# SimpleDist™ System Operation & Instruction Manual



# ENVIRONMENTAL EXPRESS

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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.

# LIMITED WARRANTY

he 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.

# REPAIR POLICY — 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.

Note: This warranty does not apply to any glassware associated with the SimpleDist System.

# REPAIR POLICY — 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.

# ENVIRONMENTAL EXPRESS

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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.

# C6000, SimpleDIST Complete System, Parts and Supplies Included:

 SIMPLEDIST SYSTEM HEATING UNIT (Catalog # C6002) includes: 12-position 50mL HotBlock, distillation block, temperature controller, fuse holder with 15 amp fuse, power relay, heater mat, and thermocouple. Feet (included) packaged separately.

- VACUUM MANIFOLD: 12-position PVC manifold includes hose barb (Manifold, Catalog # C6003B) (Hose Barb, Catalog # C6160)
- 2 BASE SUPPORTS (Catalog # C6007B)
- 12-FLOW CONTROL VALVES: Push lock design, accepts 3/8" diameter tubing (Catalog # C6005, each)
- MANUAL Includes: Operating Instructions with wiring diagram and temperature controller operating instructions.
- 12 PC TUBING KIT: 12 pieces of high temperature silicone tubing 3/8" OD X 1/4" ID to connect to the vacuum manifold and tubing adapters to connect to the collection trap (Tubing Kit, Catalog # C6142). Each length of tubing comes with a tubing adaptor (Tubing Adaptors Catalog # C6140) and a barbed fitting adapter (Catalog # C6006, 6/pk) already inserted.
- 12 BOILING TUBES: 30mm x 200mm threaded boiling tubes marked at 50mL, borosilicate glass (Catalog # C6010, each)
- TUBE RACK for handling 12 boiling tubes simultaneously, polycarbonate (Catalog # C6050)
- 12 CLOSURES: 38mm open top closures (Catalog # C6120, 6/pk)
- 12, 2-PORT CAP INSERTS (Catalog # C6130, 6/pk)
- 100 REAGENT ADDITION TUBES: polypropylene (Catalog # C6110, 100/pk)
- 100 COLLECTION TRAPS: disposable polystyrene (Catalog # C6100, 100/pk)

# C6210, 12-place Manifold Kit, Parts and Supplies Included:

- VACUUM MANIFOLD: 12-position PVC manifold (Catalog # C6003B)
- HOSE BARB (Catalog # C6160)
- 2 BASE SUPPORTS (Catalog # C6007B)
- 12 FLOW CONTROL VALVES: Push lock design, accepts 3/8" diameter tubing (Catalog # C6005, each)
- MANUAL Includes: Operating Instructions with wiring diagram and temperature controller operating instructions.
- 12-PC TUBING KIT: 12 pieces of high temperature silicone tubing 3/8" OD X 1/4" ID to connect to the vacuum manifold and tubing adapters to connect to the collection trap (Tubing Kit, Catalog # C6142). Each length of tubing comes with a tubing adaptor (Tubing Adaptors Catalog # C6140) and a barbed fitting adapter (Catalog # C6006, 6/pk) already inserted.
- 12 BOILING TUBES: 30mm x 200mm threaded boiling tubes marked at 50mL, borosilicate glass (Catalog # C6010, each)
- 12 CLOSURES: 38mm open top closures (Catalog # C6120, 6/pk)
- 12 2-PORT CAP INSERTS (Catalog # C6130, 6/pk)
- 100 REAGENT ADDITION TUBES: polypropylene (Catalog # C6110, 100/pk)
- 100 COLLECTION TRAPS: disposable polystyrene (Catalog # C6100, 100/pk)

SimpleDist System Parts and Supplies

# C6200 18-Place Manifold Kit, without HotBlock Parts and Supplies Included:

- VACUUM MANIFOLD (Catalog # C6007A): 18-position PVC manifold
- HOSE BARB (Catalog # C6160)
- 2 BASE SUPPORTS (Catalog # C6007B)
- 18 FLOW CONTROL VALVES: Push lock design, accepts 3/8" diameter tubing (Catalog # C6005, each)
- MANUAL Includes: Operating Instructions with wiring diagram and temperature controller operating instructions.
- 18-PC TUBING KIT: 18 pieces of high temperature silicone tubing 3/8" OD X 1/4" ID to connect to the vacuum manifold and tubing adapters to connect to the cyanide trap (Tubing Kit, Catalog # C6142). Each length of tubing comes with a tubing (Tubing Adaptors Catalog # C6140) and a barbed fitting adapter (Catalog # C6006, 6/pk) already inserted.
- 18 BOILING TUBES: 30mm x 200mm threaded boiling tubes marked at 50mL, borosilicate glass (Catalog # C6010, each)
- 18 CLOSURES: 38mm open top closures (Catalog # C6120, 6/pk)
- 18 2-PORT CAP INSERTS (Catalog # C6130, 6/pk)
- 100 REAGENT ADDITION TUBES: polypropylene (Catalog # C6110, 100/pk)
- 100 COLLECTION TRAPS: disposable polystyrene (Catalog # C6100, 100/pk)

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# **Unpacking and Setup:**

- 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 (2) 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 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 adapters 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 adapters into the flow control valve. Pull back on the orange locking collar of the valve and then push the adapter firmly into the valve.

### Note: See Tubing Valve Assembly, Figure 1, page 5.

- 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 6.

- 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 adapter 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 7, for complete assembly diagram.

SimpleDist System Safety Information

# **Safety Information**

- 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 Simple Cyanide 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.

## **HotBlock Temperature Settings:**

The pre-set factory "set point" temperature of your HotBlock is 106°C. Factory tests have shown that this temperature is "sea level safe." 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 method.

Note: 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.

# Adjusting the Temperature of Your HotBlock:

- 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 Blue).
- Press and hold or tap the 

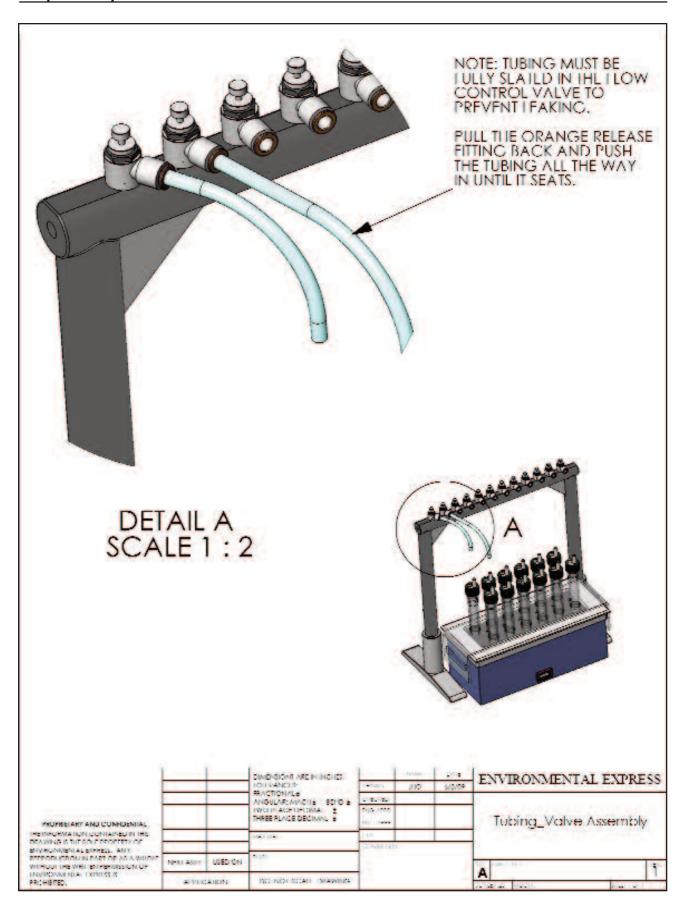
  or 

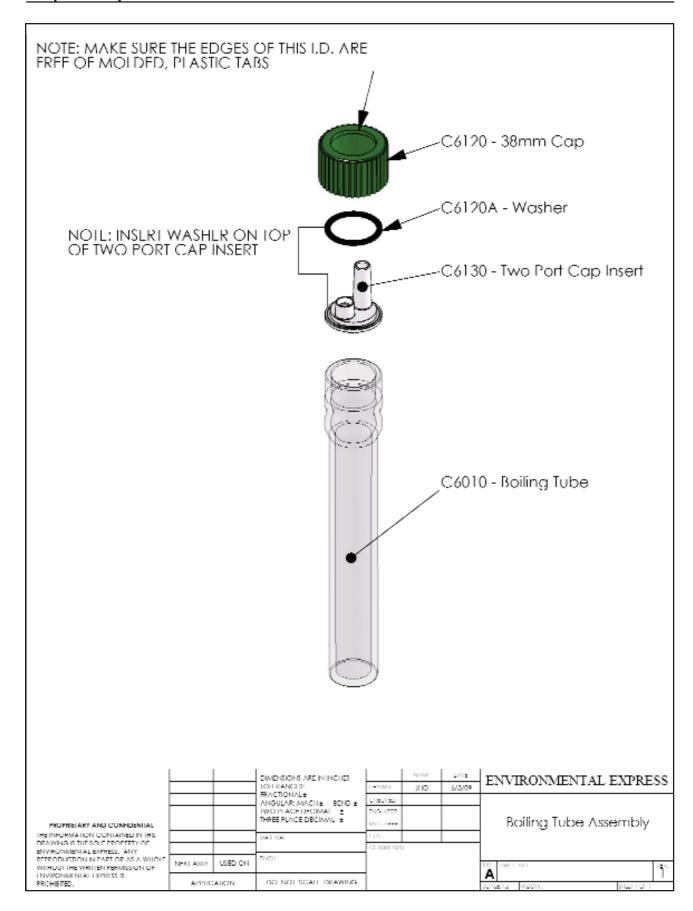
  key. The display will show the set point temperature on the right in blue. The adjustment is from ambient to 150°C in increments of 0.1°C. There is no need to press the green (advance) or 

  button.

# Safe-Sample<sup>™</sup> Temperature Protection:

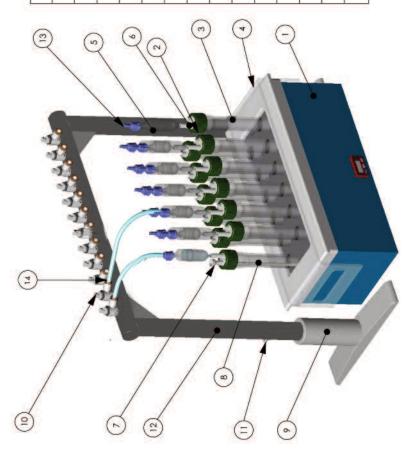
Your HotBlock is protected from runaway temperatures by a fail-safe alarm system. In the unlikely event that the heating system fails to respond to the controller, the Safe-Sample™ system will automatically shut the system off and sound an audible alarm. This alarm sequence occurs if the actual temperature 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 trouble shooting section of your HotBlock manual.





# SIMPLE DIST

	C6142	TUBING KIT, 12 PIECES
	C6002	12-WELL BLOCK ASSY
2	C6120	38MM OPEN TOP ENCLOSURE, POLYPROPYLENE
്ന	C6010	BOILING TUBE, BOROSILICATE GLASS, 38MM THREAED TOP
4	C6050	12 PLACE TUBE RACK, ACRYUC
5	C6100	25mL COLLECTION TRAP
9	C6130	TWO PORT CAP INSERT
7	C6110B	FUNNEL TIP
00	C6110A	PP TUBING - CUT TO 7" LONG
6	C6007B	SUPPORT BASE FOR 12-PLACE MANIFOLD
10	C6005	FIOW CONTROL VAIVE, SMC P/N AS3211F-NO2-11S
Ξ	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



# Total Cyanide Distillation with the SimpleDist™:

### 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  $\mu$ g/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. 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.

### 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.

### 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 1g 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.

- 5.3 Assemble inlet tube with funnel tip and insert into the smaller port on cap.
- 5.4 Pipette 25mL of 0.25N NaOH into the collection trap. 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 and 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 5mL 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 2mL. Allow a few minutes to mix.
- 5.9 Slowly add 5.0mL of 18N sulfuric acid though the reagent inlet.
- 5.10 Turn the heat ON and set the temperature of the heating block to 125°C. Heat for one hour after the block achieves this temperature (30 minutes).
- 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 The distilled cyanide in the cyanide trap is now ready for analysis. Pour contents into appropriate container. When pouring position the vent tube of the cyanide 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.

SimpleDist System Ammonia Distillation

# Ammonia Nitrogen Distillation with the SimpleDist™:

### 1.0 SCOPE AND APPLICATION

1.1 This method follows both Standard Methods 4500-NH3 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 – 2.0 mg/L NH3 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 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.

### 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.

### 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 0.04N Sulfuric acid catch solution 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.

### 5.0 PROCEDURE

- 5.1 Adjust an aliquot of at least 25 mls of sample to a pH of 9.5 using 1N Sodium hydroxide and remove any residual chlorine.
- 5.2 Pipette 25 mls of pH-adjusted sample or an aliquot diluted to 25 mls with reagent water into the SimpleDist System boiling tube. For solids weigh 1g +/- 0.01g and dilute to 25 mls. 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 Pipette 15 20 mls of the proper catch solution into the Collection Trap. Attach the collection trap to the boiling tube and attach the tubing/adaptor from the vacuum manifold to the collection trap.

- 5.6 Repeat steps 5.1 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 1.25 mls 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. Heat for 60 minutes after the HotBlock achieves this temperature.
- 5.10 Remove the collection trap from the assembly. IMPORTANT: VACUUM MUST REMAIN ON!
- 5.11 Turn air/vacuum valves to the OFF position.
- 5.12 Bring the volume of distillate in the collection trap up to the 25 ml mark with the appropriate catch solution.
- 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 After use clean exterior surfaces with a damp sponge. For acid spills sponge with a diluted solution of sodium bicarbonate followed by distilled water.

SimpleDist System Trouble Shooting

# SimpleDist System Trouble Shooting Guide:

### 1.0 SITUATION: The tubes from the manifold to the trap are falling out of the manifold.

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

### 2.0 SITUATION: The sample is boiling over or squirting out of the reagent addition tube.

- 2.1 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 insure 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.
- 2.2 Check the volume of the sample you are using as well. For cyanide distillation you should be using 50 mls of sample. For ammonia distillation you should be using 25 mls of sample.
- 2.3 Insure 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.

### 3.0 SITUATION: Recoveries are too low.

- 3.1 Check the samples for any interferences per the appropriate method.
- 3.2 Insure 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.

### 4.0 SITUATION: Recoveries are too high.

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

### 5.0 SITUATION: I cannot turn the vacuum completely off using the valves on the manifold.

5.1 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.