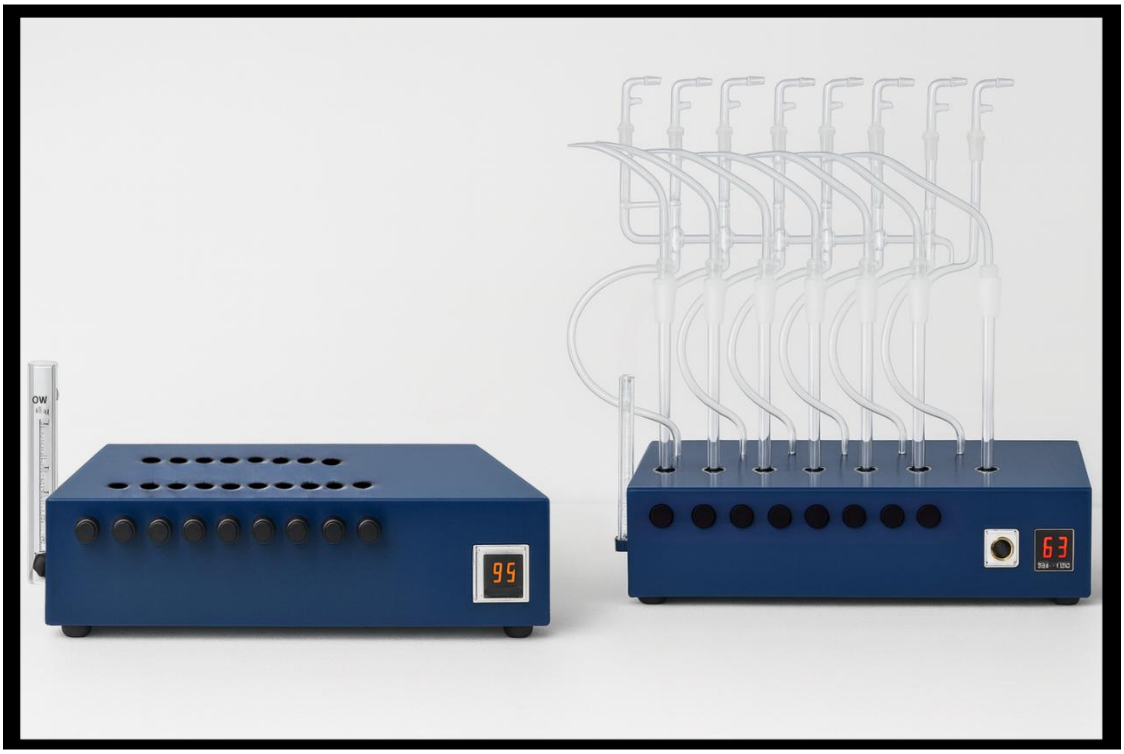


Midi-Distillation System

For 50mL Reduced Sample Volumes
USEPA / 600 Determination by Semi-Automated Colorimetry

USEPA Method	Determination
350.1	Midi-Ammonia/Phenol
420.4	
335.4	Midi-Cyanide

Instruction Manual Number 79521M0000 (Rev. October 2025)

To register your Midi Distillation System, please call customer service within 10 days of receipt. Call toll-free: 1-(800)-955-0376 Ext. 1.

Astraglass Innovations Warranty
We warrant to the original user of this product that it is free from all defects in material and workmanship under normal use and service.

All mechanical and electronic components are guaranteed for a period of **1 year unless otherwise specified.** Should any part prove to be defective within this time as a result of faulty workmanship or material, Astraglass Innovations will, at its option, repair or replace it free of charge.

This guarantee is subject to the conditions set forth under "User's Responsibility." The term "Original User" as used in this guarantee shall be deemed to mean that person, firm, association, or corporation which has made the original purchase of this instrument for his or its own use; and this guarantee shall be void if this instrument is resold. EXCEPT AS SET FORTH HEREIN, NO WARRANTY OF MERCHANTABILITY OR OF FITNESS FOR PURPOSE SHALL APPLY.
Astraglass Innovations assumes no responsibility for the cost of handling, installation, etc., or for transport charges.

Liability
While all Astraglass Innovations products are manufactured to the highest standards, we cannot accept any claims for loss, damage, or injury due to failure to operate as intended. Our responsibility is limited to repair or replacement of defective material as covered by this guarantee.

IN NO EVENT SHALL THE COMPANY BE LIABLE FOR CONSEQUENTIAL OR SPECIAL DAMAGES. This guarantee is in lieu of all other guarantees, expressed or implied, and no person is authorized to assume for us any other obligation or liability in connection with this product.

User's Responsibility - Guarantee Instructions
This product has been manufactured and tested with utmost care in order to provide the user with years of satisfactory service. To get the most satisfactory and economical results from this product, proper care should be taken of it as any other mechanism and periodic inspection should be provided.

The user should make certain that, in its installation, this product has been fully protected against improper electrical current, fluctuating voltages, or low voltages. Failures due to these conditions or any other condition not attributable to defective workmanship or materials are excluded from our guarantee.

The user is liable for any repairs or reworking due to the unit having been subjected to misapplication, misuse, damage or abusive tampering. Warranty repair units must be returned intact and are subject to inspection.

Installation of replacement parts other than standard **Astraglass Innovations** parts, or removal or defacement of the serial data plate, voids the entire guarantee.

If this product is not operating properly, call customer service at **1-(800)-955-0376 Ext. 118.**
for instructions on returned material authorization procedure. After receiving authorization to return your product, it should be carefully packed, using shock absorbing material, and insured, since **Astraglass Innovations** cannot assume responsibility for inadequate packing and damage in shipment.

NOTES:

Key Updates and Operating Instructions

(Revision — August 2025)

What's new

- Digital Read-Out (DRO) controller replaces the legacy Watlow SD display.
- Cooling-water flowmeter upgraded to a Dwyer® unit with an integrated valve; scale extended to 0 – 50 GPH.
- Heater-block temperatures — 130 – 135 °C for cyanide | 165 °C for ammonia & phenols.
- Cooling-water flow — open the water supply and allows continuous flow through the condenser.

  Product photos and diagrams now show the DRO version.

Operating highlights

1. Set the DRO Press ▲ / ▼ (ones digit flashes) → SET to advance → SET to confirm.
2. Heating status "OUT1" + amber top light = heater engaged.
3. Timer resets at power-up; starts only after the set-point is reached (0.01 s – 60 h range).
4. Flowmeter: Adjust the valve to ensure continuous cooling-water flow through the condenser.
5. Start a new cycle. Toggle the main power OFF, then ON.

For detailed DRO and timer procedures, see Appendix A.

For the MS4SM subsystem, see Appendix B.

Table of Contents

Midi-Distillation System Heater Manifold

Midi-Phenol/Ammonia

Midi-Cyanide

APPENDIX:
EPA Method 350.1, Ammonia
EPA Method 335.4, Cyanide
EPA Method 420.4, Phenols

Please document your purchase here.

Catalog No. ______________________

Serial No. ______________________

Voltage of Heater Unit ______________________

Astraglass Innovations
3740 N West Blvd. Vineland,
NJ 08360 U.S.A

Tel: (800) 995-0376
(856)-692-4435

cs@astraglassinnov.com
www.astraglassinnov.com

Instruction 9539240 Rev. 9/11
Printed in USA

Unpacking the Unit

The Midi Distillation System is shipped in 3 separate cartons. One carton contains the heater and all required tubing. The other two cartons contain sets of either Ammonia / Phenols or Cyanide glassware, depending on the item ordered. Carefully unpack the cartons and check the contents to ensure all parts are received. If you do not receive all the items listed, please contact technical support at cs@astraglassinnov.com.
Please refer to page 19 for replacement part numbers.

The Midi Distillation System Manifold Heater [795210P220 (Phenol/Ammonia 200V)] includes the following items:

1 each Manifold Heater, 200V (depending on model: Ammonia or Phenol)
1 Complete Tubing Kit (Includes tubing and connectors for all tests):

- 2 bags containing 5 ft. each of braided PVC “feed/drain” tubing for water manifold to water source or to drain
- 1 bag containing 5 ft. of clear Tygon® tubing for vacuum inlet
- 1 bag containing 20 each of silicone “condenser water manifold tubing” (17-inch length) With male quick disconnect fitting
- 1 bag containing 10 each of silicone “reaction flask to impinger” tubing (7-inch length) With slip-fit connector – for cyanide only
- 1 bag containing 10 each of silicone “absorption flask to vacuum inlet” tubing (10-inch length) with slip fit connector – for cyanide only

The Cyanide Glassware includes 5 sets each of the following items packed in foam in separate sleeves:

- 1 each cold finger condenser
- 1 each distilling head
- 1 each absorption impinger
- 2 each 50 mL reaction/absorption tubes

The Ammonia / Phenol Glassware includes 5 sets each of the following items packed in foam in separate sleeves:

- 1 each leibilg condenser
- 1 each distilling head.
- 1 each 50 mL reaction tube.
- 1 each 50 mL receiver tube.

Parts List / General Information

The Midi Distillation Systems for Cyanide and Ammonia/Phenols Reduced Sample Volumes are shipped complete with a 10-position heater block, water manifold, flowmeter, 10 complete sets of borosilicate glassware for the model ordered, all necessary tubing connections, and an instruction manual.

- The 10-Position Heater Block features a Solo SD temperature controller, 10-position vacuum manifold with needle valves, 10-position parallel feed water manifold system, and flowmeter with scale indicating gallons per hour readout.
- The Glassware Sets are listed for a specific model ordered. Refer to the diagram and the assembly set-up diagrams on pages 15 and 17.
- The 10-Position Complete Tubing Set includes the items in the list that follows. Refer to the instrument setup instructions on page 9 and the diagram on page 11.

Tubing used for Cyanide, Ammonia, and Phenols protocols:

1 each braided PVC “feed/drain” tubing, 5 ft. (water manifold to water source or to drain)

20 each of silicone “condenser to water manifold tubing” (17-inch length) with male quick disconnect fitting

Additional tubbing used with the Midi-Cyanide protocol only:

1 ea. 5 ft. of clear Tygon® tubing for vacuum inlet.
10 ea. Silicone “reaction flask to impinger” tubing (7-inch length) with slip fit connector.
10 ea. Silicone “absorption flask to vacuum inlet” tubing (10-inch length) with slip fit connector.

Safety Information

1. The MIDI DISTILLATION SYSTEM can be used for the analysis of ammonia, cyanide, or phenols. The system footprint is approximately 30” x 14.25”, and the assembled height with glassware is approximately 20”. **For maximum operational safety, the unit should be used inside a fume hood rated at 100 CFM, suitable for handling caustic and corrosive substances.**
2. Boiling chips, beads, or stones should be placed in each sample tube to minimize bumping. The ammonia/phenol reaction tube is equipped with a draft shield to optimize performance and reduce distillation time.
3. Do not attempt to move components while hot. Sudden movement may cause bumping and/or sample loss.
4. To avoid bumping or boil-over, allow the system to cool for at least 20 minutes before removing glassware.
5. **Installation of an in-line gas trap between the vacuum line and the vacuum pump is recommended to remove excess HCN vapor and to protect the vacuum pump when the unit is used for Midi-Cyanide analysis.**
6. To avoid the possibility of shock hazard, unplug the unit prior to cleaning of exterior surfaces.
7. Do not disconnect water or vacuum lines until the system has completely cooled down, as this may cause samples to boil over.
8. The heater block temperature is factory preset to 130 – 135 °C for cyanide, to 165°C for ammonia/phenols, and has a maximum temperature of 190°C for other test protocols. Caution should be exercised when preparing or removing samples. The heater should not be installed in locations where flammable materials (which have a flash point below 200°C) are present.
9. Although the case is PTFE-coated, it is not acid-proof. Wipe any spills immediately with a soft cloth, followed by rinsing with distilled water. The heater includes drain holes at the bottom to allow spills to escape. It is recommended to place small containers, such as glass Petri dishes, under these holes to collect any spilled liquid.

CAUTION!

SOME REAGENTS USED IN THE DETERMINATION OF CYANIDE ARE CAUSTIC AND MAY CAUSE SKIN IRRITATION.

ALWAYS UNPLUG THE UNIT FOR CLEANING!

Instrument Set-Up

(See diagrams on page 8 and test protocols in the Appendix)

1. Unpack the Midi-Distillation System heater. Check that all parts are present according to the list on page 6. Report any missing or damaged items immediately to Astraglass Innovations Customer Service at 1-(800)-955-0376, extension 118, or email cs@astraglassinnov.com. Retain the original packing materials in case the unit needs to be returned for repair.

2. Complete the flowmeter and water manifold tubing connections as follows:
   Connect the five-foot length of clear vacuum tubing to the brass hose barb (vacuum inlet) on the left side of the unit in front of the flowmeter. We recommend connecting the other end to a vacuum trap (vacuum filtration flask, Cat. No. 953760-series, filled with NaOH solution) and then to the vacuum source to best protect your pump.
   Connect a five-foot length of braided feed/drain tubing to the lower (water inlet) hose barb on the rear of the flowmeter. Connect the other end to a cold-water source. **A chiller is recommended to maintain a water temperature of 4°C and achieve optimal results.**
   Connect the second five-foot length of braided feed/drain tubing to the upper (water outlet) barb on the water manifold and place the other end into a drain.
   Refer to pages 15 and 17 for glassware setup and standards preparation before proceeding to step 3.

3. Plug the 100V unit power cord into a suitable 3-wire grounded electrical outlet rated at 15 Amps. **The 220V model is supplied with an unterminated international standard cord.**

4. Turn the red, lighted power switch (on top of the unit) to the "ON" position. The green light indicates power to the unit. Indicating power to the heater. The controller display will show both ambient and set-point temperatures. As the unit heats up, the temperature will rise until the set point is reached and the timing countdown sequence begins. The amber light will flicker when the set point is approached and then flash to indicate continued heating to the set point temperature. The timer will count down from the set point to the end of the test, at which time the controller will automatically shut off. Allow the unit to cool, keeping the water running for at least 15-20 minutes, and then turn off both switches.

5. Turn the unit ON. The system will run and reach operating temperature automatically

PLEASE NOTE: Any manual changes to the preset controller temperatures will automatically override (disengage) the timer function.

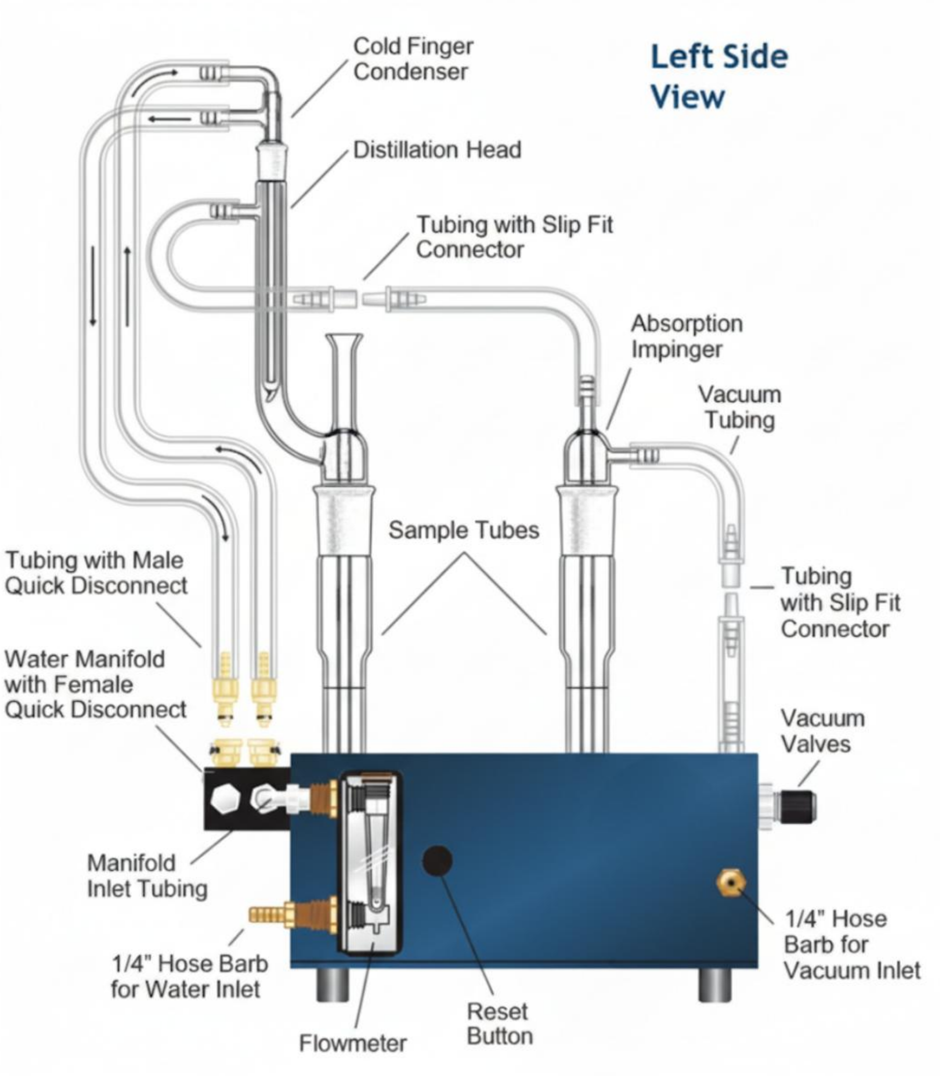
Mounting Procedure for Silicone Rubber Heaters with Pressure-Sensitive Adhesive (PSA)

Please ensure the heater is properly installed before applying power. Before removing the heater from its installation, disconnect its power supply.

1. **Surface Preparation**
   To ensure a strong bond, make sure the bonding surface is clean, dry, and free from oil, grease, soap, and dust. Avoid using cleaning solutions that could leave a film that may interfere with the adhesion of the silicone rubber heater.
2. **Liner Removal**
   When removing the release liner from the heater, peel it away from the edge using a quick, constant motion while maintaining an angle as close to 180° as possible. Avoid touching the adhesive surface during or after liner removal, and ensure that the adhesive does not lift with the liner.
3. **Adhesive Application**
   To avoid premature heater failure, press the adhesive side of the heater firmly against the bonding surface using a rubber roller or squeegee, ensuring there are no trapped air pockets. Do not re-position the heater once contact has been made, as this will significantly reduce the overall adhesive strength.
4. **Curing Time**
   Allow the lamination to set and cure for 24 to 72 hours to ensure optimum bond strength before operation.
5. **Storage Conditions**
   Store the silicone rubber heater with PSA at 70 °F (21 °C) or lower and 50% relative humidity before use. For optimal results, apply within 3 months of the purchase date.

Note: The *maximum continuous operating temperature* of silicone rubber heaters with pressure-sensitive adhesive is 400 °F (204 °C), depending upon the bonding surface material.

Heater Manifold Diagrams



Digital controlers and Power Switches

SET Key
Opens the parameter menu and confirms selections or values shown on the display.

Infinity Key (⟳ symbol)
Returns the display to the main screen. Also used to exit menus or move between parameter levels.

Up and Down Keys (▲ / ▼)
Increase or decrease the displayed value. These keys are used to adjust settings such as the temperature set point.

Note:
The controller is pre-programmed at the factory for safe operation of this unit. Changing parameters without following the official SOLO manual is not recommended, as this may affect performance or damage the system.

Instrument Controls

(refer to diagrams on page 11)

Power Switch

The unit is equipped with a single lighted power switch, located on the top right-side panel. This switch serves as the main ON/OFF control for the entire system. When turned ON, the unit powers up and is ready for operation.

Solo Controller

The Watlow SD controller is located on the front panel and is factory preset to 130 – 135 °C for cyanide and to 165°C for ammonia/phenols. It is designed to control the operation of the heater block up to 190°C by automatically turning the power to the heating element on/off. The controller may be manually adjusted by pressing the “UP arrow” key until the desired temperature is reached.

Green Pilot Light

The green pilot light located on top of the unit indicates that the main power to the unit is ON.
Note: If the light fails to come on when the main switch is ON, turn the unit OFF and then ON again.

Amber Pilot Light

The amber pilot light located on top of the unit operates during the heat cycle. The flashing light indicates power to the heating element. The flashing stops when the heater reaches the preset temperature.

Flowmeter

The flowmeter measures the amount of cooling water entering the cold finger condensers. It is calibrated to indicate gallons per hour (GPH). The water flow through the flowmeter should be maintained at 50 GPH during all procedures.

Vacuum Valves

The vacuum valves are located on the front panel, one for each position. To adjust the valve, rotate the black knob left or right. Rotating clockwise closes the valve, and counterclockwise opens the valve. Do not over-tighten the valve during shutoff, as the needle may become damaged.

Main Fuse Holder

The main fuse holder is located on the left rear of the unit. A 15-amp fuse (8-amp for 220-240V) must be in place for the unit to operate

Timer

The timer controls the duration for which the unit maintains the set temperature during a reaction. Set the desired reaction time on the timer before turning the unit ON, as this determines how long the unit will hold the target temperature once it is reached. After power is applied, the timer’s scale and time units cannot be modified. If a change in scale or units is required, the unit must be turned OFF and reset before restarting the operation.

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Troubleshooting Guide

Electrical	Recommendation
No power to unit	1. Plug the unit into a working wall outlet 2. Turn both switches to "ON" position 3. Check/replace fuse located on back of unit 15A for 100V heater unit 8A for 220V heater unit 4. Return unit for service (see page 14)
Controller error message	1. Return unit for service (see page 14)
Erratic controller temperature	1. Return unit for service (see page 14)
No heat / Samples will not boil	1. Turn the heater switch to 'ON' position 2. Check digital display for correct preset temperature 3. Check/replace fuse located on back of unit 15A for 100V heater unit 8A for 220-240V heater unit 4. Return unit for service (see page 14)
No power to controller	1. Turn the controller switch to 'ON' position 2. Return unit for service (see page 14)
Overheating	1. Check the controller set point temperature 2. Return unit for service (see page 14)

Test Procedures	Recommendation
Poor recoveries	1. Adjust cooling water to 4˚C and 50 GPH 2. Adjust the vacuum to 3 bubbles/second 3. Verify that the sample boils for 60 minutes 4. Check standard solutions 5. Check tubing connections for leaks
Condensation in tubing	1. Adjust cooling water to 4˚C and 50 GPH 2. Adjust the vacuum to 3 bubbles/second
Excessive foaming/boil over	1. Dilute the sample to reduce concentration and adjust the results

Return and Servicing Instructions

1. **Return Authorization Number**
   Contact Astraglass Innovations Customer Service at 1-(800)-955-0376 Ext. 118 or email cs@astraglassinnov.com to request a Return Authorization (RA) number. This RA number is required to return your unit for service and must appear on the outer carton shipping label. **A Certificate of Decontamination will also be issued and must be completed and returned with the heater manifold.** Please provide the serial number and date of purchase to determine warranty status. You may be referred to Technical Support for clarification. Typical turnaround time for repair is 3–5 business days from receipt. Customers will be contacted via phone or email with repair estimates for out-of-warranty units. Warranty repairs are performed free of charge.

2. **Loaner Unit Policy - Loaner units are available in the USA and Canada only.**
   For units under warranty, Astraglass Innovations provides loaner units at no charge for up to 30 days. After the 30-day period, a service charge of $130.00/month will apply. Contact Customer Service or Technical Support for more details.

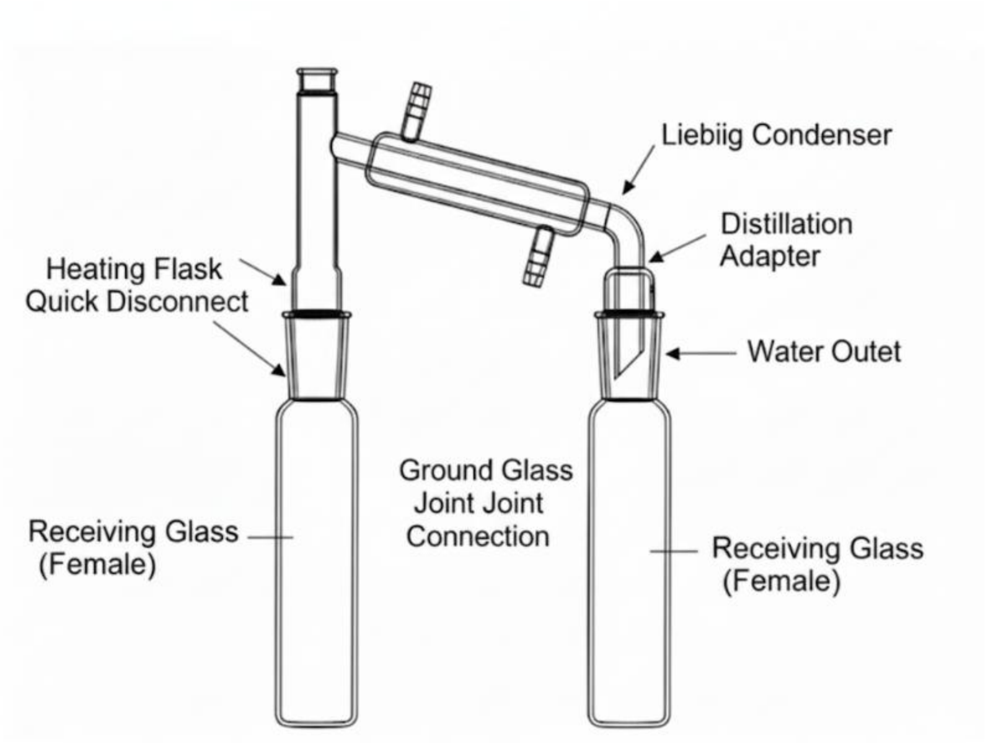
3. **Packing the Unit**
   Clean and pack the instrument securely in a sturdy box (preferably the original carton). Ensure a minimum of 2 inches of padding between the unit and all box walls, including the bottom. Use rigid packing materials to prevent shifting during transport. It is strongly recommended to ensure the return shipment to cover potential shipping damage.

4. **Return Information**
   Please include with the instrument a note with the following information: Name, telephone number, and email of the person to be contacted, along with company name, return shipping address, and method of shipment. Please also include a brief description of the problem. This information is crucial for providing prompt service.

5. **Shipping the Unit**
   Ship the unit directly to Astraglass Innovations using the address provided by your Customer Service representative. Clearly write the RA number on the outside of the package, along with your return address. Astraglass Innovations is not responsible for lost or damaged shipments.

6. **Repair / Return of the Unit**
   Once received, your unit will be evaluated, and the designated contact will be notified of any charges. A purchase order or credit card number will be required before non-warranty repairs begin. After repair, the unit will be returned to the requested address. Return shipping charges will be added to the repair invoice, unless the repair is under warranty. Warranty repairs are free of charge.

Midi-Phenol/Ammonia Glassware Set Up and Parts Diagram



Section	Piece
Glassware Component Parts	50 mL Reaction Tube *(2 supplied)* Liebig Condenser
Replacement Kits	Phenol/Ammonia Tubing Kit — 10-Position Phenol/Ammonia Tubing Kit — 4-Position *(use only if you list a 4-position version on a separate page)*

Midi-Phenol/Ammonia Assembly and System Operation

There are various forms of nitrogen reported in water analysis, typically as mg/L (parts per million).

Nitrate	NO3
Nitrite	NO2
Ammonia	NH3
Organic	N2
Total Kjeldahl	TKN

1. Organic nitrogen is calculated by subtracting the ammonia value from total Kjeldahl, which includes all forms of nitrogen, free and chemically bound. Refer to EPA method 350.1 in the appendix for the test procedure.

2. Phenols refer to a group of all compounds that contain a hydroxy or hydroxyl (-OH) substituted group as a derivative of benzene. The reaction of the distillate with the 4-aminoantipyrine and alkaline ferricyanide forms a colored complex, which is measured at 505 or 520 nm. Refer to EPA method 420.4 in the appendix for the test procedure.

3. Place one 50mL sample reaction tube containing standard or pH-adjusted sample and assembled with distillation head and cold finger condenser into each rear hole of the heater as shown in the diagram on page 11. Interferences from sulfur compounds are eliminated by adjusting the sample pH to 4.0.

4. Boiling chips, beads, or stones should be added to each sample tube to reduce bumping.

5. Attach the open ends of the 17” water manifold tubing to the top and bottom cold finger condenser inlet and outlet hose barbs so that the cooling water enters at the top and exits at the bottom. Attach the other end to the water manifold by snapping the quick disconnect fittings together. See diagram on page 9.

   Carefully introduce cooling water to the cold finger condensers and adjust for the number of positions as necessary. Check to ensure that all hoses are firmly in place and are not leaking. The recommended flow rate is 50 GPH. To ensure maximum recoveries in samples where suspected CN levels are below 100 ppb, cold water temperature should be maintained at 4°C, which will require installation of a chiller.

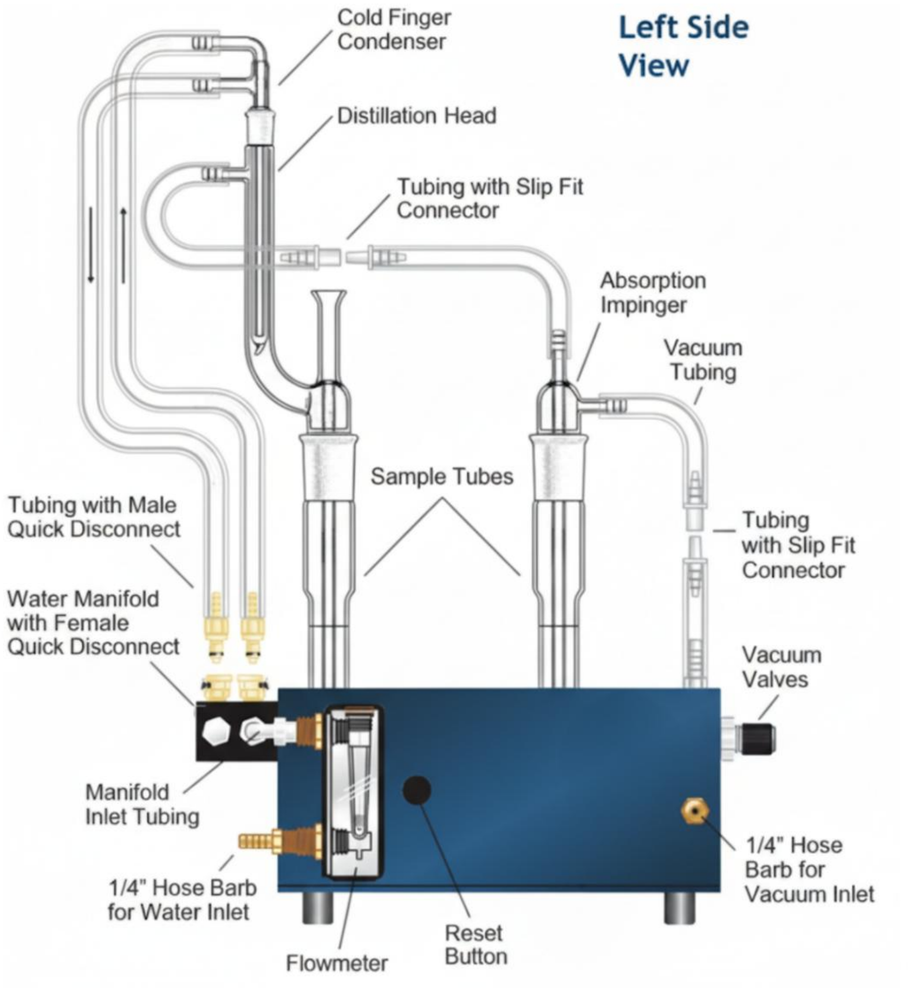
6. Place 50mL receiver tubes in front row of holes and assemble with short stem glass tube. Unscrew the red caps and remove the sealing rings, slide the tubes through the holes and reattach the caps and sealing rings to the distilling head arms.

7. The temperature for ammonia distillation is preset to 165°C. See controller setup on page 11. It will take approximately 30 minutes for the unit to reach the temperature at which time the test countdown will begin. The controller will automatically shut down the heater when the test is completed.

   NOTE: It is strongly recommended that the water temperature used during the distillation be at 4°C, which will require the use of a chiller. The water should be left running for at least 20 minutes after unit shutdown to prevent boil over and as a safety precaution.

8. Return to Instrument Set-up “Step 3” on page 10.

Midi-Cyanide Glassware Set-Up Diagram and Part List



Section	Piece
Complete Includes	Midi-Cyanide Glassware Set of 10 *(includes two 5-piece sets)* Manifold Heater only w/ tubing kit *(voltage options listed in code table)*
Glassware Component Parts	50 mL Reaction/Absorber Tube *(2 supplied)* Universal Cold Finger Condenser Dispersion Tube with Coarse Porosity Frit Distillation Absorber Flask *(2 supplied)*
Replacement Kits	Cyanide Tubing Kit — 10-Position Cyanide Tubing Kit — 4-Position

Cyanide Assembly and System Operation

1. Place one 50mL sample reaction tube containing standard or pH-adjusted sample and assembled with distillation head and cold finger condenser into each rear hole of the heater as shown in the diagram on page 17. Interferences from sulfur compounds are eliminated by adjusting the sample pH to 4.0.

2. Attach the open ends of the 17" water manifold tubing to the top and bottom cold finger condenser inlet and outlet hose barbs so that the cooling water enters at the top and exits at the bottom. Attach the other end to the water manifold by snapping the quick disconnect fittings together. See diagram on page 17.

3. Carefully introduce cooling water to the cold finger condensers and adjust the position as necessary. Check to ensure that all hoses are firmly in place and are not leaking. The recommended flow rate is 50 GPH. To ensure maximum recoveries in samples where suspected CN levels are below 100 ppb, cold water temperature should be maintained at 4°C, which will require installation of a chiller.

4. Place 50mL receiver tubes in the front row of holes and assemble with a short-stemmed glass tube. Unscrew the red caps and remove the sealing rings. Slide the tubes through the holes and reattach the caps and sealing rings to the distilling head arms.

5. The temperature for the phenols distillation is factory preset to 165°C. See instrument setup instructions on page 10. It will take approximately 30 minutes for the unit to reach the target temperature, at which time the test countdown will begin. The controller will automatically shut down the heater when the test is completed.

6. NOTE: It is strongly recommended that the water temperature used during the distillation be at 4 °C, which will require the use of a chiller. The water should be left running for at least 20 minutes after shutdown to prevent boil over and as a safety precaution.

7. Return to Instrument Set-up "Step 3" on page 10.

Component Parts

1) 4-Position MIDI Components

Item	Variant(s)	Code
4 POSITION MIDI HOUSING	Blue	7952040001
	Plain	7952040001P
4 POSITION HEATER BLOCK	Coated	7952040002
	Plain	7952040002P
LARGE INNER PANEL	Blue	7952040004
	Plain	7952040004P
4 POSITION MIDI BTM PLATE	Blue	7952040005
	Plain	7952040005P
4 POSITION MIDI TUBE RACK	Blue	7952040006
	Plain	7952040006P
4 POSITION MIDI MANIFOLD	Blue	7952040007
	Plain	7952040007P
1/16" SILICONE GASKET (4-pos)	—	7952040008
SMALL INNER PANEL (MIDI)	Blue	7952100003
	Plain	7952100003P
MIDI FLOW METER BRACKET	Blue	7952100009
	Plain	7952100009P

2) 10-Position MIDI Components

Item	Variant(s)	Code
10 POSITION MIDI HOUSING	Blue	7952100001
	Plain	7952100001P
10 POSITION HEATER BLOCK	Coated	7952100002
	Plain	7952100002P
SMALL INNER PANEL (MIDI)	Blue	7952100003
	Plain	7952100003P
LARGE INNER PANEL (10-posn)	Blue	7952100004
	Plain	7952100004P
10 POSITION MIDI BTM PLATE	Blue	7952100005
	Plain	7952100005P
10 POSITION MIDI TUBE RACK	Blue	7952100006
	Plain	7952100006P
10 POSITION MIDI MANIFOLD	Blue	7952100007
	Plain	7952100007P
1/16" SILICONE GASKET (10-pos)	—	7952100008
MIDI FLOW METER BRACKET	Blue	7952100009
	Plain	7952100009P

3) Glassware Components

Item	Code
PHENOL/AMMONIA — 50 mL Reaction/Receiver Tube	XXX80B7672
PHENOL/AMMONIA — Liebig Condenser	XXX80B7673
CYANIDE — 50 mL Reaction/Absorber Tube	QD-60-2
CYANIDE — Cold Finger Condenser	QD-60-3
CYANIDE — Fritted Dispersion Tube (coarse)	QD-60-4
CYANIDE — Distillation Absorber Flask	XXX80B7671

4) Glassware Sets

Item	Sets	Code
CYANIDE GLASSWARE	2	795204C002
	4	795204C004
	10	795210C010
	12	795210C012
PHENOL/AMMONIA GLASSWARE	2	795204P002
	4	795204P004
	10	795210P010
	12	795210P012

5) Tubing (Assemblies)

Item	Positions	Code
TUBING ASSEMBLY	4	7953010001
	10	7953010002

6) Console Only (Manifold Heater only)

Item	Voltage	Code
4 POSITION MIDI (Console Only)	110 V	7952040110
	220 V	7952040220
10 POSITION MIDI (Console Only)	110 V	7952100110
	220 V	7952100220

7) 4-Position Systems

Item	Voltage	Code
4 POSITION MIDI SYSTEM — CYANIDE	110 V	795204C110
	220 V	795204C220
4 POSITION MIDI SYSTEM — PHENOL/AMMONIA	110 V	795204P110
	220 V	795204P220
4 POSITION MIDI SYSTEM — CYANIDE SUPERVALUE	110 V	795204S110
	220 V	795204S220

8) 10-Position Systems

Item	Voltage	Code
10 POSITION MIDI SYSTEM — CYANIDE	110 V	795210C110
	220 V	795210C220
10 POSITION MIDI SYSTEM — PHENOL/AMMONIA	110 V	795210P110
	220 V	795210P220
10 POSITION MIDI SYSTEM — CYANIDE SUPERVALUE	110 V	795210S110
	220 V	795210S220

9) Vacuum Pumps

Item	Voltage	Code
VACUUM PUMP	110 V	7953105101
	220 V	7953105107

SOLO Basic

Temperature Controller User Manual

Manual Number: SLB-USER-M

SOLO BASIC TEMPERATURE CONTROLLER

USER MANUAL

Please include the Manual Number and the Manual Issue, both shown below, when communicating with Technical Support regarding this publication.

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CHAPTER

1

GETTING STARTED

In this Chapter...

Manual Overview

Overview of this Publication

The SOLO Basic Temperature Controller User Manual describes the installation, configuration, and methods of operation of the SOLO Basic Temperature Controller.

Who Should Read This Manual

This manual contains important information for those who will install, maintain, and/or operate any of the SOLO Basic Temperature Controllers.

Technical Support

By Telephone: 770-844-4200
(Mon.-Fri., 9:00 a.m.-6:00 p.m. E.T.)
On the Web: support.automationdirect.com

Our technical support group is glad to work with you in answering your questions. If you cannot find the solution to your particular application, or, if for any reason you need additional technical assistance, please call technical support at **770-844-4200**. We are available weekdays from 9:00 a.m. to 6:00 p.m. Eastern Time.

We also encourage you to visit our web site where you can find technical and non-technical information about our products and our company. Visit us at **www.automationdirect.com**.

Supplemental Manuals

If you are familiar with industrial control type devices, you may be able to get up and running with just the aid of the Quick Start Guide that is included with each SOLO Basic Temperature Controller.

Special Symbols

When you see the "notepad" icon in the left-hand margin, the paragraph to its immediate right will be a special note.

When you see the "exclamation mark" icon in the left-hand margin, the paragraph to its immediate right will be a warning. This information could prevent injury, loss of property, or even death (in extreme cases).

SOLO Basic Temperature Controller Introduction

General Description

AutomationDirect's SOLO Basic is a single loop temperature controller that can control heating or cooling processes. Depending upon the particular model of controller, the available outputs include relay, voltage pulse or current. On select models there are two alarm outputs available with nine selectable alarm types. SOLO Basic controllers have a single control output that can be used for control of a heating or cooling application. Models with alarm outputs can also be configured to use one of the alarm outputs as a second control output allowing both heating and cooling control or two stage heating or two stage cooling control. There are three types of control modes: PID, ON/OFF and Manual. SOLO Basic can accept various types of thermocouple and RTDs.

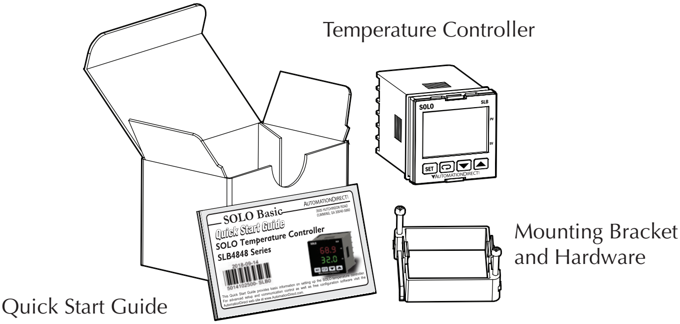
Other features include:

- Auto Tuning (AT) function with PID control
- 1/16 DIN panel size
- 2 line x 4 character 7-segment LCD display for Process Value (PV): Red color, and Set Point (SV): Green color
- Selectable display decimal point XXX.X or XXXX
- Selectable between °C and °F
- UL, CUL and CE agency approvals

Unpacking

After receiving the SOLO Basic Temperature Controller, please check for the following:

- Make sure that the package includes the Controller, the mounting bracket and hardware and the Quick Start Guide.
- Inspect the unit to insure it was not damaged during shipment.
- Make sure that the part number indicated on the serial number label corresponds with the part number of your order.



Model Number Explanation

SLB 4848 - R0

Alarm Relay Output

0: None
2: 2 Alarm outputs

Output

R: Relay output
V: Voltage Pulse output
C: DC Current output

Panel Size (W x H)

4848: 1/16 DIN W48 x H48mm

Solo Basic Temperature Controller

SOLO Basic Temperature Controller Specifications

Specifications	
Input Power Requirements	100 to 240 VAC 50 / 60 Hz
Operation Voltage Range	85 to 264 VAC
Power Consumption	5 VA Max
Control Mode	PID, ON/OFF or Manual
Input Accuracy	TC temperature indication accuracy: ±(0.3% of span + 1 digit) at 25°C ambient after 20 minutes warm up. Including NIST conformity, cold junction effect, A/D conversion errors and linearization conformity RTD temperature indication accuracy: ±(0.2% of span +1 digit)
Vibration Resistance	10 to 55 Hz, 10 m/s^2 for 10 min, each in X, Y and Z directions
Display	2 line x 4 character, 7-segment LED display PV: 11mm red SV: 9.2 mm green
Shock Resistance	Max. 300 m/s^2, 3 times in each 3 axes, 6 directions
Ambient Temperature Range	32°F to 122°F (0°C to 50°C)
Storage Temperature Range	-4°F to 149°F (-20°C to 65°C)
Altitude	2000m or less
Relative Humidity	35% to 80% (non-condensing)
IP Rating	IP66: Complete protection against dust and powerful water jets from all directions. (**inside suitable enclosure)
Agency Approvals	UL, CUL, CE (UL file number E311366)
Pollution Degree	Degree 2 - Normally, only non-conductive pollution occurs. Temporary conductivity caused by condensation is to be expected.
Input Types	
• Thermocouple*	K, J, T, E, N, R, S, B, L, U, TXK (Sampling Rate: 100 ms / per scan)
• Platinum RTD	3-wire Pt100, JPt100 (Sampling Rate: 100 ms / per scan)
• Copper, Nickel RTDs	Cu50, Ni120 (Sampling Rate: 100 ms / per scan)
Control Output Options	
• Relay (R)	SPST max. 5A @ 250 VAC resistive load
• Voltage Pulse (V)	DC 12V±15%, output current 40mA Max
• Current (C)	DC 4-20 mA output (sourcing) (Load resistance: Max 600Ω)
Alarm Output Option	(2) SPST relays with shared common, 3A @ 250VAC resistive load

***Note: Use only ungrounded thermocouples.**

**** No corrosive gases**

CHAPTER 2

INSTALLATION AND WIRING

In this Chapter...

Ambient Conditions

Ambient Conditions	
Ambient Temperature Range	32°F to 122°F (0°C to 50°C)
Storage Temperature Range	-4°F to 149°F (-20°C to 65°C)
Relative Humidity	35% to 80% (non-condensing)
Altitude	2000m or lower above sea level, keep from corrosive gas, liquid and dust
Pollution Degree	Degree 2 - Normally, only non-conductive pollution occurs. Temporary conductivity caused by condensation is to be expected.
Vibration Resistance	10 to 55 Hz, 10 m/s2 for 10 min, each in X, Y and Z directions
Shock Resistance	Max. 300 m/s2, 3 times in each 3 axes, 6 directions
IP Rating	IP66: Complete protection against dust and powerful water jets from all directions. (*inside suitable enclosure)
**** No corrosive gases***	

Installation Considerations

Improper installation of the controller will greatly reduce its life. Be sure to observe the following precautions when selecting a mounting location:

Warning: Failure to observe these precautions may damage the controller and void the warranty!

- Do not mount the controller near heat-radiating elements or in direct sunlight.
- Do not install the controller in a place subjected to high temperature, high humidity, excessive vibration, corrosive gases or liquids, or airborne dust or metallic particles.
- Do not restrict the air flow to the vent opening on the controller housing
- This controller is an open-type unit and must be placed in an enclosure to ensure proper operation and maintain the IP66 rating.

Mounting Instructions

SLB4848 Series

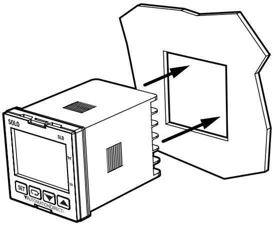
SOLO Basic temperature controllers should be mounted through a cutout in an enclosure or panel by using the dimensions shown on page 2–4. The directions for mounting the controller through a cutout are:

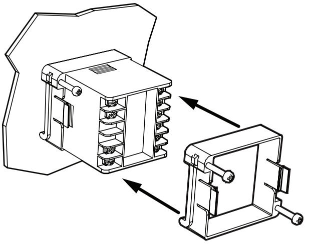
1. Insert the temperature controller through the panel cutout.

2. Slide the M3X0.5 nut into the opening in the top of the mounting bracket and insert the M3X0.5 X 30mm mounting screw in the mounting bracket.

3. Insert the mounting bracket into the mounting groove at the right and left of the controller, and push the mounting bracket forward until the bracket stops.

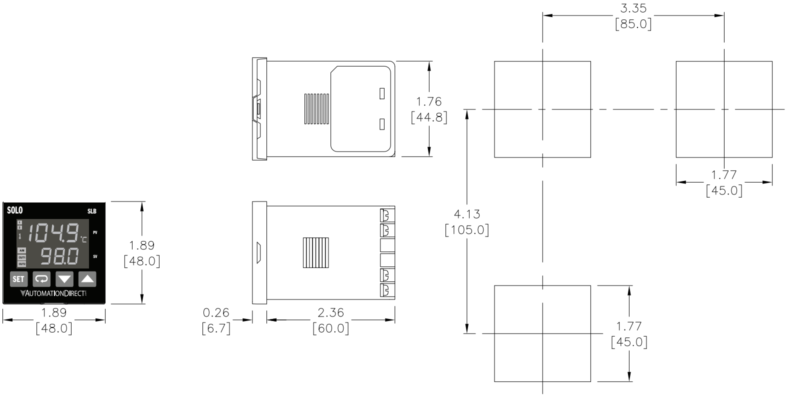
4. Tighten top and bottom screws evenly to secure temperature controller in place (The screw torque should be 0.4 to 0.5 N.m).





Controller and Panel Cutout Dimensions

SLB4848 Series Minimum Cutout and Spacing



Safety Information

DANGER!

Warning: To minimize the risk of potential safety problems, you should follow all applicable local and national codes that regulate the installation and operation of your equipment. These codes vary from area to area and it is your responsibility to determine which codes should be followed, and to verify that the equipment, installation, and operation are in compliance with the latest revision of these codes

Warning: To prevent electric shock, do not touch the AC terminals while power is supplied to the controller.

Warning: This controller is an open-type temperature controller. Make sure to evaluate any dangerous application in which a serious human injury or serious property damage may occur.

Wiring Notes: PLEASE READ PRIOR TO INSTALLATION.

Equipment damage or serious injury to personnel can result from the failure to follow all applicable codes and standards. We do not guarantee the products described in this publication are suitable for your particular application, nor do we assume any responsibility for your product design, installation, or operation.

If you have any questions concerning the installation or operation of this equipment, or if you need additional information, please call us at 1-800-633-0405 or 770-844-4200.

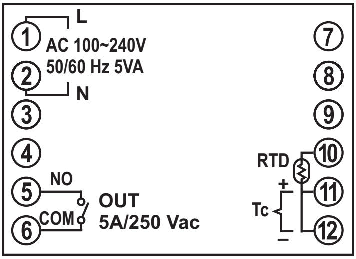
1. Always use recommended solder-less terminals: Fork terminal with isolation (M3 screw, width is 5.8mm, hole diameter 3.2mm). Screw size: M3 x 4.5 (With 6 x 6 square washer). Recommended tightening torque: 0.4 Nm (4kgfcm). Applicable wire: Solid/twisted wire 14AWG to 22AWG.
2. Protect the controller from dust or foreign objects as they can cause the controller to malfunction.
3. Never modify or disassemble the controller.
4. Do not connect anything to the “Not used” terminals.
5. Make sure all wires are connected to the correct polarity of terminals.
6. Do not install and/or use the controller in places subject to: (a) Dust or corrosive gases and liquid (b) High humidity (c) Vibration or shock (d) EMI / RFI (e) high temperature.
7. Power must be turned off when wiring or changing a sensor.
8. Be sure to use wires that match the thermocouple types when extending or connecting thermocouple wires.
9. Use wires with correct resistance when extending or connecting a RTD.
10. Keep the wire as short as possible when wiring a RTD to the controller and route power wires as far as possible from sensor wires to prevent interference and induced noise.
11. This controller is an open-type unit and must be placed in an enclosure to ensure proper operation and maintain the **IP66 rating.**
12. Make sure power cables and signals from instruments are all installed properly before energizing the controller, otherwise serious damage may occur.
13. To prevent electric shock, do not touch the terminals on the controller or try to repair the controller when power is applied.
14. Do not use acid or alkaline liquids for cleaning. Use a soft, dry cloth to clean the controller.
15. This instrument is not furnished with a power switch or fuse. Therefore, if a fuse or power switch is required, install the protection close to the instrument. Recommended fuse rating: Rated voltage 250 V, Rated current 1 A. Fuse type: Time-delay fuse. See the AutomationDirect catalog for the appropriate fuse for the specific application.
16. Note: This controller does not provide overcurrent protection. Use of this product requires that suitable overcurrent protection device(s) must be added to ensure compliance with all relevant electrical standards and codes. (Rated 250 V, 15 Amps max). A suitable disconnecting device should be provided near the controller in the end-use installation.

Terminal Identification

SLB4848 Series Wiring

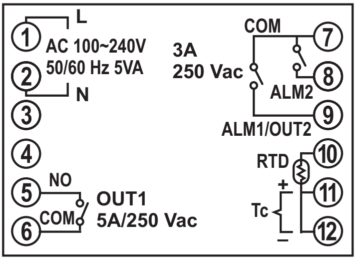
SLB4848-R0

(No alarm output)



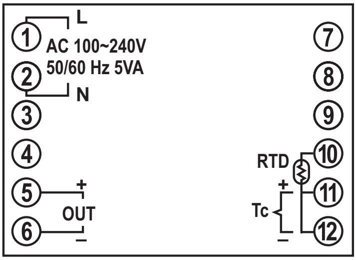
SLB4848-R2

(2 sets of alarm outputs)



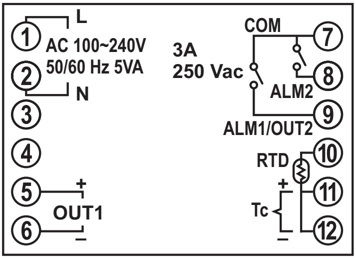
SLB4848-C0
SLB4848-V0

(No alarm output)



SLB4848-C2
SLB4848-V2

(2 sets of alarm outputs)



KEYPAD OPERATION AND SETUP PARAMETERS

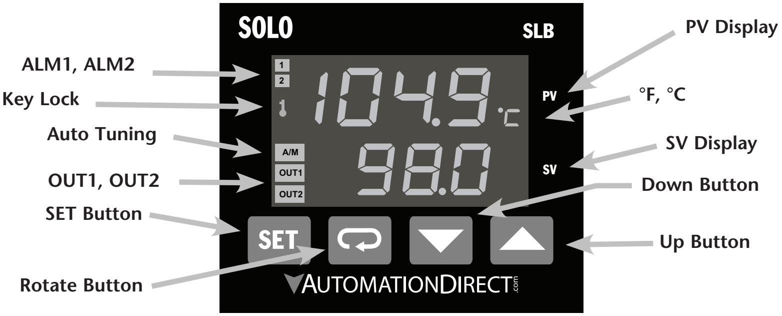
CHAPTER

3

In this Chapter...

Display, LCD and Keypad

The SOLO temperature controller has a two color seven segment LCD display and four function buttons.



PV Display

The Process Value Display. Displays the value from the input source or the parameter source.

SV Display

The Set Value Display. Displays the set point of the process, the parameter operation read value, manipulated variable, or the set value of the parameter.

A/M Indicator

A/M indicator flashes when the Auto Tuning operation is ON and is solid when the controller is in Manual Mode.

OUT1, OUT2 Indicators

Output indicators light when the output is ON.

°F, °C Indicator

Temperature unit indicator. °C: Celsius, °F: Fahrenheit

1, 2 Indicators

Alarm output indicators light when appropriate alarm is activated.

Key Indicator

Key indicator lights when key lock is enabled.

Keypad Operation

Function Buttons

SET

SET Button

Press the SET button to select the desired function mode and confirm the setting value.

↺

Rotate Button

Press the Rotate button to select parameters within the function mode.

▼

Down Button

Press the Down button to decrease values displayed on the SV display. Hold down this button to speed up the decrement.

▲

Up Button

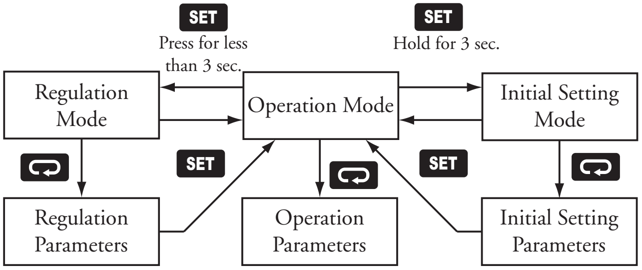
Press the Up button to increase values displayed on the SV display. Hold down this button to speed up the increment.

Initial Power up

When power is first applied to the temperature controller, the module information splash screen appears. This screen shows the firmware version on the PV displays and the output type for that particular model and the current input setting on the SV display. After three seconds, the controller will automatically proceed to the Operation mode main screen.

Keypad Operation

The temperature controller has three function modes: Initial Setting mode, Operation mode and Regulation mode. Press and hold the SET button for three seconds to go into the Initial Setting mode. Press the SET button for less than three seconds to access the Regulation mode. Press the Rotate button while inside any of the three function modes to scroll through the individual parameters for each function mode. Use the Up and Down buttons to change the individual parameter values. Pressing the SET button saves the parameter values. Press the SET button again to return the controller to the Operation mode main screen.



Setup Parameter Listing

Regulation Mode Parameters

Press the **SET** button to access these parameters.

Regulation Mode Parameter Availablity

		Controller Type						Control Mode			Heating / Cooling					
Display	**Parameter Name**	**RO**	**VO**	**CO**	**R2**	**V2**	**C2**	**PID**	**ON /OFF**	**Manual**	**Heating**	**Cooling**	**Heating/Cooling**	**Cooling/Heating**	**Heating/Heating**	**Cooling/Cooling**
At	Auto Tuning	✔	✔	✔	✔	✔	✔	✔	-	-	✔	✔	✔	✔	✔	✔
P	Proportion Band	✔	✔	✔	✔	✔	✔	✔	-	-	✔	✔	✔	✔	✔	✔
I	Integral Time	✔	✔	✔	✔	✔	✔	✔	-	-	✔	✔	✔	✔	✔	✔
d	Derivative Time	✔	✔	✔	✔	✔	✔	✔	-	-	✔	✔	✔	✔	✔	✔
PdoF	PD Control Offset	✔	✔	✔	✔	✔	✔	✔	-	-	✔	✔	✔	✔	✔	✔
o1-S	Output 1 Hysteresis	✔	✔	✔	✔	✔	✔	-	✔	-	✔	✔	✔	✔	✔	✔
o1-H	Output 1 Heating Period	✔	✔	✔	✔	✔	✔	✔	-	✔	✔	-	✔	-	✔	-
o1-C	Output 1 Cooling Period	✔	✔	✔	✔	✔	✔	✔	-	✔	-	✔	-	✔	-	✔
o2-S	Output 2 Hysteresis	-	-	-	✔	✔	✔	-	✔	-	-	-	✔	✔	✔	✔
o2-H	Output 2 Heating Period	-	-	-	✔	✔	✔	✔	-	✔	-	-	-	✔	✔	-
o2-C	Output 2 Cooling Period	-	-	-	✔	✔	✔	✔	-	✔	-	-	✔	-	-	✔
CoEF	Proportional Band Coefficient	-	-	-	✔	✔	✔	✔	-	-	-	-	✔	✔	-	-
dEAd	Deadband	-	-	-	✔	✔	✔	✔	✔	-	-	-	✔	✔	-	-
Pv-F	PV Filter	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
Pv-r	PV Filter Range	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
PvoF	PV Offset	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
PvGA	PV Gain	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
A1nA	Analog High Adjustment	-	-	✔	-	-	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
A1nL	Analog Low Adjustment	-	-	✔	-	-	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔

Operation Mode Parameters

Press the ⮌ button to access these parameters.

Operation Mode Parameter Availablity

		Controller Type						Control Mode			Heating / Cooling					
Display	Parameter Name	RO	VO	CO	R2	V2	C2	PID	ON /OFF	Manual	Heating	Cooling	Heating/Cooling	Cooling/Heating	Heating/Heating	Cooling/Cooling
r-S	Run / Stop	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
SP	Decimal Point Position	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
LoC	Lock Mode	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
AL 1H	Alarm 1 High Limit	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	-	-	-	-
AL 1L	Alarm 1 Low Limit	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	-	-	-	-
AL2H	Alarm 2 High Limit	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
AL2L	Alarm 2 Low Limit	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
oUt 1	Output 1 Level	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
oUt2	Output 2 Level	-	-	-	✔	✔	✔	✔	✔	✔	-	-	✔	✔	✔	✔
o 1nA	Output 1 Upper Limit	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
o 1nL	Output 1 Lower Limit	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
o2nA	Output 2 Upper Limit	-	-	-	✔	✔	✔	✔	✔	✔	-	-	✔	✔	✔	✔
o2nL	Output 2 Lower Limit	-	-	-	✔	✔	✔	✔	✔	✔	-	-	✔	✔	✔	✔

Initial Setting Parameters

Press the **SET** button for more than three seconds to access these parameters.

Initial Setting Mode Parameter Availablity

		Controller Type						Control Mode			Heating / Cooling					
Display	**Parameter Name**	**RO**	**VO**	**CO**	**R2**	**V2**	**C2**	**PID**	**ON /OFF**	**Manual**	**Heating**	**Cooling**	**Heating/Cooling**	**Cooling/Heating**	**Cooling/Cooling**	**Heating/Heating**
InPt	Input Type	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
tPUn	Temperature Unit	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
tP-H	Input Range High	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
tP-L	Input Range Low	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
CtrL	Control Mode	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
S-HC	Heating / Cooling	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
ALA1	Alarm 1	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	-	-	-	-
AL1o	Alarm 1 Options	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	-	-	-	-
AL1d	Alarm 1 Delay	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	-	-	-	-
ALA2	Alarm 2	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
AL2o	Alarm 2 Options	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔
AL2d	Alarm 2 Delay	-	-	-	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔	✔

Regulation Mode Parameters

Press the **SET** button for less than three seconds to access these parameters.

At

Auto Tuning

Range: On - Auto Tuning activated
Off - Auto Tuning deactivated

When this parameter is set to On, the controller begins auto tuning. After auto tuning is complete, the parameter is set to Off automatically. If this parameter is set to Off during the auto tuning process, the controller stops the auto tuning process immediately and does not change any PID parameter. A/M indicator will flash while Auto Tuning is in process.

P

Proportion Band

Range: 0.1 to 999.9

The Proportion Band is a parameter used for PID control.

I

Integral Time

Range: 0 to 9999 (Sec)

The Integral Time is a parameter used for PID control.

d

Derivative Time

Range: 0 to 9999 (Sec)

The Derivative Time is a parameter used for PID control.

PdoF

PD Control Offset

Range: 0.0 to 100.0 (%)

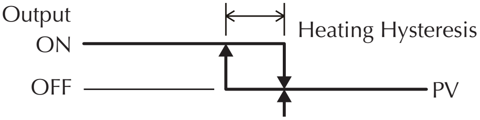
The PD Control Offset parameter is available when the P or PD control is selected [Integral Time parameter I is zero].

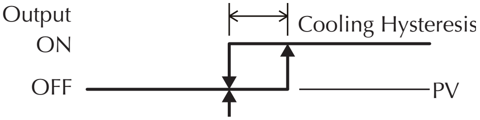
This parameter defines the offset of the output. When the P or PD control is used, the control cannot stabilize the PV at the SV because the output is zero when the PV is equal to the SV. This parameter modifies the output level when the PV is equal to the SV.

o1-S / o2-S Output 1 / Output 2 Hysteresis

Range: 0.0 to 999.9

The Output Hysteresis parameter defines the amount that the PV must go below or above the SV before the output turns on. This parameter is available only for On / Off control and hysteresis direction will be dependent on whether the output is set for heating or cooling.

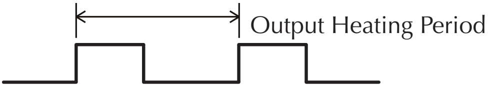




o1-H / o2-H Output 1 / Output 2 Heating Period

Range: 0.1 to 60 seconds (R0/R2 relay models - 5 to 60 seconds)

The Output Heating parameter defines one output period or the duration of one on / off cycle for the Output.

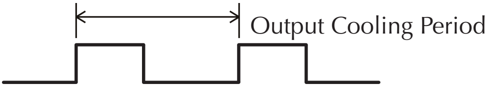


This parameter is available when the Output is programmed as a heating output. Available in PID and Manual mode.

o1-C / o2-C Output 1 / Output 2 Cooling Period

Range: 0.1 to 60 seconds (R0/R2 relay models - 5 to 60 seconds)

The Output Cooling parameter defines one output period or the duration of one on / off cycle for the Output.



This parameter is available when the Output is programmed as a cooling output. Available in PID and Manual mode.

CoEF

Proportional Band Coefficient

The PID proportional coefficient between Output 1 and Output 2 when a dual output control is selected. The P value of Output 2 equals the P value of Output 1 PID * CoEF. The I and D value of Output 2's PID are the same as Output 1's. Defualt value is 1.00.

dEAd

Deadband

The deadband around the SV value. For example, if SV = 100 degrees and dEAd = 2.0, there will be no output when the temperature is between 99~101°C. See PID and On/Off control sections in chapter 5. Default value is 0.

Pu-F

PV Filter

The process value filter takes the input and applies a rolling average to compensate for noisy signals. Filter equation: (Last displayed PV * Pu-F + Current Measurement) / (Pu-F+1). Range of 0 to 50 with a system default of 2.

Pu-r

PV Filter Range

Process value filter range is the plus or minus range from the last displayed PV that PV Filtering will be applied. For very noisy signals the number must be large enough to capture large oscillations in signal. Range of 0.10 to 10.00 degrees with a system default of 1.

PuoF

PV Offset

The PV Offset is the amount added to the current process value. Range of -99.9 to 99.9 with a system default of 0.

PuGA

PV Gain

The PV Gain is the multiplier applied to the current process value. Range of -0.999 to 0.999 with a system default of 0. Gain equation: PV = Current Measurement * (1 + PuGA / 1.000) + PuoF.

A1nA

Analog High Adjustment

The Analog High Adjustment value is added to the maximum analog output of 20ma in 1uA increments. If set to 500 the output will be 20.5ma at 100 percent output. Range of -3895 to 659 with a system default of 0. This parameter is only available on Current Output models.

A1nL

Analog Low Adjustment

The Analog Low Adjustment value is added to the minimum analog output of 4ma in 1uA increments. If set to -100 the output will be 3.9ma at 0 percent output. Range of -964 to 3895 with a system default of 0.

This parameter is only available on Current Output models.

Operation Mode Parameters

Press the button to access these parameters.

r--S

Run / Stop

The Run / Stop parameter is used to control the operational status of the SOLO Basic Controller. The controller can either be in run mode or stop mode. During stop mode the output is turned off but alarms remain active.

rUn	Run mode
StoP	Stop mode

SP

Decimal Point Position

Range: 0 (****)
1 (***.*) Note: Maximum temperature displayed (PV) will be limited to "999.9".

This parameter defines the decimal point position on the PV and SV display.

LoC

Lock Mode

Range:		
	oFF	The Lock feature is disabled.
	LoC1	Lock Mode 1
	LoC2	Lock Mode 2

Lock Mode 1: All key pad operation is ignored.

Lock Mode 2: All key pad operation is ignored except changing the SV. To unlock press the SET key and the ↻ key at the same time to display KEYP parameter. Enter the password to unlock the key. Default password is 0000.

AL1H

Alarm 1 High Limit

This parameter is used to set the high limit for Alarm 1. The range and availability are dependent on the selected alarm mode.

AL1L

Alarm 1 Low Limit

This parameter is used to set the low limit for Alarm 1. The range and availability are dependent on the selected alarm mode.

AL2H

Alarm 2 High Limit

This parameter is used to set the high limit for Alarm 2. The range and availability are dependent on the selected alarm mode.

AL2L

Alarm 2 Low Limit

This parameter is used to set the low limit for Alarm 2. The range and availability are dependent on the selected alarm mode.

oUt1 / oUt2

Output 1 / Output 2 Level

Range: 0.0 to 100 (%)

The value for this parameter can be changed in the Manual control mode. In other control modes, this parameter is read-only.

o1nA / o2nA

Output 1 / Output 2 Upper Limit

Upper output percentage. Linear calculation is done between the upper and lower limit. Range of 0.0 to 100 with a system default of 100.

o1nL / o2nL

Output 1 / Output 2 Lower Limit

Lower output percentage. Linear calculation is done between the lower and upper limit. Range of 0.0 to 100 with a system default of 0.0.

Initial Setting Parameters

Press the SET button for more than three seconds to access these parameters.

InPt Input Type

This parameter defines the input signal type.

Thermocouple* Type and Temperature Range		
Input Temperature Sensor Type	**LED Display**	**Temperature Range**
Thermocouple TXK type	tXK	-238 ~ 1472°F (-150 ~ 800°C)
Thermocouple U type	U	-328 ~ 932°F (-200 ~ 500°C)
Thermocouple L type	L	-328 ~ 1562°F (-200 ~ 850°C)
Thermocouple B type	b	212 ~ 3272°F (100 ~ 1800°C)
Thermocouple S type	S	32 ~ 3092°F (0 ~ 1700°C)
Thermocouple R type	r	32 ~ 3092°F (0 ~ 1700°C)
Thermocouple N type	n	-328 ~ 2372°F (-200 ~ 1300°C)
Thermocouple E type	E	32 ~ 1112°F (0 ~ 600°C)
Thermocouple T type	t	-328 ~ 752°F (-200 ~ 400°C)
Thermocouple J type	J	-148 ~ 2192°F (-100 ~ 1200°C)
Thermocouple K type	K	-328 ~ 2372°F (-200 ~ 1300°C)
RTD Type and Temperature Range		
Input Temperature Sensor Type	**LED Display**	**Temperature Range**
Platinum (Pt100)	Pt	-328 ~ 1562°F (-200 ~ 850°C)
Platinum (JPt100)	JPt	-148 ~ 752°F (-100 ~ 400°C)
Copper (Cu50)	CU	-58 ~ 302°F (-50 ~ 150°C)
Nickel (Ni120)	nI	-112 ~ 572°F (-80 ~ 300°C)
****Note: Use only ungrounded thermocouples.***		

tPUn Temperature Unit

Range: F, C

Sets controller units to Celsius or Fahrenheit.

tP-H Input Range High

This is the maximum value of the set point temperature range. The SV value cannot exceed the tP-H value. This parameter cannot be lower than the Input Range Low parameter (tP-L).

tP-L Input Range Low

This is the minimum value of the set point temperature range. The SV value cannot be set lower than the tP-L value. This parameter cannot be higher than the Input Range High parameter (tP-H).

CtrL

Control Mode

Range:

PId	PID control mode
onoF	On / Off control mode
MAnU	Manual control mode

This parameter is used to select one of the control modes. See Chapter 5 for a complete discription of each control mode.

S-HC

Heating / Cooling

Range:

H 1	Output = Heating	H1C2	Output 1 = Heating 2 = Cooling	H1H2	Output 1 / 2 = Heating
C 1	Output = Cooling	C1H2	Output 1 = Cooling 2 = Heating	C1C2	Output 1 / 2 = Cooling

The Heating / Cooling parameter defines whether one or two outputs will be controlled and what type of control they will perform.

ALA1
ALA2

Alarm 1
Alarm 2

Range: 0 to 9

The SOLO Basic models equipped with alarms support 2 alarm outputs. The Alarm1 and Alarm2 parameters are used to select the alarm type. Refer to Chapter 4 for details.

AL1o

Alarm 1 Options

Alarm standby until process value is within +/-5 of set point to prevent false triggers on startup xxxY (When Y=0: Normal, Y=1: Standby)

Alarm output normally open or normally closed xxYx (When Y=0: NO, Y=1: NC)

Hold alarm till power cycle or controller is stopped xYxx (When Y=0: Normal Reset, Y=1: Hold Alarm)

Not used Yxxx

AL1d

Alarm 1 Delay

Length of time alarm condition must be active in seconds before alarm condition is triggered.

AL2o

Alarm 2 Options

Alarm standby until process value is within +/-5 of set point to prevent false triggers on startup xxxY (When Y=0: Normal, Y=1: Standby)

Alarm output normally open or normally closed xxYx (When Y=0: NO, Y=1: NC)

Hold alarm till power cycle or controller is stopped xYxx (When Y=0: Normal Reset, Y=1: Hold Alarm)

Not used Yxxx

AL2d **Alarm 2 Delay**

Length of time alarm condition must be active in seconds before alarm condition is triggered.

Reset to Factory Default

Note: Resetting the Temperature Controller back to factory default erases all of the values entered by the user. Record any necessary settings before proceeding

Warning: Erasing the user entered values may result in a safety hazard and system malfunction.

The following instructions reset the controller to the factory default.

❶ Press the ⟲ button until the parameter LoC appears. Use the ▲ button to select LoC1. Press the SET button.

❷ Press and hold the SET and ▲ buttons simultaneously for three seconds and release.

❸ Use the ▲ button to change the value on the SV display to 1357. Press the SET button.

❹ Display will change from PASS to PAr-E. Press ▲ to change value from no to yes and press set button.

❺ Display will be fully lit and then controller will reboot.

Key Lock Function

Adjusting parambe of LoC to LoC1 in Operation Mode to lock all keys. LoC2 allows the SV value to be adjusted.

To unlock:

❶ Press the SET and ⟲ keys simultaneously in LOCK mode to display KEYP. Enter the password to unlock the key. Default password is 0000.

To change key lock password:

❶ Press the ⟲ key when KEYP is displayed to enter the password change screen CHGP.

❷ Enter the current password. If the password is correct, nEWP will be displayed. If the password is incorrect, the screen will return to PV/SV display mode.

③ Once nEWP is displayed enter the new password two times. The screen will return to PV/SV display mode with the keys unlocked. If two password entries do not match, the screen will return to step 2.

Note: If the password is lost or fogotten, restore the factory settings to reset lock condition.

CONTROLLER INPUTS AND OUTPUTS

CHAPTER 4

In this Chapter...

Control Input Types

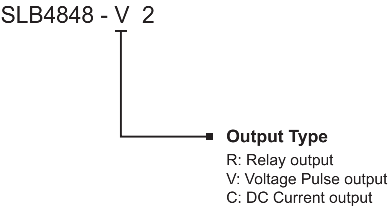
Thermocouple or RTD Input

The SOLO Basic temperature controller can accept input from eleven types of thermocouples and four types of RTD sensors.

Thermocouple* Type and Temperature Range		
Input Temperature Sensor Type	**LED Display**	**Temperature Range**
Thermocouple TXK type	tXk	-328 ~ 1472°F (-200 ~ 800°C)
Thermocouple U type	U	-328 ~ 932°F (-200 ~ 500°C)
Thermocouple L type	L	-328 ~ 1562°F (-200 ~ 850°C)
Thermocouple B type	b	212 ~ 3272°F (100 ~ 1800°C)
Thermocouple S type	S	32 ~ 3092°F (0 ~ 1700°C)
Thermocouple R type	r	32 ~ 3092°F (0 ~ 1700°C)
Thermocouple N type	n	-328 ~ 2372°F (-200 ~ 1300°C)
Thermocouple E type	E	32 ~ 1112°F (0 ~ 600°C)
Thermocouple T type	t	-328 ~ 752°F (-200 ~ 400°C)
Thermocouple J type	J	-148 ~ 2192°F (-100 ~ 1200°C)
Thermocouple K type	k	-328 ~ 2372°F (-200 ~ 1300°C)
RTD Type and Temperature Range		
Input Temperature Sensor Type	**LED Display**	**Temperature Range**
Platinum (Pt100)	Pt	-328 ~ 1562°F (-200 ~ 850°C)
Platinum (JPt100)	JPt	-148 ~ 752°F (-100 ~ 400°C)
Copper (Cu50)	CU	-58 ~ 302°F (-50 ~ 150°C)
Nickel (Ni120)	nI	-112 ~ 572°F (-80 ~ 300°C)
****Note - Use only ungrounded thermocouples***		

Control Output Types

The SOLO temperature controller supports three types of control outputs depending on the model chosen. The available outputs are Relay, Voltage Pulse and Current as shown in the controller part number.

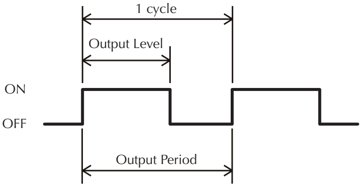

SLB4848 - V 2

Output Type
R: Relay output
V: Voltage Pulse output
C: DC Current output

Relay Output

The relay used for the relay output in the SLB4848 is rated at a maximum 250 VAC and 5A resistive load. A secondary 250 VAC 3A resistive load output is available when using a model with alarms in one of the dual output modes.

The operation cycle of the Relay output is controlled by two factors, Output Level and Output Period.



For example, when the Output Level is 60% and the Output Period is 10 seconds, the output relay is turned on for 6 seconds in the cycle.

There are six parameters that define these two factors.

Output Level

Output 1 Level (oUt1), Range: 0.0 to 100%
Output 2 Level (oUt2), Range: 0.0 to 100%

Output Period

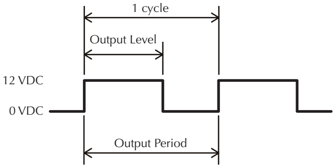
Output 1 Heating Period (o1-H), Range: 5 to 60 seconds
Output 1 Cooling Period (o1-C), Range: 5 to 60 seconds
Output 2 Heating Period (o2-H), Range: 5 to 60 seconds
Output 2 Cooling Period (o2-C), Range: 5 to 60 seconds

Note: The electrical life expectancy of the relay output is 100,000 cycles. To maximize the life of the relay output, set a longer time value for the Output Period.

Voltage Pulse Output

The Voltage Pulse output generates a high level pulse of 12 VDC nominal and low level pulse of 0VDC. It can supply up to 40ma.

The operation cycle of the Voltage Pulse output is controlled by two factors, Output Level and Output Period.



For example, when the Output Level is 60% and the Output Period is 10 seconds, the Voltage Pulse output is turned on for 6 seconds in the cycle.

There are three parameters that define these two factors.

Output Level

Output Level 1 (oUt1), Range: 0.0 to 100%

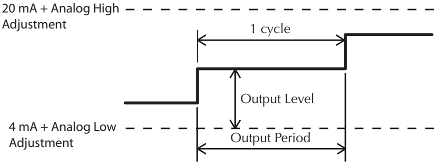
Output Period

Output 1 Heating Period (o1-H), Range: 0.1 to 60 seconds
Output 1 Cooling Period (o1-C), Range: 0.1 to 60 seconds

Current Output

The Current output generates analog DC current with a range of 4-20 mA. The maximum load resistance is 600 Ω.

The output current is controlled by four factors, Analog High Adjustment, Analog Low Adjustment, Output Level and Output Period.



The Analog High Adjustment value may be changed to adjust the output current when the Output Level is 100%.

The Analog Low Adjustment value may be changed to adjust the output current when the Output Level is 0%.

The Output Level determines the output current level between “20mA + Analog High Adjustment” and “4mA + Analog Low Adjustment”.

The Output Period sets how often the SOLO controller updates the output value.

There are seven parameters that define these four factors.

Analog High Adjustment

Analog High Adjustment (A1MA). Range -3895 to 659 uA.

Analog Low Adjustment

Analog Low Adjustment (A1ML). Range -964 to 3895 uA.

Output Level

Output 1 Level (oUt 1). Range: 0.0 to 100%
Output 1 Upper Limit (o 1nA). Range of 0.0 to 100%
Output 1 Lower Limit (o 1nL). Range of 0.00 to 100%

Output Period

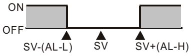
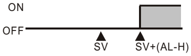
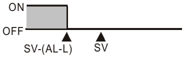
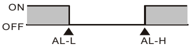
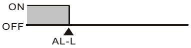
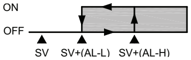
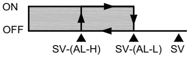
Output 1 Heating Period (o 1-H). Range: 0.1 to 60 seconds
Output 1 Cooling Period (o 1-C). Range: 0.1 to 60 seconds

Alarm Outputs

The SOLO Basic controller can support two alarms on select models.

See the Alarm Output Chart on the following page.

Alarm Output Types

Set Value	Alarm Type	Alarm Output Operation
0	Alarm function disabled	Output is OFF
1	Deviation upper- and lower-limit: Alarm output activates when PV value is higher than the setting value SV+(AL-H) or lower than the setting value SV-(AL-L).	
2	Deviation upper limit: Alarm output activates when PV value is higher than the setting value SV+(AL-H).	
3	Deviation lower limit: Alarm output activates when PV value is lower than the setting value SV-(AL-L).	
4	Absolute value upper and lower limit: Alarm output activates when PV value is higher than the setting value AL-H or lower than the setting value AL-L.	
5	Absolute value upper limit: Alarm output activates when PV value is higher than the setting value AL-H.	
6	Absolute value lower limit: Alarm output activates when PV value is lower than the setting value AL-L.	
7	Hysteresis upper limit alarm output: Alarm output activates when PV value is higher than the setting value SV+(AL-H). The alarm output turns OFF when the PV value is lower than the setting value SV+(AL-L).	
8	Hysteresis lower limit alarm output: Alarm output activates when PV value is lower than the setting value SV-(AL-H). The alarm output turns OFF when the PV value is higher than the setting value SV-(AL-L).	
9	Disconnection Alarm: This alarm output is enabled if the sensor connection is incorrect or has been disconnected.	

Note: Alarm types 1-9 will enable the alarm output if a sensor disconnect or incorrect connection is detected.

CHAPTER

5

CONTROL MODES

In this Chapter...

The SOLO Basic controller can be configured for any of the following control modes.

- PID control
- On / Off control
- Manual control

PID Control

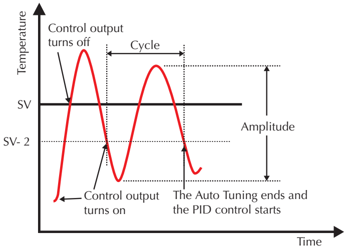
Auto Tuning

The SOLO Basic controllers support an Auto Tuning feature to set up the following PID parameters automatically.

P	Proportional Band
I	Integral Time
d	Derivative Time
PdoF	PD Control Offset

Use the ▼ and ▲ buttons to set the desired setpoint and press SET to save before starting the auto-tune process.

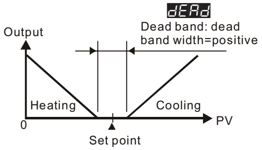
To start the Auto Tuning, set the parameter Auto Tuning (AT) to On. The controller automatically controls the output to change the PV as shown below.



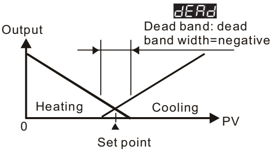
Once the Auto Tuning process is completed, the SOLO Basic controller calculates the above PID parameters and starts the PID control with the new parameter values immediately.

Deadband

The range around the PV in which the heating/cooling outputs remain off. A negative deadband has the opposite effect allowing both outputs to be on in the area around the PV.



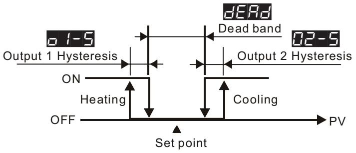
Dual control with PID
positive deadband



Dual control with PID
negative deadband

On / Off Control

In the On / Off control mode the output is controlled according to the difference between the SV and the PV. If the PV is lower than the SV and the output is set for heating, the heating output is turned on. If the PV is higher than the SV and the output is set for cooling, the cooling output is turned on. Hysteresis can be used to avoid output chatter and when in dual output mode Deadband will help prevent bouncing between heating and cooling.



Dual loop ON / OFF control output operation

Hysteresis

If heating, this is the value below the set temperature that the PV must fall to before the output will turn on. If cooling, this is the value above the set temperature that the PV must rise to before the output will turn on.

Deadband

The range around the PV in which the heating/cooling outputs remain off. A negative deadband has the opposite effect allowing both outputs to be on in the area around the PV.

Manual Control

In the Manual control, the output of the controller is manipulated manually by the operator. Adjust the values of the Output Level parameters (oUt1 , oUt2) to control the output levels.

Error Display Information

The chart below illustrates the possible error displays shown on the SOLO Basic Temperature controller.

Controller Error Display

Display Position	Display	Meaning	Cause	Corrective Action
PV	no	No sensor input	The input terminals are open.	Check the input wiring. If the problem still exists, replace the sensor or the controller.
SV	Cont			
PV	SEn	Sensor Type Error	Temperature is out of range for sensor type	Check sensor type and it's condition. If damaged or wrong replace and update settings. Check actual condition to make sure temperature is within sensor range.
SV	Err			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1-800-633-0405

Instruction Manual
Fuji Timing Relay
Type MS4SM, MS4SA, MS4SC

SAFETY PRECAUTIONS

Before installation, wiring, operation, maintenance, and inspection of the device, be sure to read the operating instructions carefully to ensure proper operation. Care should be taken that the operating instructions will be furnished to the maintenance supervisors of final users.

WARNING	:Incorrect handling of the device may result in death or serious injury.
● Turn off the power supply prior to installation, removal, wiring, maintenance or inspection to prevent electric shocks and burns due to short-circuit.	

CAUTION :Incorrect handling of the device may result in minor injury or physical damage. Even some "Caution" items may also result in a serious accident.

- ● If the device defect may cause danger to life or damage to property, careful attention must be applied to ensure the operation. It may also be necessary to incorporate fail-safe measures if connected to a critical process or safety application.
- ● Do not use one found damaged or deformed when unpacked.
- ● Installation, electric work, maintenance and inspection of the device should be performed by qualified engineers having special knowledge.
- ● Do not install the device in a place of environment with high temperature, humidity, dust, corrosive gases, excessive vibration, etc. to prevent fire accidents and malfunction of the device.
- ● Use the device in a range of the rated voltage and current.
- ● Make connection on the power supply and load in a correct manner. Or, fire accident may be occur.
- ● Care should be taken to prevent entry of foreign objects such as dust, concrete chips, Iron powder, etc., or it may result in poor contacts, defective release unless it is permitted.
- ● Make sure that terminals are fully tightened periodically to prevent malfunction.
- ● Never disassemble, modify or repair the product.
- ● When the device is to be disposed of, it should be handled as an industrial waste.

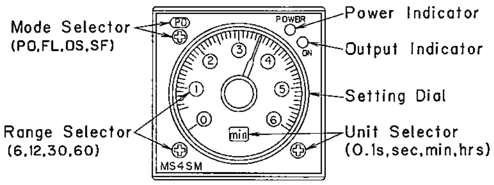
■ Specification

Type	MS4SM	MS4SA	MS4SC
Operation Mode	Multi-mode	On-delay	On-delay
Terminal Form	11-Pin	8-Pin	8-Pin
Timed Contact	DPDT	DPDT	SPDT
Instantaneous Contact	—	—	SPDT
Applicable Socket	TP411X	TP48X	TP48X

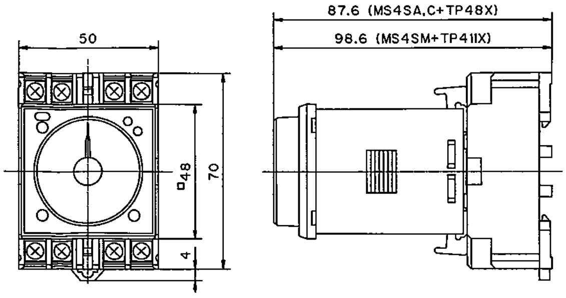
■ Timing Range Chart

Range Selection	Unit Selection			
	× 0.1s	sec	min	hrs
6	0.05-0.6s	0.5-6s	0.5-6min	0.5-6h
12	0.1-1.2s	1-12s	1-12min	1-12h
30	0.25-3s	2.5-30s	2.5-30min	2.5-30h
60	0.5-6s	5-60s	5-60min	5-60h

■ Nomenclature

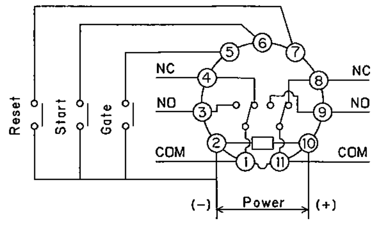


■ Dimensions. mm

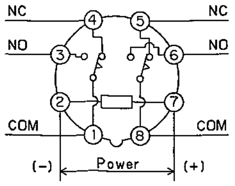


■ Wiring Diagrams

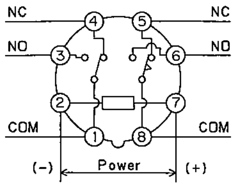
MS4SM



MS4SA



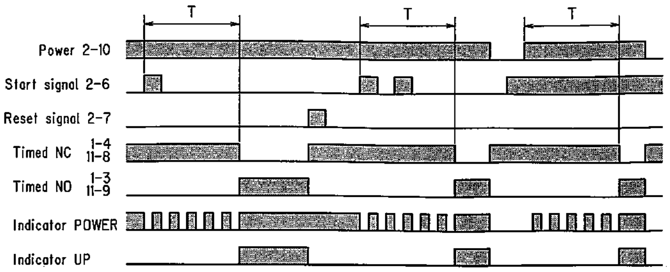
MS4SC



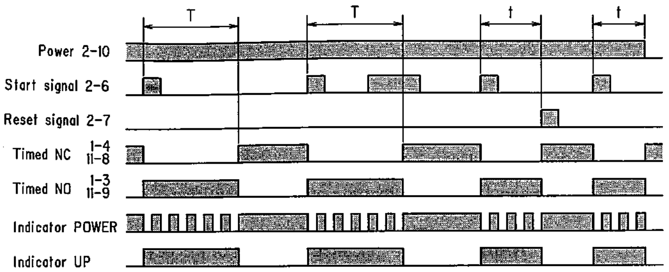
※ When the Gate is input. stops the time count.

■ Timing Diagrams (MS4SM)

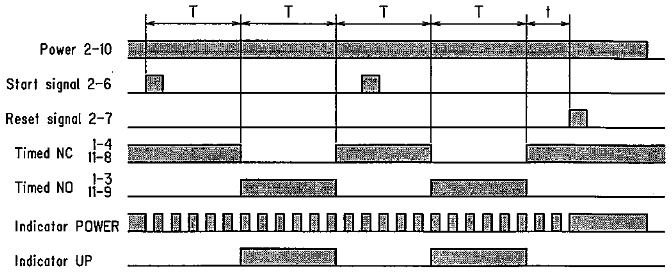
On-delay (PO)



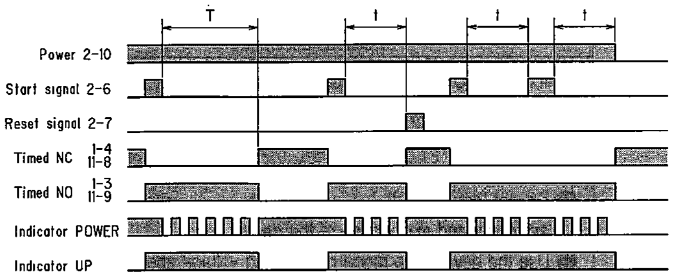
One-shot (OS)



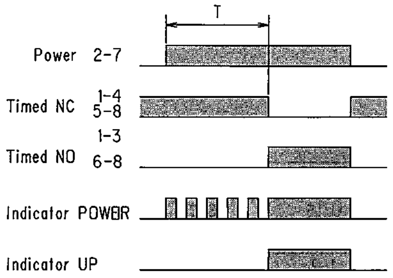
Flicker (FL)



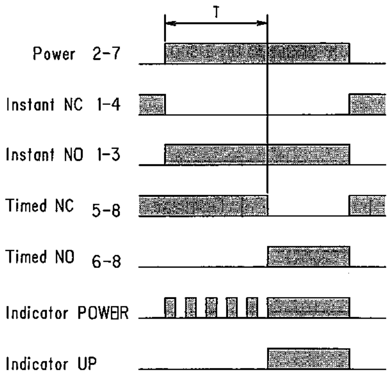
Signal off-delay (SF)



■ Timing Diagrams (MS4SA)



(MS4SC)



■ DC power supply

DC-operated timers are available for use with single-phase full-wave rectification DC power supplies.

■ Others

1. Set the time within the range of the timing scale divisions. To make a timer output a signal as soon as it is power ON, turn the dial fully counterclockwise.
2. The time setting dial can be turned while the timer is operating. However, the time for that operation will be incorrect.
3. Do not try to turn the time setting dial to outside the timing range. Turn it carefully, do not turn forcibly.
4. Do not remove the case of the timer, or operating characteristics will be changed.
5. Keep the timer away from corrosive gas, water, oil, dust, and direct sunlight. Do not use the timer in these circumstances.
6. Do not use the timer where it will be subjected to vibration or shock.
7. The case of the timer can be damaged by organic solvents such as thinner and benzene, and by corrosive substances such as strong acid or alkaloids. Keep the timer away from these substances.
8. The storage temperature must be between-25° and +60℃.

   If the timer has been left at-10℃ or below, leave it at room temperature for three hours or more before applying power.

METHOD 350.1

DETERMINATION OF AMMONIA NITROGEN BY SEMI-AUTOMATED COLORIMETRY

Edited by James W. O'Dell
Inorganic Chemistry Branch
Chemistry Research Division

Revision 2.0
August 1993

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

METHOD 350.1

DETERMINATION OF AMMONIA NITROGEN BY SEMI-AUTOMATED COLORIMETRY

1.0 SCOPE AND APPLICATION

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

1.2 The applicable range is 0.01-2.0 mg/L NH_3 as N. Higher concentrations can be determined by sample dilution. Approximately 60 samples per hour can be analyzed.

1.3 This method is described for macro glassware; however, micro distillation equipment may also be used.

2.0 SUMMARY OF METHOD

2.1 The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is distilled into a solution of boric acid. Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color formed is intensified with sodium nitroprusside and measured colorimetrically.

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.

2.4 Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Section 9.0, Quality Control.

3.0 DEFINITIONS

3.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

3.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.3 **Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.4 **Laboratory Fortified Blank (LFB)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.5 **Laboratory Fortified Sample Matrix (LFM)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.6 **Laboratory Reagent Blank (LRB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.7 **Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear.

3.8 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.9 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.10 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.11 **Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

4.0 INTERFERENCES

4.1 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.

4.2 Residual chorine must be removed by pretreatment of the sample with sodium thiosulfate or other reagents before distillation.

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

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS.

5.3.1 Sulfuric acid (Section 7.6)

5.3.2 Phenol (Section 7.7)

5.3.3 Sodium nitroprusside (Section 7.10)

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2 Glassware - Class A volumetric flasks and pipets as required.

6.3 An all-glass distilling apparatus with an 800-1000 mL flask.

6.4 Automated continuous flow analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

6.4.1 Sampling device (sampler)

6.4.2 Multichannel pump

6.4.3 Reaction unit or manifold

6.4.4 Colorimetric detector

6.4.5 Data recording device

7.0 REAGENTS AND STANDARDS

7.1 Reagent water - Ammonia free: 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. Regeneration of the column should be carried out according to the manufacturer's instructions.

Note: All solutions must be made with ammonia-free water.

7.2 Boric acid solution (20 g/L): Dissolve 20 g H_3BO_3 (CASRN 10043-35-3) in reagent water and dilute to 1 L.

7.3 Borate buffer: Add 88 mL of 0.1 N NaOH (CASRN 1310-73-2) solution to 500 mL of 0.025 M sodium tetraborate solution (5.0 g anhydrous $Na_2B_4O_7$ [CASRN 1330-43-4] or 9.5 g $Na_2B_4O_7 \bullet 10H_2O$ [CASRN 1303-96-4] per L) and dilute to 1 L with reagent water.

7.4 Sodium hydroxide, 1 N: Dissolve 40 g NaOH in reagent water and dilute to 1 L.

7.5 Dechlorinating reagents: A number of dechlorinating reagents may be used to remove residual chlorine prior to distillation. These include:

7.5.1 Sodium thiosulfate: Dissolve 3.5 g $Na_2S_2O_3 \bullet 5H_2O$ (CASRN 10102-17-7) in reagent water and dilute to 1 L. One mL of this solution will remove 1 mg/L of residual chlorine in 500 mL of sample.

7.5.2 Sodium sulfite: Dissolve 0.9 g $Na2SO_3$ (CASRN 7757-83-7) in reagent water and dilute to 1 L. One mL removes 1 mg/L Cl per 500 mL of sample.

7.6 Sulfuric acid 5 N: Air scrubber solution. Carefully add 139 mL of conc. sulfuric acid (CASRN 7664-93-9) to approximately 500 mL of reagent water. Cool to room temperature and dilute to 1 L with reagent water.

7.7 Sodium phenolate: Using a 1-L Erlenmeyer flask, dissolve 83 g phenol (CASRN 108-95-2) in 500 mL of distilled water. In small increments, cautiously add with agitation, 32 g of NaOH. Periodically cool flask under water faucet. When cool, dilute to 1 L with reagent water.

7.8 Sodium hypochlorite solution: Dilute 250 mL of a bleach solution containing 5.25% NaOCl (CASRN 7681-52-9) (such as "Clorox") to 500 mL with reagent water. Available chlorine level should approximate 2-3%. Since "Clorox" is a proprietary product, its formulation is subject to change. The analyst must remain alert to detecting any variation in this product significant to its use in this procedure. Due to the instability of this product, storage over an extended period should be avoided.

7.9 Disodium ethylenediamine-tetraacetate (EDTA) (5%): Dissolve 50 g of EDTA (disodium salt) (CASRN 6381-92-6) and approximately six pellets of NaOH in 1 L of reagent water.

7.10 Sodium nitroprusside (0.05%): Dissolve 0.5 g of sodium nitroprusside (CASRN 14402-89-2) in 1 L of reagent water.

7.11 Stock solution: Dissolve 3.819 g of anhydrous ammonium chloride, NH_4Cl (CASRN 12125-02-9), dried at 105°C, in reagent water, and dilute to 1 L.
1.0 mL = 1.0 mg NH_3-N.

7.12 Standard Solution A: Dilute 10.0 mL of stock solution (Section 7.11) to 1 L with reagent water. 1.0 mL = 0.01 mg NH_3-N.

7.13 Standard Solution B: Dilute 10.0 mL of standard solution A (Section 7.12) to 100.0 mL with reagent water. 1.0 mL = 0.001 mg NH_3-N.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.2 Samples must be preserved with H_2SO_4 to a pH <2 and cooled to 4°C at the time of collection.

8.3 Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at 4°C and may be held for up to 28 days.

9.0 QUALITY CONTROL

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

9.2.2 Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.2.3 Quality Control Sample (QCS) -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

9.2.4 Method Detection Limit (MDL) -- MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit.[9] To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

where, t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates]

S = standard deviation of the replicate analyses

MDLs should be determined every six months, when a new operator begins work or whenever there is a significant change in the background or instrument response.

9.3 ASSESSING LABORATORY PERFORMANCE

9.3.1 Laboratory Reagent Blank (LRB) -- The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

9.3.2 Laboratory Fortified Blank (LFB) -- The laboratory must analyze at least one LFB with each batch of samples. Calculate accuracy as percent recovery (Section 9.4.2). If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

9.3.3 The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = $x + 3S$
LOWER CONTROL LIMIT = $x - 3S$

The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to 10 new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to established an on-going precision statement for the level of concentrations included in the LFB. These data must be kept on file and be available for review.

9.3.4 Instrument Performance Check Solution (IPC) -- For all determinations the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, after every 10th sample (or more frequently, if required) and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within ±10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ±10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

9.4 ASSESSING ANALYTE RECOVERY AND DATA QUALITY

9.4.1 Laboratory Fortified Sample Matrix (LFM) -- The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than four times the MDL. The added analyte concentration should be the same as that used in the laboratory fortified blank.

9.4.2 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 90-110%. Percent recovery may be calculate using the following equation:

$$R = \frac{C_s - C}{s} \text{ x } 100$$

where,

R = percent recovery
C_s = fortified sample concentration
C = sample background concentration
s = concentration equivalent of analyte added to sample

9.4.3 If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Section 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.

9.4.4 Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prepare a series of at least three standards, covering the desired range, and a blank by diluting suitable volumes of standard solutions (Sections 7.12 and 7.13) to 100 mL with reagent water.

10.2 Process standards and blanks as described in Section 11.0, Procedure.

10.3 Set up manifold as shown in Figure 1.

10.4 Prepare flow system as described in Section 11.0, Procedure.

10.5 Place appropriate standards in the sampler in order of decreasing concentration and perform analysis.

10.6 Prepare standard curve by plotting instrument response against concentration values. A calibration curve may be fitted to the calibration solutions concentration/response data using computer or calculator based regression curve fitting techniques. Acceptance or control limits should be established using the difference between the measured value of the calibration solution and the "true value" concentration.

10.7 After the calibration has been established, it must be verified by the analysis of a suitable QCS. If measurements exceed ±10% of the established QCS value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis. Periodic reanalysis of the QCS is recommended as a continuing calibration check.

11.0 PROCEDURE

11.1 Preparation of equipment: Add 500 mL of reagent water to an 800 mL Kjeldahl flask. The addition of boiling chips that have been previously treated with dilute NaOH will prevent bumping. Steam out the distillation apparatus until the distillate shows no trace of ammonia.

11.2 Sample preparation: Remove the residual chorine in the sample by adding dechlorinating agent (Section 7.5) equivalent to the chlorine residual. To 400 mL of sample add 1 N NaOH (Section 7.4), until the pH is 9.5, check the pH during addition with a pH meter or by use of a short range pH paper.

11.3 Distillation: Transfer the sample, the pH of which has been adjusted to 9.5, to an 800 mL Kjeldahl flask and add 25 mL of the borate buffer (Section 7.3). Distill 300 mL at the rate of 6-10 mL/min. into 50 mL of 2% boric acid (Section 7.2) contained in a 500 mL Erlenmeyer flask.

Note: The condenser tip or an extension of the condenser tip must extend below the level of the boric acid solution.

11.4 Since the intensity of the color used to quantify the concentration is pH dependent, the acid concentration of the wash water and the standard ammonia solutions should approximate that of the samples.

11.5 Allow analysis system to warm up as required. Feed wash water through sample line.

11.6 Arrange ammonia standards in sampler in order of decreasing concentration of nitrogen. Complete loading of sampler tray with unknown samples.

11.7 Switch sample line from reagent water to sampler and begin analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Prepare a calibration curve by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.

12.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12.3 Report results in mg NH_3-N/L.

13.0 METHOD PERFORMANCE

13.1 In a single laboratory (EMSL-Cincinnati), using surface water samples at concentrations of 1.41, 0.77, 0.59, and 0.43 mg NH_3-N/L, the standard deviation was ±0.005.

13.2 In a single laboratory (EMSL-Cincinnati), using surface water samples at concentrations of 0.16 and 1.44 mg NH_3-N/L, recoveries were 107% and 99%, respectively.

13.3 The interlaboratory precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in mg NH_3-N/L.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036, (202)872-4477.

15.0 WASTE MANAGEMENT

15.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society at the address listed in Section 14.3.

16.0 REFERENCES

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17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

TABLE 1. INTERLABORATORY PRECISION AND ACCURACY DATA

Number of Values Reported	True Value (T)	Mean (X)	Residual for X	Standard Deviation (S)	Residual for S
134	0.270	0.2670	-0.0011	0.0342	0.0015
157	0.692	0.6972	0.0059	0.0476	-0.0070
136	1.20	1.2008	0.0001	0.0698	-0.0112
195	1.60	1.6095	0.0076	0.1023	0.0006
142	3.00	3.0128	0.0069	0.1677	-0.0067
159	3.50	3.4991	-0.0083	0.2168	0.0165
156	3.60	3.5955	-0.0122	0.1821	-0.0234
200	4.20	4.2271	0.0177	0.2855	0.0488
196	8.76	8.7257	-0.0568	0.4606	-0.0127
156	11.0	11.0747	0.0457	0.5401	-0.0495
142	13.0	12.9883	-0.0465	0.6961	0.0027
199	18.0	17.9727	-0.0765	1.1635	0.2106

REGRESSIONS: X = 1.003T - 0.003, S = 0.052T + 0.019

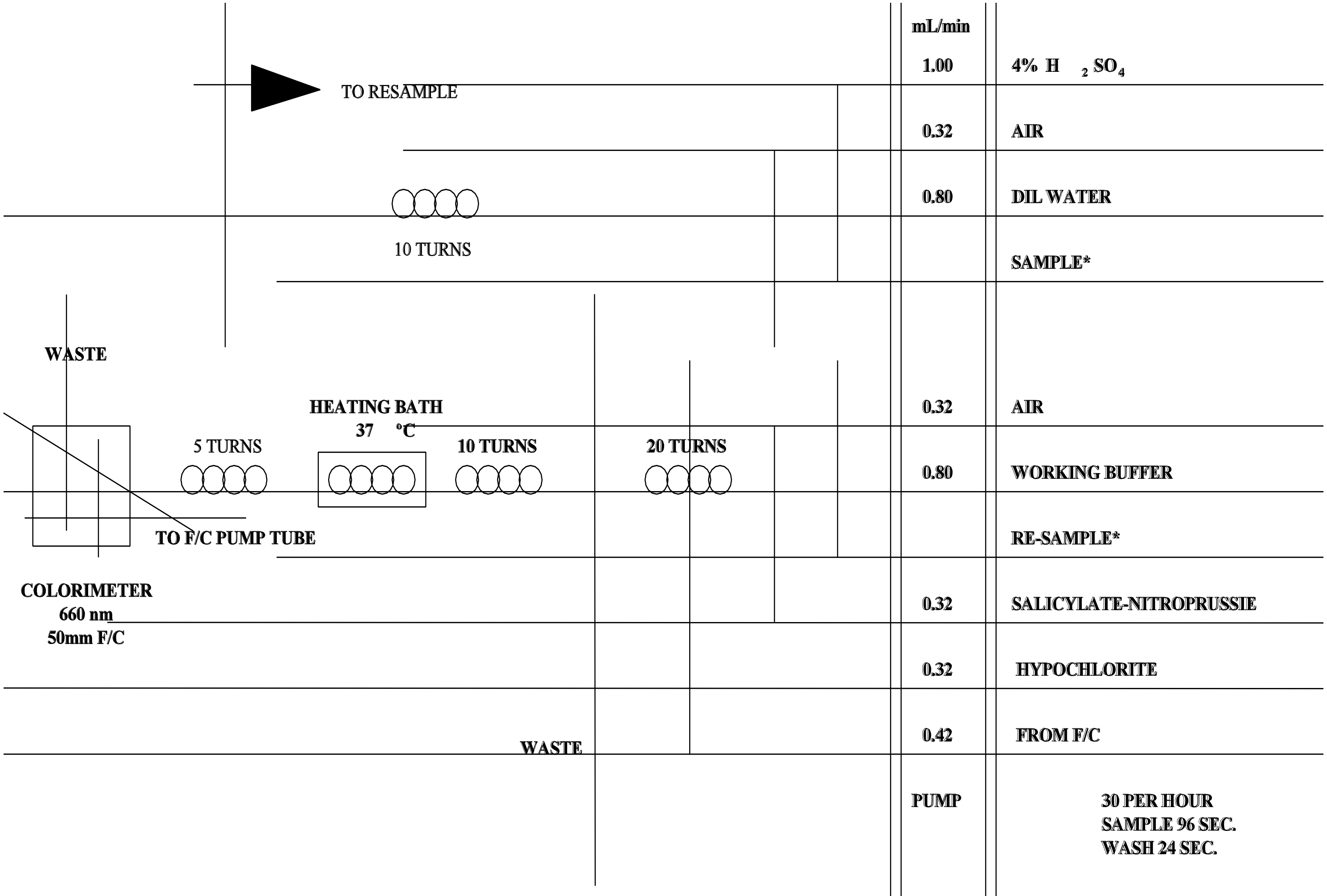

Figure 1
Ammonia Manifold

METHOD 335.4

DETERMINATION OF TOTAL CYANIDE BY SEMI-AUTOMATED COLORIMETRY

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Inorganic Chemistry Branch
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Revision 1.0
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U.S. ENVIRONMENTAL PROTECTION AGENCY
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DETERMINATION OF TOTAL CYANIDE BY SEMI-AUTOMATED COLORIMETRY

1.0 SCOPE AND APPLICATION

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

1.2 The applicable range is 5 to500 µg/L.

2.0 SUMMARY OF METHOD

2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a manual reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is converted to cyanogen chloride by reactions with chloramine-T, that subsequently reacts with pyridine and barbituric acid to give a red-colored complex.

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.

2.2 Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Section 9.0, Quality Control.

3.0 DEFINITIONS

3.1 **Calibration Blank (CB)** -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

3.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.3 **Instrument Performance Check Solution (IPC)** -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.4 **Laboratory Fortified Blank (LFB)** -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.5 **Laboratory Fortified Sample Matrix (LFM)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.6 **Laboratory Reagent Blank (LRB)** -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.7 **Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear.

3.8 **Material Safety Data Sheet (MSDS)** -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.9 **Method Detection Limit (MDL)** -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.10 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.11 **Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

4.0 INTERFERENCES

4.1 Several interferences are encountered with this method. Some of the known interferences are aldehydes, nitrate-nitrite, oxidizing agents, such as chlorine, thiocyanate, thiosulfate and sulfide. Multiple interferences may require the analysis of a series of laboratory fortified sample matrices (LFM) to verify the suitability of the chosen treatment. Some interferences are eliminated or reduced by the distillation.

4.2 Sulfides adversely affect the procedure by producing hydrogen sulfide during distillation. If a drop of the sample on lead acetate test paper indicates the presence of sulfide, treat 25 mL more of the stabilized sample (pH ≥12) than that required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate, measure the sample to be used for analysis. Avoid a large excess of cadmium and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material.

4.3 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation nitrate and nitrite will form nitrous acid that will react with some organic compounds to form oximes. These oximes will decompose under test conditions to generate HCN. The interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid.

4.4 Oxidizing agents, such as chlorine, decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch paper (KI-starch paper) at time of collection; a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper; then add an additional 0.06 g of ascorbic acid for each liter of sample volume. Sodium arsenite has also been employed to remove oxidizing agents.

4.5 Other compatible procedures for the removal or suppression of interferences may be employed provided they do not adversely effect the overall performance of the method.

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

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS.

5.3.1 Hydrochloric acid (Section 7.5)

5.3.2 Silver nitrate (Section 7.9)

5.3.3 Potassium cyanide (Section 7.10)

5.3.4 Sulfuric acid (Section 7.14)

5.4 Because of the toxicity of evolved hydrogen cyanide (HCN),distillation should be performed in a well vented hood.

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2 Glassware -- Class A volumetric flasks and pipets as required.

6.3 Midi reflux distillation apparatus including boiling flask condenser, and absorber as shown in Figure 1.

6.4 Heating mantel or heating block as required.

6.5 Automated continuous flow analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

6.5.1 Sampling device (sampler)

6.5.2 Multichannel pump

6.5.3 Reaction unit or manifold

6.5.4 Colorimetric detector

6.5.5 Data recording device

7.0 REAGENTS AND STANDARDS

7.1 Reagent water: Distilled or deionized water, free of the analyte of interest. ASTM Type II or equivalent.

7.2 Ascorbic acid: Crystal (CASRN-50-81-7)

7.3 Chloramine-T: Dissolve 2.0 g of chloramine-T (CASRN-127-65-1) in 500 mL of reagent water.

7.4 Magnesium Chloride Solution: Weigh 510 g of $MgCl_2•6H_2O$ (CASRN-7786-30-3) into a 1000 mL flask, dissolve and dilute to 1 L with reagent water.

7.5 Pyridine Barbituric Acid Reagent: Place 15 g of barbituric acid (CASRN-67-52-7) in a 1 L beaker. Wash the sides of the beaker with about 100 mL of reagent water. Add 75 mL of pyridine (CASRN-110-86-1) and mix. Add 15 mL of conc. HCl (CASRN-7647-01-0) and mix. Dilute to 900 mL with reagent water and mix until all the barbituric acid has dissolved. Transfer the solution to a 1 L flask and dilute to the mark.

7.6 Sodium dihydrogenphosphate buffer, 1 M: Dissolve 138 g of $NaH_2PO_4 \bullet H_2O$ (CASRN-10049-21-5) in 1 L of reagent water. Refrigerate this solution.

7.7 Sodium Hydroxide Solution, 1.25 N: Dissolve 50 g of NaOH (CASRN-1310-73-2) in reagent water, and dilute to 1 L with reagent water.

7.8 Sodium Hydroxide, 0.25 N: Dilute 200 mL of 1.25 N Sodium hydroxide solution (Section 7.7) to 1 L with reagent water.

7.9 Standard Silver Nitrate Solution, 0.0192 N: Prepare by crushing approximately 5 g $AgNO_3$ (CASRN-7761-88-8) crystals and drying to constant weight at 40°C. Weigh out 3.2647 g of dried $AgNO_3$, dissolve in reagent water, and dilute to 1000 mL (1 mL = 1 mg CN).

7.10 Stock Cyanide Solution: Dissolve 2.51 g of KCN (CASRN-151-50-8) and 2 g KOH (CASRN-1310-58-3) in 900 mL of reagent water. Standardize with 0.0192 N $AgNO_3$ (Section 7.9). Dilute to appropriate concentration so that 1 mL = 1 mg CN.

7.11 Standard Cyanide Solution, intermediate: Dilute 10.0 mL of stock (1 mL = 1 mg CN) (Section 7.10) to 100.0 with reagent water (1 mL = 100.0 µg CN).

7.12 Working Standard Cyanide Solution: Prepare fresh daily by diluting 20.0 mL of intermediate cyanide solution (Section 7.11) to 200.0 mL with reagent water and store in a glass stoppered bottle. 1 mL = 10.0 µg CN.

7.13 Sulfamic Acid: (CASRN-212-57-3).

7.14 Sulfuric Acid, 18N: Slowly add 500 mL of concentrated H_2SO_4 (CASRN-5329-14-6) to 500 mL of reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.2 If the sample contains chlorine or hydrogen sulfide, see Section 4.0 for treatment.

8.3 Samples must be preserved with sodium hydroxide pH ≥ 12 and cooled to 4°C at the time of collection.

8.4 Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at 4°C and may be held for up to 14 days.

9.0 QUALITY CONTROL

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, the periodic analysis of laboratory reagent blanks, fortified blanks, and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

9.2.2 Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ±10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.2.3 Quality Control Sample (QCS) -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding on with the initial determination of MDLs or continuing with on-going analyses.

9.2.4 Method Detection Limit (MDL) -- MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit.(4) To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

where, t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates]
S = standard deviation of the replicate analyses

MDLs should be determined every six months, when a new operator begins work or whenever there is a significant change in the background or instrument response.

9.3 ASSESSING LABORATORY PERFORMANCE

9.3.1 Laboratory Reagent Blank (LRB) -- The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

9.3.2 Laboratory Fortified Blank (LFB) -- The laboratory must analyze at least one LFB with each batch of samples. Calculate accuracy as percent recovery (Section 9.4.2). If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

9.3.3 The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data becomes available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S
LOWER CONTROL LIMIT = x - 3S

The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations included in the LFB. These data must be kept on file and be available for review.

9.3.4 Instrument Performance Check Solution (IPC) -- For all determinations, the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, after every tenth sample (or more frequently, if required), and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within ±10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ±10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

9.4 ASSESSING ANALYTE RECOVERY AND DATA QUALITY

9.4.1 Laboratory Fortified Sample Matrix (LFM) -- The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples. In each case, the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than four times the MDL. The added analyte concentration should be the same as that used in the laboratory fortified blank.

9.4.2 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 90-110%. Percent recovery may be calculated using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where,

R = percent recovery
C_s = fortified sample concentration
C = sample background concentration
s = concentration equivalent of analyte added to sample

9.4.3 If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Section 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.

9.4.4 Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prepare a series of at least three standards, covering the desired range, and a blank by pipetting suitable volumes of working standard solution (Section 7.12) into 100 mL volumetric flasks. To each standard (except those to be distilled) add 20 mL of 1.25 N sodium hydroxide and dilute to 100 mL with reagent water.

10.2 It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least two standards (a high and low) and a blank be distilled and compared to similar values on the standard curve to insure that the distillation technique is reliable. If distilled standards do not agree within ±10% of the undistilled standards the analyst should find the cause of the apparent error before proceeding. Before distillation, standards should contain 4 mL 0.25N NaOH (Section 7.8) per 50 mL.

10.3 Set up the manifold as shown in Figure 2 in a hood or a well-ventilated area.

10.4 Allow the instrument to warm up as required. Pump all reagents, with 0.25N NaOH in the sample line, until a stable baseline is achieved.

10.5 Place appropriate standards in the sampler in order of decreasing concentration and perform analysis.

10.6 Prepare standard curve by plotting instrument response against concentration values. A calibration curve may be fitted to the calibration solutions concentration/response data using computer or calculator based regression curve fitting techniques. Acceptance or control limits should be established using the difference between the measured value of the calibration solution and the "true value" concentration.

10.7 After the calibration has been established, it must be verified by the analysis of a suitable QCS. If measurements exceed ±10% of the established QCS value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis. Periodic reanalysis of the QCS is recommended as a continuing calibration check.

11.0 PROCEDURE

11.1 Pipet 50 mL of sample or an aliquot diluted to 50 mL into the MIDI distillation boiling flask. Add boiling chips as required. Pipet 50 mL of sodium hydroxide 0.25 N (Section 7.8) into the absorbing tube. Connect the boiling flask, condenser, and absorber in the train as shown in Figure 1.

11.2 Start a slow stream of air entering the boiling flask by adjusting the vacuum source to maintain about three bubbles per minute.

11.3 If samples contain NO_3 and/or NO_2, add 0.2 g of sulfamic acid (Section 7.13) after the air rate is set through the air inlet tube. Mix for three minutes prior to addition of H_2SO_4.

11.4 Slowly add 5 mL 18 N sulfuric acid (Section 7.14) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for three minutes. Pour 2 mL of magnesium chloride (Section 7.4) into the air inlet and wash down with a stream of water.

11.5 Heat the solution to boiling. Reflux for one and one half hours. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source and remove absorber tube.

11.6 Fill and connect reagent containers and start system. Allow the instrument to warm up as required. Pump all reagents, with 0.25N NaOH in the sample line, until a stable baseline is achieved.

11.7 Place standards, distilled standards and unknown samples (ALL in 0.25N NaOH) in sampler tray. Calibrate instrument and begin analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

12.l Prepare a calibration curve by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.

12.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

l2.3 Report results in mg/L.

13.0 METHOD PERFORMANCE

13.1 The interlaboratory precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in mg CN/L.

13.2 Single laboratory precision data can be estimated at 50-75% of the interlaboratory precision estimates.

l4.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036, (202)872-4477.

15.0 WASTE MANAGEMENT

15.1 The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess Reagents, samples, and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society at the address listed in Section 14.3.

16.0 REFERENCES

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3. USEPA Contract Laboratory Program, Document Number ILMO 1.0, Method for Total Cyanide Analysis by MIDI Distillation #335.2 CLP-M.

4. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B.

17. TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

TABLE 1. INTERLABORATORY PRECISION AND ACCURACY DATA

Number of Values Reported	True Value (T)	Mean (X)	Residual for X	Standard Deviation (S)	Residual for S
126	0.020	0.0182	0.0002	0.0055	0.0000
94	0.055	0.0501	-0.0014	0.0092	-0.0007
158	0.090	0.0843	-0.0008	0.0171	0.0027
118	0.110	0.1045	0.0003	0.0165	-0.0004
148	0.180	0.1683	-0.0030	0.0236	-0.0023
92	0.270	0.2538	-0.0038	0.0275	-0.0099
132	0.530	0.5019	-0.0049	0.0775	0.0069
119	0.540	0.5262	0.0098	0.0679	-0.0039
148	0.610	0.5803	-0.0032	0.0851	0.0043
94	0.700	0.6803	0.0105	0.1082	0.0159
92	0.800	0.7726	0.0069	0.0880	-0.0170
158	0.970	0.9508	0.0222	0.1464	0.0197

REGRESSIONS: X = 0.959T - 0.001, S = 0.128T + 0.003

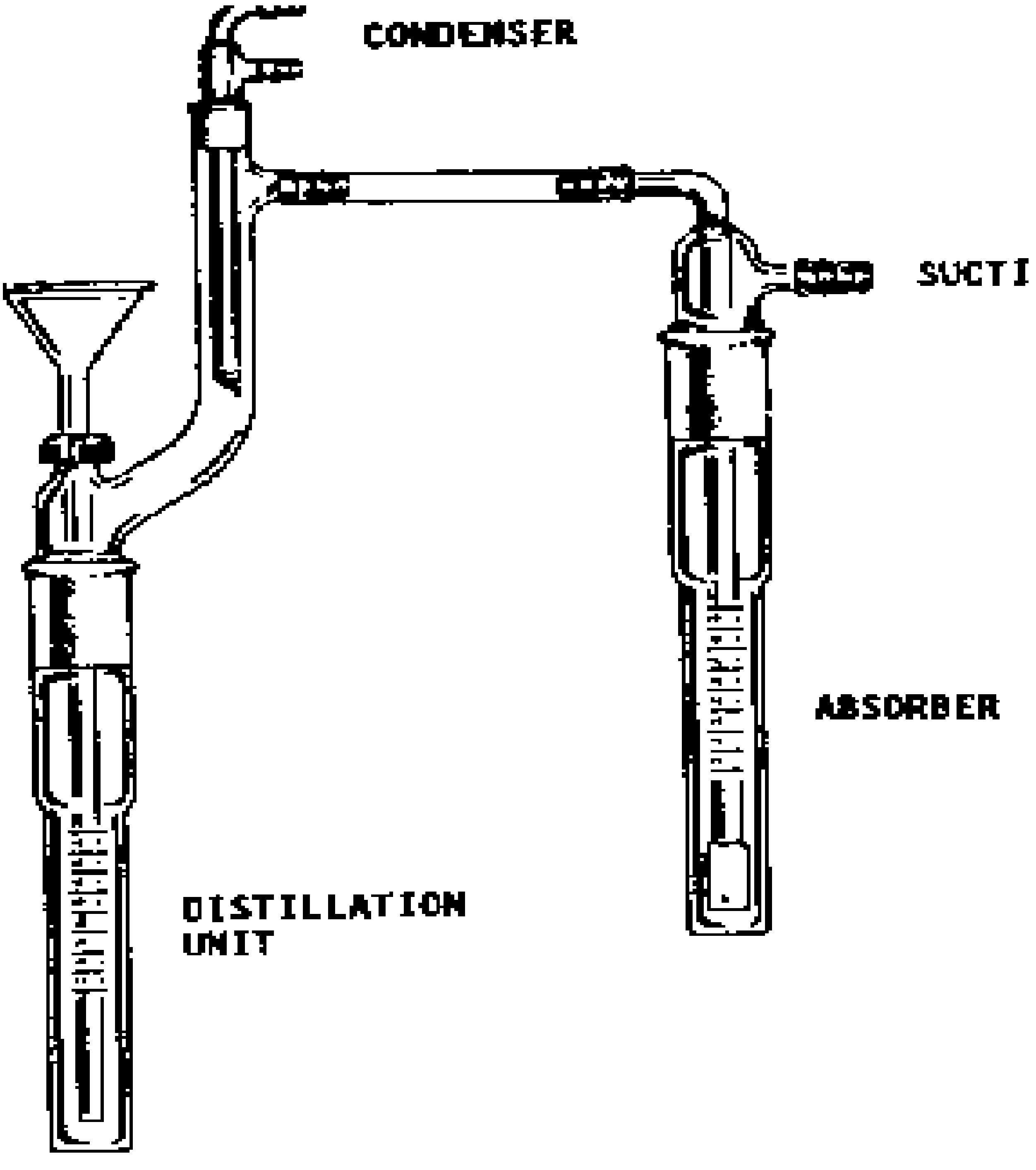

FIGURE 1. MIDI DISTILLATION APPARATUS

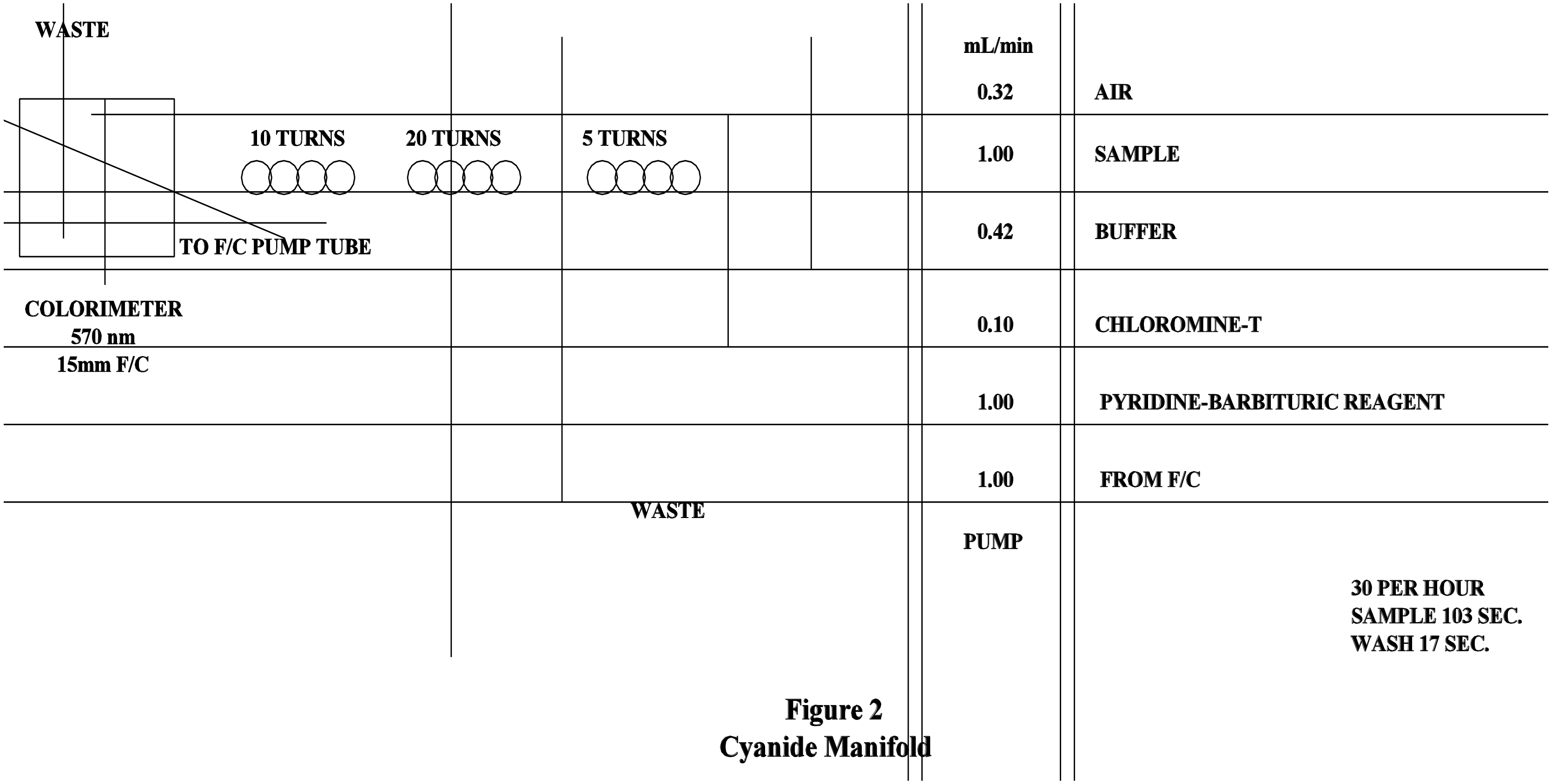

Figure 2
Cyanide Manifold

METHOD 420.4

DETERMINATION OF TOTAL RECOVERABLE PHENOLICS BY SEMI-AUTOMATED COLORIMETRY

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METHOD 420.4

DETERMINATION OF TOTAL RECOVERABLE PHENOLICS BY SEMI-AUTOMATED COLORIMETRY

1.0 SCOPE AND APPLICATION

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

1.2 The applicable range is from 2 to 500 µg/L. The working ranges are 2 to 200 µg/L and 10 to 500 µg/L.

2.0 SUMMARY OF METHOD

2.1 This semi-automated method is based on the distillation of phenol and subsequent reaction of the distillate with alkaline ferricyanide and 4-aminoantipyrine to form a red complex which is measured at 505 or 520 nm.

2.2 Color response of phenolic materials with 4-aminoantipyrine is not the same for all compounds. Because phenolic type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason, phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds 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.

2.4 Limited performance based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Sect. 9.0, Quality Control.

3.0 DEFINITIONS

3.1 CALIBRATION BLANK (CB) -- A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

3.2 CALIBRATION STANDARD (CAL) -- A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.3 INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) -- A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.4 LABORATORY FORTIFIED BLANK (LFB) -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.5 LABORATORY FORTIFIED SAMPLE MATRIX (LFM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.6 LABORATORY REAGENT BLANK (LRB) -- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

3.7 LINEAR CALIBRATION RANGE (LCR) -- The concentration range over which the instrument response is linear.

3.8 MATERIAL SAFETY DATA SHEET (MSDS) -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.9 METHOD DETECTION LIMIT (MDL) -- The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.10 QUALITY CONTROL SAMPLE (QCS) -- A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.11 STOCK STANDARD SOLUTION (SSS) -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

4.0 INTERFERENCES

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

4.2 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 an excess of ferrous ammonium sulfate (7.11). If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

4.3 Background contamination from plastic tubing and sample containers is eliminated by filling the wash receptacle by siphon (using Kel-F tubing) and using glass tubes for the samples and standards.

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

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

5.2 Each laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3 The following chemicals have the potential to be highly toxic or hazardous, consult MSDS.

5.3.1 Potassium ferricyanide (7.2)

5.3.2 Phenol (7.5)

5.3.3 Sulfuric acid (7.10)

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance -- Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2 Glassware -- Class A volumetric flasks and pipets as required.

6.3 Distillation apparatus, all glass consisting of a 1-L pyrex distilling apparatus with Graham condenser. Reduced volume apparatus also may be used.

6.4 pH meter with electrodes.

6.5 Automated continuous flow analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

6.5.1 Sampling device (sampler)

6.5.2 Multichannel pump

6.5.3 Reaction unit or manifold

6.5.4 Colorimetric detector

6.5.5 Data recording device

7.0 REAGENTS AND STANDARDS

7.1 Reagent water: Distilled or deionized water, free of the analyte of interest. ASTM type II or equivalent.

7.2 Buffered potassium ferricyanide: Dissolve 1.0 g potassium ferricyanide (CSRN 13746-66-2), 1.55 g boric acid (CASRN 10043-35-3), and 1.875 g potassium chloride (CASRN 7447-40-7) in 400 mL of reagent water. Adjust to pH of 10.3 with 1 N sodium hydroxide (CASRN 1310-73-2) (7.3) and dilute to 500 mL. Add 0.25 mL of Brij-35 (CASRN 9002-92-0). Prepare fresh weekly.

7.3 Sodium hydroxide (1N): Dissolve 20 g NaOH in 250 mL of reagent water, cool and dilute to 500 mL.

7.4 4-Aminoantipyrine: Dissolve 0.13 g of 4-aminoantipyrine (CASRN 83-07-8) in 150 mL of reagent water and dilute to 200 mL. Prepare fresh each day.

7.5 Stock phenol: Dissolve 0.50 g phenol (CASRN 108-95-2) in 500 mL of reagent water and dilute to 500 mL. Add 0.25 mL conc. H_2SO_4 (CASRN 7664-93-9) as preservative. 1.0 mL = 1.0 mg phenol.

7.6 Standard phenol solution A: Dilute 1.0 mL of stock phenol solution (7.5) to 100 mL with reagent water. 1.0 mL = 0.01 mg phenol.

7.7 Standard phenol solution B: Dilute 10.0 mL of standard phenol solution A (7.6) to 100 mL with reagent water. 1.0 mL = 0.001 mg phenol.

7.8 Standard solution C: Dilute 10.0 mL of standard phenol solution B (7.7) to 100 mL with reagent water. 1.0 mL = 0.0001 mg phenol.

7.9 Sodium hydroxide, 1+9: Dilute 10 mL of 1N NaOH (7.3) to 100 mL with reagent water.

7.10 Sulfuric acid, 1+9 : Slowly add 10 mL conc. H_2SO_4 (CASRN 7764-93-9) to 70 mL of reagent water. Cool and dilute to 100 mL with reagent water.

7.11 Ferrous ammonium sulfate: Dissolve 0.55 g ferrous ammonium sulfate in 250 mL reagent water containing 0.5 mL H_2SO_4 and dilute to 500 mL with freshly boiled and cooled reagent water.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Samples should be collected in glass bottles only. All bottles must be thoroughly cleansed and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

8.2 Samples must be preserved at time of collection with H_2SO_4 to a pH of < 2 and cooled to 4°C.

8.3 Samples should be analyzed as soon as possible after collection. If storage is required, preserved samples are maintained at 4°C and may be held up to 28 days.

9.0 QUALITY CONTROL

9.1 Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

9.2 INITIAL DEMONSTRATION OF PERFORMANCE

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

9.2.2 Linear Calibration Range (LCR) -- The LCR must be determined initially and verified every 6 months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.2.3 Quality Control Sample (QCS) -- When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ± 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

9.2.4 Method Detection Limit (MDL) -- MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit.(4) To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$\text{MDL} = (t) \times (S)$$

where, t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

MDLs should be determined every 6 months, when a new operator begins work or whenever there is a significant change in the background or instrument response.

9.3 ASSESSING LABORATORY PERFORMANCE

9.3.1 Laboratory Reagent Blank (LRB) -- The laboratory must analyze at least one LRB with each batch of samples. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

9.3.2 Laboratory Fortified Blank (LFB) -- The laboratory must analyze at least one LFB with each batch of samples. Calculate accuracy as percent recovery (Sect. 9.4.2). If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

9.3.3 The laboratory must use LFB analyses data to assess laboratory performance against the required control limits of 90-110%. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

UPPER CONTROL LIMIT = x + 3S
LOWER CONTROL LIMIT = x - 3S

The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to established an on-going precision statement for the level of concentrations included in the LFB. These data must be kept on file and be available for review.

9.3.4 Instrument Performance Check Solution (IPC) -- For all determinations the laboratory must analyze the IPC (a mid-range check standard) and a calibration blank immediately following daily calibration, after every tenth sample (or more frequently, if required), and at the end of the sample run. Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within ± 10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within ± 10%. If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift the instrument recalibrated. All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

9.4 ASSESSING ANALYTE RECOVERY AND DATA QUALITY

9.4.1 Laboratory Fortified Sample Matrix (LFM) -- The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than four times the MDL. The added analyte concentration should be the same as that used in the laboratory fortified blank.

9.4.2 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 90-110%. Percent recovery may be calculated using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where, R = percent recovery.
C_s = fortified sample concentration.
C = sample background concentration.
s = concentration equivalent of analyte added to sample.

9.4.3 If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (Sect. 9.3), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.

9.4.4 Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prepare a series of at least 3 standards, covering the desired range, and a blank by pipetting suitable volumes of working standard solutions (7.6, 7.7, 7.8) into 100-mL volumetric flasks. Suggested ranges include 1 to 5, 10 to 100, and 200 to 500 µg/L.

10.2 It is not imperative that all standards be distilled in the same manner as the samples. It is recommended that at least one standard and a blank be distilled and compared to similar values on the standard curve to insure that the distillation technique is reliable. If distilled standards do not agree within ± 10% of the undistilled standards, the analyst should find the cause of the apparent error before proceeding. Before distillation, standards should be adjusted to a pH of 4 with H_2SO_4.

10.3 Set up the manifold as shown in Figure 1 in a hood or a well-ventilated area.

10.4 Allow the instrument to warm up as required. Pump all reagents until a stable baseline is achieved.

10.5 Place appropriate standards in the sampler in order of decreasing concentration and perform analysis.

10.6 Prepare standard curve by plotting instrument response concentration values. A calibration curve may be fitted to the calibration solutions concentration/response data using computer or calculator based regression curve fitting techniques. Acceptance or control limits should be established using the difference between the measured value of the calibration solution and the "true value" concentration.

10.7 After the calibration has been established, it must be verified by the analysis of a suitable quality control sample (QCS). If measurements exceed ± 10% of the established QCS value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis. Periodic reanalysis of the QCS is recommended as a continuing calibration check.

11.0 PROCEDURE

11.1 Distillation

11.1.1 Measure 500 mL sample into a beaker. Adjust the pH to approximately 4 with 1+9 NAOH (7.9) or 1+9 H_2SO_4 (7.10), and transfer to the distillation apparatus.

11.1.2 Distill 450 mL of sample, stop the distillation, and when boiling ceases add 50 mL of warm reagent water to the flask and resume distillation until 500 mL have been collected.

11.1.3 If the distillate is turbid, filter through a prewashed membrane filter.

11.2 Set up the manifold as shown in Figure 1.

11.3 Fill the wash receptacle by siphon with reagent water. Use Kel-F tubing with a fast flow (1 L/h).

11.4 Allow the instrument to warm up as required. Run a baseline with all reagents, feeding reagent water through the sample line. Use polyethylene tubing for sample line. When new tubing is used, about 2 hours may be required to obtain a stable baseline. This two hour time period may be necessary to remove the residual phenol from the tubing.

11.5 Place appropriate phenol standards in sampler in order of decreasing concentration. Complete loading of sampler tray with unknown samples, using glass tubes.

11.6 Switch sample line from reagent water to sampler and begin analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Prepare a calibration curve by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.

12.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12.3 Report results in µg/L.

13.0 METHOD PERFORMANCE

13.1 The interlaboratory precision and accuracy data in Table 1 were developed using a reagent water matrix. Values are in mg Phenol/L.

13.2 Single laboratory precision data can be estimated at 50 to 75% of the interlaboratory precision estimates.

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

15.0 WASTE MANAGEMENT

15.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess Reagents and samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods, and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Sect. 14.3.

16.0 REFERENCES

1. Technicon AutoAnalyzer II Methodology, Industrial Method No. 127-71W, AAII.

2. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p. 574, Method 510 (1975).

3. Gales, M.E. and Booth, R.L., "Automated 4 AAP Phenolic Method," AWWA 68, 540 (1976).

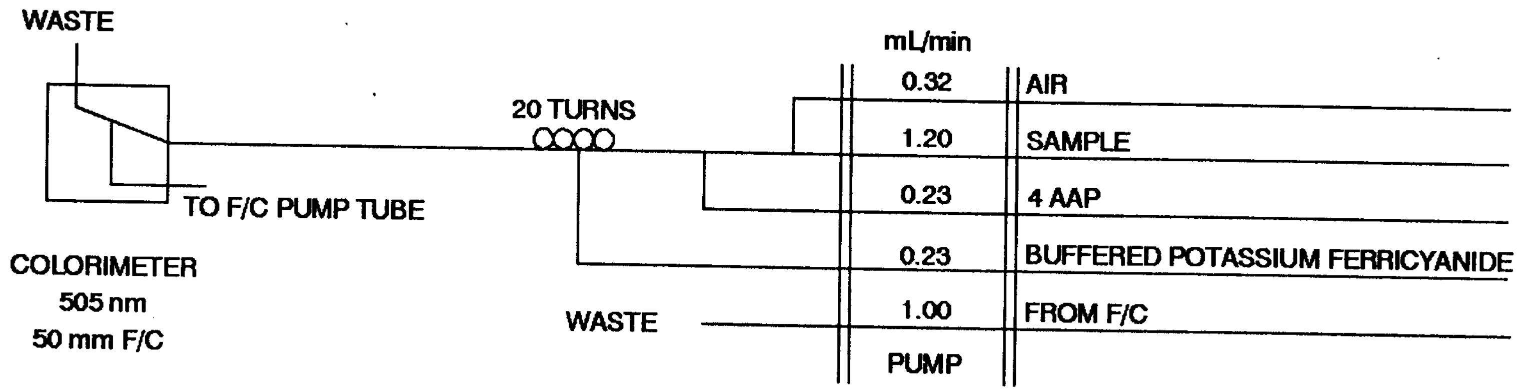
4. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B.

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

TABLE 1. INTERLABORATORY PRECISION AND ACCURACY DATA

NUMBER OF VALUES REPORTED	TRUE VALUE (T)	MEAN (X)	RESIDUAL FOR X	STANDARD DEVIATION (S)	RESIDUAL FOR S
99	0.020	0.0149	0.0000	0.0074	0.0000
87	0.250	0.1443	-0.0052	0.0268	-0.0038
76	0.400	0.2352	-0.0021	0.0422	-0.0036
110	0.545	0.3364	0.0142	0.0681	0.0076
89	0.604	0.3610	0.0043	0.0625	-0.0039
107	0.660	0.3959	0.0064	0.0894	0.0173
86	0.800	0.4627	-0.0087	0.0806	-0.0057
62	0.817	0.4692	-0.0122	0.0776	-0.0104
76	0.970	0.5680	-0.0029	0.1017	-0.0017
89	2.96	1.7734	0.0377	0.3065	0.0018
61	4.18	2.3916	-0.0582	0.4044	-0.0237
110	4.54	2.7150	0.0545	0.5382	0.0737

REGRESSIONS: $X = 0.585T + 0.003$, $S = 0.101T + 0.005$



20 PER HOUR
SAMPLE 120 SEC.
WASH 60 SEC.

Figure 1
Phenol Manifold

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