

HotBlock™ Pro Digestion Systems

Operation & Instruction Manual



ENVIRONMENTAL EXPRESS®
a Cole-Parmer company

800-343-5319 or 843-881-6560 www.environmentalexpress.com

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Limited Warranty

The Environmental Express HotBlock™ Pro 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™ Pro, the customer may, at his or her option, receive a replacement unit or a full refund. Operating the HotBlock™ Pro 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™ Pro 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™ Pro 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™ Pro 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™ Pro system.

Loaner HotBlock™ Pro 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 products, HotBlock™ Catalog 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, Inc. declares that all HotBlocks conform with the essential requirements of the applicable EC directives.

Signed: Nicole Truman

Nicole Truman, General Manager



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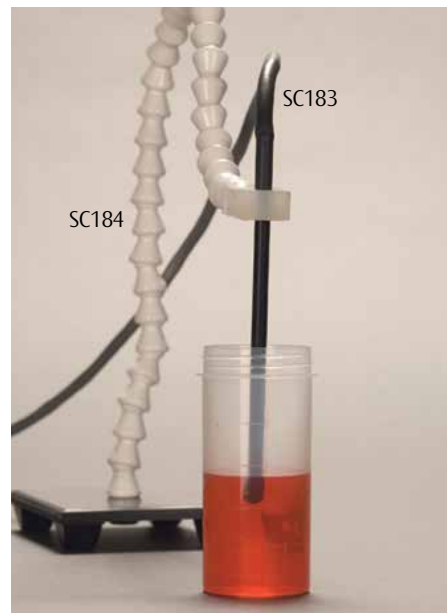
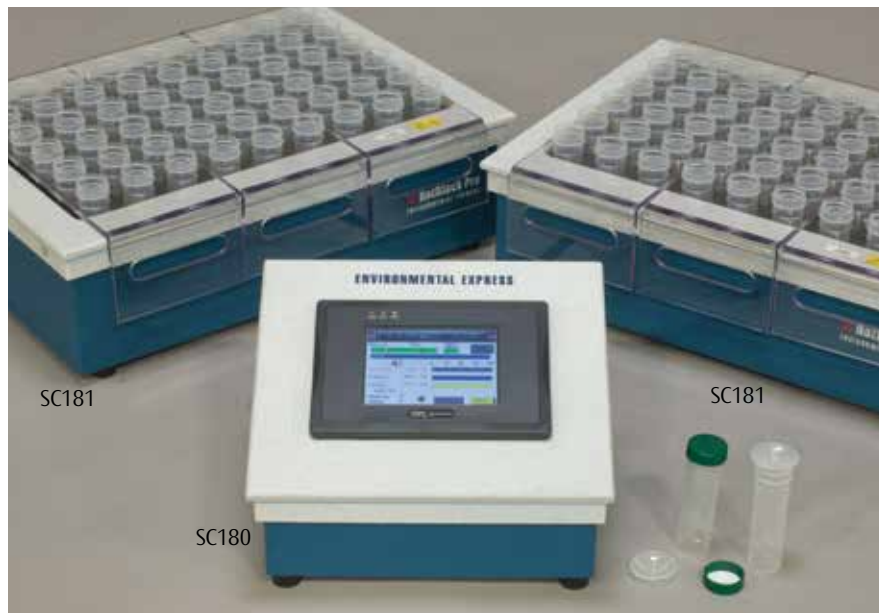
Product Information:

Item #

Date of Purchase

HotBlock™ Pro Serial #

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



The HotBlock Pro Series, offers programmable metals digestions with touch-screen remote control.

Above, the SC180 HotBlock Pro controller is shown with two 54-well HotBlock Pro blocks, SC181. The optional external thermocouple (shown right, in a specially designed holder) can be used to monitor and record sample temperatures.

The separate touch-screen controller is designed to work with one or two blocks. See individual block descriptions below. HotBlock Pro components are manufactured with the same rigorous specifications as our original HotBlocks. Polycarbonate transfer racks are included.

HotBlock Pro Heating Block Specifications:

| | |
|-------------------------|------------------------|
| Temperature Range: | to 150°C |
| Temperature Resolution: | 0.1°C |
| Temperature Uniformity: | ±0.2°C |
| Electrical: | 110V*, 9A per block |
| Internal Thermocouple: | Type K |
| External Thermocouple | SC183 |

All HotBlocks are also available in 240V.



| Description | Volume | Number of Samples | Digestion Cup Model | Racks Included | Footprint | Catalog # |
|---|--------|-------------------|---------------------|--------------------------|-----------------|-----------|
| HotBlock Pro Controller (networks any two HotBlock Pro blocks) | — | — | — | — | 9.25"W x 8.75"L | SC180 |
| 36-well HotBlock Pro Heating Block | 50mL | 36 | SC475 | 2, 18-place | 15"W x 15"L | SC191 |
| 54-well HotBlock Pro Heating Block | 50mL | 54 | SC475 | 3, 18-place | 15"W x 21.5"L | SC181 |
| 96-well HotBlock Pro Heating Block | 15mL | 96 | SC415 | 3, 32-place | 15"W x 21.5"L | SC189 |
| 25-well HotBlock Pro Heating Block | 100mL | 25 | SC490 | 1, 10-place; 1, 15-place | 15"W x 15"L | SC192 |
| 35-well HotBlock Pro Heating Block | 100mL | 35 | SC490 | 2, 10-place; 1, 15-place | 15"W x 21.5"L | SC182 |
| External Temperature Probe for HotBlock Pro | — | — | — | — | — | SC183 |
| Temperature Probe Holder | — | — | — | — | — | SC184 |

About Your Environmental Express HotBlock™ Pro Series

Environmental Express HotBlock Pro Series provides 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 using an external touch screen controller. 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™ Pro displays certain markings and symbols. All personnel working with the HotBlock™ Pro should have an understanding of the following symbols and definitions:

- | | | | | |
|---|---|---|---|--|
| <ul style="list-style-type: none"> • V = voltage • ~ = alternating current • Hz = frequency • A = amperes |  | <p>This symbol means Caution Hot Surface. The surface of the HotBlock™ may be too hot to safely touch with bare hands.</p> |  | <p>This symbol means Read and become familiar with instructions before operation of instrument.</p> |
|---|---|---|---|--|

Installation of your HotBlock™ Pro:

1. Plug the controller into an appropriate outlet. The HotBlock Pro must be plugged into a 20AMP receptacle.
2. The cord running from Block 1 plugs into the corresponding receptacle on the back of the controller. If using two blocks, attach the cord from Block 2 to the controller in the appropriate receptacle.
 - If external thermocouple probes (optional) are NOT being used, insert the two plugs included into the RTD1 and RTD2 receptacles on the back of the controller.
 - If external thermocouple probes ARE to be used, insert your probe plugs into these receptacles. RTD1 receptacle corresponds to Block 1.
3. The HotBlock or blocks should be located in a vented enclosure while the controller must remain outside the enclosure for ease of operation and to keep the controller removed from the corrosive digestion environment. See installation requirements below.

Failure to locate your HotBlock™ Pro controller outside the fume hood will void your product warranty.



Installation Requirements

Locate the HotBlock™ Pro 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: 5-45°C
- Ambient relative humidity: 0-100%RH
- Altitude: sea level to 2500 meters

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

The HotBlock Pro Controller **MUST** be located outside the fume hood. Failure to do so will void your product warranty.

Electrical Requirements

- Required Voltage 120 volts, ~60Hz, Current - 9A per block (all HotBlock™ Pro systems are also available in 240V with CE mark)
- The HotBlock™ Pro controller is supplied with a 20 amp male electrical plug. This safety feature requires that the unit is connected to a 20 amp receptacle.
- Power should not vary greater than $\pm 10\%$. Connecting to the power supply must be done with the supplied heat-resistant power cord or equivalent.
- For safety reasons, a separate power receptacle should be provided for HotBlock™ Pro system. Do not use extension cords or outlet adaptors. Make certain that power outlets are earth-grounded at the grounding pin.

Potential Hazards

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

- Use extreme caution when operating the HotBlock™ Pro digestion blocks. Plastic and graphite surfaces of the blocks may be too hot to safely touch with bare hands.
- The HotBlock™ Pro 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 each HotBlock™ Pro system. 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 HotBlock™ Pro 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

- Any service inquiries should be directed to Environmental Express Inc. 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™ Pro.
- Avoid excessive spills, as liquid allowed to overflow into the HotBlock™ Pro casing can seriously damage electronic components.

Easy, Intuitive Operation Using the HotBlock™ Pro Controller

The HotBlock Pro controller, catalog # SC180, provides operational control of either one or two HotBlock Pro digestion blocks simultaneously. The two blocks controlled may be the same style or any combination of two styles. The SC180 controller also works with just one digestion block and does not have to have two digestion blocks connected. The controller continually measures block temperature throughout the digestion process. The optional external thermocouple (SC183) may be used to measure sample temperature. These temperatures are continually displayed on the full color screen in the STATUS mode.

The full color, touch screen is easy to navigate. To move from screen to screen simply touch the name of the desired screen at the top of the touch pad with your finger (see screen illustrations below).

The color touch pad controller has four operating screens. They are the Status screen, Calibrate screen, Method screen and Timer screen.

Following are descriptions and illustrations of these screens and their functions.

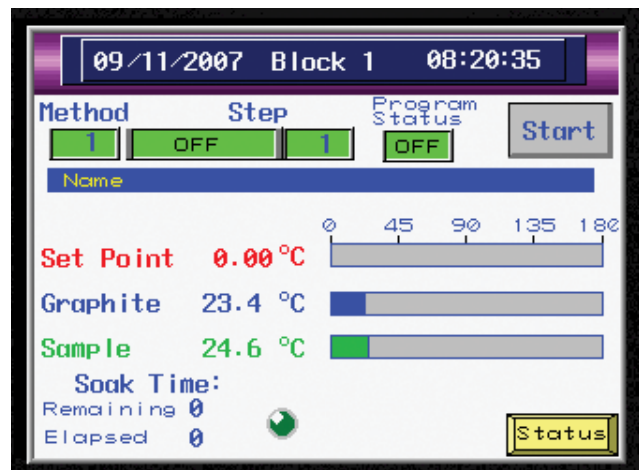
The Status Screen Continually Displays Current Operating Conditions.

The first screen to appear will give you the option of choosing BLOCK 1 or BLOCK 2. Choose the appropriate Block.

- Next, the STATUS screen will appear. When operating two blocks you can toggle between the two by pressing the strip at the top of the screen. BLOCK 1 STATUS is shown in the sample screen pictured bottom right. To view the status of two blocks simultaneously, press the STATUS key in the lower right corner of your screen. The STATUS screen continually displays current operating conditions of each digestion block throughout the digestion process. This screen allows the analyst easy access to essential information including the method in progress, block temperature, set point temperature and sample temperature. The top of the STATUS screen displays the method number and the step number of the method being performed for each block. Individual blocks may perform different methods simultaneously.
- The STATUS screen continually displays the actual temperature of each graphite block in operation as well as the set point temperature of each block. When an external thermocouple is used, the STATUS screen displays the sample temperature. When the external thermocouple is NOT in use, the Sample Temp field displays ***.
- Please note that the HotBlock Pro does not control the sample temperature directly, only the block temperature. The relation of the sample temperature to the block temperature is affected by variables including sample type, environmental conditions and use of watch glasses or similar devices.
- If there is a programmed SOAK time, the STATUS screen displays the time remaining in the SOAK period. The SOAK cycle will not begin until the temperature is within 3°C of SET POINT temperature. The flashing green light (bottom left) signals that the SOAK cycle has begun.



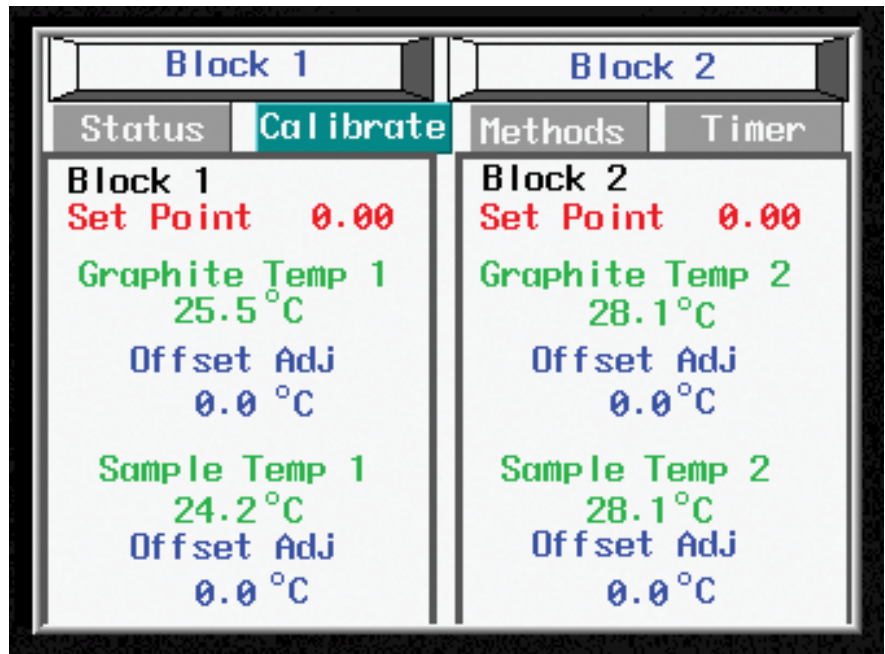
The first screen to appear when you turn on your block gives you the option of choosing Block 1 or Block 2. Choosing either block will take you to the STATUS screen.



When you choose the Block you want to monitor, The STATUS screen will appear. You have the option of monitoring either Block 1 or Block 2 or both Blocks simultaneously.

The Calibrate Screen Allows the Operator to Calibrate Block and Sample Temperatures.

- To calibrate the Graphite Block Temperature, run a method to achieve an appropriate temperature. For most labs 90°C should work. Allow the block temperature to stabilize. This may take 20-30 minutes.
- Measure the temperature of the block at several points with a surface thermometer or IR thermometer and take an average of the readings. If wells are filled with DI water please note that the temperature cannot be higher than 100°C. Do not use cups for this calibration.
- Once an average and stable temperature is achieved, press Offset Adj (Graphite Temp 1). A keypad will pop up prompting you to enter the appropriate temperature in degrees. Use your set point and your average measured temperature to calculate your Offset Adj. To decrease the temperature press (-). When you have correctly entered the appropriate temperature, press ENTER. You will automatically return to your CALIBRATION screen.



Calibration of blocks and samples is fast and easy using the CALIBRATE function of your controller.

Example: Set Point = 90°C
 Measured Temperature (Actual) = 91.2
 Offset Adj = (-) 1.2

- To calibrate the External Thermocouple, the use of a NIST traceable thermometer is recommended. First fill several digestion vessels with a representative sample or a reagent blank and place them in random wells in your block. Insert both the external thermocouple and the NIST thermometer in each sample. Calculate an average of these sample temperatures. Once an average and stable temperature is achieved, press Offset Adj (Sample Temp 1). A keypad will pop up prompting you to enter the appropriate temperature in degrees. Use your set point and your average measured temperature to calculate your Offset Adj. To decrease the temperature press (-). When you have correctly entered the appropriate temperature, press ENTER. You will automatically return to your CALIBRATION screen.

Example: Set Point = 90°C
 Measured Temperature (Actual) = 91.2
 Offset Adj = (-) 1.2

Note: Each external thermocouple-to-block connection is unique. An external thermocouple can only be calibrated to one port of one controller at a time. If the external temperature controller is used in a different port, then the calibration procedure must be repeated.

Digestion Methods Can Be Created and Stored Using the Method Configuration Screen.

Individual digestion methods can be created and stored by the HotBlock™ Pro. Each method can have up to six steps. Each step is a combination of temperature, ramp rate and soak time. The HotBlock™ Pro can store as many as 15 individual methods. Methods can be saved by number for future use.

- To program a method, select the METHODS tab on the controller. Select the Method Number you wish to program, then select View/Edit (Figure 1).
- This takes you to the screen (shown right, Figure 2) which displays six steps and programmable parameters for each step. Program each parameter individually from this screen.

To program your first step, select (Y) STEP ONE (see Figure 2). Program each parameter of Step one. As you press each parameter, a keypad will appear prompting you to enter the appropriate data.

- For example, press the TEMP field, type the appropriate temperature in the pop-up box (range 20-150°C) and press ENTER.
- Follow this procedure for RAMP (ramp must be 3.0°C/min).
- Select SOAK and type in the hours HH (99 maximum) followed by the minutes MM (59 maximum).

If no (0:00) SOAK TIME is set, the step is automatically inactive and the controller will skip to the next step in the method. If all subsequent steps have no SOAK TIME, the program is ended and the block will stop heating.

After entering all your parameters for Step 1, proceed to Step 2 of your method. Continue programming up to six steps for each method.

- If an automatic PAUSE is necessary before continuing to the next step, program the pause by changing NO to YES in the PAUSE column (see Figure 2).
- At the appropriate time in the method, the sequence will PAUSE. A pop-up box will show you that the method is paused.
- Press CONTINUE to continue to the next step.



Figure 1. Selecting Method from the main menu will take you to the screen shown above (Figure 1). Click on the Method Number you wish to program.

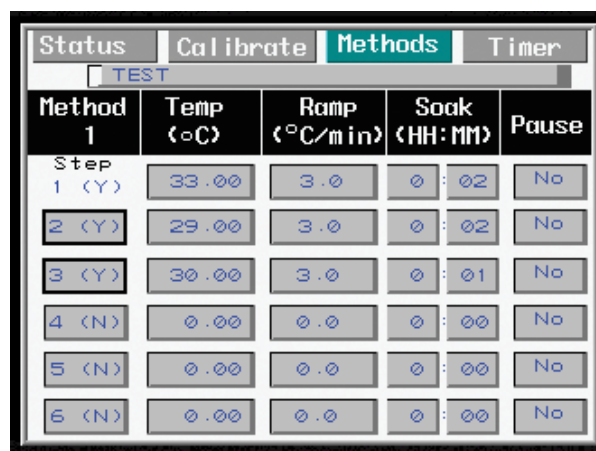
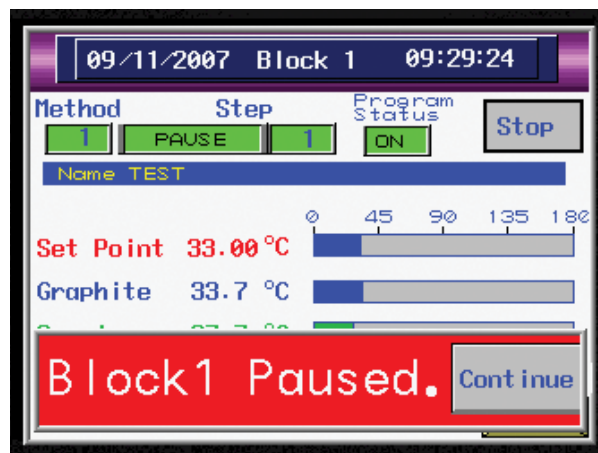


Figure 2. From this screen you will program each step of your method. Select Step 1, select the parameter you wish to set. Enter the appropriate data in the pop-up keypad and press ENTER. Continue to the next parameter.



A pop up box will show you that your method has PAUSED as programmed. Press CONTINUE to continue to the next step in your method.

Easily Name Your Programmed Methods.

Name your method AFTER programming all your steps and parameters as described on page 8. To name a method, go to the METHOD tab.

- Select the method number you wish to name, then click on VIEW/EDIT. This will take you to the screen shown here (Method Screen, Figure 1).
- Click on the GRAY BAR (indicated in Method Screen, Figure 1 by the red arrow) at the top of the screen under the TABS.
- When you click on this bar, a pop-up keypad will allow you to type in the method name.
- Then press ENTER.

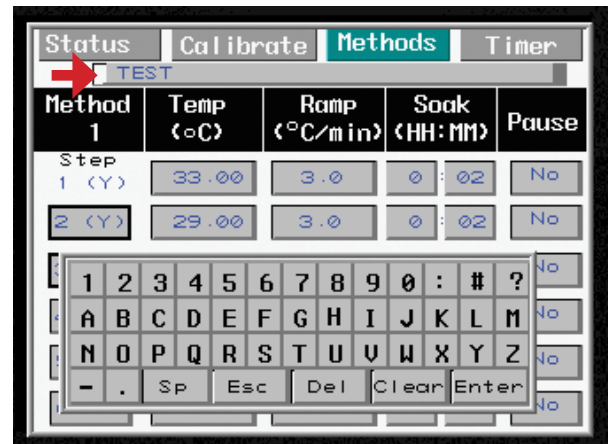
When you choose the METHOD NUMBER you wish to run, the METHOD NAME will appear in the BLUE BOX at the bottom of the METHOD screen (see METHOD screen, Figure 2).

Easily Initiate a Stored Method.

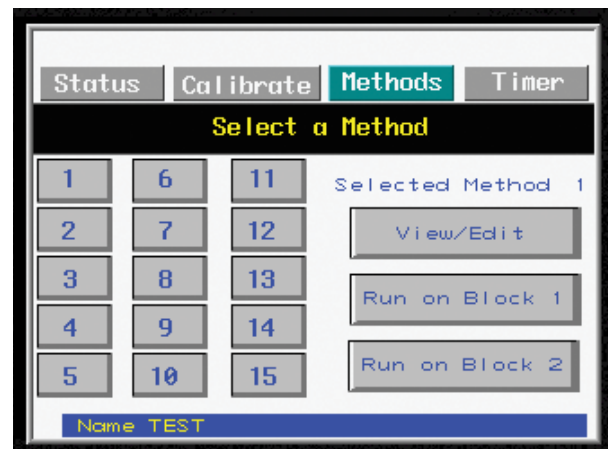
Name your method AFTER programming all your steps. To start a stored method go to the METHOD screen.

- Press the METHOD button you wish to use
- Then press RUN ON BLOCK 1 or RUN ON BLOCK 2 accordingly.

Please note that the SOAK timer will not appear on the STATUS screen or start counting down until the block temperature is within 3°C of SET POINT temperature.



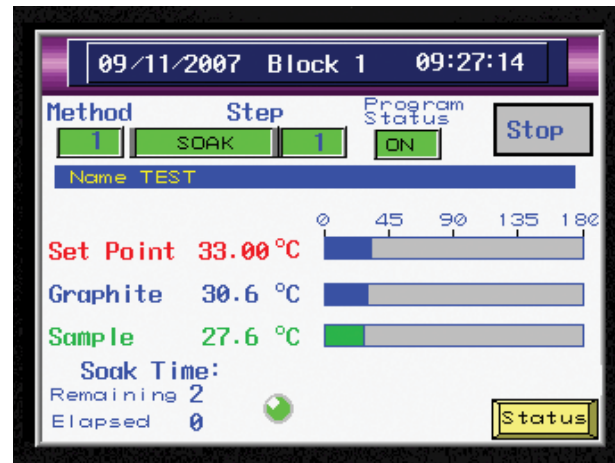
Method Screen, Figure 1. When you click on the gray bar, a pop-up keypad will enable you to type in the method name.



Method Screen, Figure 2. To initiate a stored method go to the METHOD screen and select the number of the method you wish to run. The method name will appear in the blue box at the bottom of the screen.

A Method Can Be Aborted at Any Time During the Digestion Process.

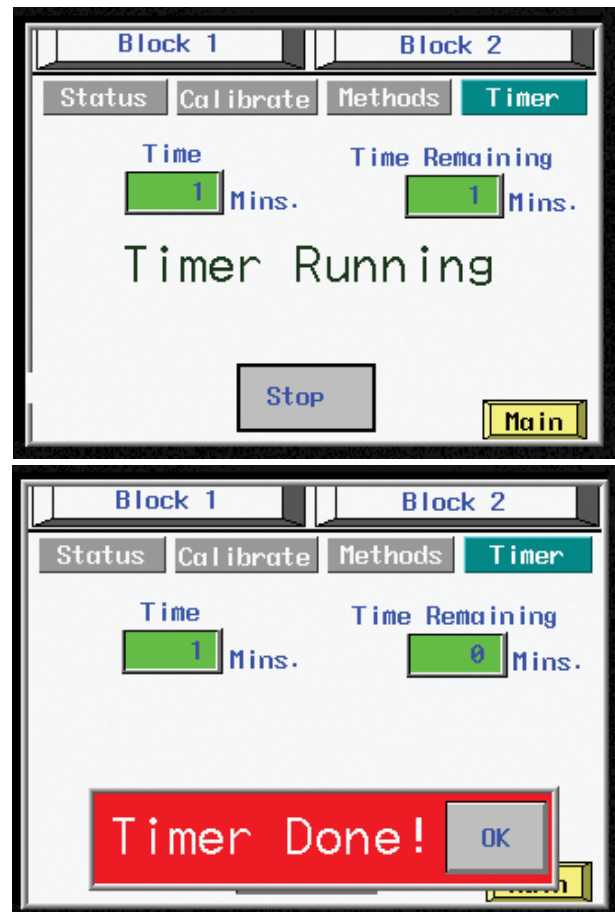
If you need to abort a digestion procedure during the program, press the STOP button on the STATUS screen.



Abort a method at any time from the STATUS screen.

The Timer Screen Can Be Used to Time Manual Digestions or Other Laboratory Functions.

The timer screen is an independent countdown timer that can be used to time manual digestions or other laboratory functions. Press the TIMER box, then press TIME. Program the time in minutes using the pop-up keypad and press ENTER. To start the timer press START. To cancel, press STOP. There is no pause function on the timer. A pop-up box will appear when the timed cycle is complete. TIME REMAINING continually shows progress of the timed cycle.



Monitor progress of a timed method on the TIMER screen. A pop up box alerts you when the timed cycle is complete.

Polypropylene Disposable Screw-cap Digestion Cups Eliminate Transfer Steps.

- Available in 15mL, 50mL and 100mL volumes
- Certified for low metals background
- Volume certified
- Lined caps ensure a leak-proof seal
- Accurate graduations eliminate the need for graduated cylinders and transfer steps

These carefully engineered digestion vessels provide premium performance in your HotBlock®, HotBlock™ Pro or AutoBlock systems. Cups are molded of clarified homopolymer polypropylene, providing a higher working temperature 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 lined with 0.040” polyethylene-faced foam liners for a leak-proof seal (except for SC415). Only the metals-free polyethylene liner comes into contact with the sample.

Each box of cups comes with a certification of volume accuracy, as well as a Metals Background Analysis Report.

Digestion Cup Specifications:

| Description | Total Capacity | Graduations | Quantity | Catalog # |
|--------------------------------|----------------|-------------|----------|-----------|
| 15mL Cup (clear cap, no liner) | 18mL | to 15mL | 1000 | SC415 |
| 50mL Cup (green cap, liner) | 68mL | to 50mL | 500 | SC475 |
| 50mL Cup (white cap, liner) | 68mL | to 50mL | 500 | SC480-W |
| 100mL Cup (white cap, liner) | 125mL | to 100mL | 225 | SC490 |

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 | Digestion Cup | Quantity | Catalog # |
|--------------------|---------------|----------|-----------|
| 19.5mm Watch Glass | SC415 | 1000 | SC417 |
| 44mm Watch Glass | SC475/SC480-W | 1000 | SC505 |
| 52mm Watch Glass | SC490 | 500 | SC610 |



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



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 — Model Number SC100, SC150*, SC154, SC181, SC182*, SC189, SC191, or SC192*
- 2.2 Polypropylene Digestion Vessels — Catalog # SC475 (50mL) or # SC490 (100mL) or # SC415 (15mL)
- 2.3 Ribbed Watch Glasses, Catalog # SC505 or Reflux Caps, Catalog # SC506 (for use with SC490 vials for HotBlock SC150, use Ribbed Watch Glasses, Catalog # SC610)
- 2.4 FilterMate — Catalog # 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 SC475 polypropylene vessel. For best results, weigh the sample directly in the vessel on a tared balance.
- 3.2 Add 1mL (1:1) HNO₃ 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.

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) 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°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% H_2O_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° less than the original set point for an additional 30 minutes. The reaction with the H_2O_2 raises the sample temperature. Boiling should not occur if the temperature of the HotBlock is lowered.
Note: H_2O_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.

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 12.13

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 — Model Number SC100, SC150*, SC154, SC181, SC182*, SC189, SC191, or SC192*
- 2.2 Polypropylene Digestion Vessels — Catalog # SC475 (50mL) or # SC490 (100mL) or # SC415 (15mL)
- 2.3 Ribbed Watch Glasses, Catalog # SC505 or Reflux Caps, Catalog # SC506 (for use with SC490 vials for HotBlock SC150, use Ribbed Watch Glasses, Catalog # SC610)
- 2.4 FilterMate — Catalog # 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, 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 HNO₃ 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 (catalog # 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 $0.5\text{g} \pm 0.01\text{g}$ of a dried sieved sample into the SC475 polypropylene vessel.
- 4.2 Add 2.0mL (1:1) HNO_3 and 5mL (1:4) HCl and swirl. Cover the vessel with a disposable watch glass (catalog # 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.

Adaptation for EPA Method 200.8, 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 200.8 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.

2.0 Apparatus and Materials

- 2.1 HotBlock for Metals Digestions — Model Number SC100, SC150*, SC154, SC181, SC182*, SC189, SC191, or SC192*
- 2.2 Polypropylene Digestion Vessels — Catalog # SC475 (50mL) or # SC490 (100mL) or # SC415 (15mL)
- 2.3 Ribbed Watch Glasses, Catalog # SC505 or Reflux Caps, Catalog # SC506 (for use with SC490 vials for HotBlock SC150, use Ribbed Watch Glasses, Catalog # SC610)
- 2.4 FilterMate — Catalog # 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 -- 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) HNO₃ 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.

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) HNO₃ 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 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.

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 SC475 polypropylene vessel.
- 5.2 Add 2.0mL (1:1) HNO₃ and 5mL (1:4) HCl and swirl. Cover the vessel with a disposable watch glass (catalog # 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.

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.

Adaptation of EPA Methods for Mercury Digestions 245.1, 7470, 7471 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 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.

2.0 Apparatus and Materials

- 2.1 HotBlock for Metals Digestions — Model Number SC100, SC150*, SC154, SC181, SC182*, SC189, SC191, or SC192*
- 2.2 Polypropylene Digestion Vessels — Catalog # SC475 (50mL) or # SC490 (100mL) or # SC415 (15mL)
- 2.3 Ribbed Watch Glasses, Catalog # SC505 or Reflux Caps, Catalog # SC506 (for use with SC490 vials for HotBlock SC150, use Ribbed Watch Glasses, Catalog # SC610)

**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 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 HNO₃.
- 3.4 Add 1.0mL of concentrated H₂SO₄.
- 3.5 Mix thoroughly. Add 3mL of 5% KMnO₄ 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.

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 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°C BEFORE placing the watch glass over the sample. Laboratory tests have proven that the addition of the ribbed watch glass will add 10°C to the sample temperature, bringing the sample temperature up to 95°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 — Model Numbers SC100, SC150*, or SC154
- 2.2 Polypropylene Digestion Vessels — Catalog # SC475 (or SC490 for use with HotBlock SC150)
- 2.3 FilterMate — Catalog # SC0401 (or appropriate FilterMate® or FlipMate®) 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 H₂SO₄ solution and swirl. (11 N H₂SO₄ — Slowly add 310mL concentrated H₂SO₄ 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.

Adaptation of EPA Method 3050B 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 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.

2.0 Apparatus and Materials

- 2.1 HotBlock for Metals Digestions — Model Numbers SC100, SC150*, or SC154
- 2.2 Polypropylene Digestion Vessels — Catalog # SC475 (or SC490 for use with HotBlock SC150)
- 2.3 Ribbed Watch Glasses, Catalog # SC505 or Reflux Caps, Catalog # SC506 (for use with SC490 vials for HotBlock SC150, use Ribbed Watch Glasses, Catalog # SC610)
- 2.4 FilterMate® — Catalog # SC0401 (or appropriate FilterMate or FlipMate®) 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, 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 SC475 polypropylene digestion vessel. For best results, weigh the sample directly in the vessel on a tared balance.
- 3.2 Add 5mL (1:1) HNO₃ + 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 HNO₃ 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% H₂O₂ 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 H₂O₂ (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.

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

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 — Model Numbers SC100, SC150*, or SC154
- 2.2 Polypropylene Digestion Vessels — Catalog # SC475 (or SC490 for use with HotBlock SC150)
- 2.3 Ribbed Watch Glasses, Catalog # SC505 or Reflux Caps, Catalog # SC506 (for use with SC490 vials for HotBlock SC150, use Ribbed Watch Glasses, Catalog # SC610)
- 2.4 FilterMate® — Catalog # SC0401 (or appropriate FilterMate® or FlipMate®) 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.
- 3.4 Remove samples from the HotBlock and cool for 5 minutes.
- 3.5 Remove watch glass and add 1.25mL HNO₃. 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.

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

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.

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).

2.0 Apparatus and Materials

- 2.1 HotBlock for Metals Digestions — Model Numbers SC100, SC150*, or SC154
- 2.2 GhostWipe wipes — Catalog # SC4210 or SC4250
- 2.3 Polypropylene Digestion Vessels — Catalog # SC475 (SC490 for HotBlock SC150)
- 2.4 Ribbed Watch Glasses, Catalog # SC505 or Reflux Caps, Catalog # SC506 (for use with SC490 vials for HotBlock SC150, use Ribbed Watch Glasses, Catalog # SC610)

- 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.*

3.0 Reagents

- 3.1 Concentrated Reagent-grade HNO₃ (5mL repipet dispenser recommended)
3.2 Concentrated Reagent-grade HCl (5mL repipet dispenser recommended)

4.0 Procedure

- 4.1 Transfer the wipe to the SC475 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 HNO₃ 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.

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 (Catalog # SC4252). The range is 175 to 200µg of Lead per wipe.

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

Revised 12.13

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.

2.0 Apparatus and Materials

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

3.0 Procedure, Solid Sample Preparation

- 3.1 Place 2.5 ± 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™.