale testing at *CTC* since it passed the selection criteria which had been established to screen candidate sorbents. This report summarizes those test results.

The cellulosic sorbent was tested for its ability to treat three types of liquid hazardous wastes: paint related waste; acidic, heavy metal containing wastes; and degreasing solvent wastes.

The results of the laboratory testing showed that the cellulosic sorbent did not sufficiently stabilize the hazardous constituents in any of the waste streams to allow landfill disposal. This performance was similar to the results for the other nonbiodegradable sorbents tested earlier by *CTC* with the exception of the clay products, which were able to stabilize the acidic heavy metal containing wastes.

2.0 TEST MATERIALS AND METHODS

Tests were conducted to help quantify three sorbent material parameters: the relative sorbency of the sorbent material, sorbent stabilization of free liquids, and sorbent stabilization of hazardous constituents within the wastes. Prior to the actual performance testing of the sorbent, laboratory tests were run to confirm vendor recommended mixing ratios, evaluate compatibility with the waste, and establish correct mixing ratios of waste to sorbent for subsequent testing.

2.1 Hazardous Waste Streams

The same three waste streams used in the earlier sorbent testing (Phase II Technical Report, August 29, 1995) were used in this study: paint related waste, aqueous heavy metals waste, and degreasing solvent waste. The paint waste was collected from the spray gun cleaning operations of the Organic Finishing Process at Concurrent Technologies Corporation's (CTC's) Environmental Technologies Facility. The aqueous heavy metals and the degreasing solvent waste streams are surrogates, which were prepared by the laboratory at CTC, as described in the Phase II Technical Report.

2.1.1 Paint Related Waste

A baseline analysis was performed on the paint related waste (Tables 1 and 2). Baseline testing consisted of a Volatile Organic Compound (VOC) scan by gas chromatograph/mass spectroscopy (GC/MS) and flash point determination using a Pensky - Martens Closed Cup Tester. The two major characteristics of interest in the waste were the high concentrations of solvents, particularly methyl ethyl ketone (MEK), and the associated low flash point of the waste.

Table 1 Baseline VOC Scan Results For Paint Related Waste Stream

Compound	Measured VOC Concentration (mg/l soln.)	Measured VOC Concentration (mg/kg soln.)†
Methyl ethyl ketone	368,000	433,000
4-Methyl-2-pentanone	6,100	7,170
Toluene	6,640	7,810
m-Xylene	4,200	4,940
p-Xylene	4,200	4,940
o-Xylene	3,420	4,020
Ethylbenzene	3,480	4,090

† Values converted from mg/l to mg/kg using the density of the sample.

Table 2 Baseline Flash Point Testing Results For Paint Related Waste Stream

Test	Measured Flash Point
Flash Point (Closed Cup)	-3.2 °C

2.1.2 Aqueous Heavy Metals Waste

A baseline analysis was performed on the aqueous heavy metals waste (Tables 3 and 4). The waste consisted of five heavy metals, cadmium (Cd), chromium (Cr), lead (Pb), nickel (Ni), and zinc (Zn) in a 10 % by volume of concentrated nitric acid (HNO₃) matrix. The aqueous heavy metals baseline testing consisted of quantification of the metals by inductively coupled plasma- atomic emission spectroscopy (ICP-AES) and pH determination by electrometric measurement.

Table 3 Baseline ICP-AES Testing Results For Aqueous Heavy Metals Waste Stream

Metal	Measured Concentration (mg/l soln.)
Cadmium	100
Chromium	506
Lead	522
Nickel	510
Zink	502

Table 4 Baseline pH Testing Results For Aqueous Heavy Metals Waste Stream

Test	Measured pH
pH (Electrometric)	Below Detection (< 0.00) (Corrosive)

2.1.3 Degreasing Solvent Waste

A baseline analysis was performed on the degreasing solvent waste (Table 5). The degreasing solvent waste stream consisted of a mixture of (by volume) 30% trichloroethylene, 30% tetrachloroethylene, 30% 1,1,1-trichloroethane, and 10% SAE 30W motor oil. The degreasing solvents baseline testing consisted of a VOC scan by GC/MS.

Table 5 Baseline VOC Scan Results For Degreasing Solvent Waste Stream

Compound	Measured VOC Concentration (mg/l soln.)	Measured VOC Concentration (mg/kg)†
Trichloroethylene	392,000	279,000
Tetrachloroethylene	626,000	445,000
1,1,1-Trichloroethane	393,000	279,000

† Values converted from mg/l to mg/kg using the density of the sample.

2.2 Sorbent Materials

To determine the most appropriate sorbent(s) for the laboratory tests, the sorbent materials identified in response to the October 1995 CBD notice were evaluated using several screening criteria. These criteria were:

1. Does the material have sorbent properties? (If "no", then eliminate from further consideration.)
2. Is the sorbent material used to absorb liquids? (If "no", then eliminate from further consideration.)
3. Was the sorbent class (i.e., peat, zeolitic, clay, polymeric) previously tested by CTC? (If "yes", then eliminate from further consideration.)
4. Does the sorbent meet the conditions of nonbiodegradability as defined by the modified Sturm Test? (If "no", then eliminate from further consideration.)
5. Is the sorbent commercially available? (If "no", then eliminate from further consideration.)

From this screening process, only one sorbent material — a cellulosic — was selected for laboratory scale testing. Physical observations of the sorbent are noted below in Table 6.

Table 6 Cellulose Sorbent Physical Observations

Sorbent Category	Sorbent Description
Cellulosic	Paper like, hard, or rigid, very coarse, particles ranging from 3 mm to 20 mm in size; light gray in color; contains particles of wood that make up a very small percentage of the sorbent.

2.3 Waste Stream/ Sorbent Physical Testing

Each of the three waste streams was treated with the cellulosic sorbent. This treatment resulted in a total of three waste/ sorbent combinations for which a total of five test procedures were performed, as explained below.

2.3.1 "L-Test" for Sorbency

Sorption capacity can be defined as the maximum mass of waste that can be absorbed per gram of sorbent. The L-Test, per Canadian General Standards Board (CGSB) Method CAN/CGSB-183.2-94, Section 9.5.2, was used to determine the sorption capacity of the cellulosic sorbent material. The L-Test is important because *CTC* showed in its previous sorbent study that a value of one-half the L-Test ratio was an appropriate waste/sorbent mix ratio, and often provided more favorable results than the vendor-recommended ratios.

2.3.2 Paint Filter Test

The Paint Filter Test (PFT) was used to determine whether or not sorbed wastes release any liquids. The procedure for the PFT is outlined in EPA Method 9095. To pass the PFT, no free liquid must pass through the paint filter during 5 minutes.

2.3.3 Waste/Sorbent Compatibility

A compatibility test was performed on the waste/sorbent samples to observe signs of incompatibility. Incompatibility was defined as the obvious visual decomposition or deterioration of a sorbent that would likely hamper the performance of the sorbent material. For these tests, waste/sorbent mixing ratios were set at twice the L-Test ratio to intentionally overexpose the sorbent. Observations were made during the initial mixing, and at 5 minute intervals for 15 minutes. After a 24 hour standing period, the mixture again was observed.

2.3.4 Volume/Temperature Change

Observations were made, after adding the sorbent, to determine if there was any change in the total volume and temperature of the resulting waste. Extreme changes in temperature can adversely affect the way in which the waste/sorbent can be handled. Changes in volume can directly impact disposal costs.

2.4 Analytical Methods

The same analytical methods used in the earlier testing were used in this testing. These analytical methods were:

- **Ignitability Testing, ASTM test method D093-90** — To perform the ignitability test, a portion of the sample was taken and exposed to a flame. If the sample ignites before the flame comes into contact with the sample or if the sample burns more vigorously than paper, the sample is considered hazardous.
- **Corrosivity Testing, EPA Method 9040A**
- **Organic Solvent Testing, EPA Method 8260A**
- **Heavy Metal Testing, EPA Methods 1311 (Toxicity Characteristic Leaching Procedure)**

3.0 BENCH SCALE TEST RESULTS AND DISCUSSION FOR CELLULOSIC SORBENT

The sections below have been organized to discuss the major results of the cellulosic sorbent testing. Section 3.1 discusses the results of the waste/sorbent dose determination testing including the "L-test", Section 3.2 shows the results of the material compatibility testing, Section 3.3 contains the results of the waste volume/mass/temperature change, Section 3.4 contains the results of the Paint Filter Test, and Section 3.5 contains the results of the post-treatment testing for landfill suitability.

3.1 Sorbent Dose Determination Testing

3.1.1 Evaluation of Vendor-Recommended Mixing Ratios

Results of the confirmation testing of vendor mixing ratios were determined for each of the three waste streams (Table 7). The cellulosic sorbent vendor provided initial recommendations for the amount to add to each waste sample, so that the sorbed waste would produce no free liquids during the Paint Filter Test. For each waste stream tested, a 3/1 ratio of waste to sorbent was used.

Table 7 Waste Stream Sorbent Testing Results at Vendor Recommended Ratios

Waste Stream	Sorbent Category: Cellulosic	Paint Filter Test
Paint Related	Vendor Ratio: 3.0g waste/ g sorbent Mixing Ratio: 3.0:1 Result: Free Liquid Description: Waste & sorbent formed a somewhat soft wet mixture.	Failed
Aqueous Heavy Metals	Vendor Ratio: 3.0g waste/ g sorbent Mixing Ratio: 3.0:1 Result: Free Liquid Description: Waste & sorbent formed a soft wet mixture.	Failed
Degreasing Solvents	Vendor Ratio: 3.0g waste/ g sorbent Mixing Ratio: 3.0:1 Result: Free Liquid Description: Waste & sorbent formed a wet mixture which remained hard.	Failed

As Table 7 shows, none of the samples passed the confirmatory Paint Filter Test (PFT) at the vendor-recommended sorbent doses. Therefore, as in CTC's earlier testing, a more appropriate mixing ratio for each waste stream/sorbent combination was determined using the "L-Test".

3.1.2 "L-Test" for Sorbency

Table 8 shows the results of the "L-Test" sorbency analysis. Ratios at one half the "L-Test" ratios were used for the volume/ physical change test and the final waste stream/ sorbent mixing. Ratios at twice the "L-Test" ratios were used for the waste stream/ sorbent compatibility testing discussed below.

Table 8 "L - Test" Ratios

Sorbent Category	Waste Streams		
	Paint Related	Aqueous Heavy Metals	Degreasing Solvents
Cellulosic	2.28 g waste : 1.00 g sorbent	2.76 g waste : 1.00 g sorbent	2.90 g waste : 1.00 g sorbent

3.2 Waste/Sorbent Material Compatibility

Table 9 shows the results from the waste stream/ sorbent compatibility screening. All sorbent/waste combinations were found to be compatible.

Table 9 Waste Stream/Sorbent Compatibility Testing Results

Waste ID	Paint Related	Aqueous Heavy Metals	Degreasing Solvents
Waste Description	Greenish-brown: cloudy gray paint pigment settles while standing	Transparent but bluish-green due to the metals present	Transparent but yellow from the oil added
Sorbent/Waste Post Mix Description	Particles remained basically the same but became somewhat soft. Free liquid remained.	Particles remained basically the same but become soft and turned a pale orange color. When the waste stream and sorbent were mixed, fuming occurred. Very little free water remained.	Particles remained basically the same (remained hard) but become wet. Free liquid remained.
Conclusion	Compatible	Compatible	Compatible

3.3 Volume/Physical Change

Table 10 shows the results for the volume/physical change testing. The aqueous heavy metals waste and sorbent mixture fumed and bubbled when the combination was mixed. The change in volume of waste after adding sorbent material was greatest in the degreasing solvent stream.

Table 10 Volume/ Physical Change Testing Results

Waste ID	Paint Related		Aqueous Heavy Metals		Degreasing Solvents	
Starting Volume of Waste (ml)	10		10		10	
Starting Mass of Waste (g)	8.588		10.589		14.135	
Time (t)	t = 0 min	t = 24 hr	t = 0 min	t = 24 hr	t = 0 min	t = 24 hr
Starting Volume of Sorbent (ml)	58		58		67	
Starting Mass of Sorbent (g)	7.531		7.675		9.746	
Final Volume of Waste/ Sorbent (ml)	57	52	58	48	71	68
Final Mass of Waste/ Sorbent (g)		15.595		17.970		23.008
Waste Volume Increase Ratio*	5.7	5.2	5.8	4.8	7.1	6.8
Max. Temp Increase (°C)	1.0		2.0		0.5	
Mass Increase Ratio		1.8		1.7		1.63

* Waste volume increase ratio defined as ratio of final volume of waste and sorbent to the starting volume of waste.

3.4 Stabilization of Free Liquid

To generate the samples for the Paint Filter Test, all wastes were treated with the appropriate sorbent at one-half the "L-Test" ratio. Table 11 shows the appropriate mixing ratio calculated for each waste stream/sorbent combination. The first bullet is the calculated mixing value at half the "L-Test", the second bullet indicates the actual mass of waste to mass of sorbent that was used for mixing.

Table 11 Waste Stream/ Sorbent Mixing Procedure Data

Sorbent Category	Waste Streams		
	Paint Related	Aqueous Heavy Metals	Degreasing Solvents
Cellulosic	<ul style="list-style-type: none"> • 1.14 g waste: 1.00 g sorbent • 213.08 g waste: 186.92 g sorbent 	<ul style="list-style-type: none"> • 1.38 g waste: 1.00 g sorbent • 231.93 g waste: 168.07 g sorbent 	<ul style="list-style-type: none"> • 1.45 g waste: 1.00 g sorbent • 236.73 g waste: 163.27 g sorbent

Once the wastes were treated with the appropriate sorbent, all mixtures were subjected to a Paint Filter Test (PFT) to determine the presence of free liquids. If the mixture failed the PFT, indicating free liquids, no further tests would be conducted. It can be seen in Table 12 that the three waste stream/sorbent combinations passed the PFT at one half the "L-Test" ratio mix.

Table 12 PFT Results

Sorbent Category	Waste Streams		
	Paint Related	Aqueous Heavy Metals	Degreasing Solvents
Cellulosic	Pass	Pass	Pass

3.5 Stabilization of Hazardous Constituents

The final (post-treatment characteristic) testing involved determining if any or all of the hazardous constituents had been stabilized by the cellulosic sorbent. For the paint related waste, the testing was designed to measure flammability and VOC concentrations. For the aqueous heavy metals waste, measurements were made of the pH of the sorbed waste and the

metals concentration in the TCLP extract. For the degreasing solvent waste, VOC tests were performed on the sorbed waste.

3.5.1 Ignitability

The paint related waste/sorbent sample ignited and thus failed the test for ignitability (Table 13). Therefore, this waste could not be landfilled.

Table 13 Post Mix Ignitability Test

Sorbent Category with Paint Related Waste	Observed Value
Cellulosic	Sample ignited while flame was at a distance of one inch from the sample. Burned more vigorously than paper. Flame was about 90% blue and 10% yellow. Same sample would burn this way at least three consecutive times.
Fail	

3.5.2 Leachable Metals

The sorbed, aqueous, heavy metals waste was subjected to a Toxicity Characteristic Leaching Procedure (TCLP) extraction to determine if any metals (Cd, Cr, Pb, Ni, and Zn) leached from the waste (Table 14). The amount of metals spiked into the sorbent is dependent on the "L-Test" ratio. The waste stream and sorbent were mixed at a ratio of 1.38 g waste to 1 g sorbent to yield a final mass of 400 grams. The milligrams of metal per gram of waste/sorbent mixture is found in Column 4. If the sorbent were to completely fail in the stabilization of hazardous constituents, then all of the metal would leach out during the TCLP, yielding the concentration given in Column 5. The observed metals concentrations are shown in Column 6, and compared to the Landfill Disposal Restrictions (LDR) in Column 7.

As the table shows, although overall metals concentrations were reduced, the TCLP results did not meet the LDR standards.

Table 14 Post Mix TCLP Metals by ICP-AES

1	2	3	4	5	6	7	8
Metal	Initial Conc. of Waste (mg/l)	Conc. of Metal In Total Waste/ Sorbent Mixture (mg/400 g)	Conc. Of Metal /g of Waste/ Sorbent Mixture (mg/g)	Theoretical Max. TCLP Conc. (mg/l)	Observed Conc., TCLP (mg/l)	LDR Phase II Std. (mg/l)	Conclusion
Cd	100	21.7	0.054	2.7	2.4	1.0	Fail
Cr	506	109.7	0.274	13.7	7.2	5.0	Fail
Pb	522	113.1	0.283	14.1	7.4	5.0	Fail
Ni	510	110.5	0.276	13.8	12.4	5.0	Fail
Zn	502	108.8	0.272	13.6	13.1	5.3	Fail

Mixing ratio 1.38 g waste: 1 g sorbent

3.5.3 pH

The sorbent treated aqueous heavy metals wastes were tested for pH (Table 15). The results showed that pH of the sorbed waste was very low (corrosive). Thus, the cellulosic sorbent only partially neutralized the waste's acidity.

Table 15 Post Mix pH Results

Aqueous Heavy Metals Waste	Measured Baseline pH
Baseline pH	Below Detection (< 0.00) (Corrosive)
Sorbent Category	Measured Final pH
Cellulosic	1.62 (Corrosive)
	Fail

3.5.4 Organic Constituent Stabilization

Both the paint related waste and degreasing solvent waste were scanned for VOCs following sorption. The results for the paint waste are shown in Table 16. The cellulosic sorbent clearly failed to reduce the VOC concentrations in this waste to appropriate levels.

Table 16 Post Mix VOC Scan Results for Sorbed Paint Related Waste

	VOC Concentration	EPA LDR Phase II Standard	
Compound	(mg VOC/kg mix)	(mg VOC/kg mix)	Conclusion
Methyl ethyl ketone	96,100	36	Fail
4-Methyl-2-pentanone	3,550	NA	Fail
Toluene	4,020	10	Fail
m-Xylene	3,260	30 (total xylenes)	Fail
p-Xylene	3,260	30 (total xylenes)	Fail
o-Xylene	2,210	30 (total xylenes)	Fail
Ethylbenzene	2,000	10	Fail

The results for the degreasing solvent waste are shown in Table 17. The cellulosic sorbent failed to reduce the VOC concentrations in this waste to appropriate levels.

Table 17 Post Mix VOC Scan Results for Sorbed Degreasing Solvent Waste

	VOC Concentration	EPA LDR Phase II Standard	
Compound	(mg VOC/kg mix)	(mg VOC/kg mix)	Conclusion
Trichloroethylene	174,000	6.0	Fail
Tetrachloroethylene	243,000	6.0	Fail
1,1,1-Trichloroethane	149,000	6.0	Fail

4.0 SUMMARY AND CONCLUSION

This testing of cellulosic sorbent was performed to complement the earlier waste/sorbent treatment tests reported in August, 1995. All of the test conditions described in the approved Test Plan were used for the cellulosic sorbent testing.

The cellulosic sorbent exhibited essentially the same performance as the nonbiodegradable peat (another organic sorbent) in the earlier testing. In all cases, the cellulosic sorbent did not sufficiently stabilize the hazardous constituents in any of the waste streams to allow landfill disposal.