

Appendix I

Toxicity Characteristics Leaching Procedure (TCLP)

1.0 SCOPE AND APPLICATION

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

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

2.0 SUMMARY OF METHOD (see Figure 1)

2.1 For liquid wastes (i.e., those containing insignificant solid material), the waste, after filtration through a 0.6- to 0.8-micron (μm) glass fiber filter, is defined as the TCLP extract.

2.2 For wastes comprised of solids or for wastes containing significant amounts of solid material, the particle-size of the waste is reduced (if necessary), the liquid phase, if any, is separated from the solid phase and stored for later analysis. 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 volatiles (see Table 1). Following extraction, the liquid extract is separated from the solid phase by 0.6- to 0.8- μm glass fiber filter filtration.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these liquids 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: An acceptable agitation apparatus is one which is capable of rotating the extraction vessel in an end-over-end fashion (see Figure 2) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessel:

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 constituents (see Table 1). The ZHE is an extraction vessel that allows for liquid/solid separation within the device, and which effectively precludes headspace (as depicted in Figure 3). This type of vessel allows for initial liquid/solid separation, extraction and final extract filtration without having to open the vessel (see Step 4.3.1). These vessels shall have an

internal volume of 500 to 600 mL and be equipped to accommodate a 90-mm filter. Suitable ZHE devices known to EPA are identified in Table 3. These devices contain viton O-rings which should be replaced frequently.

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 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.

4.2.2 When the waste is being evaluated for other than volatile contaminants, an extraction vessel that does not preclude headspace (e.g., a 2-liter bottle) is used. Suitable extraction vessels include bottles made from various materials, depending on the contaminants to be analyzed and the nature of the waste (see Step 4.3.3). It is recommended that borosilicate glass bottles be used over other types of glass, especially when inorganics are of concern. Plastic bottles may be used only if inorganics 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 Step 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.2.3 Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Section 9.0) 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.

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

4.3.1 Zero-Headspace Extractor Vessel (see Figure 3): When the waste is being evaluated for volatiles, the zero-headspace extraction vessel 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 being evaluated for other than volatile compounds, a filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation is 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 Step 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 is only recommended 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. Filter holders known to EPA to be suitable for use 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 or polyvinyl chloride 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 constituents 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-um, or equivalent. Filters known to EPA to meet these specifications are identified in Table 5. Pre-filters may not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1.0 N nitric acid followed by 3 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: Any of the commonly available pH meters are acceptable.

4.6 ZHE extract collection devices: TEDLAR® 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 non-aqueous liquid (i.e., <1% of total waste), the TEDLAR® bag should be used to collect and combine the initial liquid and solid extract. The syringe is not recommended in these cases.

4.6.2 If a waste contains a significant amount of non-aqueous initial liquid phase (i.e., >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 (e.g., a constant displacement pump, a gas tight syringe, pressure filtration unit (see Step 4.3.2) or another 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).

5.0 REAGENTS

5.1 Reagent water: Reagent water is defined as water in which an interferant is not observed at or above the method detection limit of the analyte(s) of interest. For non-volatile 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.

5.1.1 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).

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

5.1.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at $90 \pm 5^\circ \text{C}$, 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 screwcap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.2 1.0 N Hydrochloric acid (HCl) made from ACS reagent grade.

5.3 1.0 N Nitric acid (HNO₃) made from ACS reagent grade.

5.4 1.0 N Sodium hydroxide (NaOH) made from ACS reagent grade.

5.5 Glacial acetic acid (HOAc) ACS reagent grade.

5.6 Extraction fluid:

5.6.1 Extraction fluid #1: This fluid is made by adding 5.7 mL glacial HOAc to 500 mL of the appropriate water (see Step 5.1), adding 64.3 mL of 1.0 N NaOH, and diluting to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05 .

5.6.2 Extraction fluid #2: This fluid is made by diluting 5.7 mL glacial HOAc with ASTM Type II water (see Step 5.1) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05 .

Note: It is suggested that these extraction fluids be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately.

5.7 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 At least 2 separate representative samples of a waste should be collected. If volatile organics are of concern, a third sample shall be collected. The first sample is used in several preliminary TCLP evaluations (e.g., to determine the percent solids of the waste; to determine if the

waste contains insignificant solids (i.e., the waste is its own extract after filtration); to determine if the solid portion of the waste requires particle-size reduction; and to determine which of the 2 extraction fluids are to be used for the non-volatile TCLP extraction of the waste). These preliminary evaluations are identified in Section 7.0. The second and, if required, third samples are extracted using the TCLP non-volatile procedure (Section 8.0) and volatile procedure (Section 9.0), respectively.

6.3 Preservatives may not be added to samples.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste (e.g., precipitation).

6.5 When the waste is to be evaluated for volatile contaminants, care should be taken to minimize the loss of volatiles. Samples shall be taken and stored in a manner to prevent the loss of volatile contaminants. If possible, it is recommended that any necessary particle-size reduction should be conducted as the sample is being taken (see Step 8.5).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. If they need to be stored, even for a short period of time, storage shall be a 4° C, and samples for volatiles analysis may not be allowed to come into contact with the atmosphere (i.e., no headspace). See Section 10.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PRELIMINARY TCLP EVALUATIONS

The preliminary TCLP evaluations are performed on a minimum 100 gram representative sample of waste that will not actually undergo TCLP extraction (designated as the first sample in Step 6.2). These evaluations include preliminary determination of the percent solids of the waste; determination of whether the waste contains insignificant solids, and is therefore, its own extract after filtration; determination of whether the solid portion of the waste requires particle-size reduction; and determination of which of the two extraction fluids are to be used for the non-volatile TCLP extraction of the waste.

7.1 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 follows.

7.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Step 7.4.

7.1.2 If the sample is liquid or multiphase, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 7.1.3 to 7.1.9.

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

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

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

7.1.6 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.

7.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). 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 Step 7.1.5 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), filtration is stopped.

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

7.1.8 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. But even after applying vacuum or pressure filtration, as outlined in Step 7.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. The original filter is not to be replaced with a fresh filter under any circumstances. Only one filter is used.

7.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Step 7.1.3) from the total weight of the filtrate-filled container. The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Step 7.1.5 or 7.1.7. Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Step 7.1.9)}}{\text{Total weight of waste (Step 7.1.5 or 7.1.7)}} \times 100$$

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7.2 Determination of whether waste is liquid or has insignificant amounts of solid material: If the sample obviously has a significant amount of solid material, the solid phase shall be subjected to extraction; proceed to Step 7.3 to determine if the waste requires particle-size reduction (and to reduce particle-size, if necessary). Determine whether the waste is liquid or has insignificant amounts of solid material (which need not undergo extraction) as follows:

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

7.2.2 Dry the filter and solid phase at $100 \pm 20^\circ \text{C}$ until two successive weighings yield the same value within $\pm 1\%$. Record final weight.

Note: Caution should be taken to insure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or appropriate device. 7.2.3 Calculate the percent dry solids as follows:

Percent dry solids =

$$\frac{\text{Weight of dry waste and filter} - \text{tared weight of filter}}{\text{Initial weight of waste (Step 7.1.5 or 7.1.1)}} \times 100$$

7.2.4 If the percent dry solids is less than 0.5%, consult Step 6.2 and proceed to Section 8.0 if nonvolatiles in the waste are of concern, and to Section 9.0 if volatiles are of interest. In this case, the waste, after filtration is defined as the TCLP extract. If the percent dry solids is greater than or equal to 0.5%, and if the non-volatile TCLP is to be performed, return to the beginning of this Section (7.0) with a new representative waste sample, so that it can be determined if particle-size reduction is necessary (Step 7.3), and so that the appropriate extraction fluid may be determined (Step 7.4) on a fresh portion of the solid phase of the waste. If only the volatile TCLP is to be performed, see the Note in Step 7.4.

7.3 Determination of whether the wastes require particle-size reduction (particle-size is reduced during this Step): Using the solid portion of the waste, evaluate the solid for particle-size. If 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 (e.g., is capable of passing through a 9.5-mm (0.375-inch) standard sieve), particle-size reduction is not required (proceed to Step 7.4). If the surface area is smaller or the particle-size larger than described above, the solid portion of the waste is prepared for extraction by crushing, cutting or grinding the waste to a surface area or particle-size as described above.

Note: Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not required; nor is it recommended.

7.4 Determination of appropriate extraction fluid: If the solid content is greater than or equal to 0.5% of the waste and if TCLP extraction for non-volatile constituents will take place (Section 8.0), determination of the appropriate fluid (Step 5.6) to use for the non-volatiles extraction is performed as follows.

Note: TCLP extraction for volatile constituents entails using only extraction fluid #1 (Step 5.6.1). Therefore, if TCLP extraction for non-volatiles extraction is not required, proceed to section 9.0.

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

7.4.2 Add 96.5 mL of reagent water (ASTM Type II) 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, extraction fluid #1 is used. Proceed to Section 8.0.

7.4.3 If the pH from Step 7.4.2 is > 5.0, add 3.5 mL 1.0 N HCl, slurry briefly, cover with a watchglass, heat to 50° C, and hold at 50° C for 10 minutes.

7.4.4 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 8.0.

7.5 The sample of waste used for performance of this Section may not be used any further. Other samples of the waste (see Step 6.2) shall be employed for the Section 8.0 and 9.0 extractions.

8.0 PROCEDURE WHEN VOLATILES ARE NOT INVOLVED

Although a minimum sample size of 100 grams (solid and liquid phases) is required, a larger sample size may be more appropriate, depending on the solids content of the waste sample (percent solids, see Step 7.1), 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 the performance of a single TCLP extraction will not be sufficient to perform all of the analyses to be conducted, it is recommended that more than one extraction be performed and that the extracts from each extraction be combined and then aliquoted for analysis.

8.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see Step 7.1), weigh out a representative subsample of the waste (100 gram minimum) and proceed to Step 8.9.

8.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Step 4.3.2 and is outlined in Steps 8.3 to 8.8.

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

8.4 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 Step 4.4).

Note: Acid washed filters may be used for all non-volatile extractions even when metals are not of concern.

8.5 Weigh out a representative subsample of the waste (100 gram minimum) and record the weight. If the waste was shown to contain <0.5% dry solids (Step 7.2), 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 (Steps 7.1 or 7.2), use the percent solids information obtained in Step 7.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated after filtration to support the analyses to be performed on the TCLP extract.

8.6 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.

8.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Step 4.3.2). 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 Step 8.5, 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 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), filtration is stopped.

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

8.8 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 Step 8.13) 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. But even after applying vacuum or pressure filtration, as outlined in Step 8.7, 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. The original filter is not to be replaced with a fresh filter under any circumstances. Only one the filter is used.

8.9 If the waste contains <0.5% dry solids (see Step 7.2), proceed to Step 8.13. If the waste contains - 0.5% dry solids (see Step 7.1 or 7.2), and if particle-size reduction of the solid was needed in Step 7.3, proceed to Step 8.10. If particle-size reduction was not required in Step 7.3, quantitatively transfer the solid material into the extractor vessel, including

the filter used to separate the initial liquid from the solid phase. Proceed to Step 8.11.

8.10 The solid portion of the waste is prepared for extraction by crushing, cutting or grinding the waste to a surface area of particle-size as described in Step 7.3. When the surface area of particle-size has been appropriately altered, quantitatively transfer the solid material into the extractor vessel, including the filter used to separate the initial liquid from the solid phase.

Note: Sieving of the waste through a sieve that is not Teflon coated should not be done due to avoid possible contamination of the sample. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

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

Weight of extraction fluid =

$$20 \times \frac{\% \text{ solids (Step 7.1)} \times \text{weight of waste filtered (Step 8.5 or 8.7)}}{100}$$

100

Slowly add this amount of appropriate extraction fluid (see Step 7.4) 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 extractor device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction is to take place) shall be maintained at $22 \pm 3^\circ$ C during the extraction period.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., lined 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.

8.12 Following the 18 ± 2 hour extraction, the material in the extractor vessel is separated into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Step 8.7. 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 Step 4.4) if evaluating the mobility of metals.

8.13 The TCLP extract is now prepared as follows:

8.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Step 8.12 is defined as the TCLP extract. Proceed to Step 8.14.

8.13.2 If compatible (e.g., multiple phases will not result on combination), the filtered liquid resulting from Step 8.12 is combined with the

initial liquid phase of the waste as obtained in Step 8.7. This combined liquid is defined as the TCLP extract. Proceed to Step 8.14.

8.13.3 If the initial liquid phase of the waste, as obtained from Step 8.7, is not or may not be compatible with the filtered liquid resulting from Step 8.12, these liquids are not combined. These liquids, collectively defined as the TCLP extract, are analyzed separately, and the results are combined mathematically. Proceed to Step 8.14.

8.14 Following collection of the TCLP extract, it is recommended that the pH of the extract be recorded. The extract shall be immediately aliquoted for analysis and properly preserved (metals aliquots shall be acidified with nitric acid to pH < 2; all other aliquots shall 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, other than mercury, shall be acid digested. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volumeweighted average:

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

where:

V1 = The volume of the first phase (L).

C1 = The concentration of the contaminant of concern in the first phase (mg/L).

V2 = The volume of the second phase (L).

C2 = The concentration of the contaminant of concern in the second phase (mg/L).

8.15 The contaminant concentrations in the TCLP extract are compared with the thresholds identified in the appropriate regulations. Refer to Section 10.0 for quality assurance requirements.

9.0 PROCEDURE WHEN VOLATILES ARE INVOLVED

The ZHE device is used to obtain TCLP extracts for volatile analysis only. Extract resulting from the use of the ZHE may not be used to evaluate the mobility of non-volatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500-mL internal capacity. Although a minimum sample size of 100 grams was required in the Section 8.0 procedure, the ZHE can only accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no liquid (additional) 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.

The ZHE is charged with sample only once and the device is not opened until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted. The initial filtrate shall be weighed and then stored at 4° C until either analyzed or recombined with the final extract of the solid.

Although the following procedure allows for particle-size reduction during the conduct of the procedure, this could result in the loss of volatile compounds. If possible (e.g., particle-size may be reduced easily by crumbling), particle-size reduction (see Step 9.2) should be conducted on the sample as it is being taken. If necessary, particle-size reduction may be conducted during the procedure.

In carrying out the following steps, 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.

9.1 Pre-weigh the (evacuated) container which will receive the filtrate (see Step 4.6), and set aside. If using a TEDLAR® bag, all liquid shall be expressed from the device, whether it be for the initial or final liquid/solid separation, and an aliquot taken from the liquid in the bag, for analysis. The containers listed in Step 4.6 are recommended for use under the following conditions.

9.1.1 If a waste contains an aqueous liquid phase or if the waste does not contain a significant amount of non-aqueous liquid (i.e., <1% of total waste), the TEDLAR® bag shall be used to collect and combine the initial liquid and solid extract. The syringe is not recommended in these cases.

9.1.2 If a waste contains a significant amount of non-aqueous initial liquid phase (i.e., 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.

9.1.3 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 liquid expressed from the device. The remaining aliquots are used for analysis.

9.2 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 9.0, Step 7.1 and/or 7.2). 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.

9.3 If the waste is 100% solid (see Step 7.1), weigh out a representative subsample (25 gram maximum) of the waste, record weight, and proceed to Step 9.5.

9.4 If the waste was shown to contain <0.5% dry solids (Step 7.2), the waste, after filtration is defined as the TCLP extract. Enough of the sample should be filtered so that the amount of filtered liquid will support all

of the volatile analyses required. For wastes containing >0.5% dry solids (Steps 7.1 and/or 7.2), use the percent solids information obtained in Step 7.1 to determine the optimum sample size to charge into the ZHE. The appropriate sample size recommended is as follows:

9.4.1 For wastes containing >5% solids (see Step 7.1), weigh out a representative 500 gram sample of waste and record the weight.

9.4.2 For wastes containing >5% solids (see Step 7.1), the amount of waste to charge into the ZHE is determined as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\% \text{ solids (Step 7.1)}} \times 100$$

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

9.5 If particle-size reduction of the solid portion of the waste was required in Step 7.3, proceed to Step 9.6. If particle-size reduction was not required in Step 7.3, proceed to Step 9.7.

9.6 The waste is prepared for extraction by crushing, cutting or grinding the solid portion of the waste to a surface area or particle-size as described in Step 7.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4° C prior to particle-size reduction. The means used to effect particlesize reduction may 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 shall 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 Step 9.7.

9.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Wastes that settle slowly may not be centrifuged prior to filtration.

9.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens into 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 extraction 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 Step 9.4, 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 (into a hood) slowly out of the ZHE device. 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 Step 7.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Step 9.12.

9.9 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 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 any 2-minute period, filtration is stopped. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtrate collection container.

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

9.10 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. But 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 Step 7.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Step 9.15.

9.11 The liquid phase may now be either analyzed immediately (see Steps 9.13 to 9.15) or stored at 4°C under minimal headspace conditions until time of analysis. The weight of extraction fluid #1 to add to the ZHE is determined as follows:

Weight of extraction fluid =

$20 \times \% \text{ solids (Step 7.1)} \times \text{weight of waste filtered (Step 9.4 or 9.8)}$

100

9.12 The following steps 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 Step 5.6).

9.12.1 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.

9.12.2 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. Physically 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. Put 5-10 psi behind the piston (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.

9.12.3 Place the ZHE in the rotary extractor apparatus (if it is not already there) and rotate the ZHE at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction is to occur) shall be maintained at $22 \pm 3^\circ$ C during agitation.

9.13 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 Step 4.2.1, and redo the extraction 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, unless doing so would create multiple phases, or unless there is not enough volume left within the filtrate collection container. A separate filtrate collection container shall be used in these cases. Filter through the glass fiber filter, using the ZHE device as discussed in Step 9.9. All extract shall be filtered and collected in the TEDLAR[®] bag is used, if the extract is multiphase, or if the waste contained an initial liquid phase (see Steps 4.6 and 9.1).

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

9.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Step 9.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Step 9.13 and the initial liquid phase (Step 9.9) are collectively defined as the TCLP extract.

9.15 Following collection of the TCLP extract, the extract shall be immediately aliquoted for analysis and stored with minimal headspace at 4° C until analyzed. The TCLP extract will be prepared and analyzed 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 $\pm 0.5\%$), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

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

V1 = The volume of the first phases (L).

C1 = The concentration of the contaminant of concern in the first phase (mg/L).

V2 = The volume of the second phase (L).

C2 = The concentration of the contaminant of concern in the second phase (mg/L).

9.16 The contaminant concentrations in the TCLP extract are compared with the thresholds identified in the appropriate regulations. Refer to Section 10.0 for quality assurance requirements.

10.0 QUALITY ASSURANCE REQUIREMENTS

10.1 All data, including quality assurance data, shall be maintained and available for reference or inspection.

10.2 A minimum of one blank (extraction fluid #1) for every 10 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment are occurring.

10.3 For each analytical batch (up to 20 samples), it is recommended that a matrix spike be performed. Addition of matrix spikes should occur once the TCLP extract has been generated (i.e., should not occur prior to performance of the TCLP procedure). The purpose of the matrix spike is to monitor the adequacy of the analytical methods used on the TCLP extract and for determining if matrix interferences exist in analyte detection.

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

10.5 The method of standard addition shall be employed for each analyte if: 1) recovery of the compound from the TCLP extract is not between 50 and 150%, or 2) if the concentration of the constituent measured in the extract is within 20% of the appropriate regulatory threshold. If more than one extraction is being run on samples of the same

Appendix

waste (up to 20 samples), the method of standard addition need be applied only once and the percent recoveries applied to the remainder of the extractions.

10.6 Samples shall undergo TCLP extraction within the following time period after sample receipt: Volatiles, 14 days; Semi-Volatiles, 40 days; Mercury, 28 days; and other Metals, 180 days. Extraction of the solid portion of the waste should be initiated as soon as possible following initial solid/liquid separation. TCLP extracts shall be analyzed after generation and preservation within the following periods: Volatiles, 14 days; Semi-Volatiles, 40 days; Mercury, 28 days; and other Metals, 180 days.

Table 1-Volatile Contaminants*

<u>Compounds</u>	<u>CAS No.</u>
Acetone	67-64-1
n-Butyl alcohol	71-36-6
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
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
Xylene	1330-20-7

*Includes compounds identified in the Land Disposal Restrictions Rule. If any or all of these compounds are of concern, the zero-headspace extractor vessel shall be used. If other (non-volatile) compounds are of concern, the conventional bottle extractor shall be used.

Table 2. Suitable Rotary Agitation Apparatus¹

<u>Company</u>	<u>Location</u>	<u>Model</u>
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	4-vessel device, 6-vessel device
Lars Lande Manufacturing	Whitemore Lake, MI (313) 449-4116	10-vessel device, 5-vessel device,
IRA Machine Shop and Laboratory	Santurce, PR	16-vessel device (809) 752-4004
EPRI Extractor	6-vessel device ²	
REXNORD	Milwaukee, WI (414) 643-2850	6-vessel device
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel device

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

² Although this device is suitable, it is not commercially made, it may also require retrofitting to accommodate ZHE devices.

Table 3. Suitable Zero-Headspace Extractor Vessels

<u>Company</u>	<u>Location</u>	<u>Model</u>
Associated Design & Manufacturing Co.	Alexandria, VA (703) 549-5999	3740-ZHB, Gas Pressure Device
Millipore Corp.	Bedford, MA (800) 225-3384	SD1 P581 C5, Gas Pressure Device
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device

Table 4. Suitable Filter Holders¹

<u>Company</u>	<u>Location</u>	<u>Model</u>	<u>Size</u>
Nuclepore Corp.	Pleasanton, CA (800) 882-7711	425910	142mm
		410400	47mm
Micro Filtration Systems	Dublin, CA (415) 828-6010	302400	142mm
Millipore Corp.	Bedford, MA (800) 225-3384	YT30142HW	142mm
		XX1004700	47mm

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 contaminants are of concern. The 142 mm size filter holder is recommended.

Table 5. Suitable Filter Media

<u>Company</u>	<u>Location</u>	<u>Model</u>	<u>Pore Size¹</u>
Whitman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7

¹ Nominal pore size.

FIGURE 1 - TCLP FLOW CHART

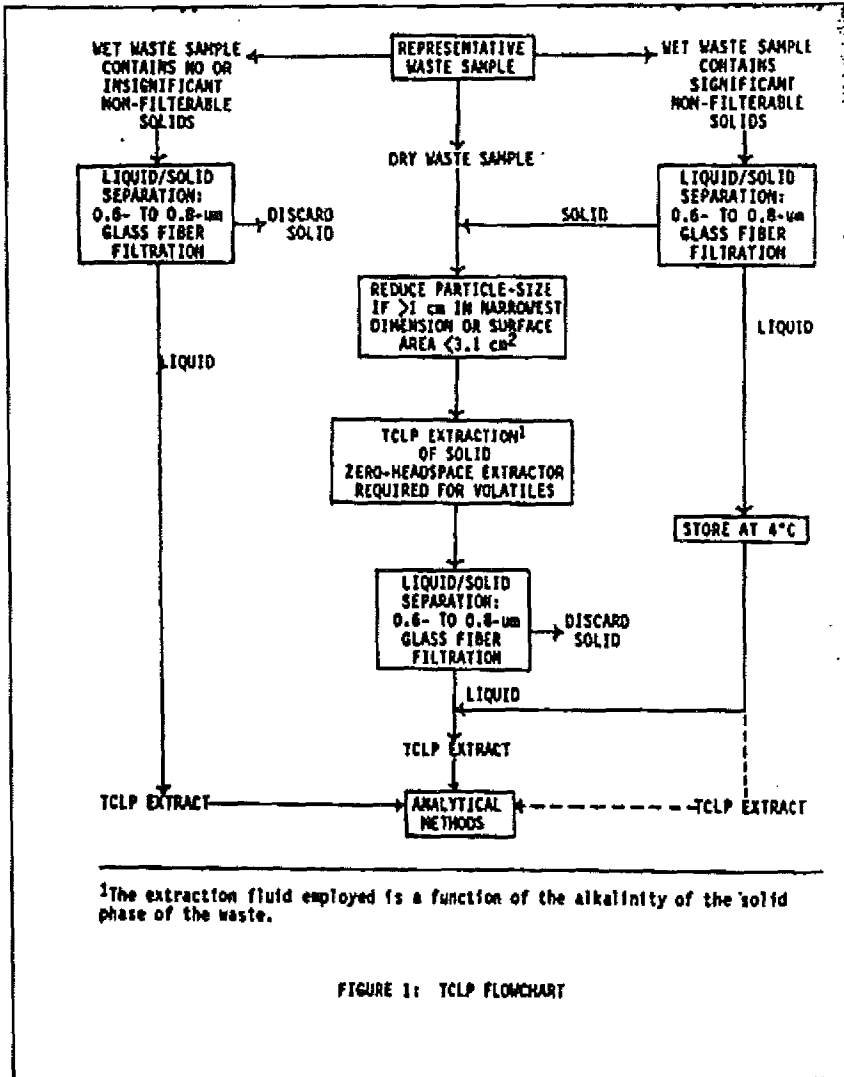


FIGURE 1: TCLP FLOWCHART

FIGURE 2 - ROTARY AGITATION

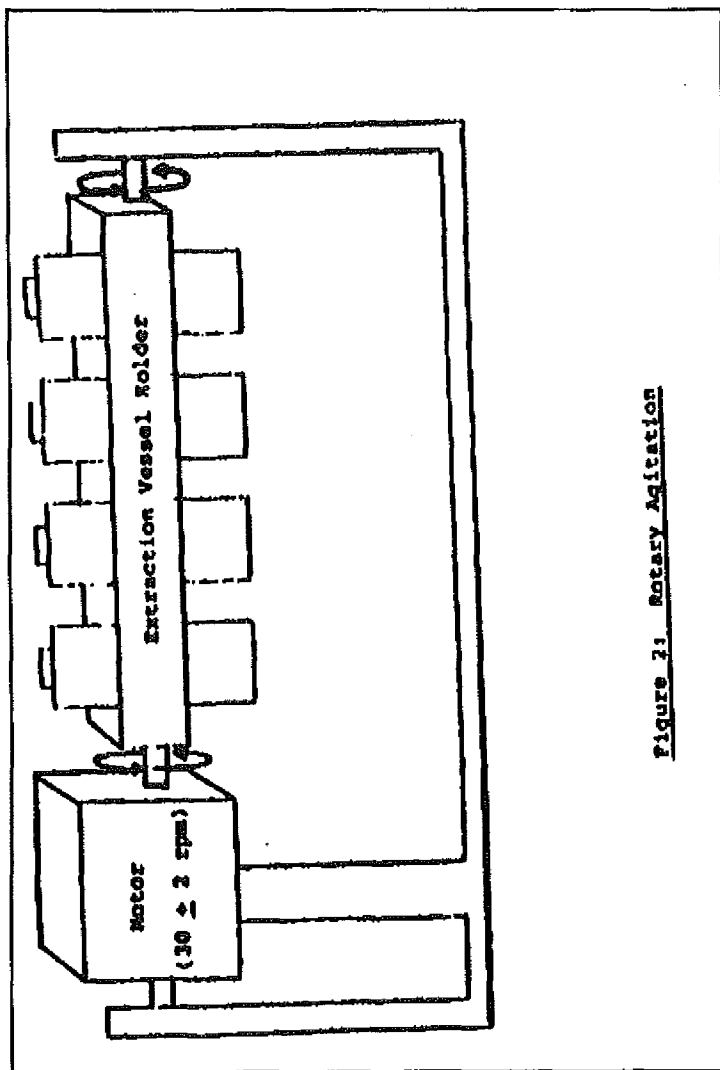
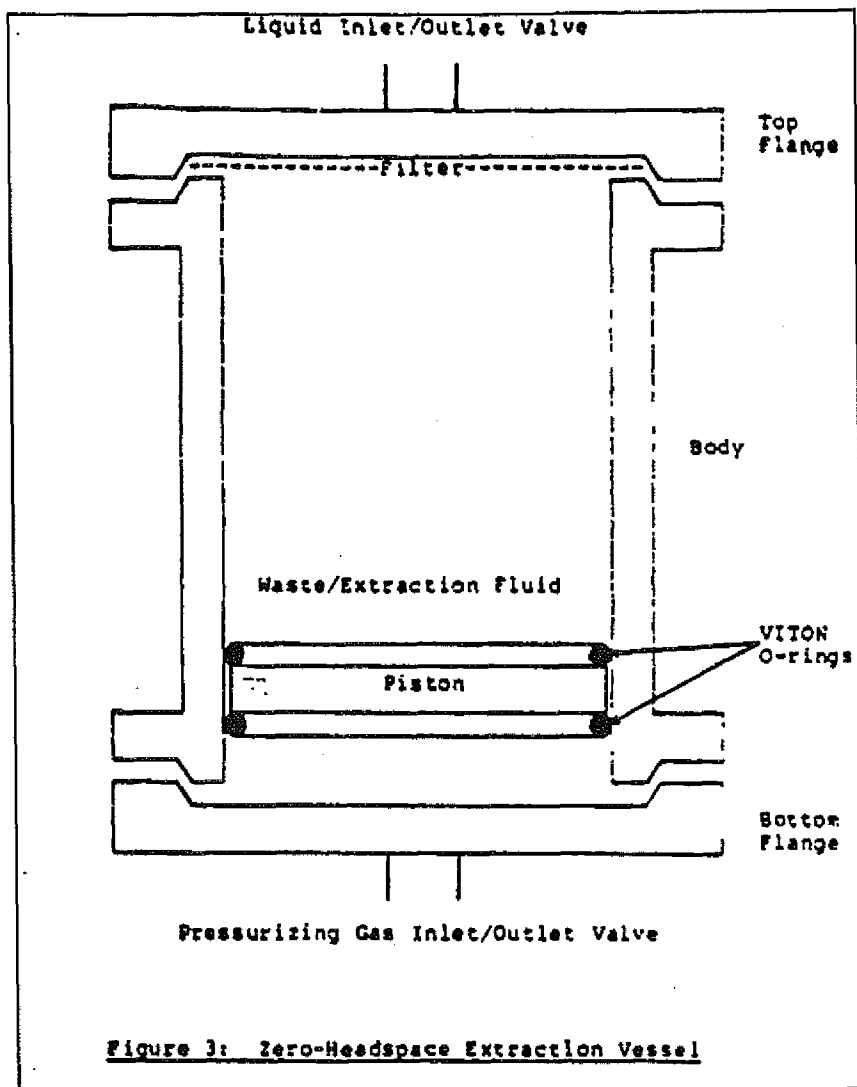


Figure 2: Rotary Agitation

FIGURE 3 - ZERO HEADSPACE EXTRACTION VESSEL



Appendix II - Treatment Standards (as concentration in the treatment residual extract)

[Note: The technologies shown are the basis of the treatment standards. They are not required to be used in meeting the treatment standards.]

Waste Treatability Groups for F001-F005 Spent Solvent Wastes (mg/l)

Constituents of F001-F005 Spent Solvent Wastes	Wastewater	TechnologyBase ¹	Wastewater Generated by Pharmaceutical Plant ²	All Other ³
Acetone	0.05	SS	0.59	
n-Butyl Alcohol	5.00	SS	5.00	
Carbon disulfide	1.05	SS	4.81	
Carbon tetrachloride	0.05	B	0.96	
Chlorobenzene	0.15	B&AC	0.05	
Cresols (cresylic acid)	2.82	AC	0.75	
Cyclohexanone	0.125	SS	0.75	
1, 2-Dichlorobenzene	0.65	B&AC	0.125	
Ethyl acetate	0.05	SS	0.75	
Ethylbenzene	0.05	B	0.053	
Ethyl ether	0.05	SS	0.75	
Isobutanol	5.00	SS	5.00	
Methanol	0.25	SS	0.75	
Methylene chloride	0.20	B	12.7	0.96
Methyl ethyl ketone	0.05	SS	0.75	
Methyl isobutyl ketone	0.05	SS	0.33	
Nitrobenzene	0.66	SS&AC	0.125	
Pyridine	1.12	B&AC	0.33	
Tetrachloroethylene	0.079	B	0.05	
Toluene	1.12	B&AC	0.33	
1,1,1-Trichloroethane	1.05	SS	0.41	
1,1,2-Trichloro-1,2,2-trifluoroethane	1.05	SS	0.96	
Trichloroethylene	0.062	B&AC	0.091	
Trichloro-fluoromethane	0.05	B	0.96	
Xylene	0.05	AC	0.15	

¹ In some instances other technologies achieved somewhat lower treatment values but waste characterization data were insufficient to identify separate treatability groups. Refer to the BDAT background document for a detailed explanation of the determination of the treatment standards.

SS = steam stripping
B = biological treatment
AC = activated carbon

² Wastewaters generated by pharmaceutical plants shall be treated to the standards given for all other wastewaters except in the case of methylene chloride.

³ The treatment standards in this treatability group are based on incineration.

Appendix

Appendix III - List of Halogenated Organic Compounds

In determining the concentration of HOCs in a hazardous waste for purposes of the California List land disposal prohibition, the following HOCs shall be included in the calculation as any compounds having a carbon-halogen bond which are listed in this Appendix. Appendix III consists of the following compounds:

Volatiles

Bromodichloromethane
Bromomethane
Carbon Tetrachloride
Chlorobenzene
2-Chloro-1,3-butadiene
Chlorodibromomethane
Chloroethane
2-Chloroethyl vinyl ether
Chloroform
Chloromethane
3-Chloropropene
1,2-Dibromo-3-chloropropane
1,2-Dibromomethane
Dibromomethane
Trans-1,4-Dichloro-2-butene
Dichlorodifluoromethane
1,1-Dichloroethane
1,2-Dichloroethane
1,1-Dichloroethylene
Trans-1,2-Dichloroethene
1,2-Dichloropropane
Trans-1,3-Dichloropropene
cis-1,3-Dichloropropene
Iodomethane
Methylene chloride
1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane
Tetrachloroethene
Tribromomethane
1,1,1-Trichloroethane
1,1,2-Trichloroethane
Trichloroethene
Trichloromonofluoromethane
1,2,3-Trichloropropane
Vinyl chloride

Semivolatiles

Bis(2-chloroethoxy)ethane
Bis(2-chloroethyl)ether
Bis(2-chloroisopropyl) ether
p-Chloroaniline
Chlorobenzilate
p-Chloro-m-cresol
2-Chloronaphthalene
2-Chlorophenol
3-Chloropropionitrile
m-Dichlorobenzene
o-Dichlorobenzene
p-Dichlorobenzene
3,3'-Dichlorobenzidine
2,4-Dichlorophenol
2,6-Dichlorophenol
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclopentadiene
Hexachloroethane
Hexachloropropene

Hexachloropropene
4,4'-Methylenebis(2-chloroaniline)
Pentachlorobenzene
Pentachloroethane
Pentachloronitrobenzene
Pentachlorophenol
Pronamide
1,2,4,5-Tetrachlorobenzene
2,3,4,6-Tetrachlorophenol
1,2,4-Trichlorobenzene
2,4,6-Trichlorophenol
2,4,6-Trichlorophenol
Tris(2,3-dibromopropyl)phosphate

Organochlorine Pesticides

Aldrin
alpha-BHC
beta-BHC
delta-BHC
gamma-BHC
Chlordane
DDD
DDE
DDT
Dieldrin
Endosulfan I
Endosulfan II
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
Isodrin
Kepone
Methoxychlor
Toxaphene

Phenoxyacetic Acid Herbicides

2,4-Dichlorophenoxyacetic acid
Silvex 2,4,5-T

PCBs

Aroclor 1016
Aroclor 1221
Aroclor 1232
Aroclor 1242
Aroclor 1248
Aroclor 1254
Aroclor 1260
PCBs not otherwise specified

Dioxins and Furans

Hexachlorodibenzo-p-dioxin
Hexachlorodibenzofuran
Pentachlorodibenzo-p-dioxin
Pentachlorodibenzofuran
Tetrachlorodibenzo-p-dioxin
Tetrachlorodibenzofuran
2,3,7,8-Tetrachlorodibenzo-p-dioxin