

# SimpleDist® Micro Heating Block

## Operating & Instruction Manual

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Call 800-343-5319 or 843-881-6560 [www.environmentalexpress.com](http://www.environmentalexpress.com)  
2345 A Charleston Regional Pkwy • Charleston, SC 29492

**Product Information:**

Item #

Date of Purchase

SimpleDist® Micro Heating Block Serial #

Please record the serial # of your SimpleDist® Micro Heating Block here for easy reference. Serial # is located on the back of your Heating Block.

## Limited Warranty

The SimpleDist® Micro Heating Block from Environmental Express, Inc. is guaranteed to heat, hold temperature, and adequately perform specified laboratory distillations 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.

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 SimpleDist® Micro Heating Block should fail to operate as warranted within the warranty period (one year from date of shipment), Environmental Express, Inc. 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, Inc. 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 SimpleDist® Micro Heating Block 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 SimpleDist® Micro Heating Block fails after the warranty period has lapsed, the repair procedure is as follows:

First, notify an Environmental Express, Inc. customer 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, Inc. 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 SimpleDist® Micro Heating Block.



## Safety & Hazard Information

- **The SimpleDist® Micro Heating Block should only be operated by properly trained personnel using standard laboratory safety practices.**
- Review Safety Data Sheets for all materials used or generated during the operation of the SimpleDist® Micro heating block.
- It is highly recommended that the SimpleDist® Micro heating block be set up and operated in a chemical fume hood with a face velocity of not less than 100 CFM.
- Wear appropriate Personal Protective Equipment (PPE) suitable for use with caustic and corrosive materials.
- Consult your in-house electrician to be certain the SimpleDist® Micro System power cord is properly grounded.
- The SimpleDist® Micro Heating Block contains electrical circuits and devices and components operating at dangerous voltages. Contact with these circuits, devices and components can cause serious injury or painful electric shock.
- Proper grounding is essential to avoid a potentially serious electric shock hazard. Ensure that there is an internal ground connection between the metal base of the system and the 3-pin, earth-grounded receptacle.
- For safety reasons a separate power outlet receptacle should be provided for each unit in the system. Do not use extension cords or outlet adaptors. Make certain each power outlet is earth-grounded at the grounding pin.
- Application of the wrong supply voltage can create a fire hazard and a potentially serious shock hazard, and could seriously damage the SimpleDist® Micro Heating Block system.
- Do not operate the SimpleDist® Micro heating block in the vicinity of combustible material.
- Users should be aware of potential dangers from heating certain types of compounds. Such dangers may include explosion or the release of toxic or flammable gases.
- **Avoid breathing any vapors that may come off of the SimpleDist® Micro heating block; they may be harmful or fatal.**
- ⚠ Use extreme caution when operating the SimpleDist® Micro Heating Block. During operation the surfaces around the heater assembly will get HOT. Do not touch the outer surface. Plastic and graphite surfaces of the SimpleDist® Micro Heating Block may be too hot to safely touch with bare hands.
- Do not attempt to operate the SimpleDist® Micro heating block over 150°C.
- ⚠ Do not move the SimpleDist® Micro heating block while hot.
- The power should be kept plugged into its outlet until the unit has cooled down.
- Unplug the SimpleDist® Micro heating block from the outlet prior to cleaning exterior surfaces. Wipe with damp sponge or towel after each use, first with mild sodium bicarbonate or similar solution followed by DI or distilled water. Avoid solutions on or near the controls.
- If boil over does occur during operation of the SimpleDist® Micro heating block, immediately wipe the system down with neutralizing solution, such as a mild solution of sodium bicarbonate.
- Always lift the SimpleDist® Micro Heating Block from the bottom of the unit.

**Note:** The above list contains some basic recommendations and safety precautions. By no measure should this list be considered complete. More rigorous enhanced precautions may be necessary while operating this equipment. Please consult your Safety Manager and Safety Data Sheets (SDS) prior to operating this equipment. Contact Environmental Express, Inc. if there are any questions. User assumes all liability for damages arising from the operation of this equipment.

## HotBlock® Declaration of Conformity

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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, IEC 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

**Note:** The SimpleDist® Micro Heating Block is manufactured using the HotBlock® technology  
and circuitry.

## Information and Markings

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

Each SimpleDist® Micro Heating Block displays certain markings and symbols. All personnel  
working with the heating block should have an understanding of the following symbols and  
definitions:

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



This symbol means **Caution hot surface**. All surfaces of the SimpleDist® Micro Heating Block may be too hot to safely touch with bare hands.



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

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## Unpacking

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1. Remove the SimpleDist® Micro Heating Block from the shipping container by lifting from the bottom of the block. The lid should not be used for lifting.
2. Your SimpleDist® Micro Heating Block is shipped with metal screws securing the bottom panel. The metal screws must be removed before operating your SimpleDist® Micro Heating Block. Remove the metal screws and replace them with the PVC screws and rubber feet included with your shipment. The corrosion-resistant PVC screws and rubber feet are designed to secure the bottom plate.  
**Important:** Do not overtighten the PVC screws!
3. Disposable distillation tubes are not included with the SimpleDist® Micro Heating Block. Order tubes separately on page 21.
4. Save original packaging material in a dry area for use if unit needs to be returned for service. Refer to warranty policy on page 2.



## Installation Requirements

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

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

This product is rated as **Pollution Degree 2** and **Installation Category 2**.

## Electrical Requirements

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Required Voltage: 120 volts, ~60 Hz, 15 A  
(Also available in 240 VAC)

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

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

See individual specifications for each SimpleDist® Micro Heating Block model.

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## Temperature Settings

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The pre-set factory “set point” temperature of your SimpleDist® Micro Heating Block is 106°C. Factory tests have shown that this temperature is “sea level safe.” Liquids in uncovered polypropylene tubes should not boil at this setting. Please note that the set point of the block is not the same as the temperature of the liquids being distilled. The block temperature should be optimized for the specific distillation. The temperature of liquid contents of the distillation micro tubes will vary according to the material being digested, the number of samples being digested, and the air movement of the distillation area.

**Note:** *The maximum sample temperature tolerance for polypropylene vessels is 130°C. Remember that the temperature display (current block temperature) is not the temperature of the sample. Sample temperature will usually be 5 to 15° less than the display temperature.*

 **Note:** *Do not attempt to operate the SimpleDist® Micro heating block over 150°C.*

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## Adjusting the Temperature Settings

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Plug the SimpleDist® Micro Heating Block into an approved receptacle. Turn the heating block on by pressing the button on the back of the unit. Wait until the display shows the current block temperature (in red) and the set point temperature (in green).

Press and hold or tap the  $\Delta$  or  $\nabla$  key. The display will show the set point temperature on the right in green. The adjustment is from ambient to 150°C in increments of 0.1°C. There is no need to press the green (advance) or  $\infty$  button.

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## Safe-Sample™ Temperature Protection

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

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

Notwithstanding the alarm system feature, Environmental Express strongly recommends that the product not be left unattended.

 If the alarm sounds, see the SimpleDist® Micro Heating Block troubleshooting guide of your manual on pages 18–20.

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## Total Cyanide Distillation with the SimpleDist® Micro Heating Block

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### 1.0 Scope and Application

- 1.1 This method follows US EPA method number 335.4 titled *Determination of Total Cyanide by Semi-Automated Colorimetry*. It is applicable for the determination of total cyanide in drinking, ground, surface, and saline waters, domestic and industrial wastes, and soils.
- 1.2 The standard range is typically 5 to 500 µg/L. Lower detection limits can be achieved by using a longer path length flow cell in the analysis step when using an automated continuous flow analyzer.

### 2.0 Summary of Method

- 2.1 The cyanide as HCN is released from metal-cyanide complexes by means of an acidic manual reflux-distillation whereby the HCN gas that is formed is separated from the sample matrix and absorbed in a dilute solution of sodium hydroxide. The distillate can be analyzed for cyanide by semi-automated colorimetry, manual colorimetry, titrimetric, or ion-selective electrode.
- 2.2 Reduced volume versions of this method that use the same reagents and molar ratios as in the original method are acceptable provided they meet the quality control and performance requirements stated in the method.

### 3.0 Interferences

There are several known interferences with this method. A few of these are:

Aldehydes	Thiocyanates
Nitrate-nitrite	Thiosulfates
Chlorine	Sulfides

Some of these interferences are reduced or eliminated by the distillation process. The addition of magnesium chloride, which acts as a catalyst, will promote the breakdown of refractory iron-cyanide complexes.

For all other pretreatment procedures refer to the US EPA method number 335.4 and/or SW846 9010C.

### 4.0 Chemicals Required: Distillation Only

- 4.1 Sodium hydroxide solution (0.95 M) - Add approximately 500 mL of reagent water to a 1000 mL volumetric flask. Add 38 g sodium hydroxide (NaOH) pellets and stir to mix. **Caution:** This reaction is exothermic and may release fumes. Allow the solution to cool to ambient temperature and fill to the 1000 mL mark with reagent water.
- 4.2 Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), 36N (Concentrated)
- 4.3 Magnesium chloride hexahydrate (MgCl<sub>2</sub>•6H<sub>2</sub>O)

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## Total Cyanide Distillation with the SimpleDist® Micro Heating Block

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- 4.4 Sulfuric acid/magnesium chloride solution - Add 277 mL of reagent water to a 500 mL beaker. Add 80.5 g  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  (4.3) and stir to completely dissolve. Slowly add 189 mL of sulfuric acid (4.2) while stirring. This should be done in a hood as toxic fumes may be released. Once the solution is cool you should have 500 mL of total volume.
- 4.5 Potassium cyanide
- 4.6 Potassium hydroxide
- 4.7 Reagent water ASTM Type II or equivalent



**Note:** The toxicity for each of the reagents used in this procedure are not fully documented. Treat each chemical as a potential health hazard and limit exposure. Exercise good laboratory technique with an emphasis on safety.

### 5.0 Procedure (Note: Standards must be distilled in a like manner as the samples for this method)

- 5.1 Set the temperature of the Heating block to 120°C and allow the heat to stabilize. This should take approximately 30 minutes. If all reagents are ready to use this should be sufficient time to set up and prepare a full batch of 20 samples and all necessary QC.
- 5.2 Place one collector tube for each sample, standard, QC, or other aliquot needing to be distilled in a test tube rack with the “Measure” end up. Add 1.59 mL of 0.95 M NaOH solution (4.1) to each tube. **Note:** After final distillate dilution this will give a concentration of 0.25 M NaOH. The initial concentration may be adjusted to provide alternate final concentrations as needed by the analytical method of choice. Cap the tubes with one membrane and cap. Ensure that the membrane is not torn and shows a visible edge all the way around the tube.
- 5.3 Place one sample tube for each sample, standard, QC, or other aliquot needing to be distilled in a test tube rack and add 6.0 mL of sample/standard to each tube. The tubes may be labeled with permanent ink to aid in sample identification. Do not add any label to the tube as this will interfere with the fit in the block as well as heat transfer during the distillation process. If solid samples are being distilled add 0.5 to 1.0 g of the sample to the tube along with approximately 5 mL of reagent water. Samples high in organic content such as sludge should tend toward the lower sample mass while samples that are relatively free of organic material can use the higher end. The total amount of cyanide present in the tube should be less than 600 µg.
- 5.4 Add 0.75 mL of the sulfuric acid/magnesium chloride solution (Step 4.4) to the first sample tube. Immediately place the ‘Distill’ end of a filled and capped collector tube (Step 5.2) over the open end of the sample tube. Press the two pieces together to form a rough fit. You will not be able to hand press a tight seal.
- 5.5 Place the distillation unit in the tube press. Hold the collection tube by the break joint to give support during the sealing procedure. Firmly press the two pieces together with one motion. The collection tube should be flush with the ring around the edge of the sample tube. If the pieces are at a visible angle the assembly will not fit all the way in the block.
- 5.6 Repeat Steps 5.4 and 5.5 for all prepared sample and collection tubes.

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## Total Cyanide Distillation with the SimpleDist® Micro Heating Block

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- 5.7 Place each assembled and pressed distillation unit in the preheated block. Ensure that the unit is completely inserted in the block by having the ring on the “Distill” side of the tube flush against the surface of the block.



*Note: Heat resistant gloves may be necessary during this stage.*

- 5.8 After 30 minutes of distillation time, remove the first distillation unit from the block.



*Note: Heat and chemical resistant gloves are needed for Steps 5.8 and 5.9.*

- 5.9 Immediately after removing the unit from the block, hold it over a chemical disposal container and pull the sample tube off of the collection tube. This is best done by twisting the two pieces in opposite directions while simultaneously pulling them apart. Rocking the sample tube from side to side may not separate the pieces in time.

**The sample tube must be removed with 4 seconds of the unit being removed from the block.** Otherwise the distillate could suck back into the sample tube, causing the sample to be lost.

- 5.10 Drop the sample tube and any remaining liquid into the disposal container. Place the collection tube with the ‘Measure’ end (the capped end) down in a test tube rack. Repeat Steps 5.8 through 5.10 for all distilled tubes.
- 5.11 Allow the tubes to cool for 10 to 15 minutes. You will notice fine condensate begin to appear on the inside walls of the collection tube.
- 5.12 After the tubes have cooled to ambient temperature, use the trapping solution inside the tube to rinse the tube walls and collect all the condensate. This can be best accomplished by holding the tube horizontally and then tilting the ‘Distill’ end a few degrees downward. Slowly roll the tube while tilting it back to level and then the ‘Measure’ end down. You may need to gently tap the tube to knock down any drops that cling to the edges.
- 5.13 With the ‘Measure’ end down snap the tube at the break joint. The ‘Distill’ end may now be discarded.
- 5.14 Use reagent water to dilute the distillate up to the 6 mL line on the ‘Measure’ end. Gently swirl the solution to mix.
- 5.15 Due to limited sample volume, the analysis is best suited to discrete or flow injection analysis. Plug the end with a stopper or cover with Parafilm® if the analysis will not be performed immediately.
- 5.16 After use, clean exterior surfaces of heating block with a damp sponge. For acid spills, sponge with a diluted solution of sodium bicarbonate followed by distilled water. For caustic spills use a dilute solution of acetic acid followed by distilled water.

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## Ammonia Nitrogen Distillation with the SimpleDist® Micro Heating Block

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### 1.0 Scope and Application

- 1.1 This method follows both Standard Methods 4500-NH<sub>3</sub> titled *Nitrogen (Ammonia)* and US EPA Method 350.1 titled *Determination of Ammonia Nitrogen by Semi-Automated Colorimetry*. These methods are applicable to the determination of ammonia nitrogen in drinking, ground, surface, and saline waters, domestic and industrial wastes, and soils.
- 1.2 The standard applicable range is 0.01 to 2.0 mg/L NH<sub>3</sub> as N. Higher concentrations can be determined by sample dilution.

### 2.0 Summary of Method

- 2.1 A sample is buffered at pH 9.5 with borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds. It is then distilled into one of two catch solutions. Boric acid is used when nesslerization or titration are used for analysis or sulfuric acid when the phenate method or ion-selective electrode method are used for analysis. The distillate is then analyzed by one of the methods listed above.
- 2.2 Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method.
- 2.3 Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed method requirements.

### 3.0 Interferences

- 3.1 Cyanate, which may be encountered in certain industrial effluents, will hydrolyze to some extent even at the pH of 9.5 at which distillation is carried out.
- 3.2 Residual chlorine must be removed by pretreatment of the sample with sodium thiosulfate or other reagent before distillation.

### 4.0 Chemicals Required: Distillation Only

- 4.1 Ammonia-free water
- 4.2 Borate buffer solution
  - 4.2.1 For unpreserved samples - Add 964 mL reagent water to a 1 L flask. Add in 2.17 g sodium tetraborate (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>); stir until dissolved. Add 36 mL 0.1 M NaOH (4g NaOH/L).
  - 4.2.2 For preserved samples - Add 964 mL reagent water to a 1 L flask. Add in 2.17 g sodium tetraborate (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>); stir until dissolved. Add in 22 g NaOH pellets; stir until dissolved.
- 4.3 Boric acid trapping solution (0.13M) - Add 995 mL reagent water to a 1 L flask. Add in 8.0 g boric acid (H<sub>3</sub>BO<sub>3</sub>); stir until dissolved.

## Ammonia Nitrogen Distillation with the SimpleDist® Micro Heating Block

- 4.4 Sulfuric acid trapping solution (0.024M) - Add approximately 500 mL reagent water to a 1000 mL volumetric flask. Add 24 mL 1.0 N sulfuric acid and swirl to mix.  
**Caution:** *This reaction is exothermic and has the potential to generate fumes. Use caution. When the solution is at ambient temperature, add reagent water to bring the solution up to the 1000 mL mark on the flask.*
- 4.5 Sodium thiosulfate (for dechlorinating).



**Note:** *The toxicity for each of the reagents used in this procedure is not fully documented. Treat each chemical as a potential health hazard and limit exposure. Exercise good laboratory technique with an emphasis on safety.*

### 5.0 Procedure

- 5.1 Set the temperature of the heating block to 120°C and allow the heat to stabilize. This should take approximately 30 minutes. If all reagents are ready to use this should be sufficient time to set up and prepare a full batch of 20 samples and all necessary QC.
- 5.2 Place one collector tube for each sample, standard, QC, or other aliquot needing to be distilled in a test tube rack with the “Measure” end up. Add 1.0 mL of 0.024 M H<sub>2</sub>SO<sub>4</sub> solution (4.4) or 2 mL 0.13 M boric acid solution (Step 4.3) to each tube. The solution to choose will depend upon the requirements of the analytical method. **Note:** *After final distillate dilution this will give a concentration of 0.04 N H<sub>2</sub>SO<sub>4</sub> or boric acid.* The initial concentration may be adjusted to provide alternate final concentrations as needed by the analytical method of choice. Cap the tubes with one membrane and cap. Ensure that the membrane is not torn and shows a visible edge all the way around the tube.
- 5.3 Place one sample tube for each sample, standard, QC, or other aliquot needing to be distilled in a test tube rack and add 6.0 mL of sample/standard to each tube. The tubes may be labeled with permanent ink to aid in sample identification. Do not add any label to the tube as this will interfere with the fit in the block as well as heat transfer during the distillation process. If solid samples are being distilled, add 0.5 to 1.0 g of the sample to the tube along with approximately 5 mL of reagent water. Samples high in organic content such as sludge should tend toward the lower sample mass while samples that are relatively free of organic material can use the higher end.
- 5.4 If the sample has been previously preserved with acid, add 1.0 mL of the borate buffer for preserved samples (Step 4.2.2) to the first sample tube. If the sample has not been previously preserved with acid instead add 0.75 mL of the borate buffer for unpreserved samples (Step 4.2.1). Immediately place the ‘Distill’ end of a filled and capped collector tube (Step 5.2) over the open end of the sample tube. Press the two pieces together to form a rough fit. You will not be able to hand press a tight seal.

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## Ammonia Nitrogen Distillation with the SimpleDist® Micro Heating Block

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- 5.5 Place the distillation unit in the tube press. Hold the collection tube by the break joint to give support during the sealing procedure. Firmly press the two pieces together with one motion. The collection tube should be flush with the ring around the edge of the sample tube. If the pieces are at a visible angle the assembly will not fit all the way in the block.
- 5.6 Repeat Steps 5.4 and 5.5 for all prepared sample and collection tubes.
- 5.7 Place each assembled and pressed distillation unit in the preheated block. Ensure that the unit is completely inserted in the block by having the ring on the “Distill” side of the tube flush against the surface of the block. (**Note:** *Heat resistant gloves may be necessary during this stage.*)
- 5.8 After 30 minutes of distillation time, remove the first distillation unit from the block. (**Note:** *Heat and chemical resistant gloves are needed for Steps 5.8 and 5.9*)
- 5.9 Immediately after removing the unit from the block, hold it over a chemical disposal container and pull the sample tube off of the collection tube. This is best done by twisting the two pieces in opposite directions while simultaneously pulling them apart. Rocking the sample tube from side to side may not separate the pieces in time. **The sample tube must be removed within 4 seconds of the unit being removed from the block.** Otherwise the distillate could suck back into the sample tube and cause the sample to be lost.
- 5.10 Drop the sample tube and any remaining liquid into the disposal container. Place the collection tube with the ‘Measure’ end (the capped end) down in a test tube rack. Repeat Steps 5.8 through 5.10 for all distilled tubes.
- 5.11 Allow the tubes to cool for 10 to 15 minutes. You will notice fine condensate begin to appear on the inside walls of the collection tube.
- 5.12 After the tubes have cooled to ambient temperature, use the trapping solution inside the tube to rinse the tube walls and collect all the condensate. This can be best accomplished by holding the tube horizontally and then tilting the ‘Distill’ end a few degrees downward. Slowly roll the tube while tilting it back to level and then the ‘Measure’ end down. You may need to gently tap the tube to knock down any drops that cling to the edges.
- 5.13 With the ‘Measure’ end down snap the tube at the break joint. The ‘Distill’ end may now be discarded.
- 5.14 Use reagent water to dilute the distillate up to the 6 mL line on the ‘Measure’ end. Gently swirl the solution to mix.
- 5.15 Due to limited sample volume the analysis is best suited to discrete or flow injection analysis. Plug the end with a stopper or cover with Parafilm® if the analysis will not be performed immediately.
- 5.16 After use, clean exterior surfaces of heating block with a damp sponge. For acid spills, sponge with a diluted solution of sodium bicarbonate followed by distilled water. For caustic spills use a dilute solution of acetic acid followed by distilled water.

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## Total Phenol Distillation with the SimpleDist® Micro Heating Block

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### 1.0 Scope and Application

- 1.1 This method follows US EPA Method 420.4 and SW-846 method 9065. These methods are applicable to the determination of phenol in drinking, ground, surface, and saline waters, domestic and industrial wastes, and soils.
- 1.2 The standard applicable range is 10 to 500 µg/L total phenols. Higher concentrations can be determined by sample dilution.

### 2.0 Summary of Method

- 2.1 A sample is pH adjusted to about 4 with sodium hydroxide or sulfuric acid to prevent interference from sulfur compounds. It is then distilled and analyzed colorimetrically.
- 2.2 Reduced volume versions of this method that use the same reagents and molar ratios are acceptable provided they meet the quality control and performance requirements stated in the method.
- 2.3 Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed method requirements.

### 3.0 Interferences

- 3.1 Sulfur compounds can interfere with the process. These compounds are removed by adjusting the pH of the sample to about 4 prior to distillation.
- 3.2 Oxidizing agents can partially or fully oxidize phenolic compounds. These are tested for at sampling and removed via appropriate measures.
- 3.3 Sample color and turbidity are removed via the distillation step. Some sample color may persist after the initial distillation. These samples may be distilled again to remove additional color. If the color persists, it must be corrected for via appropriate instrumental methods.

### 4.0 Chemicals Required: Distillation Only

- 4.1 Phenol-free reagent water
- 4.2 1 M Sodium Hydroxide (NaOH) - Add approximately 70 mL of reagent water to a 100 mL volumetric flask. Stir and dissolve 4.0 g NaOH and allow the solution to cool. Bring up to the mark with reagent water.
- 4.3 10% Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) - Add approximately 70 mL of reagent water to a 100 mL volumetric flask. Stir and dissolve 10 mL concentrated sulfuric acid and allow the solution to cool. Bring up to the mark with reagent water.



**Note:** The toxicity for each of the reagents used in this procedure is not fully documented. Treat each chemical as a potential health hazard and limit exposure. Exercise good laboratory technique with an emphasis on safety.

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## Total Phenol Distillation with the SimpleDist® Micro Heating Block

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### 5.0 Procedure

- 5.1 Set the temperature of the heating block to 130°C and allow the heat to stabilize. This should take approximately 30 minutes. If all reagents are ready to use this should be sufficient time to set up and prepare a full batch of 20 samples and all necessary QC.
- 5.2 Adjust the pH of all samples, QC, and standards to about 4 using sodium hydroxide or sulfuric acid solutions as necessary. If the amount of acid or base required changes the volume by more than 1%, stronger solutions can be made and utilized.
- 5.3 Place one collector tube for each sample, standard, QC, or other aliquot needing to be distilled in a test tube rack with the “Measure” end up. No trapping solution is necessary for phenol distillation. Cap the tubes with one membrane and cap. Ensure that the membrane is not torn and shows a visible edge all the way around the tube.
- 5.4 Place one sample tube for each sample, standard, QC, or other aliquot needing to be distilled in a test tube rack and add 6.0 mL of sample/standard to each tube. The tubes may be labeled with permanent ink to aid in sample identification. Do not add any label to the tube as this will interfere with the fit in the block as well as heat transfer during the distillation process. If solid samples are being distilled, add 0.5 to 1.0 g of the sample to the tube along with approximately 5 mL of reagent water. Samples high in organic content such as sludge should tend toward the lower sample mass while samples that are relatively free of organic material can use the higher end.
- 5.5 Immediately place the ‘Distill’ end of a filled and capped collector tube (Step 5.2) over the open end of the sample tube. Press the two pieces together to form a rough fit. You will not be able to hand press a tight seal.
- 5.6 Place the distillation unit in the tube press. Hold the collection tube by the break joint to give support during the sealing procedure. Firmly press the two pieces together with one motion. The collection tube should be flush with the ring around the edge of the sample tube. If the pieces are at a visible angle the assembly will not fit all the way in the block.
- 5.7 Repeat Steps 5.5 and 5.6 for all prepared sample and collection tubes.
- 5.8 Place each assembled and pressed distillation unit in the preheated block. Ensure that the unit is completely inserted in the block by having the ring on the “Distill” side of the tube flush against the surface of the block. (*Note: Heat resistant gloves may be necessary during this stage.*)
- 5.9 After 90 minutes of distillation time, remove the first distillation unit from the block. (*Note: Heat and chemical resistant gloves are needed for Steps 5.9 and 5.10*)
- 5.10 Immediately after removing the unit from the block, hold it over a chemical disposal container and pull the sample tube off of the collection tube. This is best done by twisting the two pieces in opposite directions while simultaneously pulling them apart. Rocking the sample tube from side to side may not separate the pieces in time. **The sample tube must be removed within 4 seconds of the unit being removed from the block.** Otherwise the distillate could suck back into the sample tube and cause the sample to be lost.
- 5.11 Drop the sample tube and any remaining liquid into the disposal container. Place the collection tube with the ‘Measure’ end (the capped end) down in a test tube rack. Repeat Steps 5.9 through 5.11 for all distilled tubes.

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## Total Phenol Distillation with the SimpleDist® Micro Heating Block

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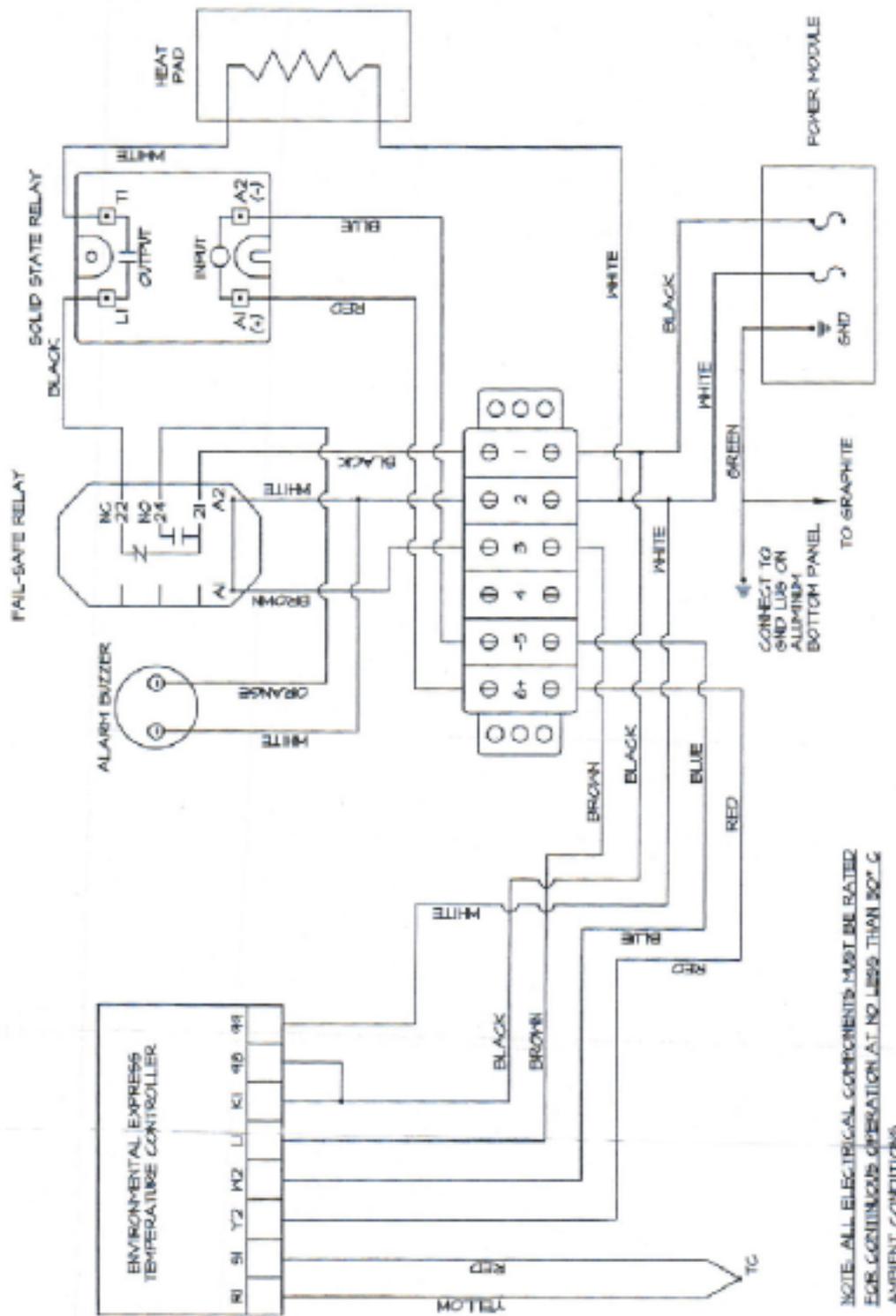
- 5.12 Allow the tubes to cool for 10 to 15 minutes. You will notice fine condensate begin to appear on the inside walls of the collection tube.
- 5.13 After the tubes have cooled to ambient temperature, use the trapping solution inside the tube to rinse the tube walls and collect all the condensate. This can be best accomplished by holding the tube horizontally and then tilting the 'Distill' end a few degrees downward. Slowly roll the tube while tilting it back to level and then the 'Measure' end down. You may need to gently tap the tube to knock down any drops that cling to the edges.
- 5.14 With the 'Measure' end down snap the tube at the break joint. The 'Distill' end may now be discarded.
- 5.15 Use reagent water to dilute the distillate up to the 6 mL line on the 'Measure' end. Gently swirl the solution to mix.
- 5.16 Due to limited sample volume, the analysis is best suited to discrete or flow injection analysis. Plug the end with a stopper or cover with Parafilm® if the analysis will not be performed immediately.
- 5.17 After use, clean exterior surfaces of heating block with a damp sponge. For acid spills, sponge with a diluted solution of sodium bicarbonate followed by distilled water. For caustic spills use a dilute solution of acetic acid followed by distilled water.

## Care & Maintenance

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- Any service inquiries should be directed to Environmental Express Technical Service Department. After each use, clean exterior surfaces with a damp sponge to remove acid residue.
- For acid spills, sponge with a diluted solution of sodium bicarbonate followed by distilled water. Acid that is spilled directly into the digestion wells should be neutralized and removed.
- Before using any cleaning or decontamination methods except those recommended, check with Environmental Express to confirm the proposed method will not damage your heating block.
- Avoid excessive spills, as liquid allowed to overflow into the SimpleDist® Micro heating block casing can severely damage electronic components.
- Unplug the SimpleDist® Micro heating block from the outlet prior to cleaning exterior surfaces. Wipe with damp sponge or towel after each use, first with mild sodium bicarbonate or similar solution followed by DI or distilled water. Avoid solutions on or near the controls.
- If boil over does occur during operation, immediately wipe the heating block system down with neutralizing solution, such as a mild solution of sodium bicarbonate.

Circuitry Diagram:



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

BLOCK DIAGRAM FOR HOT BLOCK  
 ELECTRICAL CONTROLS  
 ENVIRONMENTAL EXPRESS  
 REVISED 2-10-10  
 D043460181

## Troubleshooting Guide

Please consult the following troubleshooting guide if you experience problems with your heating block. See wiring schematic (page 17) for component identification. If you are unable to resolve the problem or if replacement components are necessary, please contact technical service at 1-800-745-8218 as component replacement varies in degree of difficulty. **Only qualified personnel should attempt troubleshooting electrical components.**

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

<b><i>Standard recovery values are consistently low or high.</i></b>	Make sure that your distillate matrix concentration matches what is required by your analytical method. Distilling your calibration standards is a good way to keep the matrix match.
<b><i>Standard recovery values are inconsistent, usually trending towards low.</i></b>	<p>Make sure that the distillation unit is properly sealed together. There should be no gap between the edge of the collection tube and the ring on the sample tube.</p> <p>Make sure that the distillation unit is completely inserted in the block. The ring on the collection tube should be flush with the surface of the block.</p> <p>Make sure that the sample tube is promptly separated from the collection tube after removing the distillation unit from the block. Small amounts of suck back can occur if the process is not performed quickly (within 4 seconds).</p>
<b><i>Solid samples coat the distillation membrane.</i></b>	The sample is high in organic content. Reduce the mass of sample used down to 0.5 g. Additional measures may include adding a layer of activated charcoal or glass wool over the sample. Reduce the amount of water added to the sample to only fill up to 6 mL of volume. Use another sample tube with 6 mL of water as a reference for the fill level.
<b><i>Violent boiling or bumping in the sample tube.</i></b>	Add one small boiling stone (Hengar stones are preferred) to the tube prior to adding the releasing agent and sealing the distillation unit. Try not to exceed 1 mm in diameter.

<p><i>The controller digital display will not illuminate.</i></p>	<p>The problem can be effectively diagnosed by determining if the controller is or is not getting voltage using the following steps:</p> <ol style="list-style-type: none"> <li>1. Confirm that the power cord is plugged securely into the SimpleDist® Micro heating block receptacle and a working outlet.</li> <li>2. Confirm that the switch is in the “on” position. Press button on the back of unit.</li> <li>3. Check the fuse located in the power socket:</li> </ol> <p> <b>Warning:</b> <i>Unplug the unit from the wall power source, ensure no voltage is present. This procedure is a potential electrical hazard and should only be performed by qualified personnel.</i></p> <ol style="list-style-type: none"> <li>4. If the controller will not illuminate and the above steps have passed, the controller is faulty. Call Environmental Express at 1-800-745-8218 for service.</li> </ol>
<p><i>The audible alarm has sounded immediately after powering on and the SimpleDist® Micro Heating Block will not heat.</i></p>	<p>There are two possible causes for your heating block to sound the alarm immediately after the controller cycles through the self test.</p> <ol style="list-style-type: none"> <li>1. Your set point has been set to a value (<math>\geq</math>) 15° less than ambient or current set point temperature. Turn the set point to within 15° of the actual temperature (blue numbers).</li> <li>2. The controller is faulty. Call Environmental Express at 1-800-745-8218 for more information.</li> </ol>
<p><i>The SimpleDist® Micro Heating Block will not heat beyond ambient temperature.</i></p>	<p>A heating block that will not heat beyond ambient temperature typically has a failed relay, heater mat or controller. Call Environmental Express at 1-800-745-8218 for service.</p>

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

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

The SimpleDist® Micro heating block can be “fooled” into fail-safe mode if the set point is manually changed to a value  $\geq 15^\circ$  below your current temperature. However, the primary cause for the runaway temperature is a failure of the internal systems.

To reset default settings:

1. Hold the up and down arrow buttons for six seconds until “Ai Set” appears.
2. Hold the down arrow until “glbl set” is in the window.
3. Press the green advance key to enter.
4. Continue pressing the green advance key until “none user” appears.
5. Press the down arrow key until “Set1 user” appears.
6. Pressing the advance key will restore default settings.
7. If the fail safe continues to trigger after the above steps have been taken, call Environmental Express at 1-800-745-8218 for service.
8. If your display continues to flash “Er.1 Attn”, the thermocouple is faulty. Call Environmental Express at 1-800-745-8218 for service.

## SimpleDist® Micro Heating Block Replacement Parts & Supplies

<i>Description</i>	<i>Catalog #</i>
SimpleDist® Micro Heating Block, 26 wells, 120 VAC	C8000
SimpleDist® Micro Heating Block, 26 wells, 240 VAC	C8000-240
15" x 5" Silicone Rubber Heater Mat for SimpleDist® Micro, 120 VAC	C6300
15" x 5" Silicone Rubber Heater Mat for SimpleDist® Micro, 240 VAC	C6300-240
Power Module (Plug Receptacle) with Push Button Switch	SC941
Power Cord - Heavy Duty	SC958
Environmental Express® Controller	SC945
110/220 V, 25 A Solid-State Relay	SC952
Type K Thermocouple	SC953
Terminal Board	SC955
Fail-Safe Relay	SC941
Alarm Buzzer	SC958
PVC Screw for Rubber Foot	SC945
Rubber Foot	SC952
Barbed Tubing Adapter, 6 pack	SC953
VICI Emitter Pad	SC955

<i>SimpleDist® Micro Disposable Distillation Tubes</i>	<i>Catalog #</i>
<b>Ammonia Tubes</b>	
10 pack	C8010A
21 pack	C8021A
26 pack	C8026A
100 pack	C8100A
<b>Cyanide Tubes</b>	
10 pack	C8010C
21 pack	C8021C
26 pack	C8026C
100 pack	C8100C
<b>Phenol Tubes</b>	
10 pack	C8010P
21 pack	C8021P
26 pack	C8026P
100 pack	C8100P