

DRAFT

METHOD 1312

SYNTHETIC PRECIPITATION LEACH TEST FOR SOILS

1.0 SCOPE AND APPLICATION

1.1 Method 1312 is designed to determine the mobility of both organic and inorganic contaminants present in soils.

1.2 If a total analysis of the soil demonstrates that individual contaminants are not present in the soil, or that they are present but at such low concentrations that the appropriate regulatory thresholds could not possibly be exceeded, Method 1312 need not be run.

2.0 SUMMARY OF METHOD

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2.1 The particle size of the soil is reduced (if necessary) and is extracted with an amount of extraction fluid equal to 20 times the weight of the soil. The extraction fluid employed is a function of the region of the country where the soil site is located. A special extractor vessel is used when testing for volatiles. Following extraction, the liquid extract is separated from the soil by 0.6-0.8 μ m glass fiber filter.

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 at 30 ± 2 rpm (see Figure 1). Suitable devices known to EPA are identified in Table 2.

4.2 Extraction vessel - acceptable extraction vessels are those that are listed below:

4.2.1 Zero Headspace Extraction Vessel (ZHE) - This device is for use only when the soil 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 1312 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 soil 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 Step 7.4) 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 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 soil is being evaluated for other than volatile compounds, a filter holder capable of

supporting a glass fiber filter and able to withstand 50 psi or more of pressure shall be used. 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 liters or greater are recommended).

4.3.3 Materials of construction - filtration devices shall be made of inert materials which will not leach or absorb soil 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 have an effective pore size of 0.6 - 0.8 μ m and shall contain no binder materials. Filters known to EPA to meet these requirements are identified in Table 5. When evaluating the mobility of metals, filters should be acid-washed prior to use by rinsing with 1.0N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1-liter 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, glass, stainless steel or PTFE gas tight syringes are used to collect the volatile extract.

4.7 Laboratory balance - any laboratory balance accurate to within ± 0.01 g may be used (all weight measurements are to be within ± 0.1 g).

4.8 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 recommended.

5.0 REAGENTS

5.1 Reagent water - reagent water is defined as water in which an interferent 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 g 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 screw-cap bottle under zero headspace and seal with a Teflon lined septum and cap.

5.2 Sulfuric acid/nitric acid (60/40 weight percent mixture) $\text{H}_2\text{SO}_4/\text{HNO}_3$. Cautiously mix 60 g of concentrated sulfuric acid with 40 g of concentrated nitric acid.

5.3 Extraction fluids:

5.3.1 Extraction fluid #1 - this fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 4.20 ± 0.05 .

5.3.2 Extraction fluid #2 - this fluid is made by adding the 60/40 weight percent mixture of sulfuric and nitric acids to reagent water until the pH is 5.00 ± 0.05 .

5.3.3 Extraction fluid #3 - this fluid is reagent water (ASTM Type II water, or equivalent) used to determine cyanide leachability.

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.

5.4 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 two separate representative samples of a soil should be collected. The first sample is used to determine if the soil requires particle-size reduction and, if desired, the percent solids of the soil. The second sample is used for extraction of volatiles and non-volatiles.

6.3 Preservatives shall not be added to samples.

6.4 Samples shall be refrigerated to minimize loss of volatile organics and to retard biological activity.

6.5 When the soil 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 be conducted as the sample is being taken.

6.6. 1312 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 at 4°C, and samples for volatiles analysis shall not be allowed to come into contact with the atmosphere (i.e. no headspace). See Section 8.0 (Quality Control) for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 The preliminary 1312 evaluations are performed on a minimum 100 g representative sample of soil that will not actually undergo 1312 extraction (designated as the first sample in Step 6.2).

7.1.1 Determine whether the soil requires particle-size reduction. If the soil passes through a 9.5 mm (0.375-inch) standard sieve, particle-size reduction is not required (proceed to Step 7.2). If portions of the sample do not pass through the sieve, then the oversize portion of the soil will have to be prepared for extraction by crushing the soil to pass the 9.5 mm sieve.

7.1.2 Determine the percent solids if desired.

7.2 Procedure when volatiles are not involved - Enough solids should be generated for extraction such that the volume of 1312 extract will be sufficient to support all of the analyses required. However, a minimum sample size of 100 grams shall be used. If the amount of extract generated by a single 1312 extract will not be sufficient to perform all of the analyses, it is recommended that more than one extraction be performed and the extracts be combined and then aliquoted for analysis.

7.2.1 Weigh out a representative subsample of the soil and transfer to the filter holder extractor vessel.

7.2.2 Determine the appropriate extraction fluid to use. If the soil is from a site that is east of the Mississippi River, extraction fluid #1 should be used. If the soil is from a site that is west of the Mississippi River, extraction fluid #2 should be used. If the soil is to be tested for cyanide leachability, extraction fluid #3 should be used.

Note: Extraction fluid #3 (reagent water) must be used when evaluating cyanide-containing soils because leaching of cyanide-containing soils under acidic conditions may result in the formation of hydrogen cyanide gas.

7.2.3 Determine the amount of extraction fluid to add based on the following formula:

$$\text{amount of extraction fluid (mL)} = 20 \times \text{weight of soil (g)}$$

Slowly add the amount of appropriate extraction fluid 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 $22 \pm 3^\circ\text{C}$ during the extraction period.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of soil (e.g. limed or calcium carbonate containing soil 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.

7.2.4 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 glass fiber filter.

7.2.5 Following collection of the 1312 extract it is recommended that the pH of the extract be recorded. The extract should be immediately aliquoted for analysis and properly preserved (metals aliquots must be acidified with nitric acid to $\text{pH} < 2$; all other aliquots must be stored under refrigeration (4°C) until analyzed). The 1312 extract shall be prepared and analyzed according to appropriate analytical methods. 1312 extracts to be analyzed for metals, other than mercury, shall be acid digested.

7.2.6 The contaminant concentrations in the 1312 extract are compared to thresholds in the clean closure guidance manual. Refer to Section 8.0 for Quality Control requirements.

7.3 Procedure when volatiles are involved:

7.3.1 The ZHE device is used to obtain 1312 extracts for volatile analysis only. Extract resulting from the use of the ZHE shall 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 g was required in the Step 7.2 procedure, the ZHE can only accommodate a maximum of 25 g of solid, due to the need to add an amount of extraction fluid equal to 20 times the

weight of the soil. The ZHE is charged with sample only once and the device is not opened until the final extract has been collected. 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 particle-size reduction (see Step 7.1.1) should be conducted on the sample as it is being taken (e.g., particle-size may be reduced by crumbling). If necessary particle-size reduction may be conducted during the procedure. In carrying out the following steps, do not allow the soil 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 the loss of volatiles. Pre-weigh the evacuated container which will receive the filtrate (see Step 4.6), and set aside. If using a TEDLAR® bag, all air must be expressed from the device.

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 it is charged with sample. 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 Quantitatively transfer 25 g of soil 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. 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 to a maximum of 50 psi to force most of the headspace out of the device.

7.3.4 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.

7.3.5 After the extraction fluid has been added, immediately close the 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.

7.3.6 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 the room in which extraction is to occur) shall be maintained at $22 \pm 3^{\circ}\text{C}$ during agitation.

7.3.7 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 and redo the extraction with a new sample of soil. If the pressure within the device has been maintained, the material in the extractor vessel is separated into its component liquid and solid phases.

7.3.8 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 inlet/outlet valve, discontinue pressure to the piston, and disconnect the filtration collection container.

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

7.3.9 Following collection of the 1312 extract, the extract should be immediately aliquoted for analysis and stored with minimal headspace at 4°C until analyzed. The 1312 extract will be prepared and analyzed according to the appropriate analytical methods.

8.0 QUALITY CONTROL

8.1 All data, including quality assurance data, should be

maintained and available for reference or inspection.

8.2 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.

8.3 For each analytical batch (up to twenty samples), it is recommended that a matrix spike be performed. Addition of matrix spikes should occur once the 1312 extract has been generated (i.e. should not occur prior to performance of the 1312 procedure). The purpose of the matrix spike is to monitor the adequacy of the analytical methods used on the 1312 extract and for determining if matrix interferences exist in analyte detection.

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

8.5 The method of standard addition shall be employed for each analyte if: 1) recovery of the compound from the 1312 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 waste (up to twenty samples), the method of standard addition need be applied only once and the percent recoveries applied on the remainder of the extractions.

8.6 Samples must undergo 1312 extraction within the following time period after sample receipt: Volatiles, 14 days; Semi-Volatiles, 40 days; Mercury, 28 days; and other Metals, 180 days. 1312 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.

9.0 METHOD PERFORMANCE

9.1 None available.

10.0 REFERENCES

10.1 None available.

TABLE 1. -- VOLATILE CONTAMINANTS

Compounds	CAS No.
Acetone.....	67-64-1
Acrylonitrile.....	107-13-1
Benzene.....	71-43-2
n-Butyl alcohol.....	71-36-6
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
1,1,1,2-Tetrachloroethane.....	630-20-6
1,1,2,2-Tetrachloroethane.....	79-34-5
Tetrachloroethylene	127-18-4
Tolulene	108-88-3
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane.....	79-00-5
Trichloroethylene	79-01-6
Trichlorofluoromethane.....	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane.....	76-13-1
Vinyl chloride.....	75-01-7
Xylene	1330-20-7

TABLE 2. -- SUITABLE ROTARY AGITATION APPARATUS¹

Company	Location	Model
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	4-vessel device, 6-vessel device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	4-vessel device, 6-vessel device
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	16-vessel device
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel device 5-vessel device
Millipore Corp.	Bedford, MA (800) 225-3384	4-vessel ZHE device or 4-one litter bottle extractor device
REXNORD	Milwaukee, WI (414) 643-2850	6-vessel device

¹Any 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

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA, (215) 343-4490	C102, Mechanical Pressure Device
Associated Design & Manufacturing Co.	Alexandria, VA (703) 549-5999	3740-ZHB, Gas Pressure Device
Lars Lande Mfg.	Whitmore Lake, MI (313) 449-4116	Gas Pressure Device
Millipore Corp.	Bedford, MA, (800) 225-3384	SD1 P581 C5, Gas Pressure Device

TABLE 4. -- SUITABLE ZHE FILTER HOLDERS¹

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

¹Any device capable of separating the liquid from the solid phase of the soil is suitable, providing that it is chemically compatible with the soil 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

Company	Location	Model	Size ¹
Millipore Corp.	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corp.	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7

¹Nominal pore size

Figure 1. Rotary Agitation

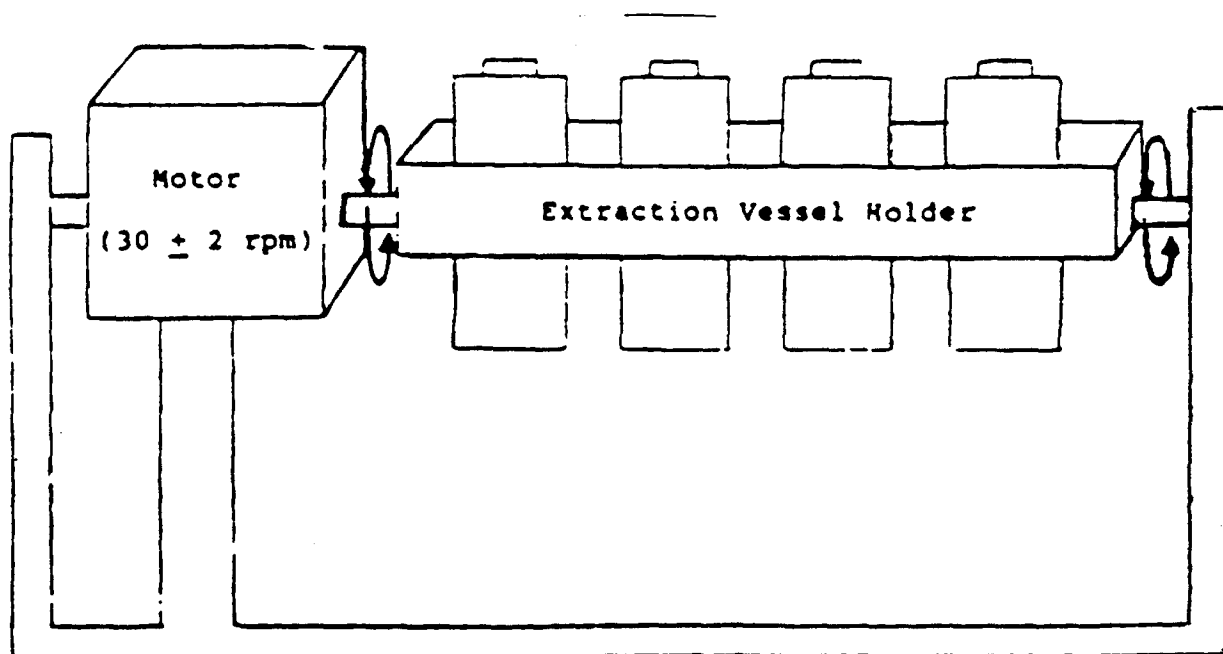
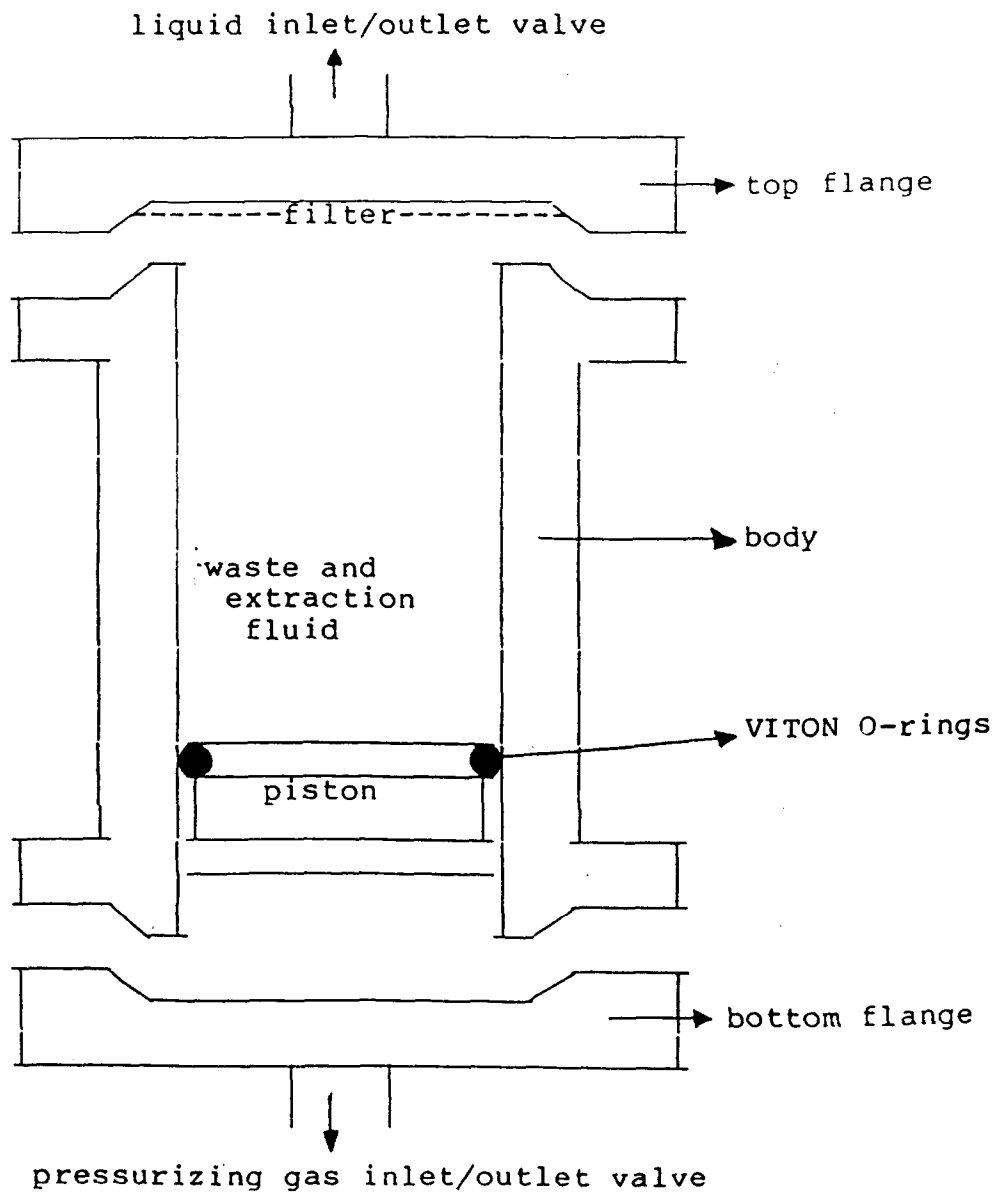


Figure 2. Zero-Headspace Extraction Vessel



METHOD 1312
SYNTHETIC ACID PRECIPITATION LEACH TEST FOR SOILS

