

# AutoBlock<sup>®</sup> Fill Filling Station

## Operation & Instruction Manual

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## Limited Warranty

The Environmental Express AutoBlock® Fill 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 AutoBlock® Fill, the customer may, at his or her option, receive a replacement unit or a full refund.

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 AutoBlock® Fill 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 AutoBlock® Fill 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 AutoBlock® Fill 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 AutoBlock® Fill system.

*Loaner AutoBlock® Fill system 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 product, AutoBlock® Fill Item Number ABF5000, is 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 the AutoBlock Fill conforms with the essential requirements of the applicable EC directives.



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**Product Information:**

Item #

Date of Purchase

AutoBlock® Fill Serial #

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



### AutoBlock® Fill Station Specifications:

Sample Capacity:	12 to 96 wells
Nominal Sample Size:	15 to 100 mL
Volume Resolution:	0.1 mL
Operating Temperature of Block:	Ambient to 300°C
Pump Accuracy:	±2% volume
Dimensions:	31"W x 27"H x 22"D
Electrical:	120/240 VAC, 10 A

<i>Description</i>	<i>Item #</i>
AutoBlock® Fill filling station with software, integrated pump, PTFE tubing, five 1-L glass reagent bottles, 100-mL waste vessel, and installation hardware	ABF5000

### Compatible HotBlock® Models

<i>Volume per Well</i>	<i>No. of Samples</i>	<i>Item #</i>
HotBlock systems		
15 mL	96	SC196, SC196-240
50 mL	36	SC100, SC100-240
50 mL	54	SC154, SC154-240
100 mL	25	SC150, SC150-240
100 mL	35	SC151, SC151-240
HotBlock Pro systems		
15 mL	96	SC189, SC189-240
50 mL	36	SC191, SC191-240
50 mL	54	SC181, SC181-240
100 mL	25	SC192, SC192-240
100 mL	35	SC182, SC182-240
HotBlock 200 systems		
15 mL	96	SC2015-96, SC2015-96V240
50 mL	36	SC2050-36, SC2050-36V240
50 mL	54	SC2050-54, SC2050-54V240
100 mL	25	SC2100-25, SC2100-25V240
100 mL	35	SC2100-35, SC2100-35V240

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## About Your Environmental Express AutoBlock® Fill Station

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AutoBlock® Fill provides a safe, efficient method of adding digestion reagents to water, wastewater, soil, and sludge samples for metals analysis. The station works with any size of HotBlock®, HotBlock® Pro, and HotBlock® 200 metals digestion systems to automate the most dangerous step of metals digestion—the reagent addition.

The station dispenses independently from up to five reagent bottles. No need to swap out reagent bottles when switching digestion methods—a five-way valve setup provides easy switching between reagents. The reagents are dispensed by an integrated Masterflex® peristaltic pump accurate to  $\pm 2\%$  volume (calibratable to any dispensing volume).

The AutoBlock® Fill can be programmed and controlled via the onboard touch screen or use the included software to connect directly to a computer. The station features preprogrammed EPA methods or the user can create customized programs.

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. The AutoBlock® Fill is designed to fit in a fume hood.

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## Definitions/Markings

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Each AutoBlock® Fill and/or HotBlock® product displays certain markings and symbols. All personnel working with the AutoBlock® Fill and/or HotBlock® 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**.

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## Installation of your AutoBlock® Fill Station

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1. The AutoBlock® Fill requires some assembly prior to operation. A video showing how to perform the assembly is located on the [AutoBlock® Fill product page](#).
2. The top section of foam packaging contains the horizontal brace, the HotBlock® rails, and the vertical brace. The bottom section of foam packaging contains the tower, base plate, XY arm assembly, and power cord and tubing. A bag of hardware to connect all the pieces together is also included.
3. Attach the tower to the base plate. Set the base plate on a secure flat surface with the cam lock lever on the left side. This will orient the base with the attached brace rail to the right, the wider track slot in the front, and the narrower track slot towards the back. The far-right side will have an outline of the tower with six screw holes. The tower should be oriented with the peristaltic pump to the front, the drain trench on the left, and the five-way valve on the right. Use six (6) #0.25-20 x 0.75 Phillips head screws. This step and the next one are best accomplished by laying the tower on its back and having the bottom panel facing you.
4. Attach the vertical brace to the bottom panel at the left rear corner. The rounded end will be up in the air and the flat end will be attached to the panel. The notched side will be at the bottom facing the rear. Use two (2) #0.25-20 x 0.75 Phillips head screws.
5. Remove the side panel from the right side of the tower. You will need a Phillips head screwdriver to remove the screws. Be sure to note which screws came from which holes. The shorter screws must go back in their specific holes to assure proper fit. Set the panel aside.

6. Attach the horizontal brace to the bottom panel, vertical brace, and tower. The open end of the horizontal brace will fit in the notch on the bottom of the vertical brace. The closed end of the horizontal brace will fit up against the side of the tower. Use five (5) #10-25 x 0.375 Phillips head screws to secure the bottom of the horizontal brace. Use three (3) #10-25 x 0.375 Phillips head screws with nuts to attach the horizontal brace to the tower. Make sure the nut is on the inside of the tower. Use two (2) #10-25 x 0.375 Phillips head screws to attach the horizontal brace to the vertical brace. Leave the middle spot empty for one (1) #0.25-20 x 0.75 Phillips head screw when attaching the XY arm assembly.
7. Remove the XY arm assembly from the box and set in place behind the unit.
8. Remove the back cover from the XY arm assembly. There are four (4) #10-24 x 0.375 Phillips head screws holding it in place; two on each side.
9. Attach the XY arm assembly to the frame. Use ten (10) #0.25-20 x 0.75 Phillips head screws to attach the XY arm assembly from the back. Use one (1) #0.25-20 x 0.25 screw inside the tower at the top.
10. Locate the two loose cable ends on the left side of the XY arm assembly and feed one through each of the two large access holes in the back of the tower. The X motor cable coming from the far-left side of the assembly should be inserted in the top hole. The Y motor cable runs inside along the length of the assembly and should be inserted in the bottom hole.
11. Attach the X and Y motor cables to the top two cables inside the tower; X to the top position and Y to the second position. The cables screw together and will be secure when you feel a physical click to lock them in place.
12. Attach the valve cables from the tower to the side panel. The cables are numbered near the connectors to match the correct ones together.
13. Replace the side panel and back cover to the appropriate parts of the unit. Make sure to place the shorter screws on the side panel in the appropriate positions. Snap the caps in place over the side panel screws.
14. Locate the wire support screws on the front left side of the tower and loosen them several full turns. There is no need to remove them from the brackets. Insert the long end of the support wire (thicker solid piece) from the spiral-wrapped assembly into the support brackets. Tighten the screws down to hold the wire in place.
15. Loosen the retaining screw on the side of the moving XY carriage, near the dispensing tip. Insert the other end of the support wire and tighten the screw to hold the wire securely in place.
16. Loosen the flange nut above the dispensing tip on the XY carriage. Insert the delivery tube into the flange nut and through the probe until it is flush with the end. Tighten the flange nut but do not clamp it down fully to pinch the tubing.
17. Lift the locking lever of the peristaltic pump on the front of the tower. Insert the peristaltic pump tubing assembly in the pump with the blue connector on the left side. Ensure that the pump tubing rests in between the clamps in the ends of the locking lever prior to securing it in place. The tubing on the right side of the pump tubing assembly attaches to the center port of the 5-way valve on the right side of the tower.
18. Attach the loose end of the delivery tubing to the left side of the pump tubing assembly. Insert the 100-mL vessel (# SC490) in the waste container holder underneath the peristaltic pump on the front of the tower.

19. For each digestion reagent bottle, you will need to cut a piece of tubing from the included coil. Use an empty bottle as a guide in determining the correct length. Insert one end of the tubing into the bottle cap and attach it to an empty bottle. Slide the tube far enough into the bottle so that the end of the tubing is near the bottom of the bottle. Do not put the tubing flush against the bottom of the container as it will not be able to draw liquid in an efficient manner. Run the tubing in a gradual bend to the 5-way valve. Make sure there are no kinks in the tubing to restrict flow. Use a ferrule and flangeless nut to secure the tubing into one of the numbered ports in the 5-way valve. Repeat for as many digestion reagents are desired.

**Note:** The bottle with DI water must always be connected to port #1.

20. Attach the 5-pin connector from the power block to the back-right corner of the AutoBlock® Fill.

## Installation of a HotBlock® in the AutoBlock® Fill Station

1. If using a new HotBlock®, remove the metal screws from the bottom plate of the unit. Attach the appropriate size rails to the bottom of the HotBlock® using the hex head screws included with the unit. An appropriate hex head driver is also included with the AutoBlock® Fill. The rails should run from side to side on the HotBlock®. If using a previously purchased HotBlock®, remove the PVC screws and rubber feet from the bottom of the unit. Attach the rails as described above.
2. Adjust the locking cam lever to accommodate the size of the HotBlock® you are using. Slide it to the right for a square block and to the left for a rectangular block. Tighten it down after adjustment.
3. Place the HotBlock® on the bottom plate of the AutoBlock® Fill with the controller facing front. Insert the power cord through the slot in the back panel of the AutoBlock® Fill.
4. Slide the HotBlock® towards the back panel of the unit until the rails slide in place in the appropriate slots on the base plate.
5. Slide the HotBlock® towards the tower until it is flush against the brace beside the tower.
6. Lock the cam lever in place against the side of the HotBlock®. It should be held securely in place and not move in any direction. The HotBlock® is now ready for positional calibration.

**Note:** See the “Using the AutoBlock® Fill Software on a PC” section for instructions on how to perform this calibration.

## Installation Requirements

If digestions are to be performed on a HotBlock® installed in the AutoBlock® Fill station, make sure to locate the entire system under a fume hood with a minimum face velocity of 100 fpm, and allow a minimum of 2” of space on all sides. The following environmental conditions should be observed:

- Ambient temperature range: 5–45°C
- Ambient relative humidity: 0–100%RH
- Altitude: sea level to 2500 meters

HotBlock® units are rated as **Pollution Degree 2 and Installation Category 2**.



## Electrical Requirements

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- Required Voltage 120/240 volts, ~50/60 Hz, Current – 10 A.
- Power should not vary greater than  $\pm 10\%$ . Connecting to the power supply must be done with the supplied power cord.
- For safety reasons, a separate power receptacle should be provided for any HotBlock® installed in the unit. Do not use extension cords or outlet adaptors. Make certain that power outlets are earth-grounded at the grounding pin.

## Potential Hazards

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

- The AutoBlock® Fill has moving parts which can pinch extremities or entangle loose hair or clothing.
- Use extreme caution when operating any attached HotBlock® digestion block. Plastic and graphite surfaces of the blocks may be too hot to safely touch with bare hands.
- All components contain 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 the AutoBlock® Fill and any attached HotBlock®. Do not use extension cords or outlet adaptors. Make certain each power outlet is earth-grounded at the grounding pin.
- Power requirements for individual blocks may be found on our website.
- Application of the wrong supply voltage can create a fire hazard and a potentially serious shock hazard, and could seriously damage the system. See specifications for individual components.
- Users should be aware of potential dangers from heating certain types of compounds. Such dangers may include the release of toxic or flammable gases or explosion.

## Maintenance

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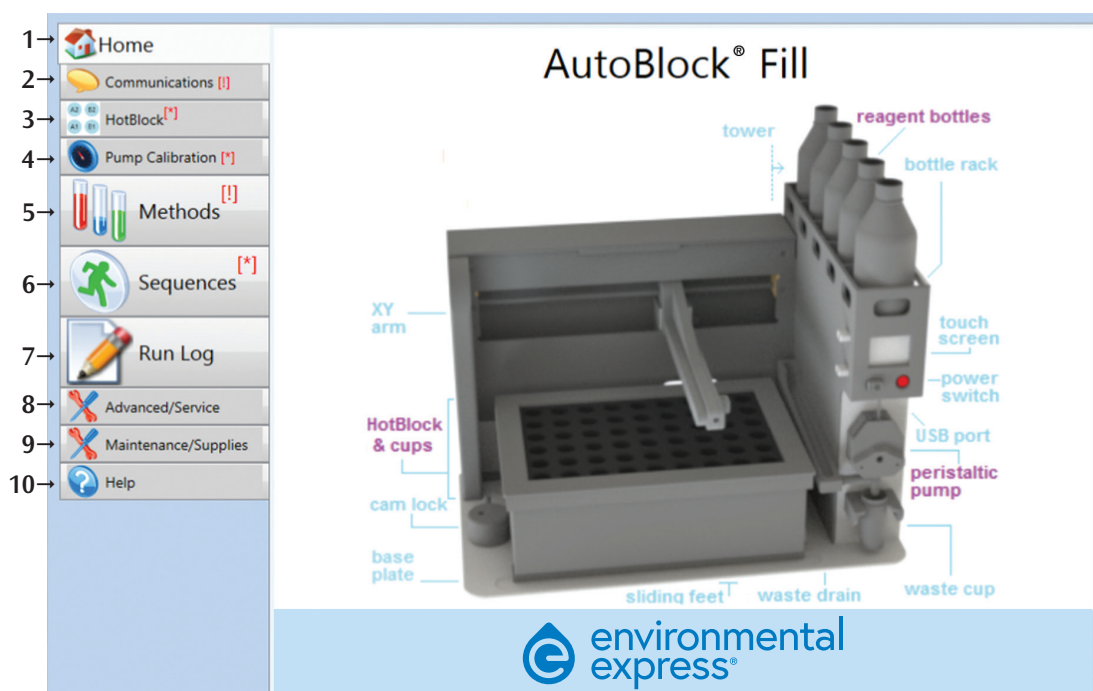
- Any service inquiries should be directed to the Environmental Express Technical Service Department at +1.800.745.8218 or +1.843.881.6560.
- After each use, clean exterior surfaces with a damp sponge to remove acid residue.
- For acid spills, sponge with a diluted solution of sodium bicarbonate followed 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 AutoBlock® Fill and/or HotBlock®.
- Avoid excessive spills; as liquid allowed to overflow into the AutoBlock® Fill and/or HotBlock® casing can seriously damage electronic components.
- Monitor the waste receptacle during use to ensure the rinses do not exceed the capacity of the 100-mL waste vessel.

## Operation Using the AutoBlock® Fill Software and Onboard Touch Screen

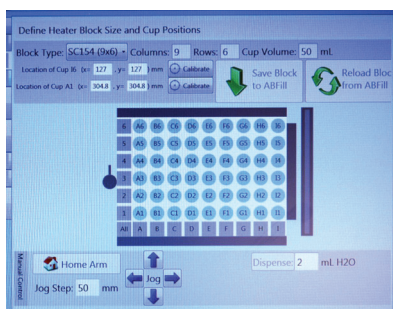
The AutoBlock® Fill can be run using either the software on a connected PC (for full control) or via the onboard touch screen (for routine, daily tasks). After initial calibrations and method development are completed, the only required function for connection to a PC is for regular pump volume calibration/verification. The software download and a video showing how to use the AutoBlock® Fill software are available on the [AutoBlock® Fill product page](#).

### Using the AutoBlock® Fill Software on a PC

The AutoBlock® Fill software consists of a primary working screen with tabs along the left side of the display. The tabs allow you to switch between functional screens and alert you to unsaved information or connection issues in the software.



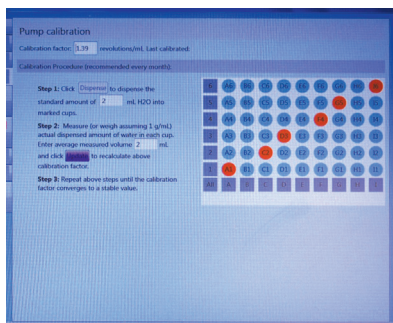
- 1. Home Screen** – The home screen provides an image of the AutoBlock® Fill and a functional link to the Environmental Express website.
- 2. Communications Screen** – This is where you will set up the serial ports for the USB connection between the PC and the instrument. The software should connect automatically once the USB cable is in place, however the first time connecting may require manual connection. Select the drop-down menu titled “Serial Port”. It defaults to “Autodetect” and will need to be changed to whichever port is populated in the menu. Then click the large button at the top labeled “Open Communications”. The software will then connect and indicate that communication has been established.



3. **HotBlock® Screen** – This screen is used to calibrate the position of the HotBlock® and check pump volumes. To calibrate the HotBlock® position, you must first choose the block type from the drop-down menu. Blocks are listed by the Environmental Express part number (e.g. SC154) as well as the well configuration (e.g. 9x6). If you are unsure of the part number, choose any option that has the correct well configuration. Once you make a selection, a map of the wells will appear onscreen. If you select the “Custom” option, you will need to specify the number of wells in each row and column. The cups’ positions are labeled in rows by number and columns by letter. You will calibrate on the front left corner cup (always labeled “A1”) and the back right corner cup.

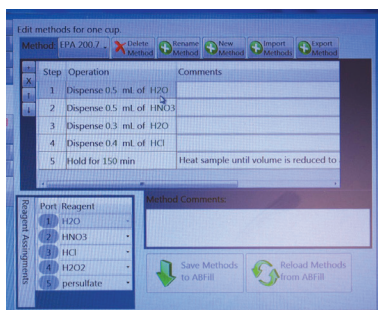
Click the “Calibrate” button on the row for “Location of Cup A1”. The button will turn red and the dispensing probe will move to a location close to where the A1 cup should be. Use the “Jog” arrows to position the arm so that the dispensing tip is centered above the A1 cup. The magnitude of the motion from the “Jog” arrows can be adjusted to smaller distances, down to 0.1 mm, by changing the field value. You can use the “Dispense” button to verify the position. If you anticipate using the disposable reflux caps, we recommend having one in position over the cup being calibrated.

Click the same “Calibrate” button as before. The button will change color again and the position will be saved to the unit. Repeat these steps for the location of the cup in the opposite corner. Once that is completed, click the “Save Block to ABFill” button. If you have not saved the block calibration to the unit, a red asterisk [\*] will appear on the tab to alert you of unsaved changes.



4. **Pump Calibration Screen** – This screen allows you to adjust the pump to account for changes in the pump tubing. The positions of the cups used to check the calibration are fixed but the volume used can be adjusted to something close to the standard amount of liquid being dispensed. Place a preweighed empty cup in each red shaded well as indicated on the map of the block. Change the amount listed in **Step 1** to the desired calibration amount; 2 mL is the default value. Click the “Dispense” button to begin the procedure. The unit will then dispense the indicated amount into each of the cups. Weigh the cups and determine the amount of water dispensed by the increase in mass. Average the values and enter in the field in **Step 2**. Click the “Update” button and the software will automatically calculate the Calibration Factor to update in the field at the top of the screen. Repeat the calibration steps until the value stabilizes within a margin of error acceptable for your laboratory.

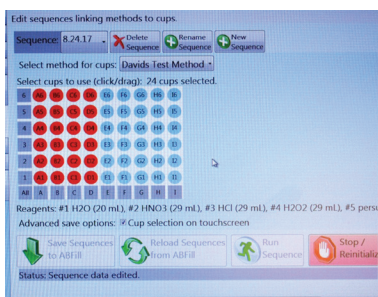
**Note:** Environmental Express recommends checking this value at least weekly and possibly more often depending on the use in your laboratory. We also recommend changing the peristaltic pump tubing at least monthly.



**5. Methods Screen** – The methods screen allows you to develop programs for dispensing of digestion reagents as well as configuration of the digestion reagents loaded onto the unit. Many of the common digestion methods from 40CFR are already preprogrammed in the software. You may edit an existing method or create a new method from scratch. The steps available in a method are Dispense, Hold, and Prompt. Dispense allows you to select a digestion reagent from those that are assigned and specify how much volume of it you wish to add to each cup. Hold will set a countdown timer for the time specified (in minutes) and wait until the timer expires before proceeding to the next step. Prompt will complete the step and then display a custom message that requires user acknowledgement prior to continuing.

The default reagents available for selection in the assignment screen are water (which must always be assigned to port #1), HCl, HNO<sub>2</sub>, H<sub>2</sub>O<sub>2</sub>, and H<sub>2</sub>SO<sub>4</sub>. If you wish to use a reagent besides one of these, select “None” for that port, highlight the text, and type in the reagent name you wish to use. That reagent will then be an option to choose in the “Dispense” command.

You must click the button “Save Methods to ABFill” in order to be able to run that method in a sequence. If you wish to save the method to the PC directory, you must click “Export Method”. If you have information on the Methods Screen that has not been saved, it will display a red exclamation mark [!] to alert you.



**6. Sequences Screen** – This screen will be the day-to-day operations screen. To set a sequence, you choose a method, choose which cups you wish to use, and give the sequence a name. If using the PC to control the unit, you can start the sequence by clicking the “Run Sequence Button”. If the touch-screen controller will be used to run the sequences, you must click “Save Sequences to ABFill” in order to have them available.

For routine operation, it is best to create a sequence using your most common digestion method with no cups selected. Make sure that the “Cup Selection” box is checked and save the sequence to the unit. This will allow you to fill in the cups needed for digestion with each batch that you run. You can save multiple sequences to the unit.

When selecting cups, you may choose individual wells by clicking each one, entire rows by selecting the appropriate letter, entire columns by selecting the appropriate number, or the entire block by selecting “All” at the bottom left corner of the block map.

The tab will show a red asterisk [\*] if there is unsaved information in the sequences screen.

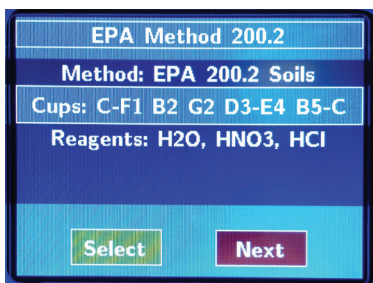
**7. Run Log Screen** – If you have checked the “Audit” box on a sequence, the unit will save basic run information for the sequence including start date/time, operator ID, Batch ID, sequence name, method used, selected wells, reagents assigned, and end time. The log must be loaded from the unit and then saved to the PC in order to retain the records.

**8. Advanced/Service Screen** – This screen should only be used under the direction of representatives from Environmental Express when troubleshooting the unit.

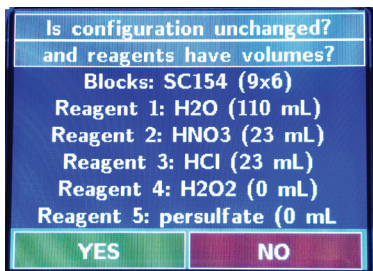
- 9. **Maintenance/Supplies Screen** – This screen contains part numbers and ordering information for common consumable items related to the AutoBlock® Fill.
- 10. **Help Screen** – Contains contact information for technical support, links to the manual, and links to support videos.

### Using the Onboard Touch-Screen Controller

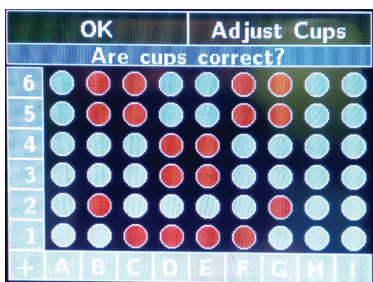
The onboard touch screen is intended for use in routine day-to-day operations of the AutoBlock® Fill. It is functional without a PC being connected to the unit. The touch screen saves a copy of the sequences and methods that have been downloaded to it from the PC software.



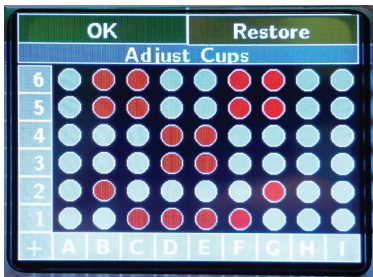
When using the touch screen, the user may cycle through all sequences that have been saved to the unit. When going through the available sequences the screen will display the method that has been assigned to that sequence, any cups that are selected, and the reagents being used.



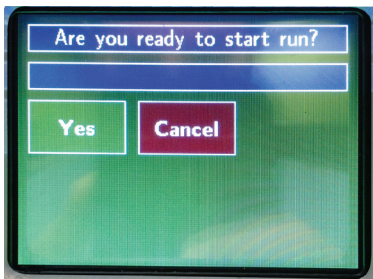
After selecting the desired sequence, it will display the block size and the volume needed for each digestion reagent in use for that sequence. The user should verify that the information is correct and that the appropriate volumes are available before proceeding.



The next screen shows a map of the block with the selected cups to be filled. The user will select "OK" or "Adjust Cups" depending on how the sequence is designed.



Adjusting the selected cups is done in a similar manner to using the PC software; individually, in rows, in columns, or the entire block. Press “OK” after the correct cups have been assigned.



It will then ask if you are ready to start the run. Press “Yes” to begin or “Cancel” to return to the main menu.



For a complete list of consumables and accessories, visit the Metals Digestions section of our website.

## Polypropylene Disposable Screw-cap Digestion Cups Eliminate Transfer Steps.

- Certified for low metals background and EPA approved† for digestions
- Volume certified

These carefully engineered digestion vessels provide premium performance in your HotBlock® systems. Cups are molded of clarified homopolymer polypropylene, providing a higher working temperature (to 130°C) and greater chemical resistance than commonly used co-polymer polypropylene.

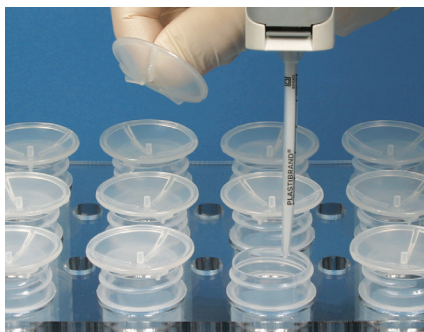
Accurate, molded-in graduations allow analysts to easily reproduce volumes to within 0.5%. Vessels serve as graduated cylinders, as well as storage containers. After digestion is complete, the sample can be brought to volume and capped for storage, eliminating time-consuming transfer steps. The result is a “one-cup” system that greatly reduces labor and costs associated with metals preparation.

Screw caps are available with polyethylene-faced foam liners for a leak-proof seal (green and white caps only) or as a plug-style closure (natural caps only).

Each box of cups comes with a Certificate of volume accuracy and a Certificate of Analysis for 68 trace metals—only metals-free materials come in contact with your sample. Both the Ultimate Cup (UC) and the Ultimate Clean Cup (UCC) vessels have been redesigned for increased durability, stability, and a tighter seal. In addition, the Ultimate Clean Cup comes in packaging to ensure ready-to-use arrival and is further trace-metal analysis certified to parts-per-billion and parts-per-trillion levels.

†Environmental Express digestion cups are approved for, but not limited to, EPA Methods 3005, 3010, 3050B, 200.2, 200.7, 200.8, 7470A, 7471A, 245.1, and 245.7.

Description	Total Capacity	Graduations	Quantity	Item #
15mL Cup (clear cap, no liner)	18mL	to 15mL	1000	SC415
100mL Cup (white cap, liner)	125mL	to 100mL	225	SC490
50mL Ultimate Cup (green cap, liner)	68mL	to 50mL	500	UC475-GN
50mL Ultimate Cup (white cap, liner)	68mL	to 50mL	500	UC475-W
50mL Ultimate Cup (natural cap, no liner)	68mL	to 50mL	500	UC475-NL
50mL Ultimate Clean Cup (natural cap, no liner)	68mL	To 50mL	150	UCC000-50



## Ribbed Watch Glasses allow partial reflux during digestion.

Ribbed watch glasses are available to fit all Environmental Express digestion vessels. Polypropylene construction is naturally free of contaminants. Watch glasses have a center stem to aid in handling and decrease the potential for contamination.

Description	For Digestion Cup	Quantity	Item #
19.5mm Watch Glass	15mL	1000	SC417
44mm Watch Glass	50mL	1000	SC505
52mm Watch Glass	100mL	500	SC610

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

Revised 12.13

### 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 # C6002, SC100, SC150\*, SC151\*, SC154, SC196, SC2015, SC2050, or SC2100\*
- 2.2 Polypropylene Digestion Vessels — 50mL, 100mL, or 15mL
- 2.3 Ribbed Watch Glasses or Reflux Caps, appropriately sized for vessel capacity
- 2.4 FilterMate — Item # SC0401 (or appropriate FilterMate® or FlipMate®) for sample filtration if necessary

*\*Note: For all procedures, when using the 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 polypropylene digestion 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.*

- 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® or FlipMate®) 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. Cover with a ribbed watch glass (SC505) or reflux cap (SC506), and place in the HotBlock at  $95^\circ\text{C}$ .
- 4.3 Heat sample for 30 minutes.
- 4.4 (Optional) Although step 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 recommended 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® or FlipMate®) 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.

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

Revised 12.13

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### 1.0 Scope and Application

- 1.1 The following procedures have been written as an aid to EPA digestion procedure 200.7 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.

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### 2.0 Apparatus and Materials

- 2.1 HotBlock for Metals Digestions — Item # C6002, SC100, SC150\*, SC151\*, SC154, SC196, SC2015, SC2050, or SC2100\*
- 2.2 Polypropylene Digestion Vessels — 50mL, 100mL, or 15mL
- 2.3 Ribbed Watch Glasses or Reflux Caps, appropriately sized for vessel capacity
- 2.4 FilterMate — Item # SC0401 (or appropriate FilterMate® or FlipMate®) for sample filtration if necessary

*\*Note: For all procedures, when using the 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, 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 polypropylene digestion 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.*

- 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® or FlipMate®) 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.*

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

- 4.1 For each digestion procedure, transfer  $0.5g \pm 0.01g$  of a dried sieved sample into the 50mL polypropylene vessel.
- 4.2 Add 2.0mL (1:1)  $HNO_3$  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.*

- 4.3 Allow to cool and bring to 50mL volume with DI water.
- 4.4 Filter with SC0401 (or appropriate FilterMate® or FlipMate®) 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 12.13

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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 # C6002, SC100, SC150\*, SC151\*, SC154, SC196, SC2015, SC2050, or SC2100\*
- 2.2 Polypropylene Digestion Vessels — 50mL, 100mL, or 15mL
- 2.3 Ribbed Watch Glasses or Reflux Caps, appropriately sized for vessel capacity
- 2.4 FilterMate — Item # SC0401 (or appropriate FilterMate® or FlipMate®) for sample filtration if necessary

*\*Note: For all procedures, when using the 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 polypropylene 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 polypropylene digestion 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.*
- 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® or FlipMate®) 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 50mL 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  $0.5g \pm 0.01g$  of a dried sieved sample into the 50mL polypropylene vessel.
- 5.2 Add 2.0mL (1:1) HNO<sub>3</sub> 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.*
- 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 12.13

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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 # C6002, SC100, SC150\*, SC151\*, SC154, SC196, SC2015, SC2050, or SC2100\*
- 2.2 Polypropylene Digestion Vessels — 50mL, 100mL, or 15mL
- 2.3 Ribbed Watch Glasses or Reflux Caps, appropriately sized for vessel capacity

*\*Note: For all procedures, when using the 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 50mL polypropylene 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  $95^{\circ}\text{C}$ .*

- 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 12.13

### 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 # C6002, SC100, SC150\*, SC151\*, SC154, SC2015, SC2050, or SC2100\*
- 2.2 Polypropylene Digestion Vessels — 50mL or 100mL
- 2.3 FilterMate — Item # SC0401 (or appropriate FilterMate® or FlipMate®) for sample filtration if necessary

*\*Note: For all procedures, when using the 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 polypropylene digestion 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.
- 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® or FlipMate®) 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 12.13

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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 # C6002, SC100, SC150\*, SC151\*, SC154, SC2015, SC2050, or SC2100\*
- 2.2 Polypropylene Digestion Vessels — 50mL or 100mL
- 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 or FlipMate®) for sample filtration if necessary

*\*Note: When using the 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 0.5-1-gram aliquot (or appropriate amount for your lab) to the 50mL 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.*
- 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. For 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® or FlipMate®) 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 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 12.13

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### 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.

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### 2.0 Apparatus and Materials

- 2.1 HotBlock for Metals Digestions — Item # C6002, SC100, SC150\*, SC151\*, SC154, SC2015, SC2050, or SC2100\*
- 2.2 Polypropylene Digestion Vessels — 50mL or 100mL
- 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® or FlipMate®) for sample filtration if necessary

*\*Note: When using the 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, Filter Preparation

- 3.1 Remove filter from cassette holder and fold into quarters taking care not to lose any sample.
- 3.2 Place in 50mL 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.*

- 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 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 with the GhostWipe® Wipes

Revised 12.13

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### Methodology Note:

The following procedure has been written as an aid for use with the Environmental Express HotBlock and strictly follows HUD (Housing and Urban Development) guidelines for Lead in Dust Wipes Appendix A-5.0 which references NIOSH 7082.

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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 from Environmental Express. The sample is heated in the presence of Nitric and Hydrochloric acids to dissolve the wipe and all lead compounds into solution. Analysis can be performed by flame atomic absorption (FLAA) or inductively coupled plasma technique (ICP).

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### 2.0 Apparatus and Materials

- 2.1 HotBlock for Metals Digestions — Item # C6002, SC100, SC150\*, SC151\*, SC154, SC2015, SC2050, or SC2100\*
- 2.2 GhostWipe wipes — Item # SC4210 or SC4250
- 2.3 Polypropylene Digestion Vessels — 50mL or 100mL
- 2.4 Ribbed Watch Glasses, Item # SC505 or Reflux Caps, Item # SC506.  
For use with 100mL vials (# SC490), use Ribbed Watch Glasses # SC610.

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- 2.5 FilterMate® or FlipMate® — optional if sample does not completely dissolve

*\*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 Reagents

- 3.1 Concentrated Reagent-grade HNO<sub>3</sub> (5mL repipet dispenser recommended)  
3.2 Concentrated Reagent-grade HCl (5mL repipet dispenser recommended)
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### 4.0 Procedure

- 4.1 Transfer the wipe to the 50mL digestion cup. It is recommended that the wipe is sent to the sampling area with a cup and wipe so no transfer steps are required. The wipe must be transported in a rigid-walled container according to the sampling procedure ASTM E1728.
- 4.2 Add 10mL DI Water, 2mL HNO<sub>3</sub> and 2mL HCl. Cover with a ribbed watch glass.
- 4.3 Heat the sample for 45 minutes at 95°C.  
*Note: When using a watch glass, adjust the set point temperature of the HotBlock so that a 50mL 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.*
- 4.4 Using the polycarbonate transfer racks, remove samples from the HotBlock and allow them to cool for approximately 10 minutes. Bring the sample to 50mL volume. If excess amounts of undigested material remain, filter the sample using a 2.0µm PTFE FilterMate®.
- 4.5 Cap and mix well.
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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 (Item # SC4252). The range is 175 to 200µg of Lead per wipe.

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

Revised 12.13

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### 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 # C6002, SC100, SC150\*, SC151\*, SC154, SC2015, SC2050, or SC2100\*
- 2.2 StirBase stirring device – Item # SC160
- 2.3 Polypropylene Digestion Vessels – 50mL or 100mL
- 2.4 Ribbed Watch Glasses – Item # SC505 for use with 50mL vessel (or SC610 for use with the 100mL vessel)
- 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 or FlipMate®) for sample filtration if necessary
- 2.8 FlipMate – Item # SC0301 (or appropriate FilterMate or 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.

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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™.