

# HotBlock® 150 Digestion System

## Operation & Instruction Manual

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+1.800.343.5319 or +1.843.881.6560 [envexp.com](http://envexp.com)

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## Limited Warranty

The Environmental Express HotBlock® is warranted against defects in materials and workmanship when used in accordance with applicable instructions, for a period of one year from the date of shipment. This warranty extends to parts, labor, and any approved transportation charges. This warranty applies only to damage or failure caused by normal laboratory use. The warranty is limited to product repair. If Environmental Express is unable to repair the HotBlock®, the customer may, at his or her option, receive a replacement unit or a full refund. Operating the HotBlock® at temperatures higher than 450°C will void the warranty.

In no event shall Environmental Express have any obligation to make repairs, replacements or corrections required, in whole or in part, as the result of (i) normal wear and tear, (ii) accident, disaster or event of force majeure, (iii) abuse, neglect, misuse, fault or negligence of or by customer, (iv) use of the product in a manner for which it was not designed, (v) causes external to the product such as, but not limited to, power failure or electrical power surges, (vi) improper storage and handling of the product, (vii) use of the product in combination with equipment or software not supplied by Environmental Express, (viii) ordinary maintenance, (ix) alterations, repairs or installations that have not been performed by Environmental Express or its authorized representative or (x) failure to maintain product in accordance with Environmental Express' written instructions.

Environmental Express makes no other warranty, expressed or implied for this product with respect to merchantability, fitness for a particular use or any other matter and expressly disclaims all other warranties. Environmental Express is not liable for any consequential, special, indirect or compensatory damages arising from use of, or in conjunction with this product. The maximum liability of Environmental Express (whether by reason of breach of contract, tort, indemnification, or otherwise, but excluding liability of seller for breach of warranty (the sole remedy for which shall be as otherwise provided herein)) shall be the invoice price of this product.

## Repair Policies

### Under Warranty Repair:

If the HotBlock® should fail to operate as warranted within the warranty period (one year from date of shipment), Environmental Express will repair it and ship it back to the customer at Environmental Express' expense. The remainder of the warranty period will be honored from the original ship date. Environmental Express will bear the cost of ground transportation both to and from the customer's location, and bear the cost of any parts, labor and cleanup required.

However, if it is determined that the damage to the HotBlock® was caused by negligence or improper use or by another excluded cause as set forth above, this warranty will not apply. The warranty is also void if the system is used beyond its intended purpose or in the event of any unauthorized repair. In such cases, reasonable and customary repair charges will apply. Repair charges will be quoted prior to work being done.

### Out of Warranty Repair:

If the HotBlock® fails after the warranty period has lapsed, the repair procedure is as follows:

First, notify an Environmental Express Technical Service Representative of product's failure and place an order for repair. Whenever possible, our customer service technician will walk you through possible troubleshooting scenarios which may enable you to repair your block on-site.

If on-site repair is not possible, the customer may return the non-working unit to Environmental Express using appropriate shipping containers and insurance. Repair charges will be assessed and estimated prior to work being done. Repair charges will include all freight costs as well as reasonable and customary charges for parts and labor.

**Note:** This warranty does not apply to any consumable items associated with the HotBlock® system.

Loaner HotBlock® MAY be available during the repair period. There are only a limited number of these units. A reasonable charge for "cleanup" will be charged if a loaner is issued. The customer will be responsible for all shipping charges associated with a loaner unit.

The manufacturer, Environmental Express, 2345A Charleston Regional Parkway, Charleston, SC 29492 declares that the following products, HotBlock® Item Numbers, SC196, SC154, SC150, SC151, SC100, and C6002, are in conformity with:



Standard for Safety Electrical Equipment for Measurement, Control, and Laboratory Use; Part 1 General Requirements, UL 61010-1, CAN/CSA-C22.2 No. 61010-1, 2nd Edition, Issued 12 July, 2004 with revisions through and including 28 October, 2008; Equipment for Measurement, Control, and Laboratory Use Part 2-010: Particular Requirements for Laboratory Equipment for the Heating of Materials, IEY 61010-2-010, 2nd Edition, Issued 1 June, 2003, Safety Requirements for Electrical Equipment for Measurement, Control, and Laboratory Use - Part 2-010: Particular Requirements for Laboratory Equipment for the Heating of Materials, CSA C22.2.61010.2.01



Environmental Express declares that all HotBlocks conform with the essential requirements of the applicable EC directives.



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**Product Information:**

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Item #	Date of Purchase
HotBlock® Serial #	

Please record the serial # of your HotBlock® here for easy reference. Your serial # is located on the back of your HotBlock®.

## HotBlocks® for 50mL Samples

### 36-Well 50mL HotBlock®



#### SC100:

Nominal Sample Size:	50mL
Temperature Range:	to 150°C
Thermocouple:	Type K
Footprint:	15" x 15"
Crated Size:	23" x 23" x 17"
Weight:	43 lb.
Shipping Weight:	58 lb.
Electrical:	120VAC*, 9A
Sample Capacity:	36
Racks Included:	SC200 (2)

### 54-Well 50mL HotBlock®



#### SC154:

Nominal Sample Size:	50mL
Temperature Range:	to 150°C
Thermocouple:	Type K
Footprint:	15" x 21.5"
Crated Size:	26" x 23" x 17"
Weight:	59 lb.
Shipping Weight:	78 lb.
Electrical:	120VAC*, 14A
Sample Capacity:	54
Racks Included:	SC200 (3)

### 12-Well 50mL HotBlock®



#### C6002:

Nominal Sample Size:	50mL
Temperature Range:	to 150°C
Thermocouple:	Type K
Footprint:	8.55" x 18.5"
Crated Size:	22" x 19" x 13"
Weight:	30 lb.
Shipping Weight:	35 lb.
Electrical:	120VAC, 13A
Sample Capacity:	12
Racks Included:	C6050 (1)

\* All HotBlocks® are also available in 240V.

## HotBlocks® for 15mL Samples

### 96-Well 15mL HotBlock®



#### SC196:

Nominal Sample Size:	15mL
Temperature Range:	to 150°C
Thermocouple:	Type K
Footprint:	15" x 21.5"
Crated Size:	26" x 23" x 17"
Weight:	59 lb.
Shipping Weight:	80 lb.
Electrical:	120VAC*, 13A
Sample Capacity:	96
Rack Set Included:	SC296

## HotBlocks® for 100mL Samples

### 25-Well 100mL HotBlock®



#### SC150:

Nominal Sample Size:	100mL
Temperature Range:	to 150°C
Thermocouple:	Type K
Footprint:	15" x 15"
Crated Size:	23" x 23" x 17"
Weight:	42 lb.
Shipping Weight:	54 lb.
Electrical:	120VAC*, 9A
Sample Capacity:	25
Rack Set Included:	SC225

### 35-Well 100mL HotBlock®



#### SC151:

Nominal Sample Size:	100mL
Temperature Range:	to 150°C
Thermocouple:	Type K
Footprint:	15" x 21.5"
Crated Size:	26" x 23" x 17"
Weight:	59 lb.
Shipping Weight:	65 lb.
Electrical:	120VAC*, 13A
Sample Capacity:	35
Rack Set Included:	SC235

\* All HotBlocks® are also available in 240V.

## HotBlock® Hot Plate

### HotBlock® Hot Plate



\* Also available in 240V.

#### SC175:

Temperature Range:	Ambient to 150°C
Thermocouple:	Type K
Footprint:	15" x 21.5"
Weight:	35 lb.
Wattage:	1065 W
Electrical:	120VAC*, 14A

### About Your Environmental Express® HotBlock®

Environmental Express HotBlocks® provide an efficient method of digesting and storing water, wastewater, soil and sludge samples for metals analysis. These innovative digestion systems allow samples to be digested in a corrosion-free environment. In addition, samples are handled in a small area with minimal radiant heat loss. Users should be aware of potential dangers from heating certain types of compounds. Such hazards may include explosion or the release of toxic or flammable gases.

### Definitions/Markings

Each HotBlock® displays certain markings and symbols. All personnel working with the HotBlocks® should have an understanding of the following symbols and definitions:

- V = voltage
- ~ = alternating current
- Hz = frequency
- A = amperes



This symbol means **Caution Hot Surface**. The surface of the HotBlock® may be too hot to safely touch with bare hands.



This symbol means **Read and become familiar with instructions before operation of instrument**.

## Unpacking Your HotBlock®:

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1. Remove the HotBlock® from the shipping container by lifting from the bottom of the block. The lid should not be used for lifting.
2. Your HotBlock® is shipped with metal screws securing the bottom panel. The metal screws must be removed before operating your HotBlock®. Remove the metal screws and replace them with the PVC screws and rubber feet included with your shipment. The corrosion-resistant PVC screws and rubber feet are designed to secure the bottom plate.

***Important: Do not over tighten the PVC screws!***

## Installation Requirements

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Locate the HotBlock® under a fume hood with a minimum face velocity of 100fpm, and allow a minimum of 2" of space on all sides. The following environmental conditions should be observed:

- Ambient temperature range: 5-30°C
- Ambient relative humidity: 0-90%RH
- Altitude: sea level to 2500 meters

HotBlocks® are rated as Pollution Degree 2 and Installation Category 2.

## Electrical Requirements

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Required Voltage: 120 volts, ~60Hz, 15A  
(all HotBlocks® are also available in 240V with CE mark)

Power should not vary greater than  $\pm 10\%$ . Use the supplied heat-resistant power cord or equivalent to connect to the power supply.

For safety reasons, a separate power receptacle should be provided for each unit in the system. Do not use extension cords or outlet adaptors. Make certain that power outlets are earth-grounded at the grounding pin.

See individual specifications for each HotBlock® model, pages 3-5.



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## HotBlock® Temperature Settings:

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The pre-set factory “set point” temperature of your HotBlock® is 106°C. Factory tests have shown that this temperature is “sea level safe.” Liquids in uncovered polypropylene tubes should not boil at this setting. Please note that the set point of the block is not the same as the temperature of the liquids being digested. The block temperature should be optimized for the specific digestion. The temperature of liquid contents of the digestion cup will vary according to:

- The material being digested
- The number of samples being digested
- The air movement of the digestion area
- The addition of a watch glass or reflux cap

If watch glasses are being used, the control point temperature should be lowered approximately 10°C to avoid boiling.

**Note:** *The maximum sample temperature tolerance for our polypropylene digestion vessels (SC475) is 130°C.*

*Remember that the temperature display (current block temperature) is not the temperature of the sample. Sample temperature will usually be 5° to 15° less than the display temperature.*

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## Adjusting the Temperature of Your HotBlock®:

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1. Plug the HotBlock® into an approved receptacle. Turn the HotBlock® on by pressing the button on the back of the unit. Wait until the display shows the current block temperature (in red) and the set point temperature (in green).
2. Press and hold or tap the  $\Delta$  or  $\nabla$  key. The display will show the set point temperature on the right in green. The adjustment is from ambient to 150°C in increments of 0.1°C. There is no need to press the green (advance) or  $\infty$  button.

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## Safe-Sample™ Temperature Protection:

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Your HotBlock® is designed to protect from runaway temperatures by a fail-safe alarm system. In the unlikely event that the heating system fails to respond to the controller, the Safe-Sample™ system will automatically shut the system off and sound an audible alarm.

This alarm sequence occurs if the actual temperature of the block reaches a temperature that is fifteen degrees higher than the set point temperature. If this should occur, the HotBlock® will stop heating, preventing the loss of samples. The HotBlock® must be turned off, then turned back on to reset the alarm.



**If the alarm sounds, see the troubleshooting section of this manual, pages 11-13.**

## Potential Hazards:

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*The HotBlock® should only be operated by properly trained personnel using standard laboratory safety practices.*

Use extreme caution when operating the HotBlock®. Plastic and graphite surfaces of the HotBlock® may be too hot to safely touch with bare hands.

The HotBlock® contains electrical circuits and devices and compounds operating at dangerous voltages. Contact with these circuits, devices and components can cause serious injury or painful electric shock.

Proper grounding is essential to avoid a potentially serious electric shock hazard. Ensure that there is an internal ground connection between the metal base of the system and the 3-pin, earth-grounded receptacle.

For safety reasons a separate power outlet receptacle should be provided for each unit in the system. Do not use extension cords or outlet adaptors. Make certain each power outlet is earth-grounded at the grounding pin.

See individual block specifications for power requirements, pages 3-4.

Application of the wrong supply voltage can create a fire hazard and a potentially serious shock hazard, and could seriously damage the HotBlock® system. See specifications for individual HotBlocks®.

Users should be aware of potential dangers from heating certain types of compounds. Such dangers may include explosion or the release of toxic or flammable gases.

Always lift the HotBlock® from the bottom of the unit.

## Maintenance:

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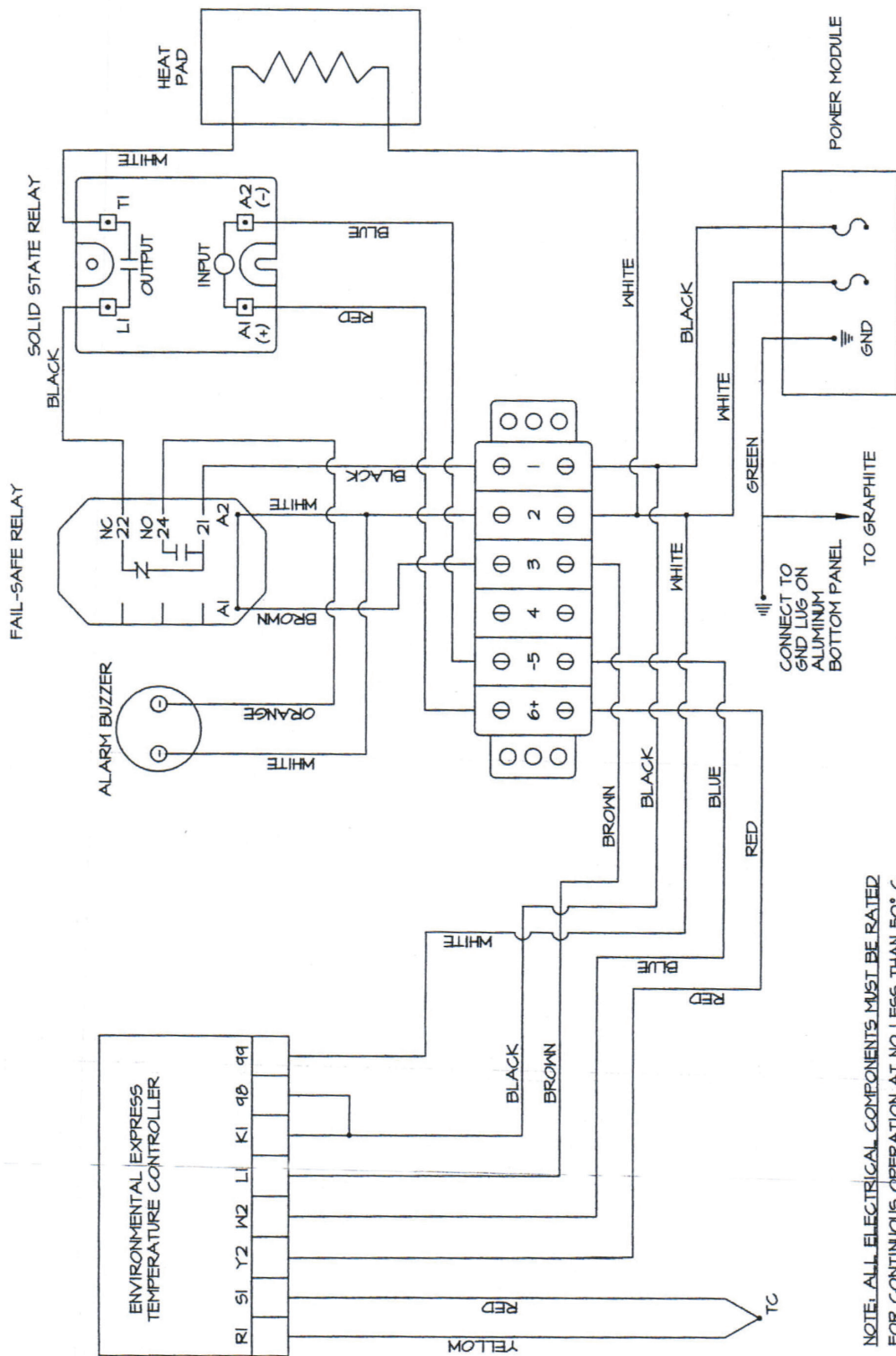
Any service inquiries should be directed to Environmental Express Technical Service Department.

After each use, clean exterior surfaces with a damp sponge to remove acid residue.

For acid spills, sponge with a diluted solution of sodium bicarbonate followed by distilled water. Acid that is spilled directly into the digestion wells should be neutralized and removed.

Before using any cleaning or decontamination methods except those recommended, check with Environmental Express to confirm the proposed method will not damage your HotBlock®.

Avoid excessive spills, as liquid allowed to overflow into the HotBlock® casing can severely damage electronic components.



NOTE: ALL ELECTRICAL COMPONENTS MUST BE RATED FOR CONTINUOUS OPERATION AT NO LESS THAN 50°C AMBIENT CONDITIONS

BLOCK DIAGRAM FOR HOT BLOCK  
ELECTRICAL CONTROLS  
ENVIRONMENTAL EXPRESS  
REVISED 2-10-10  
DOMB460IRI

## HotBlock® Replacement Parts

<i>Description</i>	<i>Item #</i>
Power module (plug receptacle) w/ push button switch	SC941
Power Cord- heavy duty	SC958
Environmental Express Controller	SC945-W
12" X 12", 120V, silicone rubber, etched-foil heater mat for models SC100 and SC150	SC951
12" X 12", 240V, silicone rubber, etched-foil heater mat for models SC100-240 and SC150-240	SC951-240*
110/220V, 25A solid state relay	SC952
Type K Thermocouple	SC953
Terminal board	SC955
Ceramic fiber insulation for models SC100 and SC150	SC959
14" X 14" Powder-coated aluminum bottom for models SC100 and SC150	SC963
12" X 18", 120V, silicone rubber, heater mat for models SC154, SC196, SC151, and SC175	SC966
12" X 18", 240V, silicone rubber, heater mat for models SC154-240, SC196-240, SC151-240, and SC175-240	SC966-240*
15" X 5" 120V silicone rubber heater mat for model C6002 (12-Place Block)	C6300
15" X 5" 120V silicone rubber heater mat for model C6002-240 (12-Place Block)	C6300-240*
Fail-Safe Relay	SC968
Alarm Buzzer	SC969
Ceramic fiber insulation for models SC154, SC196, SC151, and SC175	SC970
14" X 19", powder-coated aluminum bottom for models SC154, SC196, SC151, and SC175	SC971
PVC screw for rubber foot	SC964
Rubber foot	SC976

*\*for HotBlocks® shipped outside the United States and Canada*

## HotBlock® Troubleshooting Guide:

If you are experiencing issues and are unsure of your ability to fix AND/OR are not a qualified individual to work on electrical components and equipment, please contact our technical service team at +1.800.745.8218 and we'd be happy to help. If you are experiencing problems with your HotBlock® and you are a qualified individual, then please consult the following troubleshooting guide. See wiring schematic diagram (page 9) for component identification.

When the HotBlock® is initially powered on, the controller will cycle through a self-test sequence. It will then display the current temperature and begin heating until it reaches your set point temperature, where it will hold until the unit is powered off. The set point may be changed at any time. A change in the controller's factory default settings or a failed component may cause the HotBlock® to perform unsatisfactorily or render it inoperable.

### The controller digital display will not illuminate.

There are two possible reasons that your controller will not illuminate.

1. The controller is not getting voltage or;
2. The controller itself has failed internally.

The problem can be effectively diagnosed by determining if the controller is or is not getting voltage using the following steps:

1. Confirm that the power cord is plugged securely into the HotBlock® receptacle and a working outlet.
2. Confirm that the switch is in the "on" position. Press button on the back of HotBlock®.
3. Check the fuse located in the power module:
  - a. Locate the fuse drawing indented into the power module next to the socket.
  - b. Using a small screwdriver, pry open the fuse compartment cover.
  - c. Examine the exposed fuse for a break in the filament and if necessary, check for continuity using a volt-meter.
  - d. If the fuse is determined to be blown, replace it with the spare fuse located in the slide-out compartment beneath the operating fuse.
4. Inside the HotBlock®, check voltage leading from the power module to the controller:
  - a. Remove the bottom panel of the HotBlock® by unscrewing the rubber feet.
  - b. On the back of the controller, locate the black wire at terminal 98 and white wire at terminal 99.
  - c. Set your volt-meter on AC voltage.
  - d. Touch your red lead to the exposed white wire and black lead to the exposed black wire.
  - e. If your volt-meter reads 110-122V, the controller is receiving power but has failed internally. It must be replaced (see parts list, page 10).
  - f. If your volt-meter registers less than 110-122V, using step d above check the black and white wires at the terminal board and then at the power module to determine if there is a faulty connection.



#### CAUTION:

These procedures are a potential electrical hazard and should only be performed by a qualified individual who is trained and experienced in the repair and maintenance of electrical components, equipment and instruments.

**The audible alarm has sounded immediately after powering on and the HotBlock® will not heat.**

There are two possible causes for your HotBlock® to sound the alarm immediately after the controller cycles through the self test. These are:

1. Your set point has been set to a value ( $\geq$ ) 15° less than ambient or current set point temperature. Turn the set point to within 15° of the actual temperature (blue numbers)
2. The controller is faulty. Call Environmental Express at +1.800.745.8218 for more information.

**The temperature has overshot the set point and the audible alarm has sounded.**

The function of the fail-safe system is to cease heating of the HotBlock® in the event of a set point overshoot of 15°C and to alert the technician of the incident.

The HotBlock® can be “fooled” into fail-safe mode if the set point is manually changed to a value  $\geq 15^\circ$  below your current temperature. However, the primary cause for the runaway temperature is a faulty relay that has exceeded its useful life.



**CAUTION:**

These procedures are a potential electrical hazard and should only be performed by a qualified individual who is trained and experienced in the repair and maintenance of electrical components, equipment and instruments.

You may troubleshoot the relay by following these steps:

1. Power off your HotBlock®.
2. Remove the bottom panel of the HotBlock® by unscrewing the rubber feet.
3. Power the HotBlock® on and allow it to overshoot your set point temperature.
4. Locate the solid state relay mounted to the bottom panel.
5. Set your volt-meter to measure AC voltage.
6. Touch your red lead to terminal T1 (white wire) of the solid state relay and touch your black lead to a ground source (e.g., the green/yellow wire from the power module or an empty terminal on the terminal board).
7. If your volt-meter reads 110V-122V, then the relay is stuck in the “closed” position and it must be replaced (see parts list, page 10).

**The temperature controller is performing erratically or displays an error message.**

**-OR-**

**The temperature controller is flashing *Er.1 Attn*- Thermocouple Fault**

First reset default settings:

1. Hold the up and down arrow buttons for six seconds until ***Ai Set*** appears
2. Hold the down arrow until ***glbl set*** is in the window
3. Press the green advance key to enter
4. Continue pressing the green advance key until ***none user*** appears.
5. Press the down arrow key until ***Set1*** user appears.
6. Pressing the advance key will restore default settings.

To troubleshoot the thermocouple, follow these steps:

1. Power off your HotBlock®.
2. Remove the bottom panel of your HotBlock® by unscrewing the rubber feet.
3. Locate the thermocouple wires at terminals R1 (yellow) and S1 (red) of the controller and remove using a small screwdriver.
4. Cut the exposed ends of the two wires.
5. Strip 1/4" of insulation from each wire and reconnect them to the appropriate controller terminal and tighten.
6. Power on your HotBlock®.
7. If your display continues to flash ***Er.1 Attn***- the thermocouple is faulty and must be replaced (see parts list, page 10).

**The HotBlock® will not heat beyond ambient temperature.**



**CAUTION:**

These procedures are a potential electrical hazard and should only be performed by a qualified individual who is trained and experienced in the repair and maintenance of electrical components, equipment and instruments.

A HotBlock® that will not heat beyond ambient temperature typically has a failed relay, heater mat or controller.

**Relay:** To test the relay, the output voltage must be determined with a volt-meter.

To measure the relay voltage, follow these steps:

1. Remove the bottom panel of your HotBlock® by unscrewing the rubber feet.
2. Locate the solid state relay mounted to the bottom panel.
3. Set your volt-meter to measure AC voltage.
4. Touch your red lead to terminal T1(white wire) of the solid state relay and touch your black lead to a ground source (i.e., the green/yellow wire from the power module or an empty terminal on the terminal board).
5. If your volt-meter does not read 110V-122V, then the relay has stuck in the **open** position and it must be replaced (see parts list, page 10).

**Heater Mat:** To test the heater mat, the resistance in ohms ( $\Omega$ ) must be determined with a volt-meter. It is recommended that your heater mat be replaced if it measures 25 ohms ( $\Omega$ ) or greater. It is also recommended that the thermocouple and insulation be replaced as well, both are inexpensive parts that are not easily accessible otherwise.

To measure your heater mat resistance, follow these steps:

1. Power off your HotBlock® and remove the bottom panel of your HotBlock® by unscrewing the rubber feet.
2. Locate and disconnect the white wire connected to terminal T1 of the relay and an identical wire on terminal #2 of the terminal board (note: terminal #2 of the terminal board contains 3 white wires). To ensure you have the correct wire, trace it back and ensure it originates from the graphite portion of your HotBlock®.
3. Set your volt-meter to measure ohms ( $\Omega$ )
4. Touch the red lead to one of these wires and touch the black lead to the remaining wire.
5. If your reading is "OL" (over limit) or a value greater than 25 ohms, then the heater mat has failed and it must be replaced (see parts list, page 10).



**CAUTION:**

These procedures are a potential electrical hazard and should only be performed by a qualified individual who is trained and experienced in the repair and maintenance of electrical components, equipment and instruments.

**Controller:** To test the controller, the output voltage must be determined with a volt-meter.

To measure voltage from the controller, follow these steps:

1. Remove the bottom panel of your HotBlock® by unscrewing the rubber feet.
2. Locate the solid state relay mounted to the bottom panel.
3. Set your volt-meter to measure DC voltage.
4. Touch the red lead to terminal A1 (red) and the black lead to terminal A2 (blue) of the solid state relay.
5. Your volt-meter should read 3V-16V.
6. If your volt-meter does not read 3V-16V, perform steps 3 and 4 on the red and blue (5 and 6) wires at the terminal board and controller to determine if there is a faulty or loose connection.
7. If you do not get a reading of 3V-16V at terminals Y2 (red) and W2 (blue) of the controller then the controller has failed internally and it must be replaced (see parts list, page 10).

## Adaptation for EPA Method 200.2, Revision 2.8 for use with the Environmental Express HotBlock® Digestion System

Revised 05.05

### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to EPA digestion procedure 200.2 for use with the Environmental Express HotBlock®. EPA Method 200.2 is for the determination of total recoverable analytes in groundwater, surface water, drinking water, wastewater and (with the exception of silica) in solid samples such as sediment, sludge and soil. Use EPA Method 200.2 for reference while following the sample preparation steps outlined below. A complete list of elements appropriate for analysis is included in EPA Method 200.2. Analysis can be performed by flame atomic absorption, GF/AA, ICP and ICP-MS.

### 2.0 Apparatus and Materials

- 2.1 HotBlock® for Metals Digestions — Item # SC100, SC150\* or SC154
- 2.2 Polypropylene Digestion Vessels — Item # SC475 (or SC490 for use with HotBlock® SC150)
- 2.3 Ribbed Watch Glasses, Item # SC505 or Reflux Caps, Item # SC506 (for use with SC490 vials for HotBlock® SC150, use Ribbed Watch Glasses, Item # SC610)
- 2.4 FilterMate® — Item # SC0401 (or appropriate FilterMate) for sample filtration if necessary

*\*Note: For all procedures, when using the SC150 block with the SC490 digestion vials with a 100mL sample, double the volume of all reagents and acids added.*

### 3.0 Procedure, Aqueous Sample Preparation

- 3.1 Mix sample thoroughly to achieve homogeneity. For each digestion procedure, transfer a 50mL sample (or appropriate volume for your lab) into the SC475 polypropylene vessel. For best results, weigh the sample directly in the vessel on a tared balance.
- 3.2 Add 1mL (1:1) HNO<sub>3</sub> and 0.5mL (1:1) HCl, then swirl. Heat the sample in the HotBlock® at 95°C for 2.5 hours without boiling. Place a ribbed watch glass (SC505) on top of the digestion vessel.

*Note: When using a watch glass, adjust the control point temperature of the HotBlock® so that a 50mL, 5% acid solution is heated to 85°C BEFORE placing the watch glass over the sample. Laboratory tests have proven that the addition of the ribbed watch glass will add approximately 10°C to the sample temperature, bringing the sample temperature up to 95°C. Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.*

- 3.3 Using the polycarbonate transfer racks, remove samples from the HotBlock® and allow them to cool.
- 3.4 After cooling, dilute samples to 50mL with DI Water.
- 3.5 If necessary, filter with SC0401 (or appropriate FilterMate) to remove insoluble material.

*Note: The filtration step should be performed slowly with little pressure placed on the plunger. If excessive back pressure occurs, stop filtration and allow sediments to “settle out”. Applying pressure to the plunger may cause sample “blow through” allowing sediment to pass through the filter into the digestate.*



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#### 4.0 Procedure, Solids Sample Preparation

- 4.1 Sieve a dried sample using a 5-mesh polypropylene sieve and grind in a mortar and pestle. Weigh a representative sample of  $0.5 \pm 0.01\text{g}$  into a digestion vessel.
- 4.2 Add 2mL of (1+1)  $\text{HNO}_3$  and 5mL of (1+4) HCl+DI. Cover with a ribbed watch glass (SC505) or reflux cap (SC506), and place in the HotBlock® at  $95^\circ\text{C}$ . **Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.**
- 4.3 Heat sample for 30 minutes.
- 4.4 (Optional) Although step 4.3 is the final heating step for EPA Method 200.2, if the sample is suspected of having a high concentration of organic compounds, it is recommend to complete this step:  
Add 2mL of 30%  $\text{H}_2\text{O}_2$  to the well-cooled sample. Allow the exothermic reaction to occur (approximately 10 minutes) and place the sample back in the HotBlock® at a temperature of  $10^\circ$  less than the original set point for an additional 30 minutes. The reaction with the  $\text{H}_2\text{O}_2$  raises the sample temperature. Boiling should not occur if the temperature of the HotBlock® is lowered.

*Note:  $\text{H}_2\text{O}_2$  helps aid in the breakdown of high organic compounds in the sample thus creating a more complete digestion.*

- 4.5 Using the polycarbonate transfer racks, remove samples from the HotBlock® and allow them to cool completely.
- 4.6 Bring sample volume to 50mL with DI water.
- 4.7 If necessary, filter with SC0401 (or appropriate FilterMate) to remove insoluble material.

*Note: The filtration step should be performed slowly with little pressure placed on the plunger. If excessive back pressure occurs, stop filtration and allow sediments to “settle out”. Applying pressure to the plunger may cause sample “blow through” allowing sediment to pass through the filter into the digestate.*

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All QC samples, concentration limitations, elemental lists and reagent specifications are addressed in depth in EPA Method 200.2. Safety concerns are also part of the full method. Follow the instructions listed in EPA Method 200.2. These steps should only be used as a guide to help improve the performance of your HotBlock®.

## Adaptation for EPA Method 200.7, Revision 4.4 for use with the Environmental Express HotBlock® Digestion System

Revised 05.05

### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to EPA digestion procedure 200.7 for use with the Environmental Express HotBlock®. EPA Method 200.7 is for the determination of total recoverable analytes in groundwater, surface water, drinking water, wastewater, and (with the exception of silica) in solid samples such as sediment, sludge and soil. Use EPA Method 200.7 for reference while following the sample preparation steps outlined below. A complete list of elements appropriate for analysis is included in EPA Method 200.7. Analysis can be performed by ICP.

### 2.0 Apparatus and Materials

- 2.1 HotBlock® for Metals Digestions — Item # SC100, SC150\*, or SC154
- 2.2 Polypropylene Digestion Vessels — Item # SC475 (or SC490 for use with HotBlock® SC150)
- 2.3 Ribbed Watch Glasses, Item # SC505 or Reflux Caps, Item # SC506 (for use with SC490 vials for HotBlock® SC150, use Ribbed Watch Glasses, Item # SC610)
- 2.4 FilterMate® — Item # SC0401 (or appropriate FilterMate) for sample filtration if necessary

*\*Note: For all procedures, when using the SC150 block with the SC490 digestion vials with a 100mL sample, double the volume of all reagents and acids added.*

### 3.0 Procedure, Total Recoverable Elements, Aqueous Sample

(EPA Method 200.7, Paragraph 11.2)

- 3.1 Mix sample thoroughly to achieve homogeneity. For each digestion procedure, transfer a 50mL sample (or appropriate volume for your lab) into the SC475 polypropylene vessel. For best results, weigh the sample directly in the vessel on a tared balance.
- 3.2 Add 1mL 1:1 HNO<sub>3</sub> and 0.5mL 1:1 HCl and swirl. Heat in the HotBlock® at a sample temperature of 85°C and evaporate to about 10mL taking care that the cup does not go dry. This will take approximately 2 hours.
- 3.3 Cover the vessel with a disposable watch glass (Item # SC505) and reflux an additional 30 minutes.  
*Note: When using a watch glass, adjust the set point temperature of the HotBlock® so that a 50mL, 5% acid solution is heated to 85°C BEFORE placing the watch glass over the sample. Laboratory tests have proven that the addition of the ribbed watch glass will add approximately 10°C to the sample temperature, bringing the sample temperature up to 95°C. Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.*
- 3.4 Using the polycarbonate transfer racks, remove samples from the HotBlock® and allow them to cool. Bring samples up to volume with DI water.
- 3.5 If necessary, filter with SC0401 (or appropriate FilterMate) to remove

insoluble material.

*Note: The filtration step should be performed slowly with little pressure placed on the plunger. If excessive back pressure occurs stop filtration and allow sediments to “settle out”. Applying pressure to the plunger may cause sample “blow through” allowing sediment to pass through the filter into the digestate.*

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#### 4.0 Procedure, Total Recoverable Elements, Solids Sample (EPA Method 200.7, Paragraph 11.3)

- 4.1 For each digestion procedure, transfer  $0.5\text{g} \pm 0.01\text{g}$  of a dried sieved sample into the SC475 polypropylene vessel.
- 4.2 Add 2.0mL (1:1)  $\text{HNO}_3$  and 5mL (1:4) HCl and swirl. Cover the vessel with a disposable watch glass (Item # SC505) and reflux an additional 30 minutes at a sample temperature of  $85^\circ\text{C}$ .

*Note: When using a watch glass, adjust the set point temperature of the HotBlock® so that a 50mL, 5% acid solution is heated to  $85^\circ\text{C}$  BEFORE placing the watch glass over the sample. Laboratory tests have proven that the addition of the ribbed watch glass will add approximately  $10^\circ\text{C}$  to the sample temperature, bringing the sample temperature up to  $95^\circ\text{C}$ . Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.*

- 4.3 Allow to cool and bring to 50mL volume with DI water.
  - 4.4 Filter with SC0401 (or appropriate FilterMate) to remove insoluble material.
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All QC samples, concentration limitations, elemental lists and reagent specifications are addressed in depth in EPA Method 200.7. Safety concerns are also part of the full method. Follow the instructions listed in EPA Method 200.7. These steps should only be used as a guide to help improve the performance of your HotBlock®.

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## Adaptation for EPA Method 200.8 for use with the Environmental Express HotBlock® Digestion System

Revised 05.05

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### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to EPA digestion procedure 200.8 for use with the Environmental Express HotBlock®. EPA Method 200.8 is for the determination of total recoverable analytes in groundwater, surface water, drinking water, wastewater, and (with the exception of silica) in solid samples such as sediment, sludge and soil. Use EPA Method 200.8 for reference while following the sample preparation steps outlined below. A complete list of elements appropriate for analysis is included in EPA Method 200.8. Analysis can be performed by ICP-MS.

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### 2.0 Apparatus and Materials

- 2.1 HotBlock® for Metals Digestions — Item # SC100, SC150\*, or SC154
- 2.2 Polypropylene Digestion Vessels — Item # SC475 (or SC490 for use with HotBlock® SC150)
- 2.3 Ribbed Watch Glasses, Item # SC505 or Reflux Caps, Item # SC506 (for use with SC490 vials for HotBlock® SC150, use Ribbed Watch Glasses, Item # SC610 )
- 2.4 FilterMate® — Item # SC0401 (or appropriate FilterMate) for sample filtration if necessary

*\*Note: For all procedures, when using the SC150 block with the SC490 digestion vials with a 100mL sample, double the volume of all reagents and acids added.*

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### 3.0 Procedure, Aqueous Sample Preparation — Dissolved Analytes (EPA Method 200.8, Paragraph 11.1)

- 3.1 For the determination of dissolved analytes in ground and surface waters, pipet a 20mL or greater aliquot of filtered, acid-preserved sample into the SC475 digestion vessel.
- 3.2 Add an appropriate volume of (1+1) HNO<sub>3</sub> to adjust the acid concentration of the aliquot to approximate a 1% (v/v) nitric acid solution. If the direct addition procedure is being used, add internal standards, cap and mix.

*Note: If a precipitate is formed during acidification, transport or storage the sample aliquot must be treated using procedures for Total Recoverable Analytes.*

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### 4.0 Procedure, Aqueous Sample Preparation — Total Recoverable Analytes (EPA Method 200.8, Paragraph 11.2)

*Note: This section applies to water samples containing turbidity of greater than 1 NTU.*

- 4.1 For each digestion procedure, transfer 50mL of well-mixed, unfiltered, acid-preserved sample into the SC475 polypropylene vessel.
- 4.2 Add 1.0mL (1+1) HNO<sub>3</sub> and 0.5mL of (1+1) HCl and swirl. Heat in the HotBlock® at a sample temperature of 85°C. The HotBlock® set temperature should be approximately 105°C. The temperature of a reference blank should be tested to ensure correct temperature.
- 4.3 Reduce the volume of the sample aliquot to about 10mL at this

temperature. This should take about 2.5 hours.

- 4.4 Place a ribbed watch glass (SC505) over the digestion vessel to reduce additional evaporation.

*Note: When using a watch glass, adjust the set point temperature of the HotBlock® so that a 50mL, 5% acid solution is heated to 85°C BEFORE placing the watch glass over the sample. Laboratory tests have proven that the addition of the ribbed watch glass will add approximately 10°C to the sample temperature, bringing the sample temperature up to 95°C. Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.*

- 4.5 Gently reflux the sample for 30 minutes.
- 4.6 Using the polycarbonate transfer racks, remove samples from the HotBlock® and allow them to cool.
- 4.7 Add reagent water to bring the sample to the 50mL mark on the digestion vessel. Cap and mix.
- 4.8 If necessary, filter with SC0401 (or appropriate FilterMate) to remove insoluble material.

*Note: The filtration step should be performed slowly with little pressure placed on the plunger. If excessive back pressure occurs, stop filtration and allow sediments to “settle out.” Applying pressure to the plunger may cause sample “blow through” allowing sediment to pass through the filter into the digestate.*

- 4.9 Prior to analysis, adjust the chloride concentration by pipetting 20mL of the prepared solution into another SC475 digestion vessel and bring up to 50mL volume with reagent water. If the dissolved solids in this solution are >0.2%, additional dilution may be necessary to prevent clogging of the extraction and/or skimmer cones. Note the dilution factor for concentration calculations. If the direct addition procedure is being used, add internal standards, cap and mix.

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## 5.0 Procedure, Solid Sample Preparation

- 5.1 For each digestion procedure, transfer  $0.5\text{g} \pm 0.01\text{g}$  of a dried sieved sample into the SC475 polypropylene vessel.
- 5.2 Add 2.0mL (1:1)  $\text{HNO}_3$  and 5mL (1:4) HCl and swirl. Cover the vessel with a disposable watch glass (Item # SC505) and reflux an additional 30 minutes at a sample temperature of 85°C.

*Note: When using a watch glass, adjust the set point temperature of the HotBlock® so that a 50mL, 5% acid solution is heated to 85°C BEFORE placing the watch glass over the sample. Laboratory tests have proven that the addition of the ribbed watch glass will add approximately 10°C to the sample temperature, bringing the sample temperature up to 95°C. Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.*

- 5.3 Allow to cool and bring to 50mL volume with DI water.
- 5.4 Filter with SC0401 (or appropriate FilterMate) to remove insoluble material.

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All QC samples, concentration limitations, elemental lists and reagent specifications are addressed in depth in EPA Method 200.8. Safety concerns are also part of the full method. Follow the instructions listed in EPA Method 200.8. These steps should only be used as a guide to help improve the performance of your HotBlock®.

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## Adaptation of EPA Methods for Mercury Digestions 245.1, 7470, 7471 for use with the Environmental Express HotBlock® Digestion System

Revised 05.05

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### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to EPA Digestion Methods 245.1, 7470 and 7471 for use with the Environmental Express HotBlock®. EPA Methods 245.1 and 7470 are for the determination of mercury in aqueous samples and TCLP extracts. EPA Method 7471 is for the determination of mercury in soil, oil and sediment. Use the full EPA Methods for reference while following the sample preparation steps outlined below.

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### 2.0 Apparatus and Materials

- 2.1 HotBlock® for Metals Digestions — Item # SC100, SC150\*, or SC154
2. Polypropylene Digestion Vessels — Item # SC475 (or SC490 for use with HotBlock® SC150)
3. Ribbed Watch Glasses, Item # SC505 or Reflux Caps, Item # SC506 (for use with SC490 vials for HotBlock® SC150, use Ribbed Watch Glasses, Item # SC610)

*\*Note: For all procedures, when using the SC150 block with the SC490 digestion vials with a 100mL sample, double the volume of all reagents and acids added.*

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### 3.0 Procedure, Aqueous Sample Preparation

- 3.1 Add 20mL of well-mixed sample to a SC475 digestion vessel or 2mL of TCLP extract and 18mL water.  
*Note: This is a 10x dilution for instruments that can achieve the detection limit including the dilution, otherwise digest 20mL of TCLP extract.*
- 3.2 Add 20mL of standard solutions or appropriate amount of standard spiking solution to give desired concentration when diluted to 20mL.
- 3.3 Add 0.5mL of concentrated HNO<sub>3</sub>.
- 3.4 Add 1.0mL of concentrated H<sub>2</sub>SO<sub>4</sub>.
- 3.5 Mix thoroughly. Add 3mL of 5% KMnO<sub>4</sub> and let stand for 15 minutes. If sample does not maintain purple or brown color, add additional portions to all samples, blanks and standards until the color persists for at least 15 minutes.
- 3.6 Add 1.6mL of 5% persulfate solution. Place watch glass or reflux cap on top of digestion vessel to allow pressure to vent while minimizing evaporative loss.
- 3.7 Digest the sample for 2 hours at 95°C.
- 3.8 Remove samples and let cool to room temperature.
- 3.9 Add 1.2mL of sodium chloride/hydroxyl amine hydrochloride solution.

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### 4.0 Procedure, Soil, Oil and Sediments, 7471A:

- 4.1 Weigh 0.25-0.30g of homogenized sample into a tube. For best results, weigh the sample directly in the vessel on a tared balance.
- 4.2 Add 5mL DI water and 5mL aqua regia.
- 4.3 Heat for 2 minutes at  $95 \pm 3^\circ\text{C}$ .
- 4.4 Allow samples to cool and add 25mL DI water.
- 4.5 Add 7.5mL of  $\text{KMnO}_4$  and let stand for 15 minutes. If sample does not maintain purple or brown color, add additional portions to all samples, blanks and standards until the color persists for at least 15 minutes. Heat samples at  $95 \pm 3^\circ\text{C}$  for 30 minutes.

*Note: When using a watch glass, adjust the set point temperature of the HotBlock® so that a 50mL sample is heated to  $85^\circ\text{C}$  BEFORE placing the watch glass over the sample. Laboratory tests have proven that the addition of the ribbed watch glass will add  $10^\circ\text{C}$  to the sample temperature, bringing the sample temperature up to  $95^\circ\text{C}$ . Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.*

- 4.6 Let samples cool to room temperature and add 3mL of Sodium Chloride-Hydroxylamine Sulfate solution.

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All QC samples, concentration limitations, elemental lists and reagent specifications are addressed in depth in EPA Methods 245.1, 7470 and 7471. Safety concerns are also part of the EPA Methods. These steps should only be used as a guide to help improve the performance of your HotBlock®.

## Adaptation of EPA Method 365.1, Determination of Phosphorous by Semi-Automated Colorimetry, for use with the Environmental Express HotBlock® Digestion System Revised 05.05

### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to EPA Method 3651 for use with the Environmental Express HotBlock®. EPA Method 365.1 is for the determination of phosphorous in drinking water, surface water and saline water and in domestic and industrial wastes. Use EPA Method 365.1 for reference while following the sample preparation steps outlined below. Range is 0.01 to 0.5 mg/L.

### 2.0 Apparatus and Materials

- 2.1 HotBlock® for metals digestions — Item # SC100, SC150\*, or SC154
- 2.2 Polypropylene Digestion Vessels — Item # SC475 (or SC490 for use with HotBlock® SC150)
- 2.3 FilterMate® — Item # SC0401 (or appropriate FilterMate) for sample filtration if necessary

*\*Note: For all procedures, when using the SC150 block with the SC490 digestion vials with a 100mL sample, double the volume of all reagents and acids added.*

### 3.0 Procedure, Aqueous Sample Preparation

- 3.1 For each digestion procedure, transfer 50mL of sample (or appropriate volume for your lab) into the SC475 polypropylene vessel.
- 3.2 Add 1mL of H<sub>2</sub>SO<sub>4</sub> solution and swirl. (11 N H<sub>2</sub>SO<sub>4</sub> — Slowly add 310mL concentrated H<sub>2</sub>SO<sub>4</sub> to 600mL distilled water. When cooled dilute to 1 liter.)
- 3.3 Add 0.4g of ammonium persulfate.
- 3.4 Heat the sample in the HotBlock® at approximately 100°C for 40 minutes. The sample should slightly boil.

*Note: Adjust the HotBlock® temperature so that the sample obtains a temperature of 100°C. Please refer to page 7 of the HotBlock® Operation Manual for temperature adjustment and principles of operation.*

- 3.5 Using the polycarbonate transfer racks, remove samples from the HotBlock® and allow them to cool.
- 3.6 After cooling, dilute to 50mL with DI Water.
- 3.7 If necessary, filter with SC0401 (or appropriate FilterMate) to remove insoluble material.

*Note: The filtration step should be performed slowly with little pressure placed on the plunger. If excessive back pressure occurs, stop filtration and allow sediments to “settle out.” Applying pressure to the plunger may cause sample “blow through” allowing sediment to pass through the filter into the digestate.*

All QC samples, concentration limitations, elemental lists and reagent specifications are addressed in depth in EPA Method 365.1. Safety concerns are also part of the full method. Follow the instructions listed in EPA Method 365.1. These steps should only be used as a guide to help improve the performance of your HotBlock®.



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## Adaptation of EPA Method 3050B for use with the Environmental Express HotBlock® Digestion System

Revised 05.05

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### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to EPA Method 3050B for use with the Environmental Express HotBlock®. EPA Method 3050B is for the preparation of sediment, sludge, and soil samples for analysis. Use EPA Method 3050B for reference while following the sample preparation steps outlined below. A complete list of elements appropriate for analysis is included in EPA Method 3050B. Analysis can be performed by flame atomic absorption, GF/AA, ICP and ICP-MS.

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### 2.0 Apparatus and Materials

- 2.1 HotBlock® for Metals Digestions — Item # SC100, SC150\*, or SC154
- 2.2 Polypropylene Digestion Vessels — Item # SC475 (or SC490 for use with HotBlock® SC150)
- 2.3 Ribbed Watch Glasses, Item # SC505 or Reflux Caps, Item # SC506 (for use with SC490 vials for HotBlock® SC150, use Ribbed Watch Glasses, Item # SC610)
- 2.4 FilterMate® — Item # SC0401 (or appropriate FilterMate) for sample filtration if necessary

*\*Note: When using the SC150 block with the SC490 digestion vials with a 100mL sample, double the volume of all reagents and acids added.*

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### 3.0 Procedure, Sample Preparation

- 3.1 Mix sample thoroughly to achieve homogeneity and sieve if appropriate. For each digestion procedure, weigh to the nearest 0.01 gram and transfer a 0.5 to 1-gram aliquot (or appropriate amount for your lab) to the SC475 polypropylene digestion vessel. For best results, weigh the sample directly in the vessel on a tared balance.
- 3.2 Add 5mL (1:1) HNO<sub>3</sub> + DI Water and swirl. Cover with a ribbed watch glass or reflux cap and heat the sample in the HotBlock® at 95±5°C for 15 minutes without boiling.

*Note: If using the watch glass, adjust the HotBlock® temperature so that a 50mL, 5% acid solution is heated to 85°C. Laboratory tests have proven that the addition of the ribbed watch glass will add approximately 10°C to the sample temperature. Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.*

- 3.3 Allow the sample to cool, then add 2.5mL concentrated HNO<sub>3</sub> and reflux at 95°C for 30 minutes. Repeat this step until NO brown fumes are given off by the sample.
- 3.4 Heat sample with the ribbed watch glass to a volume of about 5mL or for 2 hours at 95±5°C. Do not allow the sample to boil or go dry. Neither of these aspects should occur if the temperature of the HotBlock® is adjusted correctly.
- 3.5 Using the polycarbonate transfer racks, remove samples from the HotBlock® and cool completely (it is very important that samples are cooled completely).
- 3.6 Add 1mL DI Water and 1.5mL of 30% H<sub>2</sub>O<sub>2</sub> slowly. Allow an exothermic

reaction to occur. Wait 5-10 minutes and place samples back in the HotBlock® with the ribbed watch glasses in place. If effervescence starts to occur lift the samples out of the HotBlock® and allow the reaction to continue. Do not let the samples foam out of the vessel. (Reducing the HotBlock® display temperature by 10°C should reduce the effervescence while maintaining the sample temperature).

- 3.7 Continue to add 0.5mL of H<sub>2</sub>O<sub>2</sub> (no more than 5mL total) until the sample remains unchanged in color (no longer than 30 minutes). Continue heating for a total of 2 hours or until volume has been reduced to approximately 5mL.
- 3.8 For the analysis of samples for FLAA or ICP-AES, add 5mL concentrated HCl to each sample and cover with a ribbed watch glass and reflux at 95°C for 15 minutes. GFAA and ICP-MS digestions, skip this step.
- 3.9 After cooling, dilute to 50mL with DI Water.
- 3.10 If necessary, filter with SC0401 (or appropriate FilterMate) to remove insoluble material.

All QC samples, concentration limitations, elemental lists and reagent specifications are addressed in depth in EPA Method 3050B. Safety concerns are also part of the full method. Follow the instructions listed in EPA Method 3050B. These steps should only be used as a guide to help improve the performance of your HotBlock®.

## Adaptation of NIOSH Method 7303 for use with the Environmental Express HotBlock® Digestion System

Revised 05.05

### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to NIOSH Method 7303 for use with the Environmental Express HotBlock®. Method 7303 is for the preparation of cellulose ester membrane filters commonly used in air sampling devices. Use EPA Method 7303 for reference while following the sample preparation steps outlined below. A complete list of elements appropriate for analysis is included in Method 7303. Analysis can be performed by ICP or AA.

### 2.0 Apparatus and Materials

- 2.1 HotBlock® for Metals Digestions — Item # SC100, SC150\*, or SC154
- 2.2 Polypropylene Digestion Vessels — Item # SC475 (or SC490 for use with HotBlock® SC150)
- 2.3 Ribbed Watch Glasses, Item # SC505 or Reflux Caps, Item # SC506 (for use with SC490 vials for HotBlock® SC150, use Ribbed Watch Glasses, Item # SC610 )
- 2.4 FilterMate® — Item # SC0401 (or appropriate FilterMate) for sample filtration if necessary

*\*Note: When using the SC150 block with the SC490 digestion vials with a 100mL sample, double the volume of all reagents and acids added.*

### 3.0 Procedure, Filter Preparation

- 3.1 Remove filter from cassette holder and fold into quarters taking care not to lose any sample.
- 3.2 Place in SC475 digestion vessel and add 1.25mL HCl.
- 3.3 Cover with plastic watch glass or reflux cap. Place in HotBlock® and heat at a sample temperature of 95°C for 15 minutes.

*Note: If using the watch glass, adjust the HotBlock® temperature so that a 50mL, 5% acid solution is heated to 85°C. Laboratory tests have proven that the addition of the ribbed watch glass will add approximately 10°C to the sample temperature. Refer to page 7 of the operations manual for instructions on HotBlock® temperature adjustment.*

- 3.4 Remove samples from the HotBlock® and cool for 5 minutes.
  - 3.5 Remove watch glass and add 1.25mL HNO<sub>3</sub>. Replace watch glass and return to HotBlock® at sample temperature of 95°C for 15 minutes.
  - 3.6 Remove the sample from the HotBlock® and cool for at least 5 minutes. Rinse watch glass into vessel. Discard watch glass.
  - 3.7 Dilute to 25mL final volume with distilled, deionized type II water.
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All QC samples, concentration limitations, elemental lists and reagent specifications are addressed in depth in NIOSH Method 7303. Safety concerns are also part of the full method. Follow the instructions listed in Method 7303. These steps should only be used as a guide to help improve the performance of your HotBlock®.

## Sample Preparation for Lead Analysis using the GhostWipe® Wipes

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### Methodology Note:

The following procedure has been written as an aid for use with the Environmental Express HotBlock® and is adapted from ASTM E-1644 Digestion of Dust Wipe Samples for the Determination of Lead. This method is intended merely as a guide for the digestion. Equipment, supplies, reagents, etc. are suggestions for use in the absence of method specified guidance. The instructions of the required method (where available) must always be used in place of any guidance in this method.

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### 1.0 Scope and Application

- 1.1 This application deals with the preparation of samples using lead wipes, specifically the GhostWipe® wipes from Environmental Express. The sample is heated with nitric acid and hydrogen peroxide to dissolve the wipe and all lead compounds into solution. Utilize the appropriate analytical technique to determine the amount of lead in solution.

## 2.0 Apparatus and Materials

- 2.1 HotBlock® for Metals Digestion – Item # SC100, SC154, or SC150. Other models, including custom sizes, are appropriate to use provided they offer the desired number of wells and sample volume needed for analysis.
  - 2.2 GhostWipe® wipes – Item # SC4210 or SC4250.
  - 2.3 Polypropylene Digestion Vessels – Item # SC475, SC490, or other part number of identical vessel in various configurations of caps and/or colors. The size of the vessel should correspond to the size of the digestion block.
  - 2.4 Ribbed Watch Glasses (Item # SC505 and SC150) or Reflux Caps (Item # SC506). Use the size appropriate for the digestion vessel.
  - 2.5 Filtration Apparatus (optional, only needed if wipe does not fully dissolve or other insoluble materials are present). Suggested options are FilterMate® system (Item # SC0407) and FlipMate® system (Item # SC0607 or SC0601).
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## 3.0 Reagents

- 3.1 Concentrated Nitric Acid – purity suitable for analytical detection limits.
  - 3.2 Hydrogen Peroxide 30% – purity suitable for analytical detection limits.
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## 4.0 Procedure

- 4.1 After sampling the appropriate area, transfer the wipe to the polypropylene digestion vessel. It is recommended that the wipe be transported from the sampling area to the analytical area in the sealed digestion vessel so that loss on transfer is prevented. Label vessels immediately after collection to minimize the potential for sample confusion.
  - 4.2 Add any needed spiking amounts to the appropriate Quality Control designated vessels.
  - 4.3 Preheat HotBlock to a temperature that will result in sample temperatures between 85°C and 100°C. **Note:** The HotBlock set point is typically 10 to 15°C above the desired sample temperature. Regular (often yearly) checks should be performed to determine the difference for each HotBlock. The watch glass and reflux cap will elevate the sample temperature by approximately 10°C over an uncovered sample.
  - 4.4 Place the digestion vessels in the appropriate size polycarbonate rack for easy transfer into and out of the HotBlock unit.
  - 4.5 Add 25 mL 1:1 nitric acid to each digestion vessel, gently swirl to mix, and cover with watch glass or reflux cap. **Caution:** When making diluted acid solutions, always add acid slowly to water in order to prevent violent exothermic reactions.
  - 4.6 Place the samples in the HotBlock and heat the samples at 85°C to 100°C for 10 to 15 minutes without boiling. As needed during the heating time, push the wipe gently with a glass rod to submerge it below the liquid level.
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- 4.7 Remove the samples from the HotBlock using the rack. Place in a safe area and allow the samples to cool to near room temperature
- 4.8 Add 10 mL of concentrated nitric acid to the samples, return samples to HotBlock, and reflux with watch glass or reflux cap for 30 minutes without boiling.
- 4.9 Remove the watch glass or reflux cap and allow the samples to evaporate to approximately 10 mL without boiling.
- 4.10 Remove samples from the HotBlock and allow them to cool to near room temperature.
- 4.11 Once samples are cooled to near room temperature, add 5 mL of reagent water and 5 mL of hydrogen peroxide to each sample. Cover samples with the watch glass or reflux cap and return to the HotBlock for warming to initiate the peroxide reaction. **Caution:** If samples have not cooled sufficiently, an excessively vigorous effervescence will occur. Similarly, rapid heating will result in the same reaction. Either case can result in loss of analyte.
- 4.12 Heat samples until the effervescence subsides. Remove the samples from the HotBlock and allow samples to cool to near room temperature.
- 4.13 Remove watch glass or reflux cap and return samples to the HotBlock. Heat the samples until the volume has been reduced to approximately 10 mL.
- 4.14 Remove the samples from the HotBlock and allow them to cool to room temperature. Remove the watch glass or reflux cap while cooling and gently rinse them into the digestion vessel. Once the samples are cool, rinse the sides of the vessel being careful not to splash any liquid out of the vessel. Bring liquid level, volumetrically, up to 100 mL and proceed with the appropriate analytical procedure. **Note:** At a final volume of 100 mL, the acid content will be approximately 10%. All standards should be made with a similar acid concentration. When using 50 mL digestion vessels you may reduce the volume of digestion reagents by 50% to keep concentrations the same. When doing so, the final volume will be 50 mL instead of 100 mL.

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According to AIHA Policies 2001- Revision Nov. 2000, one LCS, one matrix spike and one duplicate must be run every 20 samples. "The LCS shall be a solid matrix material with an established concentration obtained from a source independent of the instrument calibration and traceable to NIST or other similar reference material. Liquid spikes may not be used for preparing LCSs". Environmental Express does offer NIST traceable spiked GhostWipe® Wipes. The Item number is SC4252. The range is 175 to 200µg of Lead per wipe.

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## Adaptation of EPA Method 3060 for use with the Environmental Express® HotBlock® and StirBase™ Systems

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Revised 05.05

### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to EPA Method 3060A for use with the Environmental Express HotBlock® and StirBase. EPA Method 3060A is an alkaline digestion procedure for extracting hexavalent chromium [Cr(6+)] from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and similar waste materials. Use EPA Method 3060A for reference while following the sample preparation steps outlined below. The quantification of Cr6+ in Method 3060A digests should be performed using a suitable technique with appropriate accuracy and precision.

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### 2.0 Apparatus and Materials

- 2.1 HotBlock® for sample digestion – Item # SC100, SC154, SC150, or SC151
- 2.2 StirBase stirring device – Item # SC160
- 2.3 Polypropylene Digestion Vessels – Item # SC475 (or SC490 for use with the SC150 or SC151 HotBlocks®)
- 2.4 Ribbed Watch Glasses – Item # SC505 (or SC610 for use with the SC150 or SC151 HotBlocks®)
- 2.5 Reflux Caps – Item # SC506 (used as an alternative to the SC505 only)
- 2.6 Stir Bars – Item # SC168
- 2.7 FilterMate – Item # SC0407 (or appropriate FilterMate) for sample filtration if necessary
- 2.8 FlipMate – Item # SC0301 (or appropriate FlipMate) for sample filtration if necessary

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### 3.0 Procedure, Solid Sample Preparation

- 3.1 Place  $2.5 \pm 0.10$  g of the field-moist sample into a clean and labeled digestion vessel. The sample should have been mixed thoroughly before the aliquot is removed.
- 3.2 For the specific sample aliquot being spiked, the spike material should be added directly to the sample aliquot at this point.
- 3.3 Add  $50\text{mL} \pm 1\text{mL}$  of digestion solution to each sample using a graduated cylinder, and also add approximately 400 mg of magnesium chloride and 0.5mL of 1.0M phosphate buffer. For analytical techniques that can correct for oxidation/reduction of Cr, the addition of Mg(2+) is optional. Cover all samples with watch glasses or reflux caps.
- 3.4 Stir the samples continuously (unheated) for at least five minutes using the StirBase.
- 3.5 Heat the samples to 90 - 95°C, then maintain the samples at 90 - 95°C for at least 60 minutes with continuous stirring.
- 3.6 Gradually cool, with continued agitation, each vessel to room temperature.
- 3.7 Filter each sample using a 0.45µm FlipMate (SC0601).

- 3.8 Adjust the pH of the digestate accordingly to the method being used for analysis. If a precipitate forms, the sample should be filtered as in step 3.7.
- 3.9 Adjust the sample volume to 100mL with reagent water. Mix well. The sample digestates are now ready to be analyzed.

All QC samples, concentration limitations, interferences, and reagent specifications are addressed in depth in EPA method 3060A. Safety concerns are also part of the full method. Follow the instructions listed in EPA method 3060A. These steps should only be used as a guide to help improve the performance of your HotBlock® and StirBase.

### Accessories and Consumables for 50mL HotBlocks®



#### Polycarbonate Transfer Racks

Each sample transfer rack for the HotBlock® is designed to hold and handle eighteen SC475 50mL digestion vessels. Racks are made of polycarbonate for heat and acid resistance and fit over the HotBlock® to facilitate handling of samples and improve temperature and evaporation uniformity.

Polycarbonate Transfer Rack Each Item # SC200



#### Plastic Storage Racks

24-cup capacity plastic racks neatly hold the SC475 digestion cup. Made of polypropylene, racks accommodate any tube up to 30mm and are stackable for efficient storage.

Plastic Storage Rack Each Item # SC446



#### Urethane Foam Storage Racks

Low-cost storage system uses high density urethane foam for semi-permanent storage of digested samples. Each rack holds 25 capped SC475 digestion cups. Racks with cups may be stacked to save space. Overall size of rack is 10" x 10" x 1".

Urethane Foam Storage Racks Pack of 5 Item # SC700



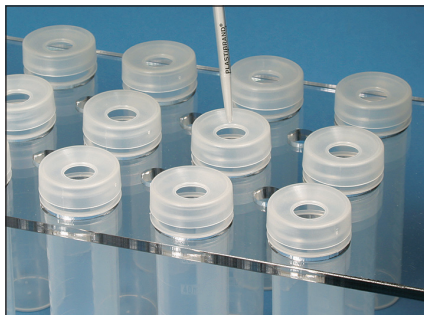
#### Temperature Probe

The temperature probe accessory allows the technician to monitor actual sample temperature in 50mL vessels. The digital thermometer is adjustable in PTFE holder to allow placement of thermometer tip just above bottom surface of digestion vessel. Thermometer has an LCD and can be set in either Fahrenheit or Celsius.

<i>Description</i>	<i>Quantity</i>	<i>Item #</i>
Temperature Probe (thermometer to 150°C) Fits SC475	Each	SC980
High Range Temperature Probe (thermometer to 250°C) Fits cups SC432, SC545, and SC475	Each	SC983







### Reflux Cap

Special cap has a 0.5" hole in its center to allow the addition of reagents while cap remains in place on the SC475 digestion vessel. The disposable reflux cap does not have to be removed during the digestion process. The cap is not ribbed, but fits over the top of the SC475 digestion vessel to maintain a centered position. Tests have shown equal reflux between SC506 reflux cap and SC505 watch glass.

Reflux Cap for SC475

Pack of 1000

Item # SC506

## Accessories and Consumables for 100mL HotBlocks®



### Polycarbonate Transfer Racks

This set of two polycarbonate racks was designed to fit the SC150 Hot Block. A 15-place rack paired with a 10-place rack give you 25 sample capacity. Racks fit the SC150 HotBlock® securely for safe, accurate digestions.

Polycarbonate Transfer Racks for SC150 HotBlock®

Set of 2

Item # SC225



### Wire Storage Rack for SC490s

Powder-coated wire rack shown above is for storing SC490 cups. Rack has an 18-cup capacity.

Wire Storage Rack

Each

Item # SC7150

### Temperature Probe

This temperature probe fits SC490 cups and allows the analyst to monitor actual temperature in a 100mL sample. The digital thermometer is adjustable in a PTFE holder to allow placement of the thermometer tip in desired position in cup. Thermometer has LCD display and may be read in either Fahrenheit or Celsius.

Temperature Probe (thermometer to 150°C) for SC490

Each

Item # SC981



### Disposable Polypropylene Digestion Vessels for use with SC150 HotBlock®

This 125mL, wide-mouth digestion vessel is supplied with a 48mm screw cap. The cap is lined with coextruded polyethylene foam for leak-proof performance. Made of homopolymer polypropylene, the SC490 digestion vessel is designed for use in the SC150 HotBlock®. Vessels are graduated at 10mL, 25mL and 50mL, 75mL and 100mL.

Disposable 125mL Digestion Cup

Pack of 225

Item # SC490

### Ribbed Watch Glass

52mm ribbed watch glass is designed for use with SC490, 125mL digestion vessel. The disposable polypropylene watch glass has a stem in its center to aid in handling.

Ribbed Watch Glass, 52mm

Pack of 500

Item # SC610

## Accessories and Consumables for 15mL HotBlocks®



### Polycarbonate Transfer Rack

These polycarbonate racks fit the SC196 HotBlock®. The racks hold our SC415 digestion vessels securely, assisting the analyst in performing safe, accurate digestions. Three 32-place racks give you 96-sample capacity.

Polycarbonate Transfer Racks for SC196 HotBlock®      Set of 3      Item # SC296



### Temperature Probe

This temperature probe fits SC415 cups and allows the analyst to monitor actual temperature in a 15mL sample. The digital thermometer is adjustable in a PTFE holder to allow placement of the thermometer tip in desired position in cup. Thermometer has LCD display and may be read in either Fahrenheit or Celsius.

Temperature Probe (thermometer to 150°C) for SC415      Each      Item # SC985

### Polypropylene Digestion Vessels for use with SC196 HotBlock®

These screw cap digestion vessels for 15mL samples, have a total volume of 18mL. Molded-in graduations are in 5mL increments up to 15mL.

Disposable 15mL Digestion Cups with Caps      Pack of 1,000      Item # SC415

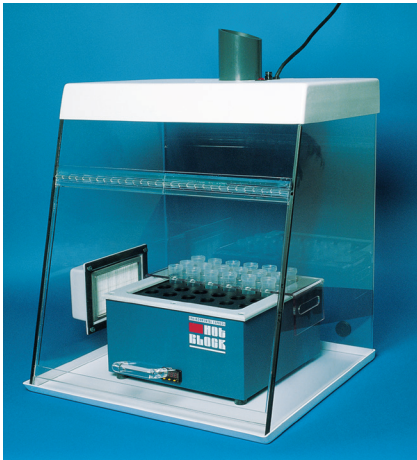


### Ribbed Watch Glass

19.5mm ribbed watch glass is designed for use with SC415, 15mL digestion vessel. The disposable polypropylene watch glass has a stem in its center to aid in handling.

Ribbed Watch Glass, 19.5mm      Pack of 1000      Item # SC417

## AirLite™ HEPA-Filtered Enclosure



<b>SC801:</b>	
Size:	25.5"h X 32"w X 24"d
Door Opening Size:	32"w x 17"h
Materials:	Acrylic walls and door, Kydex® top and bottom plates
Fan	117CFM, PVC Coated

This bench-top enclosure for the HotBlock® provides HEPA-filtered air for cleaner, more uniform digestions. The AirLite uses a quiet 117 CFM internal PVC coated fan that is easily accessible by the technician. The cabinet of the AirLite is constructed from PET-G for exceptional clarity. The door of the hood opens a full 180° to allow unrestricted access to the chamber. The

body of the enclosure rests in a thermoformed Kydex® bottom that traps spills and can easily be removed for cleaning.

The AirLite is designed to enhance the use of the HotBlock®, and can reduce sample contamination during the digestion process. The HEPA filter can be changed in minutes. The AirLite can be used anywhere a clean environment is required. It can be exhausted with standard 4" flexible duct to the outside or into existing ductwork. Spare 117CFM fan included.

<i>Description</i>	<i>Item #</i>
AirLite Enclosure; 120 VAC, 50/60 Hz	SC801
AirLite Enclosure; 240 VAC, 50/60 Hz	SC801-240
Replacement HEPA Filter	SC840
Replacement Exhaust Fan	SC830

## FlipMate® Filtration Assemblies



The FlipMate offers a quick and effective method of filtering up to 125mL of sample. The device accepts two threaded digestion cups (cups are sold separately) - one containing the sample to be filtered and an empty cup to receive the filtrate. The assembly is then “flipped” over and vacuum is applied. The sample is pulled through the filter into the empty cup. The filtered sample can then be capped and stored for later analysis. FlipMate is available with a variety of filters installed.

<i>Description</i>	<i>Sample Size</i>	<i>Filter Material</i>	<i>Porosity</i>	<i>Quantity</i>	<i>Item #</i>
FlipMate 50	50mL	PES Filter with Prefilter	0.45µm	100	SC0601
FlipMate 50	50mL	Glass Fiber	1.5µm	100	SC0602
FlipMate 100	100mL	PES Filter with Prefilter	0.45µm	50	SC0301
FlipMate 100	100mL	Glass Fiber	1.5µm	50	SC0302
FlipMate 100	100mL	PES with Prefilter	0.2µm	50	SC0303
FlipMate 100	100mL	Acid Washed Glass Fiber	0.7µm	50	SC0304
FlipMate 100	100mL	Acid Washed Filter	>10µm	50	SC0306

## FilterMate® Filtration Systems



Dirty or particulate-laden samples may require that the digested sample be filtered prior to analysis. This is especially true with sludge, wastewater and soil samples.

FilterMate Systems provide a convenient and inexpensive method of filtration that requires no transfer steps. The FilterMate fits directly into the digestion cup and is pushed through the liquid being filtered to the bottom of the cup with a detachable plunger. After the filtration is complete, the plunger is removed and discarded while the filter assembly remains in the bottom of the cup.

This arrangement does not interfere with storage or use of the cup in an autosampler. FilterMates are available with different choices of filters.

Membrane Type Membrane Characteristics Applications		Sample Size	Porosity	Lot Certified	Quantity	Item #
Membrane Type: PTFE-faced Polypropylene Characteristics: Highest dirt capacity Applications: Most laboratory applications		50mL	2µm	No	100	SC0401
Membrane Type: Acid-washed Glass Fiber Filter Characteristics: Porous Polyethylene Frit Applications: TCLP Extractions		50mL	0.7µm	No	100	SC0404
Membrane Type: PVDF with additional PTFE Prefilter Characteristics: Not for particulate-laden samples Applications: Dissolved metals only		50mL	0.45µm	No	100	SC0407
Membrane Type: PTFE Characteristics: High dirt capacity Applications: Suitable for trace level analysis		50mL	2µm	Yes	100	SC0408
Membrane Type: PVDF with additional PTFE Prefilter Characteristics: Low solids Applications: Dissolved metals only		50mL	0.45µm	Yes	100	SC0409
Membrane Type: Polyethylene Frit Characteristics: For use with 15mL digestion cups (SC415)		15mL	2µm	No	100	SC0421

### Cup Holder for FilterMate Filtrations

*This machined HDPE fixture adds stability when performing FilterMate filtrations. Digestion cup is placed into center hole of FilterMate Holder prior to beginning filtration. The holder secures the digestion cup as the FilterMate is depressed through the liquid sample.*

SC0510

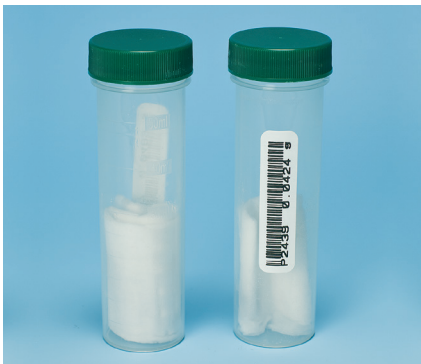
**1** Take a sample with GhostWipe.



**2** Digest as usual in acid solution.



**3** GhostWipe totally dissolves during digestion for easy analysis.



**GhostWipe® Wipes For Surface Sampling, Including Lead and Beryllium Testing**

This revolutionary product eases sample preparation and analysis of surface lead or surface dust. The GhostWipe is a sturdy wiping material moistened with DI water that holds together even on the roughest wiping surfaces. In the lab, the GhostWipe readily and completely dissolves during the digestion process. This feature provides more complete dispersion of analytes and more uniform recoveries.



In addition, there is no messy fibrous material to clog sample uptake capillary or nebulizer. GhostWipe wipes meet all ASTM Designation E 1792 specifications for sampling materials for lead in surface dust. Wipes are 15cm x 15cm and are supplied in individual sealed packets.

Description	Quantity	Item #
GhostWipe wipes	500	SC4210
GhostWipe wipes	1,000	SC4250
Dust Sampling Template, 10cm x 10cm working area	250	C1010
Dust Sampling Template, 12" x 12" working area	100	C1012

**Lead Wipe Standard in Screw-Cap, Rigid-walled Cup (SC475)**

The lead wipe standard is a Ghost Wipe spiked with in-situ dust. Using an NIST-traceable solid sample, known amounts of lead are placed on a wipe and each sample is individually labeled with the exact amount of lead the wipe contains. Each spike sample contains 175-250 micrograms of lead per wipe and comes with a certificate of analysis showing the Sample ID# and the starting concentration of the dust.

Lead Wipe Standard with 50mL Cup Box of 10 Item # SC4252