Research and Test Department

A REVIEW OF TOXICITY CHARACTERISTIC LEACHING PROCEDURE TESTING OF RAILROAD CROSSTIES

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13. ABSTRACT

Disposal of railroad crossties has represented a matter of concern to the railroad industry since the early days of environmental land disposal regulations. In 1986 EPA introduced the Toxicity Characteristic Leaching Procedure (TCLP) as a method to identify hazardous waste. To address the new methodology, the Association of American Railroads (AAR) began a testing program in 1987 to determine if crossties would be considered hazardous under the TCLP. That testing revealed that the crossties were not hazardous waste. The EPA subsequently proposed a change to the TCLP called the "cage modification" which involved using a stainless steel cage within the testing apparatus to abrade a chunk of the material being tested instead of grinding the chunky material prior to testing. The AAR continued the testing program to examine how crossties would react in the newly proposed "cage modification". Again, crossties tested not hazardous. EPA subsequently abandoned the "cage modification" and finalized the originally proposed TCLP method in 1990 with several revisions to the regulatory limits. In the intervening years, testing programs have been undertaken by railroads and others including: the Chicago and NorthWestern Railway Company (C&NW), Consolidated Rail Corporation (Conrail), the Atchison, Topeka and Santa Fe Railway (AT&SF), and the New York State Energy Research and Development Authority (NYSERDA). Data from each of these investigations draw the same conclusion: that waste crossties (new or used) are not hazardous waste.

14. SUBJECT TERMS
Railroad crossties, hazardous waste, creosote, wood preservatives, Toxicity Characteristic Leaching Procedure, TCLP, RCRA, solid waste, disposal, environmental impact

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EXECUTIVE SUMMARY

The ability to dispose of used crossties as conventional solid waste instead of regulated hazardous waste is of great importance to the railroad industry. If crossties had been determined to be hazardous waste using the testing methods required by the EPA, the cost to dispose of used railroad crossties would be enormous.

This report describes six different testing programs involving Toxicity Characteristic Leaching Procedure (TCLP) testing of railroad crossties. Data from at least 28 individual TCLP tests, representing at least two dozen new and used crossties from all over the U.S. were examined. The results from all of these tests confirm that crossties are not a hazardous waste.

Data from these TCLP testing programs are summarized in the report. Tests were conducted from 1987 through 1992. The first two studies were commissioned by the AAR. In 1987 the AAR tested chipped ties using essentially the same TCLP physical extraction protocol as exists today. In 1988 the AAR tested crossties using an alternative "cage modification" TCLP method that was proposed (and subsequently abandoned) by the Environmental Protection Agency (EPA). After the final 1990 TCLP rules were promulgated, further testing programs were undertaken by several railroads and others including: the Chicago and NorthWestern Railway Company (C&NW), Consolidated Rail Corporation (Conrail), the Atchison, Topeka and Santa Fe Railway (AT&SF), and the New York State Energy Research and Development Authority (NYSERDA); these programs, as well as the two commissioned by the AAR, are the subject of this report.

The results of all of the TCLP testing programs were similar, including those utilizing modifications of the TCLP such as the AAR's 1988 "cage modification" study, and the AT&SF 1992 study involving both the TCLP and the California Waste Extraction Test (WET). Only a small group of the 39 TCLP parameters were measurable: cresol, arsenic, barium, lead, mercury, and selenium. In most cases where present, these existed at low, near-detection-limit concentrations in the extracts. In no case did any crosstie sample approach failure of the TCLP.

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1.0 INTRODUCTION

Disposal of used railroad crossties has represented a matter of concern to the railroad industry since the early days of environmental land disposal regulations. In 1986, with the development of the Environmental Protection Agency's (EPA's) new rules and test procedures for determining whether or not a waste material is considered hazardous, it became apparent that some of the characteristics of used crossties might cause them to join a lengthening list of hazardous waste materials, the land disposal of which is either regulated or prohibited. This report describes several testing programs that were conducted by the Association of American Railroads (AAR) and others to assess the effect of the EPA mandated Toxicity Characteristic Leaching Procedure (TCLP) testing on crosstie disposal.

1.1 BACKGROUND

The EPA introduced the Extraction Procedure (EP) toxicity characteristic test in 1976 as a means for identifying hazardous wastes. That test, like its eventual successor, the TCLP, was developed with the intent of simulating the mobility of hazardous chemical compounds in a landfill environment.

The EPA's test methodology for determining whether or not a waste material is considered hazardous in terms of its toxic characteristics has undergone change and revision since its inception. On January 14, 1986, the proposed TCLP test *[1]*, also referred to as Method 1311 (see Appendix B), was introduced as a method to be used in determining whether applicable treatment standards had been met for land disposal of wastes in compliance with the new Land Disposal Restrictions Program. Subsequently, on June 13, 1986 *[2]*, the EPA proposed a new Toxicity Characteristic Rule for determining whether or not a waste is hazardous. In that rule, the EPA suggested that they would replace the EP toxicity characteristic test, then in effect, with the newly developed TCLP test. As part of the same rule-making proposal, the EPA recommended that certain additional chemicals be considered in determining if a waste was hazardous. Included in that list of TCLP extractable organics were the o-, m-, and p- isomers of cresol, along with pentachlorophenol and phenol. These organic compounds would be expected to be present to some extent in waste crossties. On November 7, 1986, the proposed TCLP test *[3]* was promulgated as part of the Land Disposal Restrictions Program which applied to specific solvent- and dioxin-containing wastes.

In the November 7, 1986 TCLP test promulgation *[3]* the EPA required particle size reduction of the waste being tested in order that the test method be environmentally conservative regarding the uncertainties over the long-term environmental stability of solid or solidified waste materials. Particle size reduction is intended to account for the worst case conditions for waste degradation by vehicular traffic, freeze-thaw cycles, wet-dry cycles, and other weathering processes in a landfill environment. Particle size reduction means grinding, chipping, or milling a waste crosstie so that the pieces are capable of passing through a ³/₈" sieve, thus exposing a large amount of surface area from which contaminants can be leached.

The EPA's proposed requirement for particle size reduction produced sufficient objection and negative comments from the regulated community that the EPA considered developing a modification to the TCLP test. This modification of the TCLP test dropped the requirement for particle size reduction and substituted a mechanical simulation of the effects of weathering and vehicular traffic on a landfill. The new method proposed by the EPA involved the development of a stainless steel "cage" which, when placed inside standard borosilicate glass extraction bottles for tumbling, would mechanically abrade large solid chunks of waste or solidified waste castings. However, after considerable testing, the EPA abandoned the TCLP "cage modification" testing method due to problems with cleaning the cages, and returned to the particle size reduction method of TCLP testing.

Additional TCLP promulgations occurred during 1990, resulting in substantially the same test procedure and regulatory standards that are still followed at the date of this writing (July 1994). In the final 1990 TCLP promulgation *[4]* several chemical parameters (including phenol) were dropped from the list, and regulatory limits were set for 39 elements and organic compounds.

1.2 PURPOSE

Over the past eight years, the Association of American Railroads (AAR), railroads, and other entities have tested waste crossties using various TCLP testing methods to determine if they are characteristically hazardous. A waste exhibiting characteristics of toxicity must be managed and disposed as a hazardous waste. With millions of crossties being removed from service every year, knowing whether or not waste crossties have to be managed as hazardous waste is very important to the railroad industry.

Crossties were determined to be non-hazardous in the initial TCLP testing program conducted by AAR in 1987. When the EPA proposed the "cage modification" TCLP method of testing, AAR again tested crossties and found them to be non-hazardous. This report summarizes those data, and the data developed by others who have conducted TCLP testing of crossties since then. The purpose of this report is to summarize and review the results of TCLP testing of crossties.

1.3 SCOPE AND APPROACH

Six different TCLP testing programs involving crossties are summarized in this report. Some of the tests were completed as early as 1987 and others as recently as 1992. AAR commissioned two of the earliest studies: one in 1987 involving chipped whole ties using essentially the same TCLP physical extraction protocol as exists today; and one in 1988 involving the proposed "cage modification" TCLP method, whose physical methodology was subsequently abandoned by the EPA. Since the 1990 TCLP rules were promulgated *[4]*, several testing programs have been undertaken by railroads and others including: the Chicago and NorthWestern Railway Company (C&NW) *[5]*, Consolidated Rail Corporation (Conrail) *[6]*, the Atchison, Topeka and Santa Fe Railway (AT&SF) *[7]*, and the New York State Energy Research and Development Authority (NYSERDA) *[8]*. These testing programs and their data are reviewed in this report as well. All of the investigations have resulted in the same conclusion: that waste crossties are not hazardous.

The approach taken by this report is to briefly summarize the sampling and analytical methods involved in each study, to present and discuss TCLP analytical results, and to summarize the conclusions.

Chapter 2 describes the TCLP method and points out some important considerations in applying the test procedure and in interpreting the results. Chapter 3 is a compilation of the available TCLP test data on creosote treated crossties; it includes a description of each testing program, a summary of the data generated, and a discussion of the results. Chapter 4 summarizes the results from all six testing programs which show that crossties should not be considered hazardous waste.

2.0 DESCRIPTION OF THE TCLP

The TCLP is a test that combines the actions of both physical and chemical forces on waste materials to simulate the mobility of toxic chemicals (which the waste materials may contain) in a landfill environment. The TCLP has been an evolving methodology and is a highly involved procedure incorporating specialized equipment and sensitive techniques. The procedure must be applied differently depending on the physical characteristics of the waste being tested, i.e., whether it is a liquid, solid, or a combination thereof; and whether or not a multiphase liquid situation exists.

2.1 BACKGROUND

The EPA initially proposed the TCLP on June 13, 1986 *[2]* and again on November 7, 1986 *[3]*, as a test procedure to be used to determine whether or not a waste material is classified as hazardous, and whether or not it can be land-disposed. A "cage modification" method of the TCLP test was proposed in 1988 *[9]*. The "cage modification" method proposed, but eventually abandoned by the EPA, would have eliminated the need for particle size reduction of many waste materials. The TCLP was promulgated in final form on March 29, 1990 *[4]*, with substantive modifications published on June 29, 1990 *[10]* (including the requirement for making matrix spike[†] corrections), and became effective on September 25, 1990. Both the original 1986, and the final March 29, 1990 *[4]*, promulgations require particle size reduction of the waste sample. Additional revisions have occurred during the intervening years, the most significant of which has been the elimination of the matrix spike correction.

2.2 THE CURRENT TCLP METHOD

In 1990 and 1991, the TCLP test method was available as Appendix B of 40 CFR, Part 261. Occasional minor revisions have continued since 1990.

[†] A matrix spike is a known amount (spike) of the specific chemical/compound of interest which is added to the unknown sample (matrix) as part of the analytical process. The analytical method compares the recovery of the spike to the recovery of the same chemical from the unspiked matrix. Matrix spikes are most commonly used in laboratory quality control programs to routinely assure accurate analyses. A matrix spike correction is a change made to the original matrix analytical result by adding to or subtracting from that result an amount determined on the basis of the amount of the spike recovered.

The most recent version (at this writing) of the TCLP test method is published in the Third Edition of *SW-846 [11]* (September, 1986) as amended by Update I (Revision 0, July 1992). The current TCLP description, as revised, is included as Appendix B of this report.

The TCLP can be broken down into three basic parts: sample collection, extraction, and analysis of the extract.

2.2.1 Sample Collection and Preparation

Sample collection is one of the most critical parts of the TCLP process and must be performed with particle size and surface area requirements in mind. Chemical parameters of concern must also be considered. Sample collection, preparation and handling methods are important because they have a direct bearing on the results that may be obtained from the TCLP testing procedure. The following information box summarizes the importance of proper sampling.

SAMPLING IS A CRITICAL PART OF THE TCLP PROCESS

- Several hundred grams of sample need to be collected.
- Sampling needs to be representative.
- Taking sub-samples from the sample container at the laboratory needs to start with a homogenous primary sample.
- Sampling must be performed with particle size and/or surface area requirements in mind.
- Sampling must be performed with parameters of interest in mind (i.e., volatiles).
- Sampling is perhaps the singular part of the TCLP test where the greatest variability in results and greatest analytical problems can be introduced.

Crossties are solid, containing no free liquid, which simplifies several aspects of the testing protocol. However, the extraction procedure requires that the solid pieces used to develop the leachate meet certain size limits or have certain surface characteristics. The procedure requires an evaluation of the particle size to determine if the surface area per gram of material is equal to or greater than 3.1 square centimeters, or if the narrowest dimension of the particles is smaller than one centimeter (capable of passing through a ³/₈" standard sieve). If the surface area is smaller or the particle size larger than previously noted, then the solids are to be cut, crushed, or ground so as to meet the surface area or particle size requirement. While the method mentions a ³/₈" sieve, it is recommended that samples not actually be sieved, or if they are sieved that the sieve be teflon coated. Actual measurement of surface area, likewise, is not recommended (nor is it required). The reason for these seemingly unlikely recommendations is to avoid excessive handling or manipulation of TCLP samples which could cause contaminants to be either lost from or introduced into the TCLP testing process. The sieve, for instance, can introduce containments such as lead which may have been trapped between the sieve wires or in other crevices or seams during previous sieve use. Based upon the results of all reported TCLP testing of crossties to-date, if a crosstie sample should fail the TCLP, it would be appropriate to question whether the sample had been contaminated by sieving or other sampling or sample handling techniques.

Because the TCLP sample has to be analyzed for volatiles, the EPA recommends that particle size reduction be conducted on the TCLP sample as it is being taken (if possible). Otherwise, any particle size reduction attempted should be conducted in such a way as to minimize atmospheric exposure of the TCLP samples and also to minimize heat generation during the particle size reduction procedure. The following information box contains the basic EPA guidelines for particle size reduction.

PARTICLE SIZE REDUCTION

- Particle size reduction is required unless the surface area per gram of material is equal to or greater than 3.1 square centimeters (about ½″ by ½″ for a "flat" solid or a little less than ³/₈″ by ³/₈″ for a "cube").
- Particle size reduction is not required if the solid is smaller than 1 centimeter (about ³/₈″) at its narrowest dimension, "(i.e., it is capable of passing through a ³/₈″ standard sieve)."
- The particle size reduction-surface area criteria are meant for filamentous materials (paper, cloth and similar). Actual measurement of surface area is not required .

2.2.2 The Extraction Process

Once particle size reduction is accomplished for a sample consisting of solid material, like crosstie chips, the remainder of the TCLP test is reasonably straightforward. A small sample is taken of the material to be tested and checked for pH to determine which extraction fluid to use in the nonvolatile extraction (for volatile extraction, Fluid No. 1 is always used). If the pH is less than 5.0 initially, or if it is over 5.0 initially, but reduces to less than 5.0 with the addition of 1 N hydrochloric acid, then Extraction Fluid No. 1 is used; otherwise, Extraction Fluid No. 2 is used.

The two different extraction fluids are each made up with glacial acetic acid (CH_3CH_2OOH). Extraction Fluid No. 1, with a pH of 4.93, is used on all volatile extractions and on all acidic (pH less than 5.0) or weakly buffered alkaline wastes. Extraction Fluid No. 2 (the more aggressive fluid with a pH of 2.88), is used on all other wastes. Refer to Method 1311 in Appendix B for a more complete description of the extraction fluids, their method of preparation, and their use.

The apparatus necessary to run the TCLP includes: 1) an agitation device capable of rotating the extraction vessel(s) in an end-over-end fashion at 30 ± 2 rpm (see Figure 1 of Appendix B); and 2) two extraction vessels. The two extraction vessels include a Zero Headspace Extraction vessel (ZHE) for use in testing the mobility of volatile constituents (see Figure 2 of Appendix B), and a borosilicate glass or plastic bottle for use in determining the mobility of nonvolatile organics and inorganics. Also needed for TCLP testing is a filtration device(s) for filtering the extract produced; borosilicate glass fiber filters for use in the ZHE vessel and/or the filtration device(s); and other laboratory devices and analytical instruments necessary to carry out TCLP evaluations.

Having selected the extraction fluid(s), as previously described, and having determined that the waste sample meets particle size and/or surface area requirements, a portion of the sample (100 grams, minimum) is weighed out and placed in the extraction vessel(s), along with a quantity of extraction fluid weighing twenty times the weight of the sample.

The extractions are run in the appropriate vessels (2-L borosilicate glass, or plastic, bottles for nonvolatiles, and the ZHE device for volatiles). After 18 hours of rotation in the agitation device, the extract is removed, filtered through a borosilicate glass fiber filter and collected in an appropriate container for the remaining analytical work.

2.2.3 Analysis of the Extract

Appropriate EPA approved analytical methods must be used to analyze the extract produced by TCLP sample preparation. Table 2-1 lists the parameters of concern and their regulatory concentration standard limits. If any contaminant listed in Table 2-1 is present in a TCLP extract at a concentration level exceeding that listed in the table, then the waste has failed the TCLP test and is characteristically hazardous. Matrix spikes are used in conjunction with the appropriate analytical methods to provide quality assurance for analytical test procedures, but the spikes are no longer used to "correct" the measured analytical values.

Table 2-1
Final Toxicity Characteristic Leaching Procedure (TCLP) Standards
40 CFR 261.24
Promulgated March 29, 1990 (FR 11798)*[4]*
Effective September 25, 1990

TCLP Metals	**Regulatory Limit**	**Hazardous Waste No.**
Arsenic	5.0	D004
Barium	100.0	D005
Cadmium	1.0	D006
Chromium	5.0	D007
Lead	5.0	D008
Mercury	0.2	D009
Selenium	1.0	D010
Silver	5.0	D011
TCLP Extractable Organics		
o-Cresol[a]	200.0	D023
m-Cresol[a]	200.0	D024
p-Cresol[a]	200.0	D025
Cresol[a]	200.0	D026
1,4-Dichlorobenzene	7.5	D027
2,4-Dinitrotoluene	0.13[b]	D030
Hexachlorobutadiene	0.5	D033
Hexachloroethane	3.0	D034
Nitrobenzene	2.0	D036
Pentachlorophenol	100.0	D037
Pyridine	5.0[b]	D038
2,4,5-trichlorophenol	400.0	D041
2,4,6-trichlorophenol	2.0	D042
TCLP Pesticides/Herbicides		
Chlordane	0.03	D020
2,4-D	10.0	D016
Endrin	0.02	D012
Heptachlor (and its expoxide)	0.008	D031
Hexachlorobenzene	0.13[b]	D032
Lindane	0.4	D013
Methoxychlor	10.0	D014
Toxaphene	0.5	D015
2,4,5-TP (Silvex)	1.0	D017
TCLP Volatile Organics		
Benzene	0.5	D018
Carbon tetrachloride	0.5	D019
Chlorobenzene	100.0	D021
Chloroform	6.0	D022
1,2-Dichloroethane	0.5	D028
1,1-Dichloroethylene	0.7	D029
Methyl ethyl ketone	200.0	D035
Tetrachloroethylene	0.7	D039
Trichloroethylene	0.5	D040
Vinyl chloride	0.2	D043

[a] If o-, m-, and p-cresol cannot be differentiated, the total cresol concentration is used. The regulatory limit of total cresol is 200.0 mg/l

[b] Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.

2.2.4 Substitution of Total Analysis or Assay for TCLP

An acceptable alternative to the TCLP is evaluation of a total analysis (or assay) of a waste. Section 1.2 of Method 1311 states:

> 1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

A dilution factor of 20 is involved in the TCLP extraction of the solid phase material, as the amount of extraction fluid used is equal to 20 times the weight of the solid phase. Therefore, if a total analysis of a sample reveals that the amount of each regulated analyte present is less than 20 times the indicated regulatory limit, then the sample could not possibly fail the TCLP, even if 100 percent of any of the analytes leached into the extraction fluid. Therefore, EPA permits a waste to be classified as non-hazardous using this evaluation.

However, if the assay results show a concentration exceeding 20 times the regulatory limit for any given parameter, then the assay is inconclusive and the TCLP extraction test must be performed to determine how much of the parameter(s) will actually leach into the extraction fluid. The following information box summarizes the considerations for assay substitution.

CONSIDERATIONS FOR ASSAY SUBSTITUTION
(Refer to Method 1311, Sections 1.2, 1.3, and 1.4
for a more complete discussion of assay substitution)

- Method 1311 allows an assay, or "total analysis" to be substituted for a TCLP.
- Consider using an assay when appropriate because it is quicker, cheaper, and simpler.
- Don't forget to collect enough sample in case the assay results are inconclusive and the use of TCLP extraction is then warranted.

An example of the "assay substitution" method as it applies to crosstie chips is presented in Chapter 3, Section 3.5, Table 3-7. That table shows the TCLP assay regulatory limits (20 times the TCLP limits) for metals. Use of the assay substitution may be appropriate under many circumstances and has the advantage of being quicker, simpler and less expensive than the full TCLP.

3.0 TCLP TESTING OF CROSSTIES

Six different testing programs have been conducted to evaluate the potential for crossties to be classified as hazardous using several versions of the TCLP. These testing programs are summarized in this chapter and include:

- AAR TCLP Testing of Chipped Crossties (1987)
- AAR Cage Method TCLP Testing of Crosstie Blocks (1988)
- C&NW TCLP Testing of Chipped Crossties (1991)
- Conrail TCLP Testing of Crossties (1991)
- AT&SF Testing of Crossties and Bridge Timbers (1992)
- NYSERDA Study (1992)

These data represent over 23 crossties and at least 28 individual TCLP analyses. Samples of the crossties were collected by somewhat different approaches in each of the testing programs, but each protocol produced representative samples. The testing was performed by different commercial laboratories throughout the United States. In some cases, only the TCLP test was run. Others involved a modification of the TCLP or additional hazardous waste characteristic testing, such as the reactive sulfide test[†] or the California Waste Extraction Test (WET)[††] procedures *[13].* Crossties that had been in service in all areas of the United States and representing a broad cross section of railroads were tested. Without exception the tests showed that crossties were not hazardous.

This chapter contains a synopsis of each testing program, along with data summaries from each program.

3.1 AAR 1987 TCLP TESTING OF CHIPPED CROSSTIES

In 1987 the AAR initiated a testing program to evaluate the potential for the EPA's proposed TCLP test to characterize crossties as hazardous waste *[12].* It involved the same

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[†] The reactive sulfide test is a hazardous waste characterization test used by the USEPA to evaluate a waste for the characteristic of reactivity.

[††] The WET is the State of California's modification of the TCLP for hazardous waste characterization.

test methodology as the final 1990 promulgation of the TCLP, including particle size reduction.

3.1.1 Sample Collection, Preparation, and Analysis

Three main line, red oak crossties were selected for testing. Each was a different age: one was new (treated but having never been installed in the track); one was ten years old; and one was approximately twenty years old. They were supplied by CSX Transportation from the central Tennessee area, and were processed, sampled, and analyzed by the environmental consulting firm of Resource Consultants, Inc. (RCI).

Each of the crossties was individually processed into chips using sawing, splitting, and chipping machinery. The chips produced from each tie were sampled representatively by three different methods that produced chip samples having slightly different physical characteristics.

One sampling method involved simply sieving the chips produced from the tree-limb chipping machinery, using a standard ⅜" sieve. This produced samples containing smaller wood pieces than the other methods. It also produced samples containing more particles of dirt or dust-like solids than the other methods. These samples are referred to as the "small sieved chips" and are designated in Table 3-1 by the letter "S".

A second sampling method involved the use of a food preparation type blender in the laboratory to reduce the size of larger chips so that all of the chips in the sample container would pass a ⅜" sieve. The blender was operated carefully to only reduce the size of wood chips to approximately ⅜" but not totally pulverize the material. This method produced some light, dust-like fines along with the ⅜" chips, but less of the sand-like or dirt-like fines than the small sieved chips. These samples are referred to as the "blender processed chips" and are designated in Table 3-1 by the letter "B".

The third sampling method involved selecting, by visual inspection, chips from a homogeneous sample of the original limb chipper's production which appeared to meet particle size and surface area restrictions. These samples contained longer, thinner slivers of wood than the others, but also contained less dirt and dust-like particles. These samples are referred to as "large sized chips" and are designated in Table 3-1 by the letter "L".

All of the samples were extracted using the TCLP methodology as described in EPA Solid Waste Manual *SW-846 [11]*, Method 1311. Volatile extractions were performed only on the small sieved chips and the large sized chips. It was decided not to perform a ZHE extraction on the blender processed chips due to the possibility that some of the volatiles would have been lost during the blending process.

Analytical methods for the specific chemical parameters or parameter groups were all performed in accordance with *SW-846 [11]*.

3.1.2 Analytical Results and Discussion

Analyses for all TCLP parameters were conducted for each crosstie for all of the chipping protocols. Most of these resulted in non-detection of the TCLP parameters. A summary of those parameters that were detected in the analyses is presented in Table 3-1.

Table 3-1
AAR 1987 TCLP Testing of Chipped Crossties
Detected TCLP Parameter Concentrations (All Ties—All Samples)

	New Crosstie			**10 Yr. Old Crosstie**			**20 Yr. Old Crosstie**			**Final 1990 TCLP Standards[b] for Detected Parameters**
	S	**B**	**L**	**S**	**B**	**L**	**S**	**B**	**L**	
o-Cresol	3.3	2.9	0.28	0.092	0.22	ND	0.28	0.28	0.14	200.0[a]
m-Cresol	5.1	4.4	ND	ND	ND	ND	ND	ND	ND	200.0[a]
p-Cresol	7.5	6.5	1.2	0.017	0.47	ND	0.93	0.93	0.37	200.0[a]
Arsenic	ND	ND	ND	0.007	ND	ND	0.54	0.044	0.01	5.0
Barium	0.4	0.3	0.4	0.7	0.5	0.3	0.6	0.4	0.5	100.0
Lead	ND	ND	ND	0.4	ND	ND	ND	ND	ND	5.0
Selenium	0.015	ND	ND	ND	0.01	ND	ND	ND	ND	1.0

Data in this table from *[12]*
All units are mg/l.
S = Small Sieved Chips
B = Blender Processed Chips
L = Large Sized Chips
ND = Not Detected at applicable quantitation limits
[a] If o-, m-, and p-cresol cannot be differentiated, the total cresol concentration is used. The regulatory limit of total cresol is 200.0 mg/l.
[b] 40 CFR 261.24

All of the data from AAR Report R-708 *[12]* are provided in Appendix A of this report. Tables A-1 through A-9 are the data tables from the AAR's 1987 testing of chipped crossties; these tables contain the analytical results, along with the final TCLP regulatory limits.

These data indicate that all analytical results are well below the regulatory limits. No volatiles were detected; nor were pesticides or herbicides. The only TCLP parameters detected in any of the nine samples analyzed were the o-, m-, and p- isomers of cresol, and the metals: arsenic, barium, lead, and selenium.

- Various isomers of cresol were present in eight of the samples. The highest cresol concentration detected was 7.5 mg/l in the extract of sieved chips from the new crosstie; the regulatory limit for cresol is 200 mg/l.
- Arsenic was present in four of the six samples from the two oldest crossties. The highest arsenic concentration was 0.54 mg/l in the extract of the sieved chips from the 20-year-old crosstie; the regulatory limit for arsenic is 5.0 mg/l.
- Barium was present in all nine of the samples. The highest barium concentration was 0.7 mg/l in the extract of the sieved chips from the 10-year-old crosstie; the regulatory limit for barium is 100 mg/l.
- Lead was present in one of the samples. The extract of the sieved chips from the 10-year-old crosstie contained a lead concentration of 0.4 mg/l. The regulatory limit for lead is 5.0 mg/l.
- Selenium was present in two of the samples. The highest selenium concentration was 0.015 mg/l in the extract of the sieved chips from the new crosstie. The regulatory limit for selenium is 1.0 mg/l.

In addition to establishing the fact that these crossties all passed the TCLP, the AAR study indicated that sieving the sample can result in a slightly higher analytical value than either grinding the sample or visually selecting appropriately sized chips for the sample. In Method 1311, EPA advises not to sieve samples.

3.2 AAR 1988 CAGE METHOD TCLP TESTING OF CROSSTIE BLOCKS

On May 24, 1988, the EPA proposed the introduction of the "cage modification" methodology to the TCLP test. The intent of the modification was to eliminate the need to do the pretest particle size reduction that is required under the current TCLP test. This was an attempt to make the TCLP test more representative of the environmental conditions to which a waste would actually be exposed. The "cage modification" method was designed so that the more structurally sound materials (such as railroad crossties) would not be broken-up into smaller pieces as much as materials of lesser structural qualities. The EPA later determined that it was difficult to have good quality control using the "cage modification" method, so it reverted back to the more aggressive particle size reduction method that is currently used in the TCLP test.

In the "cage modification" method, the waste being tested was tumbled in a similar tumbler to that used in the final TCLP procedure. The difference in this method was that the material being tested was surrounded by a steel wire cage within the tumbler. To perform this type of TCLP test on railroad crossties, a block cut from the crosstie was placed into a steel wire cage within an extraction bottle, and then into a tumbler where it was tumbled in the extraction fluid for 18 hours. The steel wire cage, in combination with the tumbling action, caused abrasion of the waste material to occur.

Shortly after the EPA proposed the "cage modification", a joint EPA/American Society of Testing and Materials (ASTM) laboratory committee was formed to evaluate the method. RCI participated in the evaluation on behalf of the AAR, and in this regard tested some crossties by the "cage modification" method *[12]*. The methods and results of the AAR's "cage modification" tests are summarized in Sections 3.2.1-3.2.2.

3.2.1 Sample Collection, Preparation, and Analysis

Three new crossties were selected for testing. New ties were chosen due to the desire to evaluate the effect of the fresh creosote preservative on the testing method. The crossties were provided by CSX Transportation and by The Koppers Company, Inc. RCI prepared the ties for processing, performed the TCLP "cage modification" method, and analyzed the extracts.

Small blocks that would fit into the cages were cut from different areas on the crossties. These areas are represented in Table 3-2 by the letters "A", "B", "C", and "D". The blocks were tumbled inside the cages for 18 hours as required by the method.

The extracts produced were processed as required by *SW-846*, Method 1311. Pesticides and herbicides were not analyzed because these were new crossties and had not been exposed to the track environment. Volatile extractions were not performed due to the protocol for volatiles determination by the "cage modification" requiring particle size reduction and use of the ZHE; AAR had previously tested for volatiles in the 1987 TCLP testing of chipped crossties. Analytical methods for the specific chemicals were all performed in accordance with *SW-846*.

3.2.2 Analytical Results and Discussion

Analyses for all TCLP parameters (except volatiles and pesticides-herbicides as discussed above) were conducted for each crosstie for all of the sample blocks taken. Most of these analyses resulted in non-detection of the TCLP parameters. A summary of those parameters that were detected in the analyses is presented in Table 3-2.

Table 3-2
AAR 1988 Cage Method TCLP Testing of Crosstie Blocks
Detected TCLP Parameter Concentrations (All Ties—All Samples)

	CSX New Tie				Koppers Petroleum/Creosote				Koppers Coal-Tar/Creosote				Final 1990 TCLP Standards[b] for Detected Parameters
	A	B	C	D	A	B	C	D	A	B	C	D	
o-Cresol	0.15	0.44	0.40	0.47	0.062	0.064	0.11	0.085	0.29	0.23	0.36	0.23	200.0[a]
p-Cresol	0.61	0.70	1.4	1.8	0.21	0.24	0.34	0.27	1.0	0.70	1.2	0.71	200.0[a]
Arsenic	ND	ND	0.002	ND	ND	0.004	0.004	0.008	0.004	ND	ND	ND	5.0
Barium	0.2	0.1	0.3	0.5	0.3	0.3	0.5	0.2	0.3	0.2	0.3	0.2	100.0
Lead	0.3	ND	0.3	ND	0.2	ND	0.1	3.9[c]	ND	ND	ND	0.2	5.0
Selenium	ND	0.01	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	1.0

Data in this table from *[12]*
All units are mg/l.
ND = Not Detected at applicable quantitation limits.

[a] If o-, m-, and p-cresol cannot be differentiated, the total cresol concentration is used. The regulatory limit of total cresol is 200.0 mg/l.

[b] 40 CFR 261.24

[c] This unusually high lead concentration is most likely a result of minute particles of leady materials that were trapped in the cage's mesh during a previous extraction test. Results such as this were not unusual in the "cage modification", and this is one of the reasons that EPA abandoned the "cage" method.

Analytical results along with the final TCLP regulatory limits are presented in Appendix A, Tables A-10 through A-21.

The results were below the TCLP regulatory limits for all samples analyzed. The only TCLP parameters detected in any of the twelve samples analyzed were the o- and p- cresol isomers, and the metals: arsenic, barium, lead, and selenium.

- Cresols were detected in all of the samples. The highest cresol isomer concentration detected was for p-cresol at 1.8 mg/l; the regulatory limit for cresol is 200 mg/l.
- Arsenic was detected in 5 of the 12 samples. The highest arsenic concentration was 0.008 mg/l; the regulatory limit for arsenic is 5.0 mg/l.
- Barium was detected in all 12 samples. The highest barium concentration was 0.5 mg/l; the regulatory limit for barium is 100 mg/l.
- Lead was detected in 6 of the samples. Lead concentrations in 5 of the extracts ranged between 0.1 and 0.3 mg/l. The highest lead concentration, 3.9 mg/l, is believed to be caused by contamination introduced by the cage apparatus. The regulatory limit for lead is 5.0 mg/l.
- Selenium was only present in one sample, at only 0.01 mg/l. The regulatory limit for selenium is 1.0 mg/l.

Even though EPA did not promulgate the "cage modification" as a final rule, these test results support the conclusion that railroad crossties are not a hazardous waste and that no significant leaching of contaminants from crossties occurs in the environment.

3.3 CHICAGO AND NORTHWESTERN RAILWAY COMPANY (C&NW) CROSSTIE TESTS

The Chicago and NorthWestern Railway Company (C&NW) collected and analyzed samples of chipped crossties during July of 1991. Estimates from C&NW indicate that these chips were composed of a mix of creosote-treated hardwoods, with 90 to 95 percent being white or red oak and possibly some red or black gum wood. The crossties were presumed to be at least 20 to 30 years old and had already been processed into piles of chips at the time of sampling.

3.3.1 Sample Collection, Preparation, and Analysis

The crosstie chip pile was sampled at four locations. Samples were collected by C&NW personnel by sieving through ³/₈″ standard sieves when collected, to satisfy the particle size reduction protocol, and shipped under chain of custody to RCI laboratories for analysis. Samples were analyzed for reactive sulfide, pH, and total solids in addition to the TCLP test procedure. The samples were extracted using the TCLP methodology as described in EPA Solid Waste Manual *SW-846*, Method 1311. Analytical methods for the specific chemical parameters or parameter groups were all performed in accordance with *SW-846*.

3.3.2 Analytical Results and Discussion

The analytical results for the C&NW tests are presented in Table 3-3. Comparison of the analytical results with the TCLP standards shows that most of the parameter results were below detection limits in the extract, and all were well below regulatory limits. The only TCLP parameters detected in any of the four samples analyzed were the m- and p-isomers of cresol, and the metals arsenic, barium and mercury.

- The m- and p- isomers of cresol were detected in only one sample, at a concentration of 0.26 mg/l. The regulatory limit for cresol is 200 mg/l.
- Arsenic was detected in only one of the samples, at a concentration of 0.009 mg/l. The regulatory limit for arsenic is 5.0 mg/l.
- Barium was detected in three of the samples. The highest barium concentration was 0.4 mg/l; the regulatory limit for barium is 100 mg/l.
- Mercury was detected in two samples, barely above the method detection limit. The highest mercury concentration was 0.006 mg/l; the regulatory limit for mercury is 0.2 mg/l.

Based upon the very low parameter concentrations detected in the extract, it can be concluded that the chipped crossties were not characterized as hazardous waste by the TCLP test.

Table 3-3
C&NW TCLP Crosstie Testing
Analytical Results

	Sample Locations				Final 1990 TCLP Standards
	1	2	3	4	
TCLP Extraction					
TCLP Metals					
Arsenic	<0.002	0.009	<0.002	<0.002	5.0
Barium	0.2	<0.1	0.4	0.3	100.0
Cadmium	<0.005	<0.005	<0.005	<0.005	1.0
Chromium	<0.02	<0.02	<0.02	<0.02	5.0
Lead	<0.1	<0.1	<0.1	<0.1	5.0
Mercury	0.006	<0.002	0.004	<0.002	0.2
Selenium	<0.01	<0.01	<0.01	<0.01	1.0
Silver	<0.01	<0.01	<0.01	<0.01	5.0
TCLP Extractable Organics					
o-cresol	<0.10	<0.10	<0.10	<0.10	200.0
m-cresol	<0.10	<0.10	<0.10	m,p-cresol	200.0
p-cresol	<0.10	<0.10	<0.10	0.26	200.0
1,4-dichlorobenzene	<0.10	<0.10	<0.10	<0.10	7.5
2,4-dinitrotoluene	<0.10	<0.10	<0.10	<0.10	0.13
Hexachlorobutadiene	<0.10	<0.10	<0.10	<0.10	0.5
Hexachloroethane	<0.10	<0.10	<0.10	<0.10	3.0
Nitrobenzene	<0.10	<0.10	<0.10	<0.10	2.0
Pentachlorophenol	<0.10	<0.10	<0.10	<0.10	100.0
Pyridine	<0.10	<0.10	<0.10	<0.10	5.0
2,4,5-trichlorophenol	<0.10	<0.10	<0.10	<0.10	400.0
2,4,6-trichlorophenol	<0.10	<0.10	<0.10	<0.10	2.0
TCLP Pesticides/Herbicides					
Chlordane	<0.015	<0.015	<0.015	<0.015	0.03
2,4-D	<5.0	<5.0	<5.0	<5.0	10.0
Endrin	<0.010	<0.010	<0.010	<0.010	0.02
Heptachlor (and its expoxide)	<0.005	<0.005	<0.005	<0.005	0.008
Hexachlorobenzene	<0.005	<0.005	<0.005	<0.005	0.13
Lindane	<0.20	<0.20	<0.20	<0.20	0.4
Methoxychlor	<1.0	<1.0	<1.0	<1.0	10.0
Toxaphene	<0.25	<0.25	<0.25	<0.25	0.5
2,4,5-TP (Silvex)	<0.5	<0.5	<0.5	<0.5	1.0
TCLP Volatile Organics					
Benzene	<0.1	<0.1	<0.1	<0.1	0.5
Carbon tetrachloride	<0.1	<0.1	<0.1	<0.1	0.5
Chlorobenzene	<0.1	<0.1	<0.1	<0.1	100.0
Chloroform	<0.1	<0.1	<0.1	<0.1	6.0
1,2-dichloroethane	<0.1	<0.1	<0.1	<0.1	0.5
1,1-dichloroethylene	<0.1	<0.1	<0.1	<0.1	0.7
Methyl ethyl ketone	<0.1	<0.1	<0.1	<0.1	200.0
Tetrachloroethylene	<0.1	<0.1	<0.1	<0.1	0.7
Trichloroethylene	<0.1	<0.1	<0.1	<0.1	0.5
Vinyl chloride	<0.1	<0.1	<0.1	<0.1	0.2
Reactive Sulfide (mg/kg)	<30 wwb	<30 wwb	<30 wwb	<30 wwb	500

All units are mg/l unless otherwise noted.
wwb: wet weight basis
NOTE: the number following < symbol also indicates method detection limit.

The reactive sulfide test is one of several test procedures used to characterize or define a waste as hazardous; it is described in detail in Section 7.3.4 of EPA Manual *SW-846*. The test measures any hydrogen sulfide gas released (reported as sulfide ion) under specific conditions that include a measured amount of sample being lowered to a pH of 2.0 and stirred for 30 minutes. The action level (failure level) is a sulfide concentration of 500 mg/kg or greater. As can be seen from the analytical report table, none of the samples released sulfide ion in amounts above the detection limit of 30 mg/kg. Thus, the crosstie chips are characterized as not hazardous by the reactive sulfide test.

3.4 CONSOLIDATED RAIL CORPORATION (CONRAIL) TCLP TESTING OF CROSSTIES

Conrail collected and analyzed samples of crossties from their Altoona Yard in Altoona, PA, during November 1991.

3.4.1 Sample Collection, Preparation, and Analysis

Crosstie samples were collected and analyzed in accordance with applicable EPA methods. Analyses were performed by Mountain Research, Inc., in Altoona. TCLP and ZHE analyses were performed in accordance with Method 1311. Metals were analyzed according to EPA Methods 6010 and 7470. Volatile organic analysis was performed by EPA Methods 601/602. Semivolatile analysis was performed by EPA Method 625. Pesticides and herbicides were analyzed by EPA Method 608.

3.4.2 Analytical Results and Discussions

Analytical results for the Conrail tests are presented in Table 3-4. No TCLP parameters were detected in the extract; all results were below method detection limits in the extract, so they were well below the regulatory limits. The analytical results confirmed that the Conrail crossties were not hazardous waste.

Table 3-4
Conrail TCLP Crosstie Testing
Analytical Results

	TCLP on Railroad Ties (mg/l)	Final 1990 TCLP Standards (mg/l)
TCLP Metals		
Arsenic	<0.08	5.0
Barium	<0.08	100.0
Cadmium	<0.004	1.0
Chromium	<0.02	5.0
Lead	<0.05	5.0
Mercury	<0.0005	0.2
Selenium	<0.1	1.0
Silver	<0.02	5.0
TCLP Extractable Organics		
o-cresol	<0.010	200.0
m-cresol	<0.010	200.0
p-cresol	<0.010	200.0
2,4-dinitrotoluene	<0.010	0.13
Hexachlorobenzene	<0.010	0.13
Hexachlorobutadiene	<0.010	0.5
Hexachloroethane	<0.010	3.0
Nitrobenzene	<0.010	2.0
Pentachlorophenol	<0.050	100.0
Pyridine	<0.010	5.0
2,4,5-trichlorophenol	<0.010	400.0
2,4,6-trichlorophenol	<0.010	2.0
TCLP Pesticides/Herbicides		
Chlordane	<0.005	0.005
2,4-D	<0.010	0.010
Endrin	<0.0002	0.0002
Heptachlor (and its expoxide)	<0.0005	0.0005
Lindane	<0.001	0.001
Methoxychlor	<0.001	0.001
Toxaphene	<0.005	0.005
2,4,5-TP (Silvex)	<0.001	0.001
TCLP Volatile Organics		
Benzene	<0.002	0.5
Carbon tetrachloride	<0.001	0.5
Chlorobenzene	<0.002	100.0
Chloroform	<0.002	6.0
1,4-dichlorobenzene	<0.001	7.5
1,2-dichloroethane	<0.001	0.5
1,1-dichloroethylene	<0.001	0.7
Methyl ethyl ketone	<0.002	200.0
Tetrachloroethylene	<0.001	0.7
Trichloroethylene	<0.001	0.5
Vinyl chloride	<0.002	0.2

NOTE: the number following < symbol also indicates method detection limit.

3.5 ATCHISON, TOPEKA AND SANTA FE RAILWAY (AT&SF) BRIDGE CROSSTIES AND BRIDGE TIMBER TESTS

In October of 1992, the Atchison, Topeka and Santa Fe Railway (AT&SF) collected and analyzed samples of creosote-treated crossties and bridge timbers from the Middle River Bridge in the Sacramento Delta west of Stockton, California. The AT&SF bridge was originally constructed in 1930 with creosote-treated bridge timbers, as well as creosote-treated crossties.

3.5.1 Sample Collection, Preparation, and Analysis

Crosstie samples were collected by Environmental Solutions, Inc. from two different crossties by removing slices from the center of the crossties; the samples were subdivided and composited into one sample for analysis. Bridge timbers including deck planking, piling, stringers, and caps were sampled and composited into one sample for analysis. The samples were transported under chain of custody to Chromalab, Inc., for analysis.

This testing program was performed to characterize the wood material for waste disposal purposes, so the testing protocols followed the State of California waste characterization regulations which are similar to, but not identical to, USEPA hazardous waste characterization regulations. TCLP testing was limited to the parameters of cresols and pentachlorophenol. Metals analyses were performed in accordance with the *California Assessment Manual (CAM) [13]* for California's list of 17 metals (CAM-17 metals). All of the TCLP metals are included in the *CAM* list, so with the inclusion of TCLP testing for cresols as previously noted, all of the TCLP parameters detected to date in the testing of crossties are included in the AT&SF testing program.

The regulatory limits for the CAM-17 metals are the California Soluble Threshold Limit Concentrations (STLC) and the Total Threshold Limit Concentrations (TTLC). The TTLC are assay/total analysis based numbers. The STLC are extraction procedure based numbers applicable to California's Waste Extraction Test (WET) Procedures *[14]*. The California regulatory limits are shown in the right-hand columns of Table 3-5. The following information box lists the differences between the WET and TCLP methodology.

DIFFERENCES IN WET AND TCLP METHODOLOGY

- The WET test involves milling of solid samples, to pass a No. 10 (two millimeter) sieve in the case of metals, and a one millimeter standard sieve in the case of organics; whereas the TCLP involves cutting, crushing, or grinding if the solids are not capable of passing a 9.5 mm sieve.
- The WET test uses 0.2 M sodium citrate at pH of 5.0 ± 0.1 as an extraction solution, compared to two different glacial acetic acid fluids (pH 4.93 and 2.88) for the TCLP test.
- The WET test uses a 50 gm sample size, whereas the TCLP test requires a 100 gm sample size.
- The WET test uses 50 ml of extraction solution rather than the two liters commonly used for solid samples in the TCLP test.
- The WET test allows the use of other types of mixing systems (other than a rotary tumbler), whereas the TCLP requires a rotary tumbler at 30±2 rpm.
- The WET test requires extracting for 48 hours, whereas the TCLP test requires extracting for 18 ± 2 hours.
- The WET test requires deaeration of the extraction solution with nitrogen gas which is not required by the TCLP test.

Analytical methods for the specific chemical parameters were all performed in accordance with the *CAM*, which incorporates the analytical methods in *SW-846*

3.5.2 Analytical Results and Discussion

The AT&SF test results are shown in Table 3-5 and Table 3-6.

Differences exist between the USEPA and the California sampling, sample preparation, and analytical protocols, but there are also similarities. The California "Total" or "TTLC" test is an assay of a sample for the various parameters of concern. The USEPA TCLP test also allows an assay to be substituted for the extraction method, as explained in Section 2.2.4. In the TCLP test a dilution factor of 20 is produced within the extraction process, so that regulatory limits for a TCLP assay substitution can be written as the TCLP standard value multiplied by the factor of 20. Table 3-7 compares the TCLP assay regulatory limits for metals (based upon the dilution factor of 20) with the California TTLC limits for the same metals.

Table 3-5
AT&SF Middle River Bridge—Crossties
Analytical Results

COMPOUND	CONCENTRATION			REGULATORY LIMIT	
	Total (mg/kg)	TCLP (mg/l)	STLC (mg/l)		
BTEX					
Analytical Method(s)	8020	---	---	---	
Benzene	0.031	---	---	---	
Toluene	0.102	---	---	---	
Ethylbenzene	0.43	---	---	---	
Xylenes	1.6	---	---	---	
PHENOLS					
Analytical Method(s)	---	TCLP 8270	---	TCLP[b] mg/l	
o-cresol (2-methylphenol)	---	0.086	---	200	
m-cresol (3-methylphenol)	---	<0.005	---	200	
p-cresol (4-methylphenol)	---	0.21	---	200	
Pentachlorophenol	---	<0.025	---	100	
METALS					
Analytical Method(s)	TTLC 3050 6010 7471	---	WET-STLC 3010 6010 7470	TTLC[a] (mg/kg)	STLC[a] (mg/l)
Antimony	<1.00	---	<0.20	500	15
Arsenic	<0.25	---	<0.05	500	5.0
Barium	3.8	---	<0.05	10,000	100
Beryllium	<0.05	---	<0.01	75	0.75
Cadmium	<0.05	---	<0.01	100	1.0
Cobalt	<0.50	---	<0.10	8,000	80
Chromium	<0.50	---	<0.10	2,500	5
Copper	1.3	---	<0.05	2,500	25
Lead	2.3	---	<0.10	1,000	5.0
Mercury	<0.05	---	<0.01	20	0.2
Molybdenum	<0.25	---	<0.05	3,500	350
Nickel	1.5	---	<0.20	2,000	20
Selenium	10	---	<0.10	100	1.0
Silver	<0.25	---	<0.05	500	5
Thallium	<2.00	---	<0.10	700	7.0
Vanadium	0.60	---	<0.10	2,400	24
Zinc	4.4	---	<0.05	5,000	250

NOTE: the number following < symbol also indicates method detection limit.
TTLC = Total Threshold Limit Concentration = assay
STLC = Soluble Threshold Limit Concentration
WET = Waste Extraction Test
[a] = applicable in California only
[b] = applicable USEPA limit

Table 3-6
AT&SF Middle River Bridge—Bridge Timbers
Analytical Results

COMPOUND	CONCENTRATION			REGULATORY LIMIT	
	Total (mg/kg)	TCLP (mg/l)	STLC (mg/l)		
BTEX					
Analytical Method(s)	8020	---	---	---	
Benzene	0.33	---	---	---	
Toluene	2.1	---	---	---	
Ethylbenzene	2.0	---	---	---	
Xylenes	8.6	---	---	---	
PHENOLS					
Analytical Method(s)	---	TCLP 8270	---	TCLP^{b} mg/l	
o-cresol (2-methylphenol)	---	2.8	---	200	
m-cresol (3-methylphenol)	---	<0.005	---	200	
p-cresol (4-methylphenol)	---	5.6	---	200	
Pentachlorophenol	---	0.025	---	100	
METALS					
Analytical Method(s)	TTLC 3050 6010 7471	---	WET-STLC 3010 6010 7470	TTLC^{a} (mg/kg)	STLC^{a} (mg/l)
Antimony	<1.00	---	<0.20	500	15
Arsenic	0.85	---	<0.05	500	5.0
Barium	<0.25	---	<0.05	10,000	100
Beryllium	<0.05	---	<0.01	75	0.75
Cadmium	<0.05	---	<0.01	100	1.0
Cobalt	<0.50	---	<0.10	8,000	80
Chromium	<0.50	---	<0.10	2,500	5
Copper	1.2	---	<0.05	2,500	25
Lead	<0.50	---	<0.10	1,000	5.0
Mercury	<0.05	---	<0.01	20	0.2
Molybdenum	<0.25	---	<0.05	3,500	350
Nickel	0.59	---	<0.20	2,000	20
Selenium	14	---	0.14	100	1.0
Silver	<0.25	---	<0.05	500	5
Thallium	8.0	---	<0.10	700	7.0
Vanadium	<0.50	---	<0.10	2,400	24
Zinc	5.7	---	0.07	5,000	250

NOTE: the number following < symbol also indicates method detection limit.
TTLC = Total Threshold Limit Concentration = assay
STLC = Soluble Threshold Limit Concentration
WET = Waste Extraction Test
[a] = applicable in California only
[b] = applicable USEPA limit

Table 3-7
AT&SF Crosstie Testing
Comparison with TCLP and TTLC Assay Regulatory Limits for the Metals-in-Common

Metal	AT&SF Crossties	AT&SF Bridge Timbers	TCLP Assay Regulatory Limits	TTLC* Regulatory Limits
Arsenic	<0.25	0.85	100	500
Barium	3.8	<0.25	2,000	10,000
Cadmium	<0.05	<0.05	20	100
Chromium	<0.50	<0.50	100	2,500
Lead	2.3	<0.50	100	1,000
Mercury	<0.05	<0.05	4	20
Selenium	10	14	20	100
Silver	<0.25	<0.25	100	500

NOTE: All concentrations are mg/kg.
The number following < symbol also indicates method detection limit.
* Total Threshold Limit Concentration Assay

It is evident from comparison of the values in Tables 3-5, 3-6, and 3-7, that the AT&SF crossties and bridge timbers passed the hazardous waste characterization tests by large margins.

The only metals detected in the crosstie samples were barium (3.8 mg/kg), copper (1.3 mg/kg), lead (2.3 mg/kg), nickel (1.5 mg/kg), selenium (10 mg/kg), vanadium (0.5 mg/kg), and zinc (4.4 mg/kg). None of these metals were present in sufficiently high enough concentrations to fail the TCLP test for metals, nor were the regulated metals present in high enough concentrations to fail the California TTLC test limits. The metal selenium, which assayed at 10 mg/kg (with the regulatory limits at 20 mg/kg for the TCLP test and 100 mg/kg for the TTLC test), came the closest to causing the crossties samples to exceed the TCLP regulatory limits. The cresols were extracted at minimal concentrations of 0.086 mg/l for o-cresol and 0.21 mg/l for p-cresol, with the TCLP regulatory limits at 200 mg/l for each of these.

The only metals detected in the bridge timber samples were arsenic (0.85 mg/kg), copper (1.2 mg/kg), nickel (0.59 mg/kg), selenium (14 mg/kg), thallium (8.0 mg/kg), and zinc

(5.7 mg/kg). None of these metals were present in sufficiently high enough concentrations to cause failure of the TCLP test for metals. Neither were the regulated metals present in high enough concentrations to fail the California TTLC test limits. The closest the bridge timbers came to exceeding any regulatory limit also involved the metal selenium, which assayed at 14 mg/kg with the regulatory limits at 20 mg/kg for the TCLP test and 100 mg/kg for the TTLC test. The cresols were extracted at minimal concentrations of 2.8 mg/l for o-cresol, and 5.6 mg/l for p-cresol, with the TCLP regulatory limits at 200 mg/l for each of these.

Based upon the analytical regimen and the results, both the crossties and the bridge timbers were determined to be non-hazardous waste. This is the case whether using the USEPA-TCLP parameters or the State of California-WET/TTLC-STLC parameters *[14]*.

3.6 NEW YORK STATE ENERGY RESEARCH AND DEVELOPMENT AUTHORITY STUDY OF 1992

A study prepared in 1992 for the New York State Energy Research and Development Authority (NYSERDA), and co-sponsored by the U.S. Environmental Protection Agency; Canadian Department of Energy, Mines, and Resources; U.S. Department of Energy's Regional Biomass Program; and Virginia Department of Mines, Minerals and Energy, conducted TCLP and other testing on used railroad crossties and telephone poles. The purpose of the NYSERDA project and report was to provide environmental regulators, project developers, and others with data to make informed decisions on the use of waste wood materials as a combustion resource. Potential environmental problems and solutions were identified. This section summarizes the important and relevant results of the NYSERDA study with regard to hazardous waste testing of creosote-treated wood products.

3.6.1 Sample Collection, Preparation, and Analysis

Samples of creosote-treated railroad crossties and telephone poles were collected and analyzed separately. Ten railroad crossties from 30 to 50 years old were sampled; their condition varied from rotting to no structural disintegration. The text of the NYSERDA report states that, "from 10 to 15 telephones (sic) were hogged for this project." However, the report does not discuss how the crossties were processed or sampled. It is assumed that the crossties were also "hogged" (ground or chipped whole in a heavy-duty chipper/grinder). The report continues, "approximately 100 pounds of chips were collected and quartered into a ten pound sample for analysis." Complete TCLP tests were performed on the ground wood samples. Assays were also performed for selected parameters.

3.6.2 Analytical Results and Discussion

The data tables that follow contain the assay results presented in the NYSERDA study report, as well as the complete TCLP extraction results. Table 3-8 contains the assay results, and Table 3-9 presents the TCLP extraction results.

Table 3-8 shows two TCLP extractable organic parameters: 2-4-6-trichlorophenol, which has a TCLP standard of 2.0 mg/l, and pentachlorophenol, which has a TCLP regulatory limit of 100 mg/l. Converting these extract standards to assay standards, on the basis of the dilution factor of 20, the assay standard for 2-4-6-trichlorophenol is 40 mg/kg, and the assay standard for pentachlorophenol is 2,000 mg/kg. Comparing the NYSERDA assay values of <2.97 mg/kg for 2-4-6-trichlorophenol, and <15.3 mg/kg for pentachlorophenol, the crossties pass the extractable organics limits for these two TCLP parameters by wide margins.

Table 3-8
NYSERDA TCLP Parameter Testing
of Creosote-Treated Wood
Total Analysis (Assay) Results

Chemical Parameter	Telephone Poles	Railroad Crossties	Regulatory Limits
Pentachlorophenol	<11.9	<15.3	2,000
2-4-6-Trichlorophenol	<2.31	<2.97	40
Arsenic	0.75	1.5	100
Barium	14.5	25.2	2,000
Cadmium	0.14	0.1	20
Chromium	<5	<5	100
Lead	39.5	8	100
Mercury	<0.25	<0.25	4
Silver	0.05	<0.05	100

NOTE: All values are mg/kg, dry weight basis.
The number following < symbol also indicates method detection limit.

Table 3-9
NYSERDA TCLP Parameter Testing
TCLP Extraction Results

TCLP Extraction	Telephone Poles	Railroad Crossties	Final 1990 TCLP Standards
TCLP Metals			
Arsenic	<0.005†	0.018	5.0
Barium	0.076	0.140	100.0
Cadmium	<0.001†	<0.001†	1.0
Chromium	0.010	<0.005†	5.0
Lead	0.042	0.010	5.0
Mercury	<0.005	<0.005	0.2
Selenium	0.084	0.031	1.0
Silver	<0.001	<0.001	5.0
TCLP Extractable Organics			
Cresol	<0.1†	0.1	200.0
1,4-dichlorobenzene	<0.005	<0.005	7.5
2,4-dinitrotoluene	<0.01	<0.01	0.13
Hexachlorobutadiene	<0.005	<0.005	0.5
Hexachloroethane	<0.005	<0.005	3.0
Nitrobenzene	<0.1	<0.1	2.0
Pentachlorophenol	<1.0	<1.0	100.0
Pyridine	<0.5	<0.5	5.0
2,4,5-trichlorophenol	<0.1	<0.1	400.0
2,4,6-trichlorophenol	<0.1	<0.1	2.0
TCLP Pesticides/Herbicides			
Chlordane	<0.005	<0.005	0.03
2,4-D	<0.01	<0.01	10.0
Endrin	<0.001	<0.001	0.02
Heptachlor (and its expoxide)	<0.0005	<0.0005	0.008
Hexachlorobenzene	<0.01	<0.01	0.13
Lindane	<0.001	<0.001	0.4
Methoxychlor	<0.01	<0.01	10.0
Toxaphene	<0.05	<0.05	0.5
2,4,5-TP (Silvex)	<0.002	<0.002	1.0
TCLP Volatile Organics			
Benzene	<0.005	<0.005	0.5
Carbon tetrachloride	<0.005	<0.005	0.5
Chlorobenzene	<0.005	<0.005	100.0
Chloroform	<0.005	<0.005	6.0
1,2-dichloroethane	<0.005	<0.005	0.5
1,1-dichloroethylene	<0.005	<0.005	0.7
Methyl ethyl ketone	<0.1	<0.1	200.0
Tetrachloroethylene	<0.005	<0.005	0.7
Trichloroethylene	<0.005	<0.005	0.5
Vinyl chloride	<0.005	<0.005	0.2

All units are mg/l unless otherwise noted.

NOTE: the number following < symbol also indicates the detection limit as reported by the analytical laboratory. Those numbers followed by the † symbol were reported by the laboratory (Environmental Laboratories, Inc.) as BDL=Below Detection Limit. It is assumed that the laboratory felt that those parameters were present, but at concentrations less than the estimated quantitation limit (EQL). All other numbers which follow the < symbol were reported by the laboratory as ND=None Detected.

Examining the assay concentrations for seven of the TCLP metals as shown in Table 3-8, the crossties again pass the metals limits by wide margins. Lead comes the closest to the regulatory limit, and it is still more than a factor of 10 below the assay standard.

The TCLP extraction results shown in Table 3-9, also show that the crossties pass the TCLP by wide margins. Six TCLP metals parameters were detected in the extracts from the crossties: arsenic, barium, cadmium, chromium, lead, and selenium. Cresol was the only organic parameter detected.

- Arsenic was detected at only 0.018 mg/l; the regulatory limit for arsenic is 5.0 mg/l.
- Barium was detected at only 0.140 mg/l; the regulatory limit for barium is 100 mg/l.
- Cadmium's presence was detected but its concentration was below the estimated quantitation limit (EQL)[†]; its concentration was <0.001 mg/l. The regulatory limit for cadmium in 1.0 mg/l.
- Chromium's presence was detected but its concentration was below the EQL; its concentration was <0.005 mg/l. The regulatory limit for chromium is 5.0 mg/l.
- Lead was detected at only 0.010 mg/l; the regulatory limit for lead is 5.0 mg/l.
- Selenium was detected at 0.031 mg/l; the regulatory limit for selenium is 1.0 mg/l.
- Cresols were extracted at the detection limit concentration of 0.1 mg/l; the regulatory limit for cresols is 200 mg/l.

A factor of 100 or more separates the extraction results from the regulatory limits for all parameters except selenium which still passed by a factor of about 32. The results all indicate that the tested crossties are not hazardous waste.

[†] Estimated quantitation limit (EQL) as defined in *SW-846 [11]*, is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.

4.0 SUMMARY AND CONCLUSIONS

Six different testing programs involving TCLP testing of railroad crossties have consistently shown that crossties pass the hazardous waste characterization test. Data from at least 28 individual TCLP tests, representing at least two dozen crossties from all over the U.S. support the conclusion that crossties are not a hazardous waste.

Table 4-1 provides a qualitative summary of the results of the various testing programs. Each program took a slightly different approach to sampling and preparation of the sample for analysis, but the analytical results consistently showed that all crossties tested were below regulatory limits. This was the case even with modifications of the TCLP, including both EPA's proposed "cage modification", and California's Waste Extraction Test (WET) *[14]*.

Table 4-1
Summary of TCLP Testing of Crossties

TCLP Chemical		AAR Chips	AAR Cage	C&NW	Conrail	AT&SF	NYSERDA
Cresol	Detected above EQL	Y	Y	Y	N	Y	Y
	Detected above regulatory limit	N	N	N	N	N	N
Arsenic	Detected above EQL	Y	Y	Y	N	Y	Y
	Detected above regulatory limit	N	N	N	N	N	N
Barium	Detected above EQL	Y	Y	Y	N	Y	Y
	Detected above regulatory limit	N	N	N	N	N	N
Lead	Detected above EQL	Y	Y	N	N	Y	Y
	Detected above regulatory limit	N	N	N	N	N	N
Mercury	Detected above EQL	N	N	Y	N	N	N
	Detected above regulatory limit	N	N	N	N	N	N
Selenium	Detected above EQL	Y	Y	N	N	Y	Y
	Detected above regulatory limit	N	N	N	N	N	N
Other TCLP Parameters Detected above EQL		N	N	N	N	N	N
TCLP Pass/Fail		**Pass**	**Pass**	**Pass**	**Pass**	**Pass**	**Pass**

Y = Yes N = No

Out of the 39 chemicals for which the TCLP lists regulatory standards, only eight (the three cresol isomers, plus arsenic, barium, lead, mercury, and selenium) were detected above the EQL in the testing. In most instances, these have only been present at low, near-detection-limit concentrations in the extracts. The worst case concentrations involved lead in the "cage modification" (believed to have been imported by a contaminated cage) at 3.9 mg/l, or 78 percent of the regulatory limit; and selenium in the AT&SF bridge timbers at 14 mg/kg, or 70 percent of the regulatory limit. In all other cases, no regulated parameter exceeded even 50 percent of the regulatory limit.

Results of TCLP testing of over two dozen crossties of different hardwoods and softwoods, of ages from zero to over 50 years old, and from different environments, showed that none of the ties exceeded regulatory limits. These data, representing crossties from a variety of locations and services, show that, in general, railroad crossties would not be classified as hazardous waste.

REFERENCES

[1] 40 CFR Part 260 et al; Hazardous Waste Management System; Land Disposal Restrictions; Proposed Rule, *Federal Register* Vol.51, No. 9, January 14, 1986, pp. 1602-1766.

[2] 40 CFR Parts 261, 271 and 302; Hazardous Waste Management System; Identification and Listing of Hazardous Waste; Notification Requirements; Reportable Quantity Adjustments; Proposed Rule, *Federal Register* Vol. 51, No. 114, June 13, 1986, pp. 21648-21693.

[3] 40 CFR Part 260 et al.; Hazardous Waste Management System; Land Disposal Restrictions; Final Rule, *Federal Register* Vol. 51, No. 216, November 7, 1986, pp. 40572-40654.

[4] 40 CFR 261 et al.; Hazardous Waste Management System; Identification and Listing of Hazardous Waste; Toxicity Characteristics Revisions; Final Rule, *Federal Register* Vol. 55, No. 61, March 29, 1990, pp. 11798-11877.

[5] Chicago & NorthWestern Railway Company letter dated June 2, 1993, and attachments.

[6] Consolidated Rail Corporation letter dated June 18, 1993, and attachments.

[7] The Atchison, Topeka and Santa Fe Railway (AT&SF) letter dated June 22, 1993, and attachments.

[8] New York State Energy Research and Development Authority's Report entitled: "Wood Products in the Waste Stream: Characterization and Combustion Emissions", Energy Authority Report 92-8, Vol. 1, November 1992.

[9] 40 CFR Part 268; Land Disposal Restrictions; Proposed Rule, *Federal Register* Vol. 53, No. 100, May 24, 1988, pp. 18792-18797.

[10] 40 CFR Parts 261, 264, 265, 268, 271 and 302; Hazardous Waste Management System; Identification and Listing of Hazardous Waste; toxicity Characteristic Revisions; Final Rule, *Federal Register* Vol. 55, No. 126, June 29, 1990, pp. 26986-26998.

[11] "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication *SW-846* [Third Edition (September, 1986), as amended by Update I (July 1992)].

[12] *Toxicity Characteristic Leaching Procedure Testing of Railroad Crossties*, Association of American Railroads Report No. R-708, August 1, 1988.

[13] *The California Assessment Manual for Hazardous Waste*, California Department of Health Services, Hazardous Materials Management Section, June 1981 (out of print—currently incorporated in reference *[14]*).

[14] *California Code of Regulations, Title 22*, "Hazardous Waste Management Regulations," Section 66261.24 and Appendix II.

APPENDIX A

Data Tables from AAR Report R-708

The data tables that follow are revised versions of the original data tables from AAR Report R-708. The difference between the original data tables and these is that the original tables showed the proposed EPA TCLP regulatory standards, while these revised tables show the final, 1990 TCLP regulatory standards.

NON-VOLATILES		ID. 38256
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.3	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.12	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	2.9	200.0
m-Cresol	4.4	200.0
p-Cresol	6.5	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	4.9	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-1

AAR/RCI

TCLP TESTING OF NEW CROSSTIE -BLENDED

NON-VOLATILES		ID. 38255
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.5	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	0.010	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.19	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.22	200.0
m-Cresol	ND	200.0
p-Cresol	0.47	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	4.9	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-2

AAR/RCI

TCLP TESTING OF 10 YEAR OLD CROSSTIE-BLENDED

TABLE A-3

AAR/RCI

TCLP TESTING OF 20 YEAR OLD CROSSTIE-BLENDED

NON-VOLATILES		ID. 38254
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.044	5.0
Barium	0.4	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.34	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.28	200.0
m-Cresol	ND	200.0
p-Cresol	0.93	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	.28	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

NON-VOLATILES		ID. 38005
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.4	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	0.015	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.32	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	3.3	200.0
m-Cresol	5.1	200.0
p-Cresol	7.5	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	5.7	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-4

AAR/RCI

TCLP TESTING OF NEW CROSSTIE - SMALL CHIPS

VOLATILES		ID. 38006
Parameter	Analytical Results	1990 Final TCLP Standards
VOLATILE ORGANICS		
Acrylonitrile	ND	NA
Benzene	ND	0.5
Carbon disulfide	ND	NA
Carbon tetrachloride	ND	0.5
Chlorobenzene	ND	100.0
Chloroform	ND	6.0
1,2-Dichloroethane	ND	0.5
1,1-Dichloroethylene	ND	0.7
Isobutanol	ND	NA
Methylene chloride	ND	NA
Methyl ethyl ketone	ND	200.0
1,1,1,2-Tetrachloroethane	ND	NA
1,1,2,2-Tetrachloroethane	ND	NA
Tetrachloroethylene	ND	0.7
Toluene	ND	NA
1,1,1-Trichloroethane	ND	NA
1,1,2-Trichloroethane	ND	NA
Trichloroethylene	ND	0.5
Vinyl Chloride	ND	0.2

All units are mg/l unless otherwise noted.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-4a

AAR/RCI

TCLP TESTING OF NEW CROSSTIE - SMALL CHIPS

NON-VOLATILES		ID. 38007
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.007	5.0
Barium	0.7	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	0.4	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.34	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.092	200.0
m-Cresol	ND	200.0
p-Cresol	0.017	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	ND	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-5

AAR/RCI

TCLP TESTING OF 10 YEAR OLD CROSSTIE-SMALL CHIPS

VOLATILES		ID. 38008
Parameter	Analytical Results	1990 Final TCLP Standards
VOLATILE ORGANICS		
Acrylonitrile	ND	NA
Benzene	ND	0.5
Carbon disulfide	ND	NA
Carbon tetrachloride	ND	0.5
Chlorobenzene	ND	100.0
Chloroform	ND	6.0
1,2-Dichloroethane	ND	0.5
1,1-Dichloroethylene	ND	0.7
Isobutanol	ND	NA
Methylene chloride	ND	NA
Methyl ethyl ketone	ND	200.0
1,1,1,2-Tetrachloroethane	ND	NA
1,1,2,2-Tetrachloroethane	ND	NA
Tetrachloroethylene	ND	0.7
Toluene	ND	NA
1,1,1-Trichloroethane	ND	NA
1,1,2-Trichloroethane	ND	NA
Trichloroethylene	ND	0.5
Vinyl Chloride	ND	0.2

All units are mg/l unless otherwise noted.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-5a

AAR/RCI

TCLP TESTING OF 10 YEAR OLD CROSSTIE-SMALL CHIPS

NON-VOLATILES		ID. 38009
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.54	5.0
Barium	0.6	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.29	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.28	200.0
m-Cresol	ND	200.0
p-Cresol	0.94	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.30	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-6

AAR/RCI

TCLP TESTING OF 20 YEAR OLD CROSSTIE-SMALL CHIPS

VOLATILES		ID. 38009
Parameter	Analytical Results	1990 Final TCLP Standards
VOLATILE ORGANICS		
Acrylonitrile	ND	NA
Benzene	ND	0.5
Carbon disulfide	ND	NA
Carbon tetrachloride	ND	0.5
Chlorobenzene	ND	100.0
Chloroform	ND	6.0
1,2-Dichloroethane	ND	0.5
1,1-Dichloroethylene	ND	0.7
Isobutanol	ND	NA
Methylene chloride	ND	NA
Methyl ethyl ketone	ND	200.0
1,1,1,2-Tetrachloroethane	ND	NA
1,1,2,2-Tetrachloroethane	ND	NA
Tetrachloroethylene	ND	0.7
Toluene	ND	NA
1,1,1-Trichloroethane	ND	NA
1,1,2-Trichloroethane	ND	NA
Trichloroethylene	ND	0.5
Vinyl Chloride	ND	0.2

All units are mg/l unless otherwise noted.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-6a

AAR/RCI

TCLP TESTING OF 20 YEAR OLD CROSSTIE-SMALL CHIPS

NON-VOLATILES		ID. 38261
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.4	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.19	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.28	200.0
m-Cresol	ND	200.0
p-Cresol	1.2	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.22	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-7

AAR/RCI

TCLP TESTING OF NEW CROSSTIE - LARGE CHIPS

VOLATILES		ID. 38262
Parameter	Analytical Results	1990 Final TCLP Standards
VOLATILE ORGANICS		
Acrylonitrile	ND	NA
Benzene	ND	0.5
Carbon disulfide	ND	NA
Carbon tetrachloride	ND	0.5
Chlorobenzene	ND	100.0
Chloroform	ND	6.0
1,2-Dichloroethane	ND	0.5
1,1-Dichloroethylene	ND	0.7
Isobutanol	ND	NA
Methylene chloride	ND	NA
Methyl ethyl ketone	ND	200.0
1,1,1,2-Tetrachloroethane	ND	NA
1,1,2,2-Tetrachloroethane	ND	NA
Tetrachloroethylene	ND	0.7
Toluene	ND	NA
1,1,1-Trichloroethane	ND	NA
1,1,2-Trichloroethane	ND	NA
Trichloroethylene	ND	0.5
Vinyl Chloride	ND	0.2

All units are mg/l unless otherwise noted.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-7a

AAR/RCI

TCLP TESTING OF NEW CROSSTIE - LARGE CHIPS

NON-VOLATILES		ID. 38260
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.3	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.13	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	ND	200.0
m-Cresol	ND	200.0
p-Cresol	ND	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	ND	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-8

AAR/RCI

TCLP TESTING OF 10 YEAR OLD CROSSTIE-LARGE CHIPS

VOLATILES		ID. 38258
Parameter	Analytical Results	1990 Final TCLP Standards
VOLATILE ORGANICS		
Acrylonitrile	ND	NA
Benzene	ND	0.5
Carbon disulfide	ND	NA
Carbon tetrachloride	ND	0.5
Chlorobenzene	ND	100.0
Chloroform	ND	6.0
1,2-Dichloroethane	ND	0.5
1,1-Dichloroethylene	ND	0.7
Isobutanol	ND	NA
Methylene chloride	ND	NA
Methyl ethyl ketone	ND	200.0
1,1,1,2-Tetrachloroethane	ND	NA
1,1,2,2-Tetrachloroethane	ND	NA
Tetrachloroethylene	ND	0.7
Toluene	ND	NA
1,1,1-Trichloroethane	ND	NA
1,1,2-Trichloroethane	ND	NA
Trichloroethylene	ND	0.5
Vinyl Chloride	ND	0.2

All units are mg/l unless otherwise noted.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-8a

AAR/RCI

TCLP TESTING OF 10 YEAR OLD CROSSTIE-LARGE CHIPS

NON-VOLATILES		ID. 38259
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.010	5.0
Barium	0.5	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
Zinc	0.25	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.14	200.0
m-Cresol	ND	200.0
p-Cresol	0.37	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.072	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0
PESTICIDES/HERBICIDES		
Chlordane	ND	0.03
2,4-D	ND	10.0
Endrin	ND	0.02
Heptachlor (& its hydroxide)	ND	0.008
Hexachlorobenzene	ND	0.13
Lindane	ND	0.4
Methoxychlor	ND	10.0
Toxaphene	ND	0.5
2,4,5-TP (Silvex)	ND	1.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-9

AAR/RCI

TCLP TESTING OF 20 YEAR OLD CROSSTIE-LARGE CHIPS

VOLATILES		ID. 38257
Parameter	Analytical Results	1990 Final TCLP Standards
VOLATILE ORGANICS		
Acrylonitrile	ND	NA
Benzene	ND	0.5
Carbon disulfide	ND	NA
Carbon tetrachloride	ND	0.5
Chlorobenzene	ND	100.0
Chloroform	ND	6.0
1,2-Dichloroethane	ND	0.5
1,1-Dichloroethylene	ND	0.7
Isobutanol	ND	NA
Methylene chloride	ND	NA
Methyl ethyl ketone	ND	200.0
1,1,1,2-Tetrachloroethane	ND	NA
1,1,2,2-Tetrachloroethane	ND	NA
Tetrachloroethylene	ND	0.7
Toluene	ND	NA
1,1,1-Trichloroethane	ND	NA
1,1,2-Trichloroethane	ND	NA
Trichloroethylene	ND	0.5
Vinyl Chloride	ND	0.2

All units are mg/l unless otherwise noted.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-9a

AAR/RCI

TCLP TESTING OF 20 YEAR OLD CROSSTIE-LARGE CHIPS

TABLE A-10

AAR/RCI

TCLP CAGE METHOD TESTING OF
SAMPLE A-CSX NEW CROSSTIE

CSX New Mainline Crosstie Sample A

NON-VOLATILES		ID. 40769/40776
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.4	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.3	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.15	200.0
m-Cresol	ND	200.0
p-Cresol	0.61	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.22	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

NON-VOLATILES		ID. 40770/40777
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.1	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.44	200.0
m-Cresol	ND	200.0
p-Cresol	0.70	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.33	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

CSX New Mainline Crosstie Sample B

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-11

AAR/RCI

TCLP CAGE METHOD TESTING OF
SAMPLE B-CSX NEW CROSSTIE

CSX New Mainline Crosstie Sample C

NON-VOLATILES		ID. 40771/40778
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.002	5.0
Barium	0.3	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	0.3	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.40	200.0
m-Cresol	ND	200.0
p-Cresol	1.4	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.30	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-12

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE C-CSX NEW CROSSTIE

NON-VOLATILES		ID. 40772/40779
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.5	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.47	200.0
m-Cresol	ND	200.0
p-Cresol	1.8	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.30	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

CSX New Mainline Crosstie Sample D

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-13

AAR/RCI

TCLP CAGE METHOD TESTING OF
SAMPLE D-CSX NEW CROSSTIE

NON-VOLATILES		ID. 42335/42336
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.5	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	0.2	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether.	ND	NA
o-Cresol	0.062	200.0
m-Cresol	ND	200.0
p-Cresol	0.21	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.10	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

Koppers Co., North Little Rock, AR Railroad
Tie Sample A treated with petroleum/creosote
preservative. (Initial pH = 4.8, Extraction
Fluid #1 Used, Final pH = 4.9)

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-14

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE A
KOPPERS PETROLEUM/CREOSOTE
NEW CROSSTIE

NON-VOLATILES		ID. 42337/42338
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.004	5.0
Barium	0.3	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.064	200.0
m-Cresol	ND	200.0
p-Cresol	0.24	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.094	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

Koppers Co., North Little Rock, AR Railroad Tie Sample B treated with petroleum/creosote preservative. (Initial pH = 4.85, Extraction Fluid #1 Used, Final pH = 4.85)

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-15

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE B
KOPPERS PETROLEUM/CREOSOTE
NEW CROSSTIE

NON-VOLATILES		ID. 42339/42340
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.004	5.0
Barium	0.5	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.11	200.0
m-Cresol	ND	200.0
p-Cresol	0.34	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.18	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

Koppers Co., North Little Rock, AR Railroad Tie Sample C treated with petroleum/creosote preservative. (Initial pH = 4.8, Extraction Fluid #1 Used, Final pH = 4.95)

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-16

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE C
KOPPERS PETROLEUM/CREOSOTE
NEW CROSSTIE

NON-VOLATILES		ID. 42341/42342
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.008	5.0
Barium	0.2	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	3.9	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.085	200.0
m-Cresol	ND	200.0
p-Cresol	0.27	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.14	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

Koppers Co., North Little Rock, AR Railroad Tie Sample D treated with petroleum/creosote preservative. (Initial pH = 4.8, Extraction Fluid #1 Used, Final pH = 4.95)

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-17

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE D
KOPPERS PETROLEUM/CREOSOTE
NEW CROSSTIE

NON-VOLATILES		ID. 42343/42344
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	0.004	5.0
Barium	0.3	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.29	200.0
m-Cresol	ND	200.0
p-Cresol	1.0	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.54	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

Koppers Co., Grenada, MS Railroad Tie Sample A treated with Coal-Tar/Creosote preservative. (Initial pH = 4.8, Extraction Fluid #1 Used, Final pH = 4.9)

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-18

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE A
KOPPERS COAL-TAR/CREOSOTE
NEW CROSSTIE

NON-VOLATILES		ID. 42345/42346
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.2	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.23	200.0
m-Cresol	ND	200.0
p-Cresol	0.70	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.42	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

Koppers Co., Grenada, MS Railroad Tie Sample B treated with Coal-Tar/Creosote preservative. (Initial pH = 4.8, Extraction Fluid #1 Used, Final pH = 4.9)

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-19

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE B
KOPPERS COAL-TAR/CREOSOTE
NEW CROSSTIE

NON-VOLATILES		ID. 42347/42348
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.3	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.36	200.0
m-Cresol	ND	200.0
p-Cresol	1.2	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.91	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

Koppers Co., Grenada, MS Railroad Tie Sample C treated with Coal-Tar/Creosote preservative. (Initial pH = 4.8, Extraction Fluid #1 Used, Final pH = 4.95)

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-20

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE C
KOPPERS COAL-TAR/CREOSOTE
NEW CROSSTIE

NON-VOLATILES		ID. 42349/42350
Parameter	Analytical Results	1990 Final TCLP Standards
METALS		
Arsenic	<0.002	5.0
Barium	0.2	100.0
Cadmium	<0.005	1.0
Chromium	<0.02	5.0
Lead	<0.1	5.0
Mercury	<0.0002	0.2
Nickel	<0.02	NA
Selenium	<0.005	1.0
Silver	<0.01	5.0
Thallium	<0.002	NA
EXTRACTABLE ORGANICS		
Bis(2-chloroethyl)ether	ND	NA
o-Cresol	0.23	200.0
m-Cresol	ND	200.0
p-Cresol	0.71	200.0
1,2-Dichlorobenzene	ND	NA
1,4-Dichlorobenzene	ND	7.5
2,4-Dinitrotoluene	ND	0.13
Hexachlorobutadiene	ND	0.5
Hexachloroethane	ND	3.0
Nitrobenzene	ND	2.0
Pentachlorophenol	ND	100.0
Phenol	0.49	NA
Pyridine	ND	5.0
2,3,4,6-Tetrachlorophenol	ND	NA
2,4,5-Trichlorophenol	ND	400.0
2,4,6-Trichlorophenol	ND	2.0

Koppers Co., Grenada, MS Railroad Tie Sample D treated with Coal-Tar/Creosote preservative. (Initial pH = 4.8, Extraction Fluid #1 Used, Final pH = 4.95)

All units are mg/l unless otherwise noted.
*Standards not proposed.
ND = None detected at the appropriate quantitation limit (0.010 mg/l in most cases).
NA = Standards not promulgated

TABLE A-21

AAR/RCI

TCLP CAGE METHOD TESTING OF SAMPLE D
KOPPERS COAL-TAR/CREOSOTE
NEW CROSSTIE

APPENDIX B

METHOD 1311

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (<u>i.e.</u>, those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

2.3 If compatible (<u>i.e.</u>, multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device is for use only when the waste is being tested for the mobility of volatile analytes (i.e., those listed in Table 1). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see Section 4.3.1). The vessels shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON®[1] O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Section 7.3) refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

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[1] VITON® is a trademark of Du Pont.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Section 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Section 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in Section 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered (see Section 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known to EPA are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 μm, or equivalent. Filters known to EPA which meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to ± 0.05 units at 25 °C.

4.6 ZHE Extract Collection Devices: TEDLAR®[2] bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (<u>i.e.</u>, <1% of total waste), the TEDLAR® bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (<u>i.e.</u>, >1% of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (<u>e.g.</u>, a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (see Section 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, 500 mL.

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

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[2] TEDLAR® is a registered trademark of Du Pont.

4.11 Magnetic stirrer.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent Water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 ± 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

5.4 Nitric acid (1N), HNO_3, made from ACS reagent grade.

5.5 Sodium hydroxide (1N), NaOH, made from ACS reagent grade.

5.6 Glacial acetic acid, CH_3CH_2OOH, ACS reagent grade.

5.7 Extraction fluid.

5.7.1 Extraction fluid # 1: Add 5.7 mL glacial CH_3CH_2OOH to 500 mL of reagent water (See Section 5.2), add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

5.7.2 Extraction fluid # 2: Dilute 5.7 mL glacial CH_3CH_2OOH with reagent water (See Section 5.2) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.8 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction (see Section 1.4 concerning the use of this extract for volatile organics). If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4 °C. Samples should be opened only immediately prior to extraction).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Section 8.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 Preliminary Evaluations

Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (Section 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 7.1.4).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Section 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Section 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Section 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Section 7.1.1.9)}}{\text{Total weight of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in Section 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Section 7.1.3 to determine whether the solid material requires particle size reduction or to Section 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 7.1.1.9 is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed and to Section 7.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at 100 ± 20 °C until two successive weighing yield the same value within ± 1%. Record the final weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{(\text{Wt. of dry waste + filter}) - \text{tared wt. of filter}}{\text{Initial wt. of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed, and to Section 7.3 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Section 7.1.3) and determine the appropriate extraction fluid (Section 7.1.4). If only the volatile TCLP is to be performed, see the note in Section 7.1.4.

7.1.3 Determination of whether the waste requires particle size reduction (particle size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Section 7.3.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 7.2), determine the appropriate fluid (Section 5.7) for the nonvolatiles extraction as follows:

NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 5.7.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH from Section 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50 °C, and hold at 50 °C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 7.2.

7.1.5 If the aliquot of the waste used for the preliminary evaluation (Sections 7.1.1 - 7.1.4) was determined to be 100% solid at Section 7.1.1.1, then it can be used for the Section 7.2 extraction (assuming at least 100 grams remain), and the Section 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Section 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 7.3. The aliquot of the waste subjected to the procedure in Section 7.1.1.7 might be appropriate for use for the Section 7.2 extraction if an adequate amount of solid (as determined by Section 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Section 7.2.10 of the nonvolatile TCLP extraction.

7.2 Procedure When Volatiles are not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, See Section 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see Section 7.1.1), weigh out a subsample of the waste (100 gram minimum) and proceed to Section 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (Section 7.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (Sections 7.1.1 or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Section 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.2.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Section 7.2.12) or stored at 4 °C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the waste contains <0.5% dry solids (see Section 7.1.2), proceed to Section 7.2.13. If the waste contains >0.5% dry solids (see Section 7.1.1 or 7.1.2), and if particle size reduction of the solid was needed in Section 7.1.3, proceed to Section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 7.2.11.

7.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Section 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2 °C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Section 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.2.12 is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.2.12 with the initial liquid phase of the waste obtained in Section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Section 7.2.7, is not or may not be compatible with the filtered liquid resulting from Section 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4 °C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to $\pm$ 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\ (C_1) + (V_2)\ (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes (<u>e.g.</u>, metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4 °C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (See Section 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Section 4.6 are recommended for use under the conditions stated in Sections 4.6.1 - 4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the waste is 100% solid (see Section 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 7.3.5.

7.3.4 If the waste contains < 0.5% dry solids (Section 7.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing ≥ 0.5% dry solids (Sections 7.1.1 and/or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For wastes containing < 5% solids (see Section 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing ≥ 5% solids (see Section 7.1.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent solids (Section 7.1.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle size reduction of the solid portion of the waste was required in Section 7.1.3, proceed to Section 7.3.6. If particle size reduction was not required in Section 7.1.3, proceed to Section 7.3.7.

7.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 7.1.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4 °C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Section 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 7.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see Section 7.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (See Sections 7.3.13 through 7.3.15) or stored at 4 °C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.3.4 or 7.3.8)}}{100}$$

7.3.12 The following Sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Section 5.7).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction occurs) shall be maintained at 23 ± 2 °C during agitation.

7.3.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 7.3.9. All extract shall be filtered and collected if the TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Sections 4.6 and 7.3.1).

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.3.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Section 7.3.13 and the initial liquid phase (Section 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4 °C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)\ (C_1) + (V_2)\ (C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

8.0 QUALITY ASSURANCE

8.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

8.2 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.2.2 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$\%R\ (\%\text{Recovery}) = 100\ (X_s - X_u)/K$

where:
X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1. The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The forth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo TCLP extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES [Days]

	From: Field collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 METHOD PERFORMANCE

9.1 Ruggedness. Two ruggedness studies have been performed to determine the effect of various perturbations on specific elements of the TCLP protocol. Ruggedness testing determines the sensitivity of small procedural variations which might be expected to occur during routine laboratory application.

9.1.1 Metals - The following conditions were used when leaching a waste for metals analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Extraction time	16 hours vs. 18 hours
Headspace	20% vs. 60%
Buffer #2 acidity	190 meq vs. 210 meq
Acid-washed filters	yes vs. no
Filter type	0.7 μm glass fiber vs. 0.45 μm vs. polycarbonate
Bottle type	borosilicate vs. flint glass

Of the seven method variations examined, acidity of the extraction fluid had the greatest impact on the results. Four of 13 metals from an API separator sludge/electroplating waste (API/EW) mixture and two of three metals from an ammonia lime still bottom waste were extracted at higher levels by the more acidic buffer. Because of the sensitivity to pH changes, the method requires that the extraction fluids be prepared so that the final pH is within ± 0.05 units as specified.

9.1.2 Volatile Organic Compounds - The following conditions were used when leaching a waste for VOC analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Headspace	0% vs. 5%
Buffer #1 acidity	60 meq vs. 80 meq
Method of storing extract	Syringe vs. Tedlar• bag
Aliquotting	yes vs. no
Pressure behind piston	0 psi vs. 20 psi

None of the parameters had a significant effect on the results of the ruggedness test.

9.2 Precision. Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of waste materials).

9.2.1 Metals - The results of a multi-laboratory study are shown in Table 6, and indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements.

9.2.2 Semi-Volatile Organic Compounds - The results of two studies are shown in Tables 7 and 8. Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20 - 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (i.e., column degradation of the gas chromatograph) for the analytical conditions used.

9.2.3 Volatile Organic Compounds - Eleven laboratories participated in a collaborative study of the use of the ZHE with two waste types which were fortified with a mixture of VOCs. The results of the collaborative study are shown in Table 9. Precision results for VOCs tend to occur over a considerable range. However, the range and mean RSD compared very closely to the same collaborative study metals results in Table 6. Blackburn and Show concluded that at the 95% level of significance: 1) recoveries among laboratories were statistically similar, 2) recoveries did not vary significantly between the two sample types, and 3) each laboratory showed the same pattern of recovery for each of the two samples.

10.0 REFERENCES

1. Blackburn, W.B. and Show, I. "Collaborative Study of the Toxicity Characteristics Leaching Procedure (TCLP)." Draft Final Report, Contract No. 68-03-1958, S-Cubed, November 1986.

2. Newcomer, L.R., Blackburn, W.B., Kimmell, T.A. "Performance of the Toxicity Characteristic Leaching Procedure." Wilson Laboratories, S-Cubed, U.S. EPA, December 1986.

3. Williams, L.R., Francis, C.W.; Maskarinec, M.P., Taylor D.R., and Rothman, N. "Single-Laboratory Evaluation of Mobility Procedure for Solid Waste." EMSL, ORNL, S-Cubed, ENSECO.

Table 1.
Volatile Analytes[1,2]

Compound	CAS No.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1,-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

[1] When testing for any or all of these analytes, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

[2] Benzene, carbon tetrachloride, chlorobenzene, chloroform, 1,2-dichloroethane, 1,1-dichloroethylene, methyl ethyl ketone, tetrachloroethylene, and vinyl chloride are toxicity characteristic constituents.

Table 2.
Suitable Rotary Agitation Apparatus[1]

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel extractor (DC20S) 8-vessel extractor (DC20) 12-vessel extractor (DC20B) 24-vessel extractor (DC24C)
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	2-vessel (3740-2-BRE) 4-vessel (3740-4-BRE) 6-vessel (3740-6-BRE) 8-vessel (3740-8-BRE) 12-vessel (3740-12-BRE) 24-vessel (3740-24-BRE)
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	8-vessel (08-00-00) 4-vessel (04-00-00)
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	8-vessel (011001)
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel (10VRE) 5-vessel (5VRE) 6-vessel (6VRE)
Millipore Corp.	Bedford, MA (800) 225-3384	4-ZHE or 4 2-liter bottle extractor (YT31ORAHW)

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[1] Any device that rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

Table 3.
Suitable Zero-Headspace Extractor Vessels[1]

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device
Lars Lande Manufacturing[2]	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device
Millipore Corporation	Bedford, MA (800) 225-3384	YT30090HW, Gas Pressure Device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Pressure Device
Gelman Science	Ann Arbor, MI (800) 521-1520	15400 Gas Pressure Device

[1] Any device that meets the specifications listed in Section 4.2.1 of the method is suitable.

[2] This device uses a 110 mm filter.

Table 4.
Suitable Filter Holders[1]

Company	Location	Model/ Catalogue No.	Size
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

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[1] Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic analytes are of concern. The 142 mm size filter holder is recommended.

Table 5.
Suitable Filter Media[1]

Company	Location	Model	Pore Size (μm)
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	GF75	0.7
Gelman Science	Ann Arbor, MI (800) 521-1520	66256 (90mm) 66257 (142mm)	0.7

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[1] Any filter that meets the specifications in Section 4.4 of the Method is suitable.

Table 6. Multi-Laboratory TCLP Metals, Precision

Waste	Extraction Fluid	Metal	$\overline{X}$	S	%RSD
Ammonia Lime Still Bottoms	#1	Cadmium	0.053	0.031	60
	#2		0.023	0.017	76
	#1	Chromium	0.015	0.0014	93
	#2		0.0032	0.0037	118
	#1	Lead	0.0030	0.0027	90
	#2		0.0032	0.0028	87
API/EW Mixture	#1	Cadmium	0.0046	0.0028	61
	#2		0.0005	0.0004	77
	#1	Chromium	0.0561	0.0227	40
	#2		0.105	0.018	17
	#1	Lead	0.0031	0.0031	100
	#2		0.0124	0.0136	110
Fossil Fuel Fly Ash	#1	Cadmium	0.080	0.069	86
	#2		0.093	0.067	72
	#1	Chromium	0.017	0.014	85
	#2		0.070	0.040	57
	#1	Lead	0.0087	0.0074	85
	#2		0.0457	0.0083	18

%RSD Range = 17 - 118
Mean %RSD = 74

NOTE: $\overline{X}$ = Mean results from 6 - 12 different laboratories
Units = mg/L
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 7. Single-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	Phenol	#1	19000	2230	11.6
		#2	19400	929	4.8
	2-Methylphenol	#1	2000	297	14.9
		#2	1860	52.9	2.8
	4-Methylphenol	#1	7940	1380	17.4
		#2	7490	200	2.7
	2,4-Dimethylphenol	#1	321	46.8	14.6
		#2	307	45.8	14.9
	Naphthalene	#1	3920	413	10.5
		#2	3827	176	4.6
	2-Methylnaphthalene	#1	290	44.8	15.5
		#2	273	19.3	7.1
	Dibenzofuran	#1	187	22.7	12.1
		#2	187	7.2	3.9
	Acenaphthylene	#1	703	89.2	12.7
		#2	663	20.1	3.0
	Fluorene	#1	151	17.6	11.7
		#2	156	2.1	1.3
	Phenanthrene	#1	241	22.7	9.4
		#2	243	7.9	3.3
	Anthracene	#1	33.2	6.19	18.6
		#2	34.6	1.55	4.5
	Fluoranthrene	#1	25.3	1.8	7.1
		#2	26.0	1.8	7.1
API/EW Mixture	Phenol	#1	40.7	13.5	33.0
		#2	19.0	1.76	9.3
	2,4-Dimethylphenol	#1	33.0	9.35	28.3
		#2	43.3	8.61	19.9
	Naphthalene	#1	185	29.4	15.8
		#2	165	24.8	15.0
	2-Methylnaphthalene	#1	265	61.2	23.1
		#2	200	18.9	9.5
				%RSD Range = 1 - 33 Mean %RSD = 12	

NOTE: Units = μg/L
Extractions were performed in triplicate
All results were at least 2x the detection limit
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 8. Multi-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms (A)	BNAs	#1	10043	7680	76.5
		#2	10376	6552	63.1
API/EW Mixture (B)	BNAs	#1	1624	675	41.6
		#2	2074	1463	70.5
Fossil Fuel Fly Ash (C)	BNAs	#1	750	175	23.4
		#2	739	342	46.3
					Mean %RSD = 54

NOTE: Units = μg/L
$\bar{X}$ = Mean results from 3 - 10 labs
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

%RSD Range for Individual Compounds

A, #1	0 - 113
A, #2	28 - 108
B, #1	20 - 156
B, #2	49 - 128
C, #1	36 - 143
C, #2	61 - 164

Table 9. Multi-Laboratory (11 Labs) VOCs, Precision

Waste	Compound	$\bar{X}$	S	%RSD
Mine Tailings	Vinyl chloride	6.36	6.36	100
	Methylene chloride	12.1	11.8	98
	Carbon disulfide	5.57	2.83	51
	1,1-Dichloroethene	21.9	27.7	127
	1,1-Dichloroethane	31.4	25.4	81
	Chloroform	46.6	29.2	63
	1,2-Dichloroethane	47.8	33.6	70
	2-Butanone	43.5	36.9	85
	1,1,1-Trichloroethane	20.9	20.9	100
	Carbon tetrachloride	12.0	8.2	68
	Trichloroethene	24.7	21.2	86
	1,1,2-Trichloroethene	19.6	10.9	56
	Benzene	37.9	28.7	76
	1,1,2,2-Tetrachloroethane	34.9	25.6	73
	Toluene	29.3	11.2	38
	Chlorobenzene	35.6	19.3	54
	Ethylbenzene	4.27	2.80	66
	Trichlorofluoromethane	3.82	4.40	115
	Acrylonitrile	76.7	110.8	144
Ammonia Lime Still Bottoms	Vinyl chloride	5.00	4.71	94
	Methylene chloride	14.3	13.1	92
	Carbon disulfide	3.37	2.07	61
	1,1-Dichloroethene	52.1	38.8	75
	1,1-Dichloroethane	52.8	25.6	49
	Chloroform	64.7	28.4	44
	1,2-Dichloroethane	43.1	31.5	73
	2-Butanone	59.0	39.6	67
	1,1,1-Trichloroethane	53.6	40.9	76
	Carbon tetrachloride	7.10	6.1	86
	Trichloroethene	57.3	34.2	60
	1,1,2-Trichloroethene	6.7	4.7	70
	Benzene	61.3	26.8	44
	1,1,2,2-Tetrachloroethane	3.16	2.1	66
	Toluene	69.0	18.5	27
	Chlorobenzene	71.8	12.0	17
	Ethylbenzene	3.70	2.2	58
	Trichlorofluoromethane	4.05	4.8	119
	Acrylonitrile	29.4	34.8	118

%RSD Range = 17 - 144
Mean %RSD = 75

NOTE: Units = µg/L

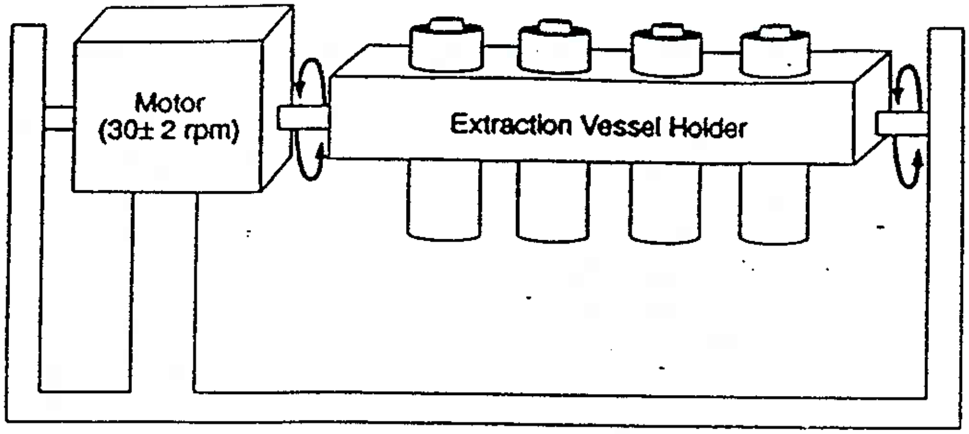


Figure 1. Rotary Agitation Apparatus

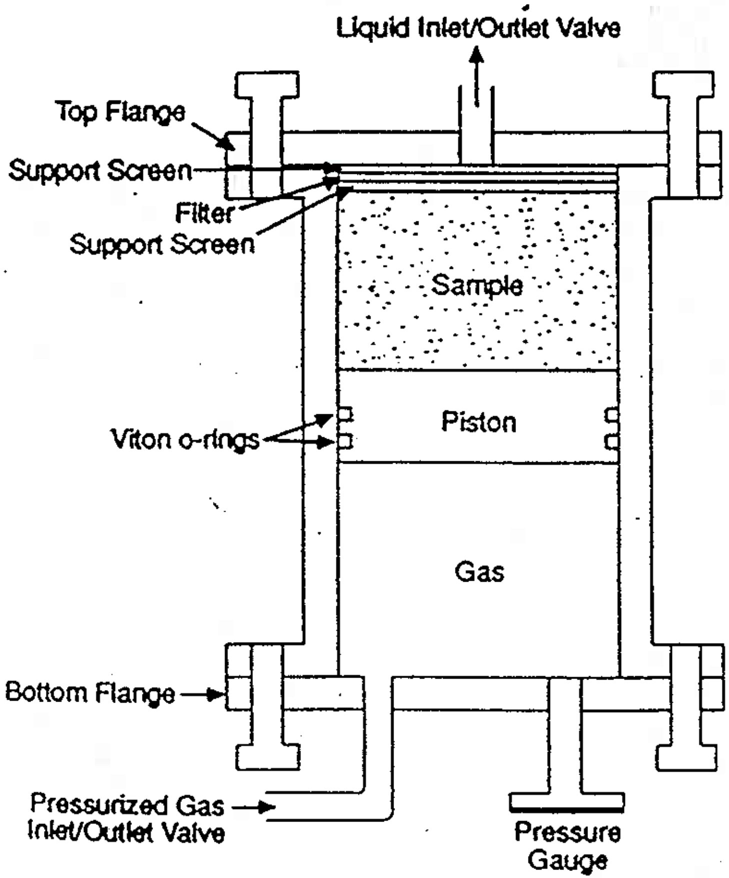
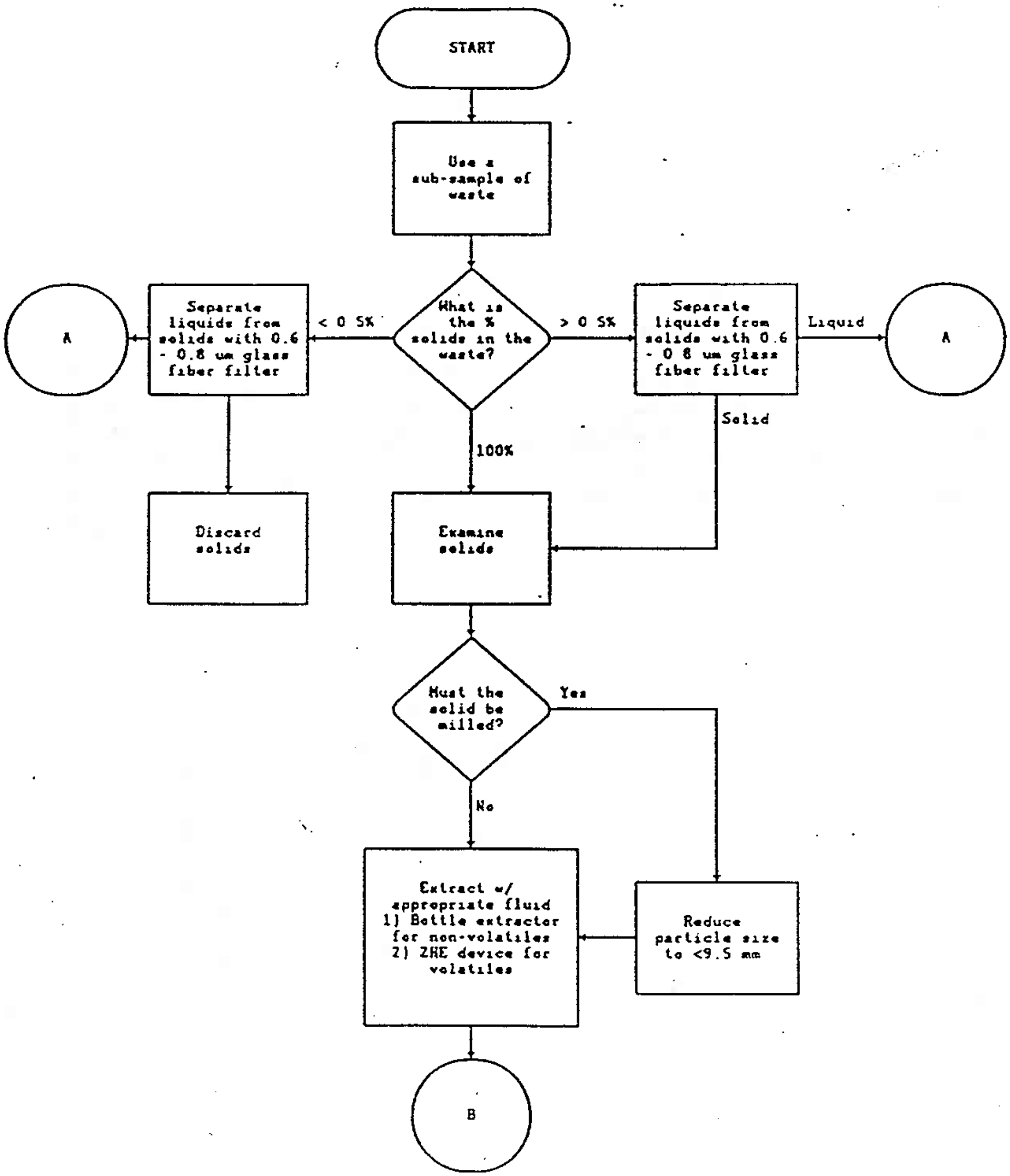


Figure 2. Zero-Headspace Extractor (ZHE)

METHOD 1311

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



METHOD 1311 (CONTINUED)

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE