User Guide

Zero Head Space Extractor

YT30090HW

About the Zero Head Space Extractor

The Zero Head Space Extractor (ZHE) meets the U.S. EPA'S Toxicity Characteristics Leaching Procedure (TCLP) requirements for volatiles. The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid and multiphasic wastes.

- The design eliminates the need to introduce air into the sample, thus eliminating the loss of volatiles.
- The vent-relief valve on the base plate automatically opens at 125 psi to prevent excessive pressure build-up. The valve also permits manual venting of the system.
- As a safety precaution, the ZHE cannot be pressurized unless completely assembled, eliminating the possibility of propelling the piston out of the unit.

The ZHE barrel is sealed to the bottom and top plates with Viton®-A O-rings. The top plate is fitted with an inlet/outlet sample valve to which is connected a female Luer port. The plate is secured to the barrel with three hand wheels. Internally, two stainless steel screens support the filter.

The stainless steel piston is fitted with two Viton®-A sealing O-rings and a Viton®-A wiper ring (on the top edge of the piston). The 500 mL ZHE barrel is made of Type 316 stainless steel, mounted on a rigid base supported by three legs. The ZHE unit may be used in a standard rotary agitator (legs intact), or in a box type agitator (legs removed).

Caution: All the O-rings in this unit are Viton® material and will stand up to most chemicals under normal operating conditions. However, to be certain of the chemical compatibility for your particular application, check the materials compatibility guide for O-rings at SigmaAldrich.com.

Preparing for Use

Inspecting the Components

Prior to shipment, the Zero Head Space Extractor is leak tested. However, we recommend that you inspect the ZHE components before each use. Damage to any of the components or extraneous debris in the O-ring grooves may cause the system to leak.

1. Remove the ZHE housing barrel.
2. Remove the O-rings from the top and bottom of the barrel. Inspect the O-ring grooves for damage and extraneous debris.
3. Inspect each O-ring for nicks or damage. If the O-rings are damaged, replace them with new ones.
4. Inspect the O-rings and grooves of the top plate.
5. Remove the piston from the package and inspect the wiper O-rings and sealing O-rings for damage and extraneous debris.
6. Wrap all threaded connections with Teflon® tape. Remove and discard any residual tape on fittings and re-wrap the components with new tape (1 or 2 turns only).

Assembling the System

1. Place the O-rings in the top plate groove and the bottom barrel groove. Sect the O-rings firmly to prevent a loss of seal integrity.

   Note: You might need to wet the O-rings and groove slightly with Milli-Q® or laboratory-grade water. If the O-rings are new, it may be necessary to stretch them slightly before installing.

2. Place the ZHE barrel, top end up, onto the edge of a flat laboratory bench, with approximately one half inch of the barrel extending over the edge of the surface of the bench. This will allow air to escape from the barrel when inserting the piston.
3. Wet the wiper ring and piston O-rings slightly with Milli-Q® or laboratory-grade water. With the wiper ring facing up, place the piston into the barrel.
4. Using the piston pusher, move the piston down to the bottom of the barrel, then remove the piston pusher.
5. Center the barrel, bottom side down, onto the bottom plate, making sure the O-ring groove is firmly seated on the O-ring in the bottom plate.

Preparing Extraction Fluid

Extract all waste to be evaluated for volatile organics with an amount of extraction fluid #1 (equal to 20 times [20X] the weight of the dry solid phase of the waste being tested). Prepare extraction fluid #1 using the following steps. Use only reagent-grade water generated by Milli-Q® or Milli-Q® Plus water purification system.

1. Add 5.7 mL of glacial HOAc to 500 mL of reagent-grade water.
2. Add 64.3 mL of 1.0 N NaOH to the HOAc/water solution and dilute to a volume of 1 L with reagent-grade water.
3. Check the pH of the extraction fluid before using. When prepared correctly, the pH of this fluid will be 4.93 ± 0.05.

   Note: Monitor the extraction fluid frequently for impurities.

Basic TCLP Guidelines

The following steps are intended as basic guidelines on how to use the ZHE for TCLP evaluations of waste samples. Please refer to U.S. EPA method 1311 for more detailed specifications.

Preliminary Evaluations

1. Determine the particle size of the sample.
2. Determine the pH of the sample.
3. Determine the percent dry solids of the sample. Percent dry solids is defined as the percentage of a waste sample from which no liquid may be forced out by an applied pressure.

$$\text{Percent dry solids} = \frac{\text{weight of solids}}{\text{total weight of waste}} \times 100$$

   Note: Before measuring the percent dry solids, dry the sample in a drying oven until two consecutive weights are within ± 1%.

Loading the Sample

1. Add the pre-evaluated sample (minimum of 25 grams of dry solid) into the ZHE barrel.
2. Remove the 2 support screens from their package. Place a 90 mm type-AP40, binderless, glass fiber filter between them. Center this assembly inside the top plate.
3. Place the heavy-top O-ring into the top plate to hold the filter and support screen in place.
4. Center the top plate securely on top of the barrel.
5. Tighten the hand wheels by turning them to the right as you face the front of the unit. Use the enclosed hand-wheel wrench to tighten the hand wheels as securely as possible.

Pressurizing the Unit

See EPA method 1311 for more detailed TCLP specifications before proceeding with the following general instructions for sample collection and analysis.

Caution: Do not exceed a maximum pressure of 50 psi (3.5 kg/cm^2). Never pressurize the unit unless it is fully assembled and secured; otherwise, the piston could be damaged.

1. Attach a suitable 1/4 in. hose to an appropriate source of compressed air or nitrogen (a pressurized gas cylinder with a regulator or house-compressed air are suitable sources.)
2. Use the 1/4 in. male quick-disconnect fitting (provided) to attach the tubing from your gas source to the ZHE.
3. Fit the male quick-disconnect fitting into the female quick-disconnect fitting on the bottom plate of the ZHE. When the quick-disconnect fittings are properly connected, you will hear a click.
4. To expel any air trapped in the cylinder, open the inlet/outlet sample valve at the top of the unit.
5. Slowly pressurize the ZHE unit. Beginning at a pressure of 0 psi, gradually increase the pressure until the piston break-force is reached and the piston begins to move. This break force should be less than 15 psi.
6. As soon as you see liquid exiting from tine inlet/outlet sample valve, close the valve.

Collecting the Primary Leachate

1. Attach a Tedlar® bag (using the ZHE Tedlar bag fitting) or a gas-tight syringe to the Luer fitting on the sample valve at the top of the ZHE. Do not leave any headspace in the Tedlar® bag or syringe.
2. Reopen the inlet/outlet sample valve.
3. Starting at the pressure used to eliminate the head space, gradually increase the pressure by 10 psi and begin collecting fluid. Hold at that pressure for 2 minutes, collecting any fluid expelled out of the ZHE.
4. At the end of 2 minutes, increase the pressure an additional 10 psi and continue to collect fluid for another 2 minutes.
5. Collect fluid at each pressure increment for 2 minutes until a maximum pressure of 50 psi is reached. At 50 psi, close the inlet/outlet valve and turn off the pressure source.
6. Release the air pressure behind the piston by opening the vent relief valve on the base plate.
7. Disconnect the tubing from the quick-disconnect valve at the base of the unit.
8. Detach the Tedlar® bag or syringe which contains the primary leachate. Remove the fitting and cap the bag with the cap provided. Evaluate this primary leachate immediately or store at 4 °C (39.2 °F) for future analysis.

Repressurizing the Unit

The following instructions assume the use of our Dispensing Pressure Vessel and ZHE accessory kit.

1. Preassemble the pressure vessel, with the accessory kit, according to the instructions included with the pressure vessel.
2. Pour the 20X volume of extraction fluid into the vessel. Add an additional 40-60 mL to prime the lines and fill the void volume in the bottom of the 5-liter pressure vessel.
3. Connect the male quick-disconnect fitting on the pressure source tubing to the female quick disconnect fitting at the inlet fitting on the pressure vessel.

   Note: The same male quick-disconnect fitting can be attached to both the dispensing pressure vessel during extraction and to the bottom of the ZHE during filtration.
4. Gently pressurize the vessel and open the ball valve to purge any air trapped in the tubing between the vessel and the ZHE.
5. Close the ball valve of the pressure vessel and pressurize the vessel to 15 psi.
6. Attach the outlet tubing from the pressure vessel to the female Luer fitting at the inlet/outlet valve on the top of the ZHE. Open the inlet/outlet valve.

Extracting and Agitating the Sample

1. Open the ball valve of the pressure vessel and dispense the extraction fluid into the ZHE.
2. After the ZHE is filled with fluid, the pressure gauge on the pressure vessel will rise rapidly. Turn off the pressure at the source, close the inlet/outlet valve, and close the ball valve on the pressure vessel.
3. Release the pressure in the vessel and the tubing by opening the vent-relief valve on the pressure vessel. Disconnect the tubing from the top of the ZHE.
4. Agitate the ZHE cylinder by hand, end-over-end, for 3-4 seconds, then re-open the valve on the top plate to allow any trapped air to escape. Close the valve.
5. Reattach the pressure source to the female, quick-disconnect fitting at the bottom of the ZHE. Slowly pressurize the ZHE unit, with 5-10 psi of pressure, making sure all valves are closed.

6. Clamp the ZHE into a rotor agitator, making sure it is properly balanced within the agitator.

   Note: Consult the instructions included with the rotary agitator for proper operating procedures.

7. Close the cover of the rotary agitator and agitate the ZHE end-over-end at 30 rpm ± 2 rpm for 18 hours.
8. Remove the ZHE from the rotary agitator.

Collecting the Secondary Leachate

1. Reattach the pressure source to the ZHE, using the 1/4 in. male, quick-disconnect fitting. Fit the male, quick-disconnect fitting into the female, quick-disconnect fitting on the bottom plate of the ZHE.
2. Reattach a Tedlar® bag (using the ZHE Tedlar® bag fitting) or a gas-tight syringe to the Luer fitting on the sample valve at the top of the ZHE. Do not leave any headspace in the Tedlar® bag or syringe.
3. Reopen the inlet/outlet sample valve.
4. Gradually add 10 psi of pressure and hold for 2 minutes. Collect any fluid expelled out of the ZHE.
5. At the end of 2 minutes, increase the pressure an additional 10 psi and continue to collect the fluid for another 2 minutes.
6. Collect the fluid at each pressure increment for 2 minutes until a maximum pressure of 50 psi is reached. At 50 psi, close the inlet/outlet valve and turn off the pressure source.
7. Release the air pressure behind the piston by opening the vent relief valve on the base plate.
8. Disconnect the tubing from the quick-disconnect valve at the base of the unit.
9. Detach the Tedlar® bag or syringe which contains the secondary leachate. Remove the fitting and cap the bag with the cap provided. Evaluate immediately or store at 4 °C (39.2 °F) for future analysis.

If the secondary leachate and the primary leachate are compatible, they can be physically combined and analyzed. If they are not miscible, they should be analyzed separately and the results mathematically combined.

Maintainence

Cleaning the ZHE

Wash the ZHE unit with mild detergents and soft brushes. To remove stubborn residues from any stainless steel surfaces, use a non-chlorinated, non-scratching cleanser. If residues persist, soak in Everite solution for 5-10 minutes. Rinse well with Milli-Q® or laboratory-grade water and allow to air dry.

Use a stiff, short-bristle brush to clean debris from the threads. To clean valves, disassemble before cleaning. After cleaning, rinse with Milli-Q® or laboratory-grade water and re-assemble.

Testing for Leaks

Periodic leak tests should be performed on the ZHE unit to ensure optimum performance and results. To perform a leak test, use the following steps.

1. Disassemble the ZHE unit and thoroughly clean all parts.
2. Remove the O-rings and clean the O-ring grooves. Reseat the O-rings into the correct O-ring grooves.
3. Reassemble and load the ZHE unit, substituting water for sample. Make sure that the O-rings are properly seated and that the top plate is secured firmly by the hand wheels.
4. Connect the gas source to the quick-disconnect gas valve at the bottom of the ZHE. When the quick disconnect nipple is fitted into the valve, you should hear a click. The valve is now open.
5. Using a parallel connection, connect the inlet/outlet sample valve to the gas source. Open the valve.
6. Pressurize both sides of the ZHE piston to 20-50 psi.
7. Close the sample valve and disconnect the pressure source tubing from the ZHE unit.
8. Allow the ZHE to stand for 5 minutes, then check the pressure gauge. If the pressure in the ZHE remains constant, there are no leaks in the system.
9. If the pressure in the ZHE drops, indicating a leak in the system, continue with the following section, Locating a Leak, for leak-testing at threaded connections, valves and sealing O-rings.

Locating a Leak

1. Dispense a small amount of water mixed with a mild detergent on all threaded connections, valves and seals. (Do not submerge the ZHE.) Small, continuously-forming bubbles indicate a leak at that fitting.

   Note: Small bubbles trapped on the surfaces of the Teflon® tape covering the threads are natural. Only continuously forming bubbles indicate a leak.

2. If none of the connections, valves or seals indicate a leak, continue with the instructions for leak testing the piston O-rings.
3. If small, continuous bubbles form at any fitting, continue with the instructions for leak testing at that particular type of fitting.

Threaded Connections

If you find leaks at any of the threaded connections:

1. Depressurize the ZHE and re-tighten the fittings firmly with an open-end or adjustable wrench.
2. Pressurize the ZHE and retest the fitting with a small amount of water.
3. If the unit leaks again, unscrew the fittings, remove all Teflon® tape wrapped on the threads and clean the fittings thoroughly.
4. Retape using one or two turns of tape only and reassemble the fitting.
5. Insert the fittings tightly with a wrench, pressurize the unit, and retest as above. If leaks persist, the threads might be damaged and the fittings should be repaired or replaced.

Valves

If you find leaks in the body of any of the valves:

1. Depressurize the ZHE and unscrew the valve.
2. Inspect and clean the valve gaskets and seat; rinse well with Milli-Q® or laboratory-grade water and reinstall.
3. Pressurize the unit and test for leaks as described above.

Sealing O-Rings

If you find leaks at any of the O-ring sealing points:

1. Depressurize the ZHE unit, disassemble, and remove the O-ring.
2. Clean out the O-ring groove and examine the O-ring. If undamaged (no nicks or cuts), clean the O-ring and reseat it firmly within the groove.
3. Reassemble the system tightly, pressurize the unit to 20-50 psi and retest. If leakage persists, replace the O-rings.

Piston O-Rings

If the pressure in the ZHE drops during initial leak testing, but no leaks are apparent at any threaded connections, valves or sealing O-rings, use the following instructions to test for leaks at the piston O-rings.

1. Connect a piece of small O.D. tubing to the sample valve and immerse the tubing into a beaker of water.
2. Pressurize the ZHE unit gradually up to 50 psi and open the sample valve. Any residual air will escape. If the seals leak, you will see a continuous flow of bubbles.
3. If you see a leak, depressurize the ZHE unit and disassemble.
4. Remove the O-rings from the piston, leave the wiper in place, and examine the grooves and O-rings for damage or debris. Clean the O-ring grooves and reseat the O-rings tightly.
5. Reassemble the unit, adding water in place of sample, and retest for leaks.
6. If leaks persist, replace the O-rings with new piston O-rings.

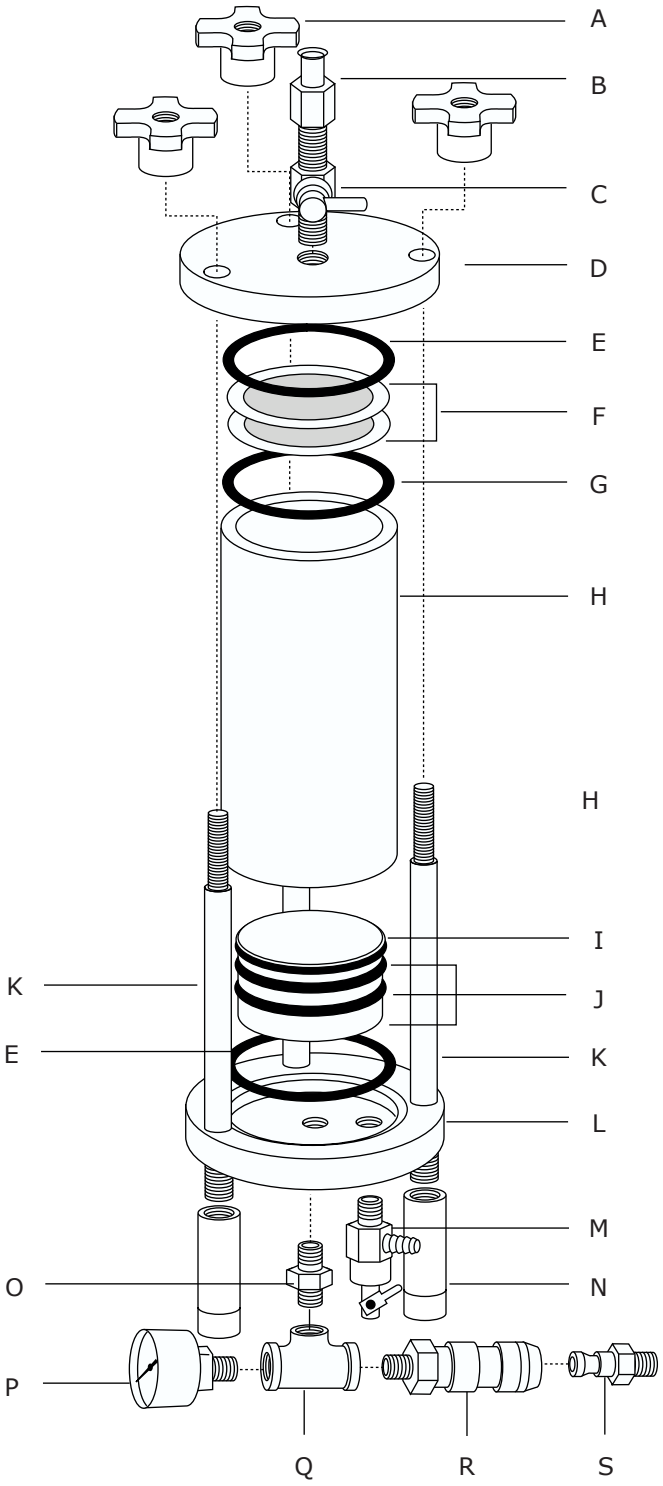
Technical Information

Specifications

Materials	
Body	316 Stainless steel
Hand wheel insert	Brass
Hand wheels	Molded polypropylene
O-rings	Viton™-A
Connections	1/8 in. NPTM inlet/outlet sample valve 1/8 in. NPTF female Luer 1/4 in. NPTM to 1/8 in. NPTF reducer 1/8 in. NPTF T-fitting 1/4 in. NPT quick-disconnect male 1/8 in. NPT quick-disconnect female 1/4 in. NPTM relief valve
Working Pressure	Up to 50 psi (3.5 kg/cm^2) maximum
Filter/prefilter Size	90 mm
Effective Filtration Area	63.6 cm^2
Piston Brake Force	5-10 psi (0.35-0.7 kg/cm^2)
Dimensions	6-1/4 in. diameter (15.9 cm) 11-1/2 in. tall (28.58 cm)

Ordering Information

Description	Key	Catalogue/ Part No.
ZHE Hazardous Waste Filtration System		YY30090HW
Replacement parts		
Hand wheel knob, 1 pk	A	YY2214257
Luer fitting (1/8 in. NPTF)	B	YT3009002
Two-way stainless steel valve (1/8 in. NPTM)	C	XX2702512
Top plate	D	
Top and bottom plate O-rings, 2, 6 pk	E	YY2209068
90 mm stainless steel screens, 2	F	YY2209064
Cylinder O-ring, 3 pk	G	XX6700010
Cylinder	H	YT3009010
Piston	I	YT3009001
Piston wiper and O-ring set	J	YT3009003
Cylinder support posts, 3 pk	K	XX4209002
Bottom plate	L	
Vent relief valve, 1/4”, NPTM	M	XX6700024
Support legs, 316 stainless steel, 1 pk	N	15017
Reducer, 1/4 in. NPTM to 1/8 in. NPTF with 1/8 in., NPT nipple	O	XX6700105
Pressure gauge	P	P16938
Bottom plate T-fitting, 1/4 in. NPTF to 1/8 in. NPTF	Q	P16939
Quick-Disconnect, female Swagelok, 1/8 in. NPT	R	P16940
Quick-Disconnect, male Swagelok, 1/4 in., NPT	S	YT3009014
Piston pusher (not pictured)		13899
Adapter, 1/8 in. NPTM to 1/4 in. tube (not pictured)		YT3009018
O-ring kit for two-way valve (XX2702512)		P30906



Accessories	Catalogue/ Part No.
Forceps, stainless steel	XX6200006
AP40, 0.7 µm glass filter, 100 pk	AP4009000
ZHE Tedlar® bags, 10 pk	YT3009016
Tedlar® bag connector	YT3009017
ZHE glass-tight syringe, 50 mL	YT3009015
Street elbow, 1/4 in. NPTM to 1/4 in. NPTF	XX6700104
Quick-disconnect, female Swagelok, 1/4 in. NPT	YT3009013
Vacuum pressure oump, 115V, 60 Hz	XX5500000
Pressure vessel, ASME, 5L	XX6700P05
Pressure vessel accessories kit (recommended for use with the Millipore ZHE; contains 1 of each of the following):	XX6700P 00
Adapter, 1/8 in. NPTF to male Luer	XX3002567
Adapter, 1/2 in. NPTM to 1/4 in. tube	XX6702507
Ball valve, 1/4 in. NPTF	YY2029348
Hex nipple, 1/4 in. NPTM, 51 mm (2 ft length)	XX6700125
Pressure gauge, 0-7 bar (0-100 psig)	YY1301015
Quick-disconnect, female Swagelok, 1/4 in. NPT	YT3009013
Quick-disconnect nipple and coupling, 1/4 in. NPT	XX6700030
Tubing, polypropylene, 6 mm (1/4 in.) O.D.,3 m (10 feet)	XX6702508
Vent relief valve, 1/4 in. NPT	XX6700024

Notice

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