

# SimpleDist™ System

## Operating & Instruction Manual

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 **ENVIRONMENTAL EXPRESS®**  
a Cole-Parmer company

800-343-5319 or 843-881-6560 [www.environmentalexpress.com](http://www.environmentalexpress.com)

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The C6000 SimpleDist™ Complete System is shown here. The 12-place Manifold Kit is also sold separately, C6210, and fits the 36-well SC100 HotBlock™ or the 12-place HotBlock™, C6002.



Pictured above, the 18-place SimpleDist™ Manifold Kit, C6200, fits the SC154 HotBlock™.

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## Limited Warranty

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The SimpleDist™ System 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.

Environmental Express, Inc. makes no other warranty, expressed or implied for this product with respect to merchantability, fitness for a particular use or any other matter. Environmental Express, Inc. is not liable for any consequential or compensatory damages arising from use of, or in conjunction with, this product. The maximum liability shall be the invoice price of this product.

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## Repair Policies

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### Under Warranty Repair:

If the SimpleDist™ should fail to operate within the warranty period (one year from date of shipment) Environmental Express, Inc. will repair it and ship it back to the customer at our 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™ System was caused by negligence or improper use, 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™ System 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 glassware associated with the SimpleDist™ System.**



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

HotBlock™ Serial # (if applicable)

If you ordered the C6000 please record the serial # of your HotBlock™ here for easy reference. Your serial # is located on the back of your HotBlock™.

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## Parts and Supplies

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### SimpleDist™ Complete System

C6000

Parts and Supplies Included:

<i>Description</i>	<i>Catalog #</i>
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12-well HotBlock™	C6002
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12-valve Manifold Kit	C6210
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SimpleDist™ Manual

Includes operating Instructions with wiring diagram and temperature controller operating instructions.

### 12-well HotBlock™

C6002

Parts and Supplies Included:

The 12-place HotBlock™ is a compact block for laboratories digesting small batches of 50mL samples. Each block comes with a specially designed 12-place polycarbonate tube rack (C6050) and has the same sturdy construction and features of our other HotBlock™ Systems.

<i>Description</i>	<i>Catalog #</i>
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Tube Rack	C6050
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For handling 12 boiling tubes simultaneously, polycarbonate

Power Cord	SC958
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### 12-valve Manifold Kit

C6210

Parts and Supplies Included:

<i>Description</i>	<i>Catalog #</i>
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12-valve PVC Vacuum Manifold	C6003B
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Base Supports, 2 piece set	C6007B
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12 Flow Control Valves	C6005 each
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Push lock design, accepts 3/8" diameter tubing

1 PVC Hose Barb for SCS, 1/4"	C6160
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12 Boiling Tubes	C6010 each
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30mm x 200mm threaded boiling tubes marked at 50mL, borosilicate glass

1 pack Collection Traps	C6100 100pk
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Disposable polystyrene

1 pack Polypropylene Reagent Addition Tubes	C6110 100pk
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2 packs Caps	C6120 6pk
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38mm open top closures

2 packs Black Neoprene Washers	C6120A 6pk
--------------------------------	------------

2 packs 2-Port Cap Inserts	C6130 6pk
----------------------------	-----------

1 12-Piece Tubing Kit	C6142
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Tubing kits come with 12 lengths of tubing (3/8" OD X 1/4" ID). Six pieces of tubing are 8" and the other 6 are 10.25". The longer pieces extend from the manifold valves to the front row of sample collection flasks while the shorter lengths go to the back row of collection flasks. Each length of tubing comes with a tubing adaptor and a barbed fitting adaptor already inserted.

Tubing Adaptors	C6140 12pk
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Barbed Tubing Adaptor	C6006 6pk
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**18-valve Manifold Kit**

C6200

## Parts and Supplies Included:

*Description**Catalog #*

18-valve PVC Vacuum Manifold

C6007A

Base Supports, 2 pieces

C6007B

18 Flow Control Valves

Push lock design, accepts 3/8" diameter tubing

C6005 each

1 PVC Hose Barb for SCS, 1/4"

C6160

18 Boiling Tubes

30 mm. x 200 mm. threaded boiling tubes marked at 50mL, borosilicate glass

C6010 each

1 pack Collection Traps

Disposable polystyrene

C6100 100pk

1 pack Polypropylene Reagent Addition Tubes

C6110 100pk

3 packs Caps

38 mm. open top closures

C6120 6pk

3 packs Black Neoprene Washers

C6120A 6pk

3 packs 2-Port Cap Inserts

C6130 6pk

2 12-Piece Tubing Kits

Tubing kits come with 12 lengths of tubing (3/8" OD X 1/4" ID). Six pieces of tubing are 8" and the other 6 are 10.25". The longer pieces extend from the manifold valves to the front row of sample collection flasks while the shorter lengths go to the back row of collection flasks. Each length of tubing comes with a tubing adaptor and a barbed fitting adaptor already inserted.

C6142

Tubing Adaptors

C6140

Barbed Tubing Adaptors

C6006 6pk

Snip & Pour, 0.2N H<sub>2</sub>SO<sub>4</sub>

C7005 50x5mL

Snip &amp; Pour, 1.25N NaOH

C71155 50x5mL

Snip &amp; Pour, 20g/L Boric Acid

C7125 50x5mL

SimpleDist Manual

Includes operating Instructions with wiring diagram and temperature controller operating instructions.

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## Safety Information

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1. The Environmental Express SimpleDist™ System should be set up and operated in a chemical fume hood with a face velocity of not less than 100 CFM.
2. Wear appropriate Personal Protective Equipment (PPE) suitable for use with caustic and corrosive materials.
3. Do not operate the SimpleDist™ System in the vicinity of combustible material.
4. Consult your in-house electrician to be certain the SimpleDist System power cord is properly grounded.
5. During operation the surfaces around the heater assembly will get HOT. Do not touch the outer surface.
6. Do not move the SimpleDist™ System while hot.
7. Do not attempt to operate the SimpleDist™ System block over 180°C.
8. Review Material Safety Data Sheets for all materials used or generated during the operation of the SimpleDist™ System.



***Avoid breathing any vapors that may come off of the SimpleDist™ System; they may be harmful or fatal.***

9. Vacuum should be continued until the SimpleDist™ System has cooled down and the collection trap is removed from the assembly.
10. The power should be kept plugged into its outlet until the unit has cooled down.
11. If boil over does occur during operation of the SimpleDist™ System operation, immediately wipe the system down with neutralizing solution, such as a mild solution of sodium bicarbonate.
12. Unplug the SimpleDist™ System 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.
13. Install an excess gas trap in the vacuum line to remove excess vapors.
14. Use of micro-porous boiling chips in each distillation flask may help to prevent bumping.

***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 Material Safety Data Sheets 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.***

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## Setup and Assembly:

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1. Unpack the heating unit and place it in a chemical fume hood with the temperature controller facing outward. (If you are purchasing a manifold kit to use with a previously purchased HotBlock™, skip to step 2).
2. Unpack boiling flasks. The boiling flasks should be cleaned according to laboratory protocol and assembled in the SimpleDist™ block.
3. Unpack the bags of tubing which were packed with the heating unit.
4. Review the diagrams and familiarize yourself with parts, names, and locations of the parts for the chemistry you are doing.
5. Save original packaging material in a dry area for use if unit needs to be returned for service. Refer to warranty policy on page 1.
6. Assemble the vacuum manifold and base supports and then tighten 4" set screws using an 3/16" Allen wrench.
7. Place vacuum manifold containing flow control valves behind the heating block. Slide the manifold forward until base supports surround the heating block on both sides.
8. Connect vacuum tubing to the PVC barb located on the left leg of the vacuum manifold. An excess vapor trap containing NaOH solution may be inserted between the manifold and the vacuum source.
9. Open the C6142 Tubing Kit to find the lengths of silicone tubing with tubing on one end and barbed fitting adaptors into the other end. Depending on the manifold purchased, there are 6 or 9 long tubes for the front positions and 6 or 9 shorter tubes for the back positions. Insert the end with the barbed fitting adaptors into the flow control valve. Pull back on the orange locking collar of the valve and then push the adaptor firmly into the valve.  
**Note:** See *Tubing Valve Assembly, Figure 1, page 11.*
10. The glassware/plasticware for the SimpleDist™ System is assembled as follows:
  - a. Place a boiling tube into one of the left-most distillation block positions.
  - b. Place a washer (Catalog # 6120A) on top of a 2-port cap insert. Then, place a 2-port cap insert into one of the closures (green cap).  
**Note:** See *Boiling Tube Assembly drawing, Figure 2, page 12.*
  - c. Thread a closure/cap insert assembly onto the boiling tube.
  - d. Insert an assembled disposable reagent addition tube/tip into the smaller port on the cap insert.
  - e. Fit a collection trap into the larger top port of the cap insert.
  - f. Connect tubing/tubing adaptor onto the top of the collection trap.
  - g. Repeat steps A through F for the remaining glassware, working from left to right.
  - h. To remove the glassware, reverse the above procedure.  
**Note:** See *drawing #C6000, Figure 3, page 13, for complete assembly diagram.*

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## 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:



4000040

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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

A handwritten signature in black ink, appearing to read 'Al Jurgela', is written over a horizontal line.

Signed: \_\_\_\_\_

Al Jurgela, Chief Executive Officer



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## HotBlock™ Information and Markings

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

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

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



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



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

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## HotBlock™ Unpacking and Installation

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### Unpacking Your HotBlock™:

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

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

### Installation Requirements

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

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

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

### Electrical Requirements

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

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

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

See individual specifications for each HotBlock™ model.

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## HotBlock™ Temperature Information:

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

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

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

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

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

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## Adjusting the Temperature of Your HotBlock™:

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

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## Safe-Sample™ Temperature Protection:

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

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

If the alarm sounds, see the HotBlock™ troubleshooting guide of your manual on pages 20-22.

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## HotBlock™ Potential Hazards and Maintenance

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### Potential Hazards:

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

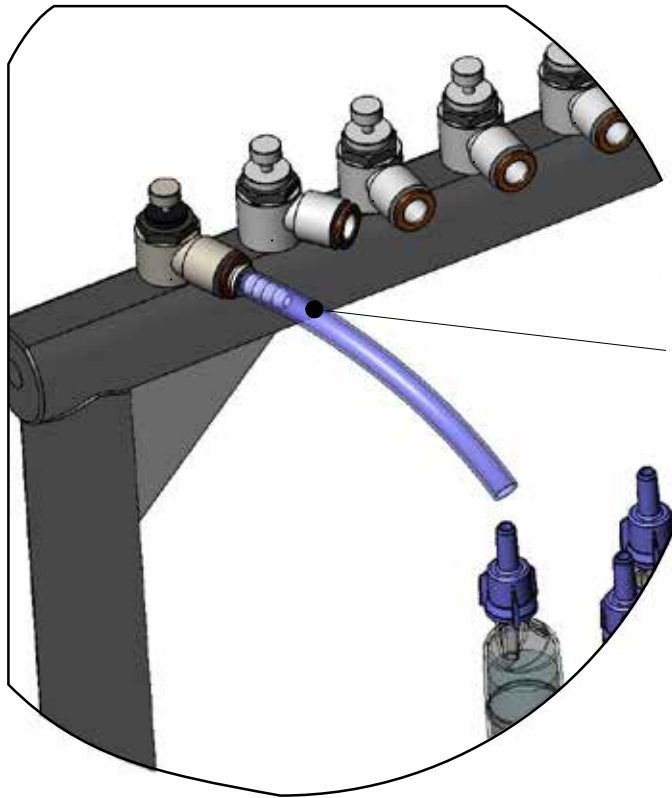
1. Use extreme caution when operating the HotBlock™. Plastic and graphite surfaces of the HotBlock™ may be too hot to safely touch with bare hands.
2. The HotBlock™ contains electrical circuits and devices and compounds operating at dangerous voltages. Contact with these circuits, devices and components can cause serious injury or painful electric shock.
3. 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.
4. 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.
5. See individual block specifications for power requirements.
6. Application of the wrong supply voltage can create a fire hazard and a potentially serious shock hazard, and could seriously damage the HotBlock™ system. See specifications for individual HotBlocks™.
7. 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.
8. Always lift the HotBlock™ from the bottom of the unit.

### Maintenance:

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1. Any service inquiries should be directed to Environmental Express Technical Service Department.
2. After each use, clean exterior surfaces with a damp sponge to remove acid residue.
3. 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.
4. Before using any cleaning or decontamination methods except those recommended, check with Environmental Express to confirm the proposed method will not damage your HotBlock™.
5. Avoid excessive spills, as liquid allowed to overflow into the HotBlock™ casing can severely damage electronic components.

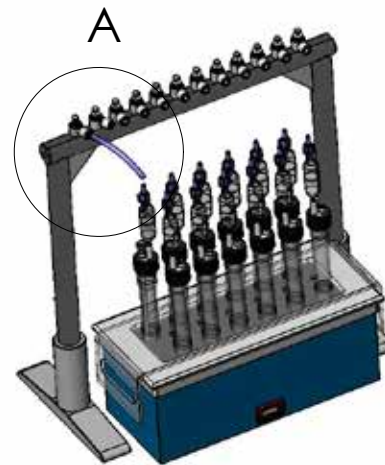
**Tubing Valve Assembly, Figure 1**



NOTE: BARBED FITTING ADAPTER MUST BE FULLY SEATED IN THE FLOW CONTROL VALVE TO PREVENT LEAKING.

PULL THE ORANGE RELEASE FITTING BACK AND PUSH THE ADAPTER ALL THE WAY IN UNTIL IT SEATS.

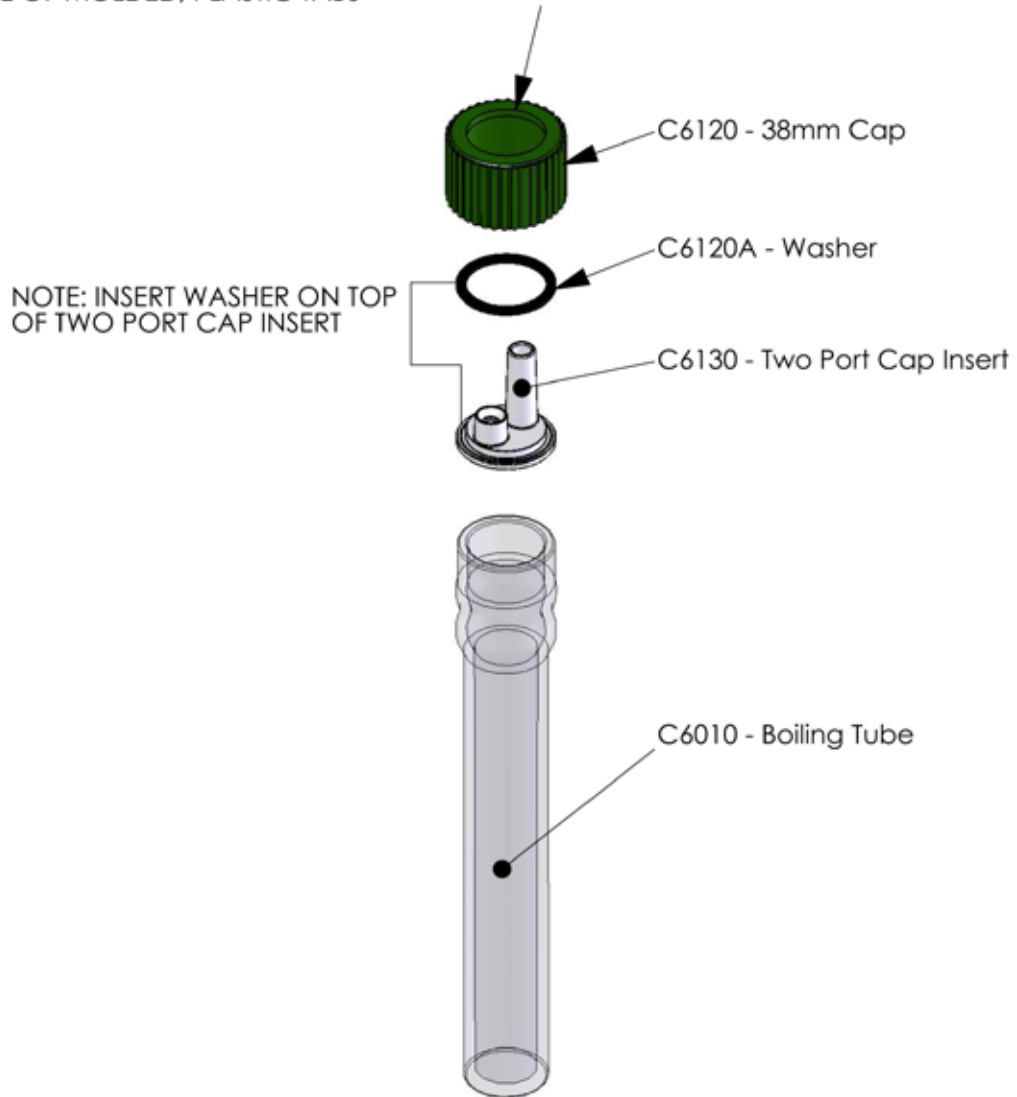
DETAIL A  
SCALE 1 : 2



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		TOLERANCES:	DRAWN	JHO		5/21/10
		FRACTIONAL ±	CHECKED			
		ANGULAR: MACH ± BEND ±	ENG APPR.			
		TWO PLACE DECIMAL ±	MFG APPR.			
		THREE PLACE DECIMAL ±	Q.A.			
		MATERIAL	COMMENTS:			
	NEXT ASSY	USED ON	FINISH		SIZE	
	APPLICATION	DO NOT SCALE DRAWING			DWG. NO.	
					SCALE:1:20	WEIGHT:
						SHEET 1 OF 1
						REV. 2

**Boiling Tube Assembly, Figure 2**

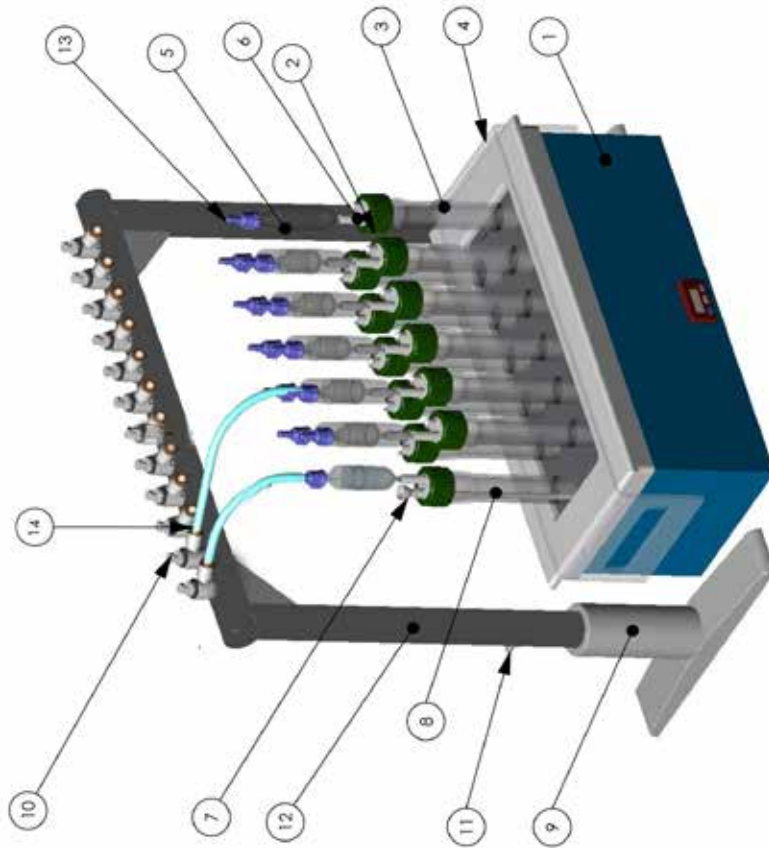
NOTE: MAKE SURE THE EDGES OF THIS I.D. ARE FREE OF MOLDED, PLASTIC TABS



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				DRAWN	JHO 6/3/09	
				CHECKED		
				ENG APPR.		
				MFG APPR.		
			MATERIAL	Q.A.		
			FINISH	COMMENTS:		
	NEXT ASSY	USED ON				
	APPLICATION		DO NOT SCALE DRAWING			
				SCALE: 1:5	WEIGHT:	SHEET 1 OF 1

Assembly Complete System, Figure 3

SimpleDist™



	C6142	TUBING KIT, 12 PIECES
1	C6002	12-WEEL BLOCK ASSY
2	C6120	38MM OPEN TOP ENCLOSURE, POLYPROPYLENE
3	C6010	BOILING TUBE, BOROSILICATE GLASS, 38MM THREADED TOP
4	C6050	12 PLACE TUBE RACK, ACRYLIC
5	C6100	25mL COLLECTION TRAP
6	C6130	TWO PORT CAP INSERT
7	C6110B	FUNNEL TIP
8	C6110A	PP TUBING - CUT TO 7" LONG
9	C6007B	SUPPORT BASE FOR 12-PLACE MANIFOLD
10	C6005	FLOW CONTROL VALVE, SMC P/N AS3211FN02-11S
11	C6160	1/4" BARB CONNECTOR FOR VACUUM INLET
12	C6003B	12 PORT MANIFOLD W/O VALVES, PVC (FULLER MFG)
13	C6140	TUBING ADAPTER - FITS 1/4" ID TUBING AND COLLECTION TRAP
14	C6006	BARBED FITTING ADAPTER - 1/4" BARB TO 3/8" STRAIGHT

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## Total Cyanide Distillation with the SimpleDist™:

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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 500µg/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.

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### 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.

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### 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. For example, the nitrate-nitrite interference is eliminated by addition of sulfamic acid during the distillation step. Further, the addition of magnesium chloride, which acts as a catalyst, will promote the breakdown of refractory iron-cyanide complexes.

The reagent preparations for these two interferences will be summarized in the Reagent Preparation section. For all other pretreatment procedures refer to the US EPA method number 335.4 and/or SW846 9010C.

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### 4.0 Chemicals Required: Distillation Only

- 4.1 Sodium hydroxide
- 4.2 Sulfuric acid, 18N
- 4.3 Magnesium chloride, if refractory iron-cyanide complexes are present
- 4.4 Sulfamic acid – if nitrates and nitrites are present
- 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.*

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### 5.0 Procedure

- 5.1 Pipette 50mL of sample or an aliquot diluted to 50mL with reagent water into the SimpleDist™ System boiling tube. For solid samples weigh 1.0g or less to the nearest 0.01g, dilute to 50mL.
- 5.2 Insert inlet port liner into the green screw cap and thread assembly onto top of glassware. Be sure that the “step” on the inlet port liner is flush with the top opening of the green cap.



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## Total Cyanide Distillation with the SimpleDist™ Continued:

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- 5.3 Assemble inlet tube with funnel tip and insert into the smaller port on cap.
- 5.4 Pour the entire contents of the 1.25 N NaOH Snip & Pour tube into the collection trap. If Snip & Pour tubes are not being utilized add a sufficient volume of NaOH solution that will result in a final concentration of 0.25 N NaOH after adding DI water to 25 mL. (Example: 10 mL of 0.625 N NaOH solution). For best results wait until the distillation is complete to bring the trapping solution to final volume. The trap may be attached to the glassware at this point or it may be filled off-line and then placed onto the glassware. Attach tubing/adaptor to the collection trap.
- 5.5 Repeat 5.1 through 5.4 for all samples to be distilled.
- 5.6 Turn on the vacuum and slowly adjust each valve to provide an air flow bubble rate of 5-10 bubbles per second for each position as viewed in collection trap. Vacuum should be sufficient to maintain slight negative pressure on the assembly throughout the distillation.
- 5.7 If nitrate-nitrite is known to be present add 5.0mL of sulfamic acid reagent through the reagent inlet. Allow the air flow to mix for several minutes.
- 5.8 If iron cyanide complexes are known to be present add 2.0mL of magnesium chloride reagent. If excess foaming occurs, add an additional 2.0mL. Allow a few minutes to mix.
- 5.9 Slowly add 5.0mL of 18N sulfuric acid through the reagent inlet.
- 5.10 Turn the heat on and set the temperature of the heating block to 125°C. The block will achieve this temperature in approximately 30 minutes. Heat for an additional 60 minutes after the block achieves this temperature.
- 5.11 Remove the collection trap from the assembly.
- 5.12 Turn air/vacuum valves to the **off** position. Repeat for each sample position.
- 5.13 Bring the volume of liquid in the collection trap up to the 25 mL line with DI water (a graduated cylinder, volumetric flask, or other volumetric glassware may be used for volume measurements). The distilled cyanide in the trap is now ready for analysis. Pour contents into an appropriate container. When pouring, position the vent tube of the trap up to prevent trapping of air.
- 5.14 After use clean exterior surfaces with a damp sponge. For acid spills sponge with a diluted solution of sodium bicarbonate followed by distilled water.

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## Ammonia Nitrogen Distillation with the SimpleDist™:

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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 – 2.0mg/L NH<sub>3</sub> as N. Higher concentrations can be determined by sample dilution.

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### 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 into 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.

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## Ammonia Nitrogen Distillation with the SimpleDist™ Continued:

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### 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 pre-treatment of the sample with sodium thiosulfate or other reagents before distillation.

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### 4.0 Chemicals Required: Distillation Only

- 4.1 Ammonia-free water
- 4.2 Borate buffer solution
- 4.3 Sodium hydroxide, 1N
- 4.4 Boric acid catch solution (20g/L) – for use with the nesslerization and titration methods
- 4.5 Sulfuric acid catch solution, 0.04N – for use with the phenate and ion selective electrode methods
- 4.6 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.*

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### 5.0 Procedure

- 5.1 Adjust an aliquot of at least 25mL of sample to a pH of 9.5 using 1N sodium hydroxide and remove any residual chlorine.
- 5.2 Pipette 25mL of pH-adjusted sample or an aliquot diluted to 25mL with reagent water into the SimpleDist™ System boiling tube. For solids weigh 1.0g +/- 0.01g and dilute to 25mL. Add boiling chips to the boiling tube.
- 5.3 Insert the inlet part liner into the green screw cap and thread assembly onto the top of the boiling tube.
- 5.4 Assemble inlet tube with funnel tip and insert into the smaller port on cap.
- 5.5 Pour the entire contents of the appropriate Snip & Pour trapping solution into the collection trap. The final analytical method will determine which trapping solution you use. Generally, titration methods use boric acid while other methods use sulfuric acid. If Snip & Pour tubes are not being utilized add a sufficient volume of the appropriate trapping solution that will result in the proper final concentration after adding DI water to 25 mL. (Example: 10 mL of 0.1 N H<sub>2</sub>SO<sub>4</sub> solution) For best results wait until the distillation is complete to bring the trapping solution to final volume. The trap may be attached to the glassware at this point or it may be filled off-line and then placed onto the glassware. Attach tubing/adaptor to the collection trap.
- 5.6 Repeat steps 5.1 through 5.5 for all samples to be distilled.
- 5.7 Turn on the vacuum and adjust each valve to provide an air flow bubble rate of 10-15 bubbles per second for each position as viewed in the collection trap. Vacuum should be sufficient to maintain slight negative pressure on the assembly throughout the distillation.



**Important: Monitor the vacuum to insure a back pressure does not build up in the boiling tube!**

- 5.8 Add 1.25mL of borate buffer to the sample through the reagent inlet tube.
- 5.9 Turn the heat on and set the temperature of the HotBlock™ to 135°C. The block will achieve this temperature in approximately 30 minutes. Heat for an additional 60 minutes after the block achieves this temperature.
- 5.10 Remove the collection trap from the assembly.



**Important: Vacuum must remain on!**

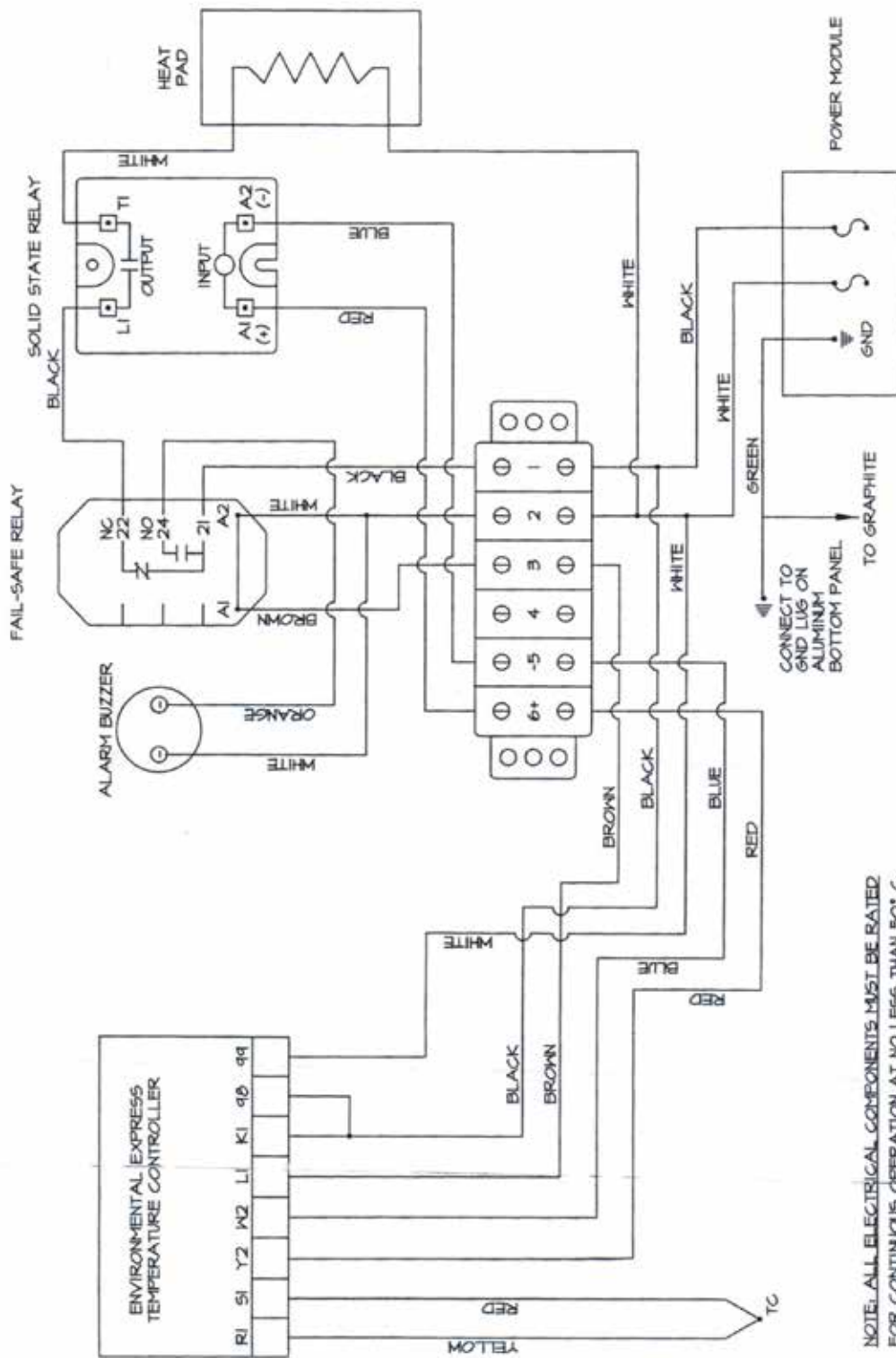
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**Ammonia Nitrogen Distillation with the SimpleDist™ Continued:**

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- 5.11 Turn air/vacuum valves to the *off* position.
- 5.12 Bring the volume in the collection trap up to the 25 mL line with DI water (a graduated cylinder, volumetric flask, or other volumetric glassware may be used for volume measurements).
- 5.13 The distillate in the collection trap is now ready for analysis. Pour contents into an appropriate container. When pouring, position the vent tube of the collection trap up to prevent trapping of air.
- 5.14 Bring the volume in the collection trap up to the 25 mL line with DI water (a graduated cylinder, volumetric flask, or other volumetric glassware may be used for volume measurements).

**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  
D08B46CIRI

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**SimpleDist™ System Troubleshooting Guide:**

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**The tubes from the manifold to the trap are falling out of the manifold.**

1. Ensure that the tubing adaptors are completely inserted into the valves of the manifold. These adaptors may not protrude far enough out of the tubing to allow for this. Work the adaptors out of the tubing to allow them to properly fit into the manifold valves. If the adaptors need to be replaced, order part # C6006.

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**The sample is boiling over or squirting out of the reagent addition tube.**

1. This is due to a back pressure buildup within the boiling tube. During the first couple of distillations one should continue to monitor the bubble rate in the trap as the HotBlock™ comes to temperature. To ensure that you maintain a bubble rate of 10 – 15 bubbles per second you may need to increase the amount of vacuum applied to the traps. Open the valves on the top of the manifold to increase the vacuum to maintain the proper bubble rate.
2. Check the volume of the sample you are using as well. For cyanide distillation you should be using 50mL of sample. For ammonia distillation you should be using 25mL of sample.
3. Ensure that the vacuum you are using will pull at least 15" of Hg. Anything less will not provide adequate vacuum to overcome the back pressure.

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**Recoveries are too low.**

1. Check the samples for any interferences per the appropriate method.
2. Ensure you have the proper sample temperature and vacuum pull. Also make sure you are distilling for the appropriate amount of time. The recommended distillation times start when the HotBlock™ comes to the required temperature.

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**Recoveries are too high.**

1. Clean the boiling tubes to insure there is no carryover from previous samples.
2. Do not reuse any traps—these are designed for one-time use.
3. Check the samples for any interferences per the appropriate method.

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**I cannot turn the vacuum completely off using the valves on the manifold.**

1. Make sure the black washer is flush with the top white knob. If it is not the washer will prevent you from closing the valve all the way.

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## HotBlock Troubleshooting Guide:

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Please consult the following troubleshooting guide if you experience problems with your HotBlock™. See wiring schematic (page 19) 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. We recommend that only qualified personnel attempt troubleshooting electrical components.

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

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### The controller digital display will not illuminate.

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

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

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

1. Confirm that the power cord is plugged securely into the HotBlock receptacle and a working outlet.
2. Confirm that the switch is in the "on" position. Press button on the back of HotBlock™.
3. Check the fuse located in the power module:
  - a. Locate the fuse drawing indented into the power module next to the socket.
  - b. Using a small screwdriver, pry open the fuse compartment cover.
  - c. Examine the exposed fuse for a break in the filament and if necessary, check for continuity using a volt-meter.
  - d. If the fuse is determined to be blown, replace it with the spare fuse located in the slide-out compartment beneath the operating fuse.



**Caution: This procedure is a potential electrical hazard and should only be performed by qualified personnel.**

4. Inside the HotBlock™, check voltage leading from the power module to the controller:
  - a. Remove the bottom panel of the HotBlock by unscrewing the rubber feet.
  - b. On the back of the controller, locate the black wire at terminal 98 and white wire at terminal 99.
  - c. Set your volt-meter on AC voltage.
  - d. Touch your red lead to the exposed white wire and black lead to the exposed black wire.
  - e. If your volt-meter reads 110-122V, the controller is receiving power but has failed internally. It must be replaced (see parts list, page 23).
  - f. If your volt-meter registers less than 110-122V, using step d above check the black and white wires at the terminal board and then at the power module to determine if there is a faulty connection.

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

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

1. Your set point has been set to a value ( $\geq$ ) 15° less than ambient or current set point temperature. Turn the set point to within 15° of the actual temperature (blue numbers)
2. The controller is faulty. Call Environmental Express at 1-800-745-8218 for more information.

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

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

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

You may troubleshoot the relay by following these steps:



**Caution: This procedure is a potential electrical hazard and should only be performed by qualified personnel.**

1. Power off your HotBlock™.
2. Remove the bottom panel of the HotBlock by unscrewing the rubber feet.
3. Power the HotBlock™ on and allow it to overshoot your set point temperature.
4. Locate the solid state relay mounted to the bottom panel.
5. Set your volt-meter to measure AC voltage.
6. Touch your red lead to terminal T1 (white wire) of the solid state relay and touch your black lead to a ground source (e.g., the green/yellow wire from the power module or an empty terminal on the terminal board).
7. If your volt-meter reads 110V-122V, then the relay is stuck in the “closed” position and it must be replaced (see parts list, page 23).

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

**-OR-**

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

First reset default settings:

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

To troubleshoot the thermocouple, follow these steps:

1. Power off your HotBlock™.
2. Remove the bottom panel of your HotBlock™ by unscrewing the rubber feet.
3. Locate the thermocouple wires at terminals R1 (yellow) and S1 (red) of the controller and remove using a small screwdriver.
4. Cut the exposed ends of the two wires.
5. Strip 1/4” of insulation from each wire and reconnect them to the appropriate controller terminal and tighten.
6. Power on your HotBlock™.
7. If your display continues to flash ***Er.1 Attn***- the thermocouple is faulty and must be replaced (see parts list, page 23).

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

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

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



**Caution: This procedure is a potential electrical hazard and should only be performed by qualified personnel.**

To measure the relay voltage, follow these steps:

1. Remove the bottom panel of your HotBlock by unscrewing the rubber feet.
2. Locate the solid state relay mounted to the bottom panel.
3. Set your volt-meter to measure AC voltage.
4. Touch your red lead to terminal T1(white wire) of the solid state relay and touch your black lead to a ground source (i.e., the green/yellow wire from the power module or an empty terminal on the terminal board).
5. If your volt-meter does not read 110V-122V, then the relay has stuck in the **open** position and it must be replaced (see parts list, page 23).

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

To measure your heater mat resistance, follow these steps:

1. Power off your HotBlock and remove the bottom panel of your HotBlock™ by unscrewing the rubber feet.
2. Locate and disconnect the white wire connected to terminal T1 of the relay and an identical wire on terminal #2 of the terminal board (note: terminal #2 of the terminal board contains 3 white wires. To ensure you have the correct wire, trace it back and ensure it originates from the graphite portion of your HotBlock™).
3. Set your volt-meter to measure ohms ( $\Omega$ )
4. Touch the red lead to one of these wires and touch the black lead to the remaining wire.
5. If your reading is "OL" (over limit) or a value greater than 25 ohms, then the heater mat has failed and it must be replaced (see parts list, page 23).

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



**Caution: This procedure is a potential electrical hazard and should only be performed by qualified personnel.**

To measure voltage from the controller, follow these steps:

1. Remove the bottom panel of your HotBlock™ by unscrewing the rubber feet.
2. Locate the solid state relay mounted to the bottom panel.
3. Set your volt-meter to measure DC voltage.
4. Touch the red lead to terminal A1 (red) and the black lead to terminal A2 (blue) of the solid state relay.
5. Your volt-meter should read 3V-16V.
6. If your volt-meter does not read 3V-16V, perform steps 3 and 4 on the red and blue (5 and 6) wires at the terminal board and controller to determine if there is a faulty or loose connection.
7. If you do not get a reading of 3V-16V at terminals Y2 (red) and W2 (blue) of the controller then the controller has failed internally and it must be replaced (see parts list, page 23).



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## HotBlock™ Replacement Parts

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<i>Description</i>	<i>Catalog #</i>
Power Module (Plug Receptacle) w/ Push Button Switch	SC941
Power Cord - Heavy Duty	SC958
Environmental Express Controller	SC945
110/220V, 25A Solid State Relay	SC952
Type K Thermocouple	SC953
Terminal Board	SC955
15" X 5" 120V Silicone Rubber Heater Mat for model C6002 (12-Place Block)	C6300
15" X 5" 240V Silicone Rubber Heater Mat for model C6002-240 (12-Place Block)	C6300-240*
Fail-Safe Relay	SC968
Alarm Buzzer	SC969
PVC Screw for Rubber Foot	SC964
Rubber Foot	SC976
Barbed Tubing Adaptor, 6 pack	C6006
VICI Emitter Pad	SC978

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