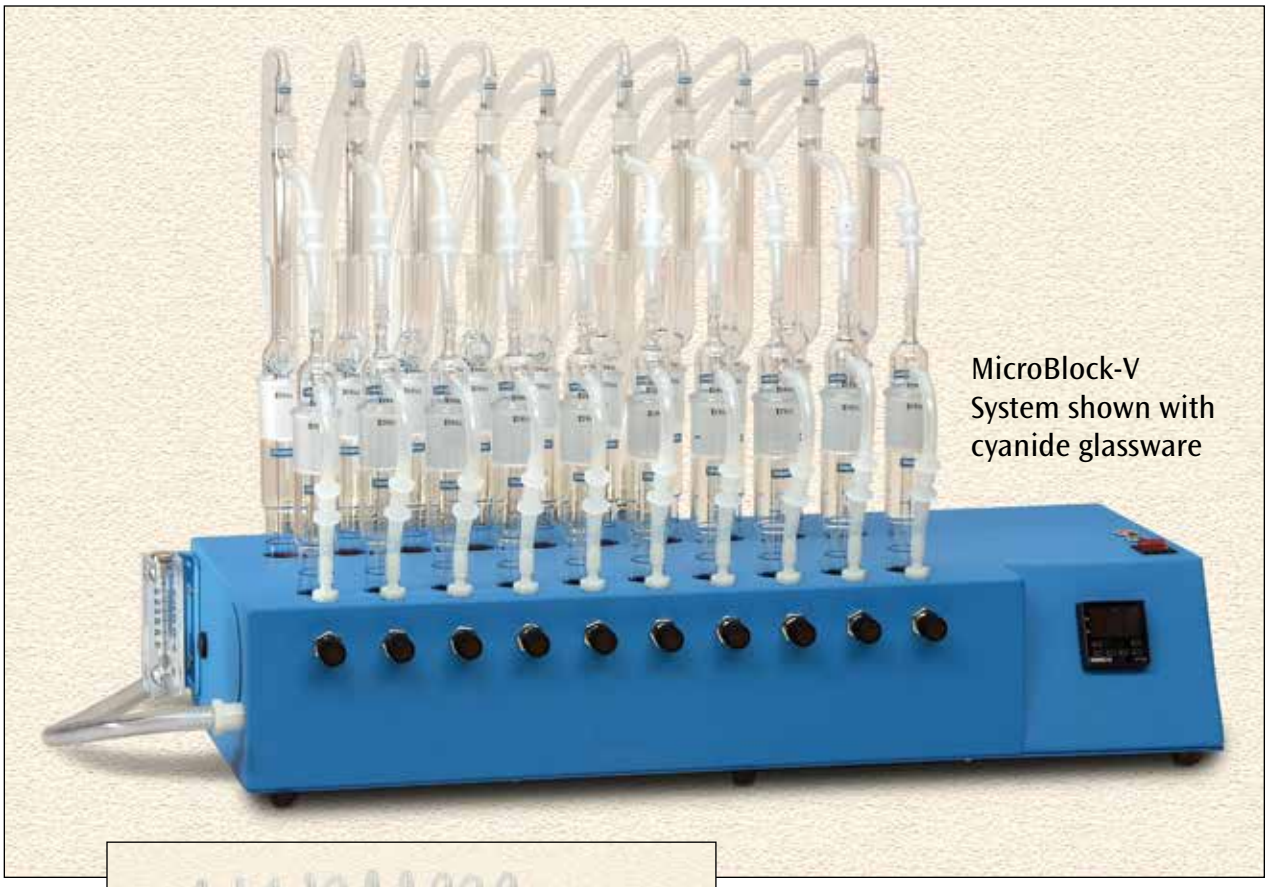
MicroBlock™ Distillation Systems
Operation & Instruction Manual



MicroBlock-V
System shown with
cyanide glassware

MicroBlock-R
System shown with
ammonia glassware

ENVIRONMENTAL EXPRESS®
a Cole-Parmer company

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

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Contents

Limited Warranty

The MicroBlock 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 MicroBlock should fail to operate within the warranty period (one year from date of shipment) Environmental Express, Inc. will repair 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 MicroBlock 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 MicroBlock System.

Out of Warranty Repair

If the MicroBlock 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. A loaner MicroBlock MAY be available during the repair period. There are only a limited number of these units. A reasonable charge for "cleanup" will be charged if a loaner is issued. The customer will be responsible for all shipping charges associated with a loaner unit.

Product Information:

Item # Date of Purchase

MicroBlock™ Serial #

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

Safety Information

1. The Environmental Express MicroBlock 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 MicroBlock in the vicinity of combustible material.
4. Be certain the MicroBlock power cord is properly grounded by consulting your in-house electrician.
5. During operation the surfaces around the heater assembly will get HOT. Do not touch the outer surface.
6. Do not move the MicroBlock system while hot.
7. Do not attempt to operate the MicroBlock system over 190°C.
8. Allow the MicroBlock to cool for 20 minutes before removing glassware. Separation of the "hot" glassware components could result in bumping, boil over, and/or spraying of hot corrosive/caustic materials.
9. Review Material Safety Data Sheets for all materials used or generated during the operation of the MicroBlock. Avoid breathing any vapors that may come off of the MicroBlock system, they may be harmful or fatal.
10. Water and vacuum (if used) should be continued until the MicroBlock has cooled down to prevent boil over. The electric cord should be kept plugged into its outlet until the unit has cooled down.
11. If boil over does occur during operation of the MicroBlock operation, immediately wipe the system down with neutralizing solution, such as a mild solution of sodium bicarbonate.
12. Unplug the MicroBlock 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. If a MicroBlock system with a vacuum is used, install an excess gas trap in the vacuum line to remove excess vapors.
14. Use micro-porous boiling chips in each distillation flask to prevent bumping.
15. It is recommended that a tray or petri dishes be used under the drain outlets to catch any spills from boil over and/or breakage.

Caution: 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.

Parts Included

MICROBLOCK HEATING UNIT

Includes:
Heater Block, Temperature Controller with Timer, Fuse Holder with 15 amp Fuse, Flowmeter, Power Relay, Fan with Thermostat, Water Manifold with Quick Disconnect Fittings, High Temperature Switch and Glass Breakage Drain Outlets.

VACUUM FRONT SECTION

Includes:
(if purchased for cyanide operation) Vacuum Valves, Vacuum Barbs and Vacuum Manifold.
If purchased for ammonia/phenol/fluoride operation only, vacuum components are not included.

MANUAL

Includes:
Operating Instructions with Wiring Diagram and Temperature Controller Operating Instructions.

COMPLETE TUBING SET

Includes:
20 Water Manifold-to-Cold Finger Tubes with Quick Disconnect fittings, 10 Reaction Flask-to-Absorption Impinger Tubes (if cyanide glassware purchased), 10 Absorption Flask-to-Vacuum Tubes (if cyanide glassware purchased), 1 Manifold Water Inlet Tube, 1 Manifold Water Drain Tube and 1 Vacuum Tube (if cyanide glassware purchased).

GLASSWARE

Includes:
If cyanide glassware purchased: 20 Reaction/Absorption Flasks, 10 Distillation Heads, 10 Cold Fingers and 10 Absorption Impingers.
If Ammonia/Phenol/Fluoride glassware purchased: 10 Reaction Tubes, 10 Distillation Heads, 10 Cold Fingers, 10 Receiver Tubes, 10 Ammonia Outlet Tubes and 10 Phenol/Fluoride Outlet Tubes.

BOILING CHIPS:

Includes:
A small sample of boiling chips, Environmental Express # C5249.

Parts Not Included

TRAY OR PETRI DISHES:

It is recommended that a tray or two petri dishes or equal be used under the drain outlets.

MicroBlock™ Cyanide Blocks and Systems

Part number	*Description:*
C5200	MicroBlock-V system (air/vacuum manifold) with 10 sets of cyanide glassware, 120 VAC
C5200-240	MicroBlock-V system (air/vacuum manifold) with 10 sets of cyanide glassware, 240 VAC (CE marked)
C5201	MicroBlock-V block only, 120 VAC
C5201-240	MicroBlock-V block only, 240 VAC (CE marked)

MicroBlock™ Ammonia/Phenol Blocks and Systems

Part number	*Description:*
C5203	MicroBlock-V system (air/vacuum manifold) with 10 sets of ammonia/phenol glassware, 120 VAC
C5201	MicroBlock-V block only, 120 VAC
C5230	MicroBlock-R system with 10 sets of ammonia/phenol glassware, 120 VAC
C5230-240	MicroBlock-R system with 10 sets of ammonia/phenol glassware, 240 VAC (CE marked)
C5231	MicroBlock-R block only, 120 VAC
C5231-240	MicroBlock-R block only, 240 VAC (CE marked)

Glassware and Parts

Part number	*Description:*
C5012	Reaction/absorption flask for MicroBlock-V cyanide distillation
C5210	Cold finger for distillation glassware
C5212	Cyanide glassware for MicroBlock-V, 1 unit (without tubing)
C5213	Ammonia/phenol pierced cap, 10 pk
C5214	Ammonia/phenol glassware for MicroBlock-V/-R, 1 unit (without tubing)
C5214B	Ammonia/phenol reaction tube, skirted
C5216	Cyanide distillation head, Style 2
C5217	Absorption impinger for cyanide
C5218	PTFE-faced septa for distillation cap, 20 pk
C5219	Ammonia/phenol distillation head
C5221	Tubing and quick-connect fittings for cyanide glassware, 10 sets
C5222	Tubing and quick-connect fittings for ammonia/phenol and cyanide glassware, 10 sets
C5225	Silicon sealing ring, 10 pk
C5226	Outlet tube for ammonia distillation, long stem
C5227	Outlet tube for phenol distillation, short stem
C5228	Receiver tube for ammonia/phenol distillation
C5249	Carbon boiling chips, 8 oz
C5253	Upgrade heater mat replacement kit for MicroBlock
C5257	Replacement fan for MicroBlock
C5260	Female quick-disconnect body for water manifold, 2 pk
C5261	Needle valve for MicroBlock-V

Unpacking and Setup

I. Unpack the heating unit and place it in a chemical fume hood with the temperature controller facing outward.

2. Unpack 10 glassware sets. The glassware is packed 5 sets per box. The glassware should be cleaned according to the protocol and assembled in the MicroBlock unit. If the chemistry being done requires the use of a frit, refer to page 12, paragraph 7.17 for frit cleaning.

3. Unpack the bags of tubing which were packed with the heating unit.

4. Review the diagrams. 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 to be used if item needs to be returned for service. Refer to warranty policy on page 1.

6. Connect tubing to the MicroBlock unit as follows:

   A. Connect a 5-foot reinforced tube to the hose barb on the outlet water manifold (right side, away from flowmeter). Place the other end into a suitable drain.

   B. Connect the other 5-foot reinforced tube to the lower flowmeter hose barb and the other end to a suitable needle valve cold water source. This needle valve is used to control the flow of water and is not supplied with the MicroBlock.

   C. If the chemistry is for Cyanide, connect the 5-foot vacuum tube to the white hose barb located on the left side of the unit (behind the vacuum knobs). Connect the other end to an excess gas trap (scrubber flask filled with NaOH solution). Connect gas trap to your vacuum source.

   Note: The excess gas trap and vacuum source are not supplied with the MicroBlock.

7. Assemble the glassware into the MicroBlock in the following manner (reference Figures 2 - 4)

   Note: Names used here are for the Cyanide design of glassware.

   A. Place a Reaction/Absorption Flask into the left-most heat block position. This is the Reaction Flask.

   B. Place the Distillation Head into the Reaction Flask.

   C. Place the Cold Finger into the joint at the top of the Distillation Head.

   D. Place a Reaction/ Absorption Flask into the left-most front position. This is the Absorption Flask.

   E. Place the Absorption Impinger into the Absorption Flask.

   F. Repeat steps A through E for the remaining glassware, working from left to right.

   G. To remove the glassware, reverse the above procedure.

8. Connect tubing to the glassware and MicroBlock as follows:

   Note: 20 Manifold-to-Cold Finger tubes are supplied at the same lengths. These tubes are long enough to remove the Cold Finger from the Distillation Head without disconnecting the Quick Disconnect Fitting. You may find them long if you are going to use the Quick Disconnect Fitting to remove the Cold Finger, if so the tubing can be easily cut.

   A. The Cold Finger tubing lines are the lines having a quick disconnect fitting attached. Connect one end of a line to the upper barb on the Cold Finger. Insert the Quick Disconnect Fitting into the forward adapter on the water manifold (water inlet), squeezing the metal tab first. Push in until the tab clicks out. Repeat the process for the other nine Lower Cold Finger Condensers.

   B. Repeat step A using the other 10 tubing lines going to the lower Cold Finger barb and the back adapter on the water manifold (water drain).

   C. The Distillation Head-to-Absorption Impinger tubing bag contains ten lengths with a slip connector in the middle. Connect one open end to the barb at the top of the Distillation Head, and the other end to the barb on the Absorption Impinger .

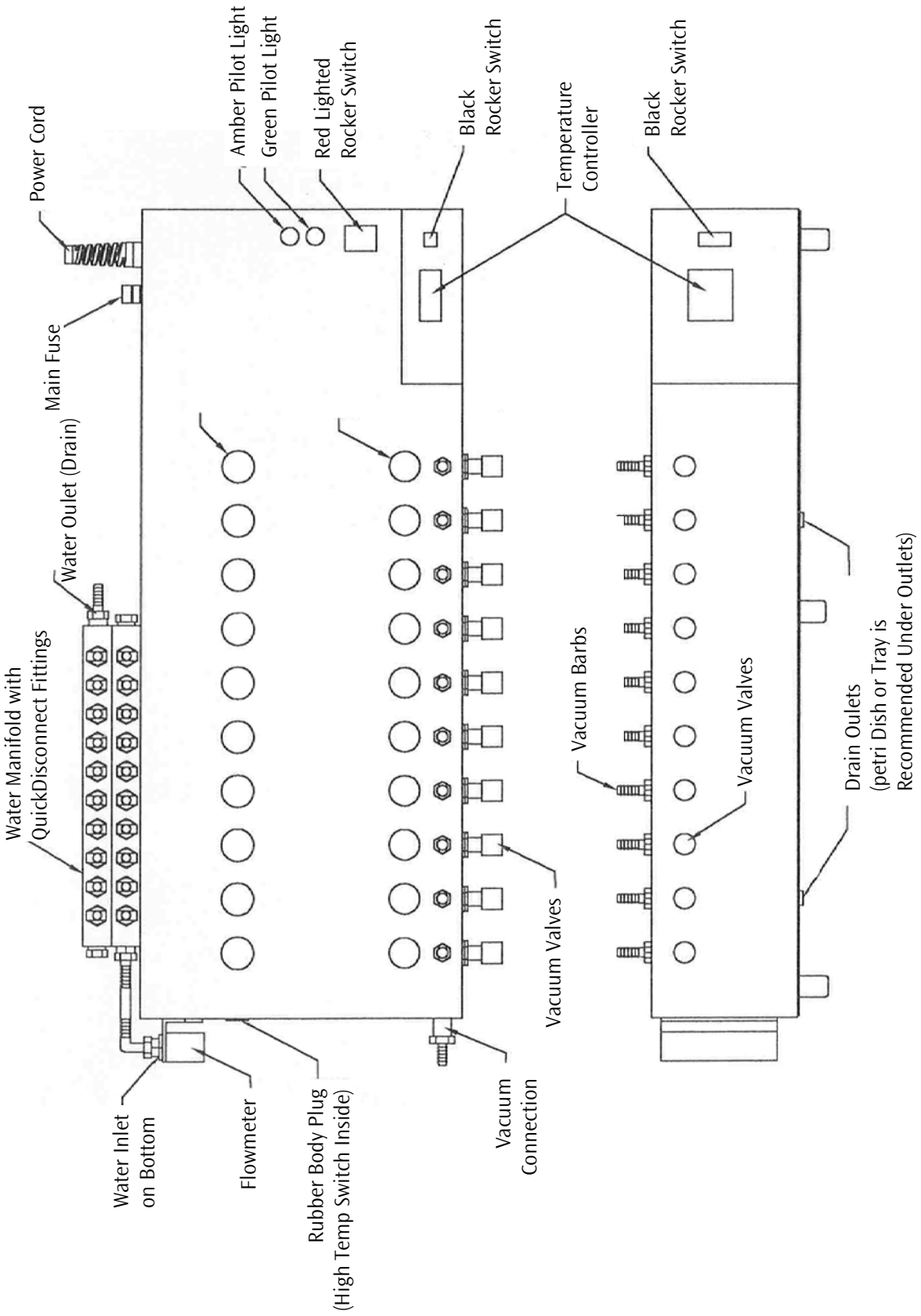
   D. The Absorption Impinger-to-Vacuum tubing bag contains ten lengths, each with a slip connector near one end. Connect the longer tube to the side barb on the Absorption Impinger, and the shorter tube to the vacuum barb on the MicroBlock unit.

9. Plug the power cord into an outlet rated for 15 amps. If the Red Rocker Switch glows, turn it off.

Instrument Controls and Description

Control	*Location*	*Description*
Red Lighted Rocker Switch	Top Right on Unit	A DPST switch that turns power on and off. ***Note: This switch does not control the cooling fan.***
Black Rocker Switch	Right Side of Temperature Controller	Enables the heater block circuit. When turned on, the Green Pilot Light is illuminated and heating is enabled. When turned off, the Green Pilot Light is off and the heater is disabled. The Temperature Controller will remain on, temperature to be monitored during idle or cooldown periods.
Green Pilot Light	Top Right on Unit	Operates when the power is turned on by the Red Lighted Rocker Switch and heating is enabled by the Black Rocker Switch. If the Green Pilot Light does not illuminate when Red Lighted Rocker Switch is illuminated and the Black Rocker Switch is turned on, the High Temperature Switch is not closed and must be re-set.
Amber Pilot Light	Top Right on Unit	Operates during the heating cycle. Light turns on and off indicating heating element is getting power.
Temperature Controller/Timer	Front Right on Unit	Controls the operation of the heater block up to its maximum temperature of 190°C. Turns the power on and off to the heating elements and can be programmed to heat at slower rate than maximum and programmed to automatically shut the power off at the selected time. See Temperature Controller Operating Instructions on page 8.
Main Fuse Holder	Right Rear of Unit	A 15 amp fuse must be in place for unit to operate. ***Note: The Temperature Controller and Fan are fused separately.***
High Temp Switch	Left End of Heater Block Inside Unit	Protects unit from burnout caused by failure of the Temperature Controller and/or Power Relay. May be re-set by removing rubber body plug on side of unit and pushing the re-set button inside of unit. Always use a non-metallic device to make this adjustment.
Power Relay	Right Rear Inside	Controls power between the temperature controller and the heating elements.

Figure 1 - Wiring Diagram



Simplified Temperature Controller Instructions

Front Panel Key Functions

The decimal point flashes when Self Tune is operating

Keys are illuminated when pressed. Key functions are as follows:

INDEX: Menu Navigation. Pressing the INDEX key advances the display to the next menu item. May also be used in conjunction with other keys as noted below.

UP ARROW: Increments a value, changes a menu item, or selects the item to ON. The maximum value obtainable is 9999 regardless of decimal point placement.

DOWN ARROW: Decrements a value, changes a menu item, or selects the item to OFF. The minimum value obtainable is -1999 regardless of decimal point placement.

ENTER: Pressing ENTER stores the value or the item changed. If not pressed, the previously stored value or item will be retained. The display will flash once when ENTER is pressed.

AUTO/MANUAL (CT16A3): This key toggles the control output between Automatic mode and Manual mode. Press and hold key for three seconds to activate. See section on AUTO/MANUAL operation on Page 9.

RUN/HOLD (CT16A3): This key toggles the Ramp/Soak program functions between Run mode (program runs as set up) , and Hold mode (program functions are suspended). Press and hold key for three seconds to activate. See section on Ramp/Soak (Page 9) for further details.

UP ARROW & ENTER: Menu Access. Pressing these keys simultaneously brings up the secondary menu starting at the alarm, tune, or cycle item (depending on programming). Pressing these keys for 5 seconds will bring up the secure menu.

INDEX & DOWN ARROW: Menu navigation. Pressing these keys simultaneously will allow back-up of one menu item, or if at the first menu item, will cause the display to return to the primary menu.

Manual Operation

1. Press the INDEX key to change to set-point display, the SV display will have SPI displayed.
2. Change the process set point (PV display) by pressing the UP ARROW or DOWN ARROW key to change the value, and press the ENTER key to retain it.
3. Press the INDEX key to return to the process / set point display. The unit will heat to this temperature and hold.

Automatic Operation

Note: These instructions are for a ramp, hold and shut off, you can have up to 16 ramps and hold settings. The example is for a 20 minute ramp to 125°C hold at 125°C for 60 minutes and turn off.

1. Press the UP ARROW & ENTER keys simultaneously for one second only to get into the Secondary Menu.
2. Press the INDEX key 10 times until the SV display shows PROg. If the PV display is ON it must be turned OFF by pressing the UP ARROW or DOWN ARROW key and press ENTER to retain it.
3. Press the INDEX key once until PSET is displayed.
4. Press the UP ARROW key to turn the Program Function ON if it is not on and press ENTER to retain it.
5. Press the INDEX key once, the SV display should now be STAT. Press the UP ARROW to turn on the Primary Menu if it is not ON and press ENTER to retain it.
6. Press the INDEX key once, the SV display should now be TBAS. Press the UP ARROW to change the Ramp/Soak Time Base to minutes. The PV display should now be 60 S, press ENTER to retain the setting.
7. Press the INDEX key once, the SV display should now be IT. Press the UP ARROW or DOWN ARROW to set 20minutes for the first segment and press ENTER to retain the setting.
8. Press the INDEX key once, the SV display should now be ISP. Press the UP ARROW or DOWN ARROW to set 125 value for the temperature and press ENTER to retain the setting.
9. Press the INDEX key once, the SV display should now be ZT. Press the UP ARROW or DOWN ARROW to set 60 minutes for the (hold) second segment and press ENTER to retain the setting.
10. Press the INDEX key once, the SV display should now be ZSP. Press the UP ARROW or DOWN ARROW to set 125 value for the hold temperature and press ENTER to retain the setting.
11. Press the INDEX key 29 times until the SV display has PEND on it. Press the UP ARROW or DOWN ARROW key until the display shows 0OFF (Program End Action) and press ENTER to retain the setting.
12. Return to the HOME position by waiting for the Secondary Menu display to time out or by pressing the UP ARROW & ENTER keys and then the DOWN ARROW & INDEX keys.
13. Press the UP ARROW & ENTER keys to get into the Secondary Menu.
14. Press the INDEX key 10 times until the SV display is PRO9. Press the UP ARROW or DOWN ARROW key to turn ON Ramp/Soak Feature and press ENTER to retain the setting.
15. Return to the HOME position by waiting for the Secondary Menu display to time out or pressing the UP ARROW & ENTER keys and then the DOWN ARROW & INDEX keys
16. The unit when turned on will be in the hold mode. To run the program press the HOLD key for 3 seconds. The unit will now heat to 125°C in 20 minutes, hold at 125°C for 60 minutes and turn the heat off.
17. To re-start the program, turn the main switch off and on, press the RUN/HOLD key for 3 seconds.

Total Cyanide Distillation with the MicroBlock™

1.0 SCOPE AND APPLICATION

1.1 This method replicates 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, as well as, domestic and industrial wastes, and soils.

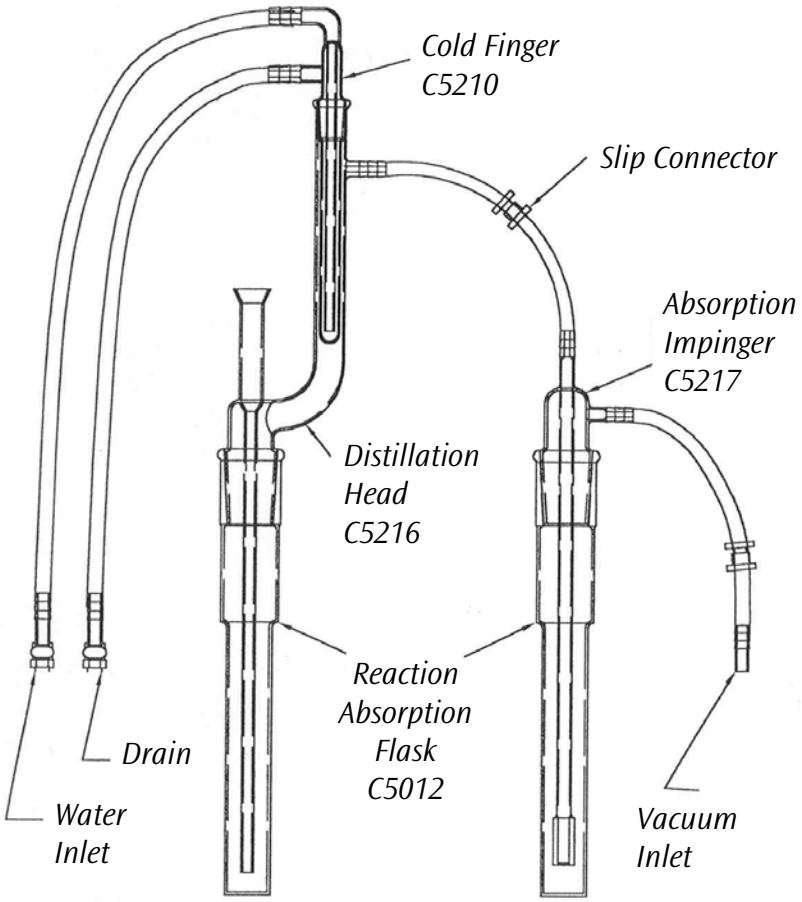
1.2 The standard range is typically 5 to 500µg/L. Lower detection limits can be achieved by using a longer path length flow cell in the analysis step when using an automated continuous flow analyzer.

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.

Figure 2 - Cyanide Glassware



3.0 INTERFERENCES

There are several known interferences with this method. A few of these are Aldehydes, Thiocyanates, Nitrate-nitrite, Thiosulfates, Chlorine, and 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 335.4.

Total Cyanide Distillation with the MicroBlock™ (Continued)

4.0 CHEMICALS REQUIRED – DISTILLATION ONLY

Caution: The toxicities 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.

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

4.8 Boiling Chips – Environmental Express # C5249

5.0 EQUIPMENT AND SUPPLIES

5.1 The MicroBlock distillation glassware setup and tubing connections are shown in Figure 2 (Cyanide Style #1).

Part Number	*Description*
C5012	Reaction/Absorption Flask
C7110	Snip & Pour® Premeasured Reagent, 1.25N NaOH, 50 x 10mL
C5210	Cold Finger
C5217	Absorption Impinger
C5216	Distillation Head

5.2 Helpful Accessories (Part# BT844004)
5mL delivery Repipette for 18N H_2SO_4 addition
5mL delivery Repipette for Mg_2Cl addition

6.0 REAGENT PREPARATION – DISTILLATION ONLY

6.1 Sodium Hydroxide Solution, Stock, 1.25N
Dissolve 50g of NaOH in reagent water and dilute to 1L.

6.2 Sodium Hydroxide Solution, Working, 0.25N
Dilute 200mL of 1.25N stock solution to 1L with reagent water.

6.3 Sulfuric Acid, 18N
Place a 1L glass container in an ice bath with a magnetic or mechanical stirrer. Add 500mL of reagent water. Slowly add 500mL of concentrated Sulfuric Acid drop-wise while stirring. Replenish ice in the ice-bath if required. Exercise caution when preparing this reagent because of the exothermic nature of the reaction. After all the sulfuric acid has been added, dilute to 1L with reagent water and mix thoroughly. Cool to room temperature before use.

6.4 Magnesium Chloride Reagent
Dissolve 510g of $MgCl_2$:6 H_2O in reagent water and dilute to 1L.
Note: This reagent is required if it is known that refractory iron-cyanide complexes are present.

6.5 Sulfamic Acid
Dissolve 20g of reagent grade sulfamic acid in reagent water and dilute to 1L.
Note: *This reagent is required if it is known that nitrates-nitrites are present.*

Total Cyanide Distillation with the MicroBlock™ (Continued)

6.6 Potassium Cyanide, Stock Standard
Dissolve 2.51g of KCN in reagent water containing 2.0g of KOH. Dilute to 1L. Concentration of 1 mL = 1mg CN. Standardize with 0.0192 N $AgNO_3$ on a monthly basis. Refer to US EPA method 335.4, paragraph 7.9 for the $AgNO_3$ preparation.

6.7 Potassium Cyanide, Intermediate Standard
Dilute 10mL of Stock Cyanide Standard to 100mL with reagent water. Concentration of 1mL = 100.0µg CN.

6.8 Potassium Cyanide, Working Standard
Prepare a series of standards covering the working range. Dilute 10mL of the Intermediate Standard to 100µL with reagent water. Concentration of 1mL = 10µg CN. Prepare fresh daily or before each use.

7.0 PROCEDURE

7.1 Pipette 50mL of sample or an aliquot diluted to 50mL with reagent water into the MicroBlock Reaction Flask. Add a pinch of boiling chips. For solid samples weigh 1g or less to the nearest 0.01g, dilute to 50mL and add boiling chips.

7.2 Pipette 50mL of 0.25N NaOH into the Absorption Flask. If using the Snip & Pour concentrate pour the entire 10mL of the 1.25N NaOH into the absorption flask. Dilute to 50mL using DI Water. Refer to the schematic (Figure 2, page 10) for the appropriate placement positions and flexible tubing connections to cool the Cold Finger with running tap water. All air/vacuum valves are in the OFF position.

7.3 Repeat steps 7.1 and 7.2 for all samples to be distilled up to a maximum of 10.

7.4 Position the Cold Finger and Absorber Impingers.

7.5 Turn tap water ON after assuring all tubing connections are firmly placed. A water flow rate of 50 to 60 gallons per hour is required.

7.6 Turn ON the vacuum and slowly adjust each valve to provide an air flow bubble rate of several bubbles per second for each position as viewed in the absorption vessel.

7.7 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.

7.8 Slowly add 5.0mL of 18N sulfuric acid though the reagent inlet.

7.9 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.

7.10 Turn the heat ON and set the temperature of the heating block to 150°C.

7.11 Set the timer for 1 hour.

7.12 Timer will automatically turn OFF power to the heater block at the pre-selected time.

7.13 Disconnect the Distillation Head and Absorption Impinger by separating the Slip Connector. Turn air/vacuum valves to the OFF position. Repeat for each sample position.

7.14 Allow the tap water to continue circulating through the Cold Fingers which will cool the heater block more rapidly in preparation for the next run.

7.15 The distilled cyanide in the 0.25N NaOH solution is now ready for analysis.

7.16 Immediately after the run is finished, place the Absorption Impinger in distilled water. Do not let the impinger dry out since the NaOH may clog the frit.

7.17 If the frit becomes clogged, clean with concentrated HCl using an ultrasonic bath. If this procedure is not successful use aqua regia.

7.18 After use clean exterior surfaces with a damp sponge. For acid spills sponge with a diluted solution of sodium carbonate followed by distilled water.

Ammonia Distillation with the MicroBlock™

1.0 SCOPE AND APPLICATION

1.1 This method covers the determination of ammonia in drinking, ground, surface, and saline waters and domestic and industrial wastes.

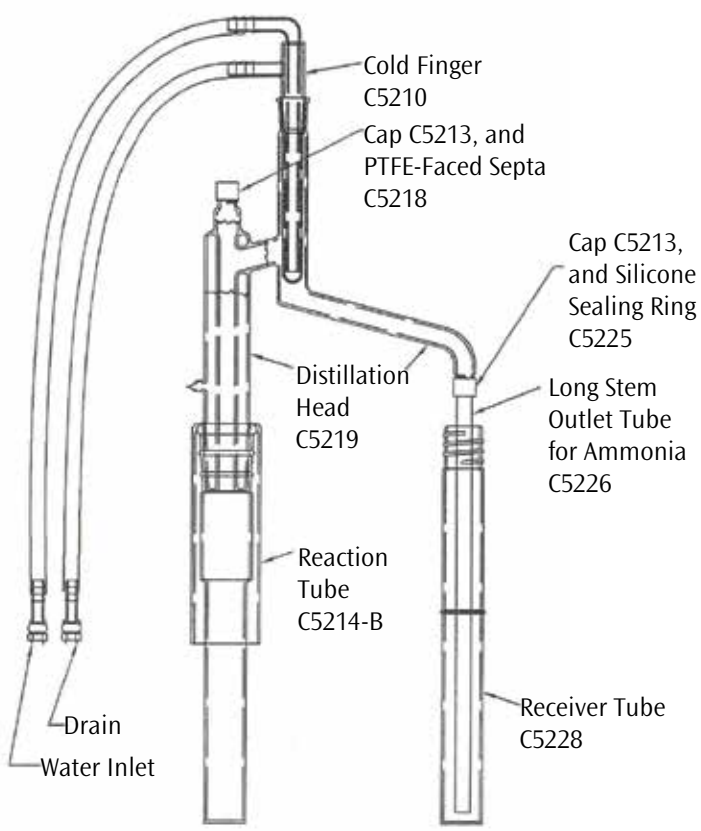
1.2 The applicable range is 0.01 to 2.0mg/L ammonia as N. Higher concentrations can be determined by dilution.

2.0 SUMMARY OF METHOD

2.1 The sample is buffered at pH 9.5 with borate buffer for aqueous samples to decrease hydrolysis of cyanates and other organic nitrogen compounds. Under acidic conditions these compounds can form ammonia. The sample is distilled into 0.04N sulfuric acid or 2% boric acid depending upon the analytical technique used to quantitate for ammonia.

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.

Figure 3 - Ammonia Glassware



3.0 INTERFERENCES

3.1 Cyanate in certain industrial wastes will hydrolyze to some extent even at pH 9.5, which is recommended for distillation.

3.2 Residual chlorine must be removed prior to distillation by treatment with sodium thiosulfate or sodium sulfite.

4.0 CHEMICALS REQUIRED – DISTILLATION ONLY

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

4.1 Sodium Hydroxide

4.2 Sodium Tetraborate

4.3 Sulfuric Acid

4.4 Sodium Thiosulfate

4.5 Sodium Sulfite

4.6 Ammonium Chloride

Ammonia Distillation with the MicroBlock™ (Continued)

4.7 Reagent Water ASTM Type II or Equivalent, especially ammonia-free
Note: Such water is best prepared by passage through an ion-exchange column containing a strongly acidic cation exchange resin mixed with a strongly basic anion exchange resin.

4.8 Boiling Chips, Environmental Express # C5219

5.0 EQUIPMENT AND SUPPLIES

5.1 The MicroBlock distillation glassware setup and tubing connections are shown in Figure 3 with long outlet tube.

Part Number	Description
C5214B	Reaction Tube, Skirted
C5219	Distillation Head
C5210	Cold Finger
C5228	Receiver Tube
C5226	Long Stem Outlet Tube
C7010	Snip & Pour Premeasured Reagent, 0.2N H_2SO_4, 50 x 10mL
C7120	Snip & Pour Premeasured Reagent, 20g/L Boric Acid, 50 x 10mL

6.0 REAGENT PREPARATION – DISTILLATION ONLY

6.1 Sodium Hydroxide, 0.1N

Dissolve 4g of NaOH in reagent water and dilute to 1L.

6.2 Sodium Tetraborate Solution, 0.025 M

Dissolve 5.0g of $Na_2B_4O_7$ [anhydrous] or 9.5g of $Na_2B_4O_7$:$10H_2O$ and dilute to 1L with reagent water.

6.3 Borate Buffer Solution

Add 88mL of 0.1N Sodium Hydroxide solution to 500mL of 0.025 M sodium tetraborate and dilute to 1L with reagent water.

6.4 Sodium Thiosulfate Solution

Dissolve 3.5g $Na_2S_2O_3$: $5H_2O$ to 1L of reagent.

Note: One mL of this solution will remove 1mg/L of residual chloride in 500mL of sample.

6.5 Sodium Sulfite Solution

Dissolve 0.9g Na_2SO_3 :$5H_2O$ to 1L of reagent water.

Note: One mL of this solution will remove 1mg/L of residual chlorine in 500mL of sample.

6.6 Stock Ammonium Chloride

Dissolve 3.819g of anhydrous ammonium chloride, previously dried at 105°C, and dilute to 1L with reagent water.

1.0mL = 1.0mg NH_3-N.

6.7 Standard Ammonium Chloride, Solution A

Dilute 10.0mL of Stock Solution to 1L with reagent water.

1.0mL = 0.01mg NH_3-N.

6.8 Standard Ammonium Chloride, Solution B

Dilute 10.0mL of Stock Solution A to 100mL with reagent water.

1.0mL = 0.001mg NH_3-N.

Ammonia Distillation with the MicroBlockTM (Continued)

7.0 PROCEDURE

7.1 Preparation of distillation glassware. Steam out the distillation glassware using 50mL reagent water with a pinch of boiling chips until no trace of ammonia can be detected.

7.2 If residual chlorine is present add sodium thiosulfate or sodium sulfite equivalent to the chlorine level.

7.3 To a 50mL sample add 1N NaOH drop-wise with mixing and adjust to pH 9.5.

7.4 Transfer sample to the Reaction Tube, skirted, and add 2.5mL of borate buffer followed by a pinch of boiling chips.

7.5 To the Receiver Tube add 5-10mL of 20g/L Boric Acid or 0.2N H_2SO_4. If using the Snip & Pour concentrate pour the entire contents of the tube in the Reaction Tube. The tip of the long stem must be below the level of boric acid or sulfuric acid.

7.6 Repeat for all samples to be distilled up to a maximum of 10.

7.7 Assemble the distillation glassware setup as shown in Figure 3 (page 13).

7.8 Turn on the tap water to cool the Cold Finger after ensuring all tubing connections are tight. Check for any leaks.

7.9 Turn the power ON and set heater block temperature to 190°C and the timer to 60 minutes.

7.10 Collect a minimum of 30mL of distillate.

7.11 Turn power OFF.

7.12 Unscrew the cap holding the Long Stem in the receiver tube and remove from all distillation positions to prevent any suck back of the distilled sample. When the Long Stem is disconnected, gently let it rest at the bottom of the Receiver Tube.

Total Recoverable Phenolics with the MicroBlock™

1.0 SCOPE AND APPLICATION

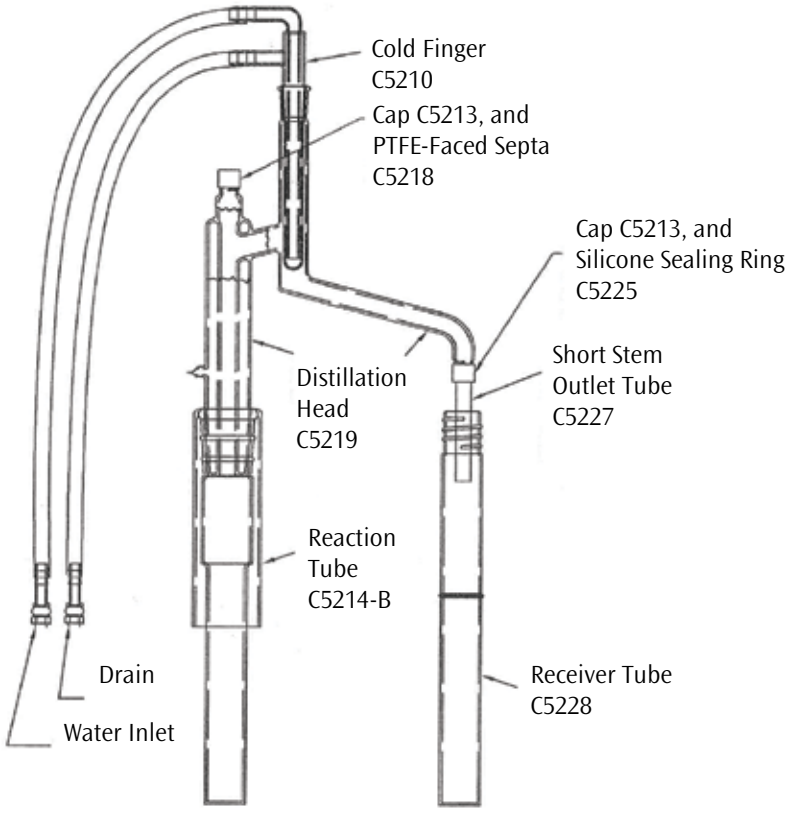
1.1 This method is for the determination of phenolic compounds in drinking, ground, surface, and saline waters, and domestic and industrial wastes.

1.2 The applicable range is 2-500µg/L. The working ranges are 2-200µg/L or 10-500µg/L.

2.0 SUMMARY OF METHOD

2.1 This method is based on the distillation of phenol followed by the reaction with alkaline ferricyanide and 4-aminoantipyrine. The color complex is measured at 505 or 520nm. Whether the analysis is performed by manual or automated continuous flow analyzers, a 50mm path length measuring optical cell is required to achieve a detection limit of 2µg/L.

Figure 4 — Phenol glassware



Total Recoverable Phenolics with the MicroBlock™ (Continued)

2.2 Phenol is used as the standard even though the various forms of phenol react differently with the colorimetric reaction. The result obtained represents the minimum concentration present in the sample.

2.3 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.

3.0 INTERFERENCES

3.1 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH 4.0 and aerating briefly by stirring.

3.2 Oxidizing agents such as chlorine detected by the liberation of iodine upon acidification in the presence of potassium iodide are removed immediately after sampling by the addition of ferrous ammonium sulfate. If chlorine is not removed, the phenolic may be partially oxidized and the results may be low.

3.3 Background contamination from plastic tubing and sample containers must be avoided. Use glass tubes wheneverpossible.

3.4 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other processing apparatus that bias the analyte response.

4.0 CHEMICALS REQUIRED – DISTILLATION ONLY

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

4.1 Sodium Hydroxide

4.2 Sulfuric Acid.

4.3 Ferrous Ammonium Sulfate – if chlorine is present at the time of sampling.

4.4 Phenol

4.5 Reagent Water ASTM Type II or Equivalent

4.6 Boiling Chips, Environmental Express # C5219

5.0 EQUIPMENT AND SUPPLIES

5.1 The MicroBlock distillation glassware setup and tubing connections are shown in Figure 4 with short outlet tube.

Part Number	*Description*
C5214B	Reaction Tube, Skirted
C5219	Distillation Head
C5210	Cold Finger
C5228	Receiver Tube
C5227	Short Stem Outlet Tube

Total Recoverable Phenolics with the MicroBlock™ (Continued)

6.0 REAGENT PREPARATION – DISTILLATION ONLY

6.1 Stock Phenol

Dissolve 0.50g phenol in 500mL reagent water. Add 0.25mL of concentrated sulfuric acid as a preservative.

1.0mL = 1.0mg phenol.

6.2 Standard Phenol, Solution A

Dilute 1.0mL of the Stock Phenol Solution to 100mL with reagent water.

1.0mL = 0.01mg phenol6.3 Standard Phenol, Solution B

Dilute 10.0mL of Standard Phenol Solution A to 100mL with reagent water.

1.0mL = 0.001mg phenol.

6.4 Standard Phenol, Solution C

Dilute 10.0mL of Standard Phenol Solution B to 100mL with reagent water.

1.0mL = 0.0001mg phenol.

6.5 Sodium Hydroxide, 0.1 N [1+9]

Dissolve 4.0g of NaOH in 1L of reagent water.

6.6 Sulfuric Acid, 10% [1+9]

Slowly add 10mL of concentrated sulfuric acid to 70mL of reagent water. Cool and dilute to 100mL.

6.7 Ferrous Ammonium Sulfate

Dissolve 0.55g of ferrous ammonium sulfate in 250mL reagent water containing 0.5mL sulfuric acid and dilute to 500mL.

7.0 PROCEDURE

7.1 Pipette 50mL of sample into a beaker. Adjust pH to 4 with NaOH [1+9] or with Sulfuric Acid [1+9]. Transfer to the skirted Reaction Tube. For soil samples, weigh 1g or less into beaker and 50mL reagent water. Adjust pH to 4 with NaOH [1+9] or sulfuric acid [1+9] while stirring and transfer to the skirted Reaction Tube. Add a pinch of boiling chips.

7.2 Repeat Step 7.1 for all samples to be distilled up to a maximum of 10.

7.3 Assemble the distillation glassware setup as shown in Figure 4 (page 16) and position in the heater block.

7.4 Turn on the tap water to cool the Cold Finger after ensuring all tubing connections are tight. Check for leaks.

7.5 Turn power ON and set heater block temperature to 190°C and timer to 60 minutes.

7.6 When approximately 45mL of sample is distilled add 5mL of reagent water using a syringe by piercing the rubber septum located on the top of the Distillation Head.

7.7 When 50mL of sample is collected in the Receiver Tube remove by unscrewing the cap containing the Short Stem.

7.8 When all samples have distilled turn the power OFF.

7.9 Add 5mL increments of reagent water to the Reaction Tube by piercing the septum using a syringe. Since the heater block is still hot the distillation process will continue. Place a collection tray or beakers to catch any further distillate.

7.10 After each use clean exterior with a damp sponge.

7.11 When the heater temperature falls below 100°C, the next run can be started.

Note: If boil over occurs, add 2-3 drops of Antifoam B agent to the sample prior to distillation.

Troubleshooting Guide

Problem	*Possible Cause*	*Solution*
Temperature Controller Display Blank		
Red Lighted Rocker Switch not lit	Unit turned off	Turn on Red Lighted Rocker Switch
	Unit unplugged or no power to unit	Plug unit into 120 VAC/20A outlet
	Main fuse blown	Replace 15A/250V fuse
	Internal failure	Call for service
Red Lighted Rocker Switch lit	Units with S/N 2056 and below High Temp Switch tripped	Reset High Temp Switch
Green Pilot Light not lit	High Temp Switch tripped	Reset High Temp Switch
	Units with S/N 2057 and above heater turned off	Turn on Black Rocker Switch
	Internal failure	Call for service
Unit does not heat to operating temperature	Green Pilot Light lit	See above
	Process Temperature not set	Set correct operating temperature
	High Temp Switch tripped	Reset High Temp Switch
	Units with s/n 2057 and above heater turned off	Turn on Black Rocker Switch
	High Temp Switch failure	Call for service
High Temp Switch trips repeatedly	Block temperature above safe range	Set operating temperature to 190°C or below
	High Temp Switch failure	Call for service
Block temperature exceeds set value by more than 1°C	Internal failure	Call for service
Temperature Controller displays error message	Refer to Controller Manual (available upon request)	Call for service