

Distillation Apparatus USER MANUAL

02/2013 Edition 5



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Section 1 General Description

1.1 Introduction

The Hach Distillation Apparatus adapts easily for a variety of distillation needs. It is suitable for wastewater, water and seawater testing requiring distillation pretreatment. Applications for the General Purpose Apparatus include: albuminoid nitrogen, ammonia nitrogen, fluoride, phenols, selenium, and volatile acids.

In addition to the General Purpose Apparatus, arsenic requires the Arsenic Distillation Apparatus Set and cyanide requires the Cyanide Distillation Apparatus Set. All connecting glassware is manufactured with threaded connectors for easy use and safety. The General Purpose Heater and Support Apparatus provide efficient heating and anchoring of the glassware.

1.1.1 Heater

The heater is a single-element, 200-watt mantle with adjustable heat and a variable speed, bi-directional magnetic stirring motor. The heater block is sized for a 500-mL distillation flask. Operating controls include a heat control, a stir speed control, and a power switch. Indicator lights show when the power and heater circuits are energized. The power requirements are 115 or 230 Vac, 50-60 Hz, 200 W.

1.1.2 Operator Controls and Indicators

Figure 1 on page 6 illustrates the heater assembly and control indicators. A functional description of the controls is given in Table 1.

Table 1

Name	Function
Power Indicator	Indicates when the heater is plugged in.
Heat Indicator	Indicates when the heater circuit is operating
Stir Speed Control	Controls the rotation speed of the stir bar. Counter-clockwise (1-10) rotation of the knob increases the rotation speed. The power indicator must be on to operate the stir motor.
Stir Mode Switch	Selects for 3 functions: 1 (highest position) Bi-direction with a reversing period of about 20 seconds 2 (middle position) One direction up to about 150 rpm
	3 (lowest position) Manual capture. If stirring action is lost due to excessive rotation, reduce the stir speed and depress the stir mode switch to this position to recapture the stir bar in the magnetic field.
Stir Indicator	Indicates when the magnetic stirring motor is on.
Stir On/Off Switch	Turns stirring motor on or off. The stir light will light when the switch is on.
Heater Control	Controls electrical current to the heating element. The off position is marked 0 and the temperature increases as the knob is turned clockwise to 10. The heater indicator will light when the heating element is on and go off when the selected temperature level is reached.

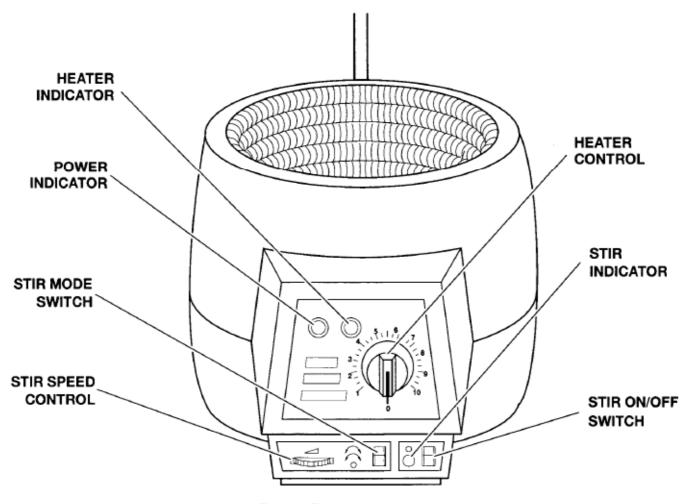


Figure 1 Electromantle Heater

1.2 Heater and Support Apparatus

The heater comes with a support rod, three universal clamps and three clamp holders. The support rod is inserted into the holder on the back of the heater base and is secured with a hand-operated screw. Clamp holders and clamps are positioned on the support rod to secure the distillation glassware.

1.2.1 General Purpose Apparatus

The General Purpose glassware is used for all distillations except arsenic and cyanide (additional glassware is necessary for those tests).

- The heater comes with a support rod, 3 universal clamps and 3 clamp holders. Insert the support rod into the holder on the back of the heater base and secure it with a hand-operated screw set.
- **2.** Position the clamp holders on the support rod to secure the glassware.
- 3. Assemble the general purpose glassware as shown in Figure 2.

4. Assemble the condenser so cold water enters through the lower port and leaves from the upper port. Secure tubing to both ports with cable ties.

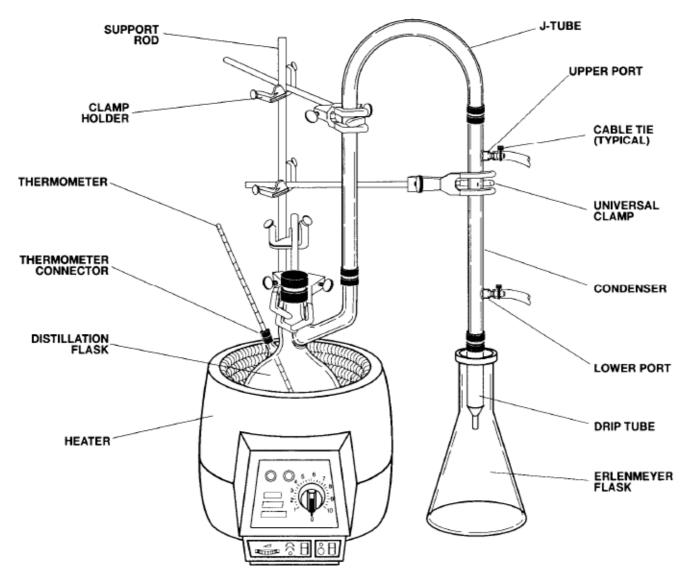


Figure 2 General Purpose Distillation Apparatus Assembly

1.2.2 Arsenic Distillation Apparatus

Figure 3 shows the distillation apparatus setup for arsenic. Assemble as shown, using these instructions:

- **1.** Complete Steps 1 and 2 from the General Purpose assembly procedure.
- 2. Use the distillation flask and J-tube from the General Purpose Apparatus, along with the gas scrubber, gas bubbler, and cylinder from the Arsenic Distillation Apparatus to assemble the glassware as illustrated in Figure 3.

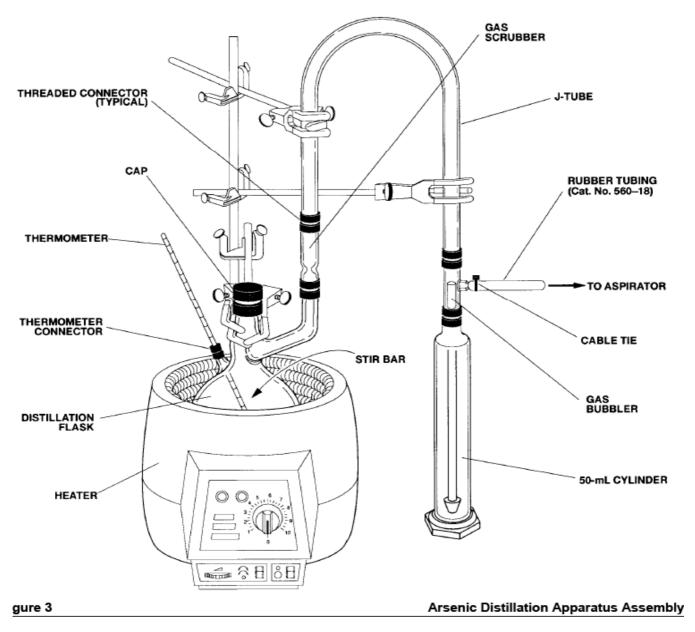


Figure 3 Arsenic Distillation Apparatus Assembly

1.2.3 Cyanide Distillation Apparatus

The distillation setup for cyanide is shown in Figure 4. Assemble as shown, using the following instructions:

- **1.** Complete Steps 1 and 2 from the General Purpose glassware assembly procedure.
- 2. Use the distillation flask, condenser and J-tube from the General Purpose Apparatus along with the gas bubbler, cylinder, flow meter, filter flask, and aspirator from the Cyanide Distillation Apparatus to assemble the Cyanide Glassware as illustrated.

- **3.** Assemble the condenser so the cold water enters through the lower port and leaves from the upper port. Secure tubing to both ports with cable ties.
- **4.** The aspirator requires a 3/8-inch NPT inside thread. Adapters for other thread connectors may be purchased commercially.
- **5.** Secure all tubing connections with cable ties except at the gas bubbler port.

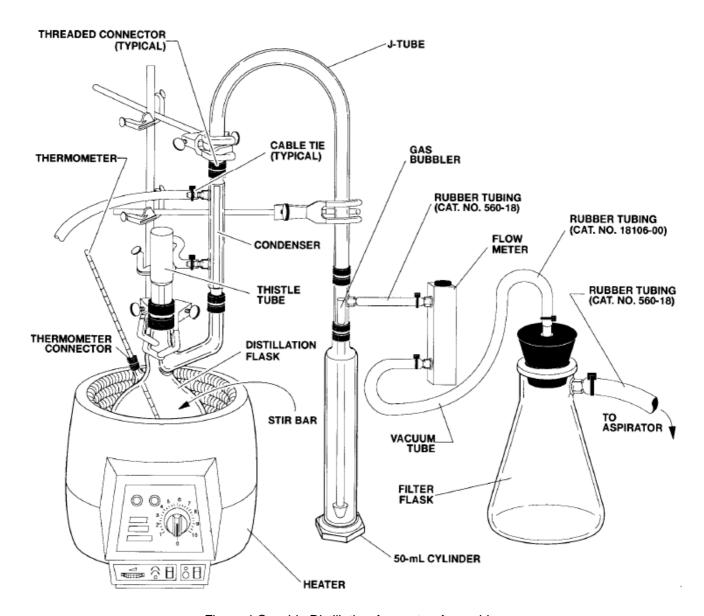


Figure 4 Cyanide Distillation Apparatus Assembly

1.3 Using the Distillation Apparatus

1.3.1 Burn In

When first used, the surface of the heating element will smoke slightly as the fabric dressing burns away (burn-off). The element will become discolored and then gradually revert back to white. It is

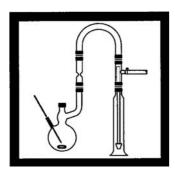
advisable to burn off the mantle before inserting any glassware to avoid staining the flask.

1.3.2 Operational Notes

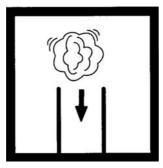
Wipe the distillation flask thoroughly to ensure it is clean and dry before placing it on the mantle. Be sure the bottom of the flask makes good contact with the heating element. Use a slight rotary action when installing or removing the flask from the mantle.

If a spill occurs, turn off the heater immediately and allow it to cool. The mantle must be cleaned and dried before using it again.

Arsenic Distillation¹



1. Prepare the Arsenic Distillation Apparatus as shown in the Assembly section. Place a stir bar into the distillation flask. Place it in a hood to vent toxic fumes.



2. Dampen a cotton ball with 10% Lead Acetate Solution. Place it in the gas scrubber, making sure the cotton seals against the glass.

Note: 2 N Zinc Acetate Solutinon may be used in place of the Lead Acetate Solution if the results are not used for EPA reporting purposes.



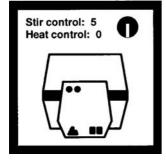
3. Fill a 25-mL graduated cylinder to the 25-mL mark with the prepared Arsenic Absorber Solution. Pour the solution into the cylinder/gas bubbler assembly of the distillation apparatus. Attach the assembly to the distillation apparatus.

Note: See Preparing Arsenic Absorber Solution following these steps



4. Measure 250 mL of water sample in a 250-mL graduated cylinder. Transfer it to the round-bottom distillation flask.

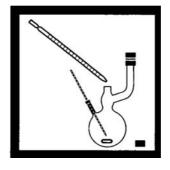
Note: For proof of accuracy, use a 0.12 mg/L arsenic standard (see ACCURACY CHECK) in place of the sample.



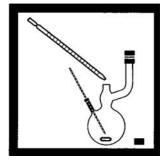
5. Turn the stirrer power switch on and set the stir control to 5.



6. Measure 25 mL of hydrochloric acid, ACS, in a 25-mL graduated cylinder and add it to the distillation flask.



7. Using a pipet filler and 5-mL serological pipet, add 1 mL of Stannous Chloride Solution to the distillation flask.

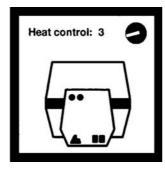


8. Add 3 mL of Potassium lodide Solution to the flask. Screw the cap on and allow to mix for 15 minutes.

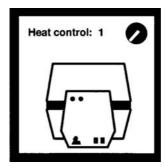
¹Adapted from Standard Methods for Examination of Water and Wastewater.



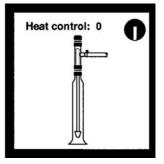
9. After 15 minutes, weigh out 6.0 g of 20-mesh zinc and add it to the flask. Cap immediately.



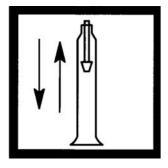
10. Turn the heat control to 3 and heat for 15 minutes.



11. After 15 minutes, turn the heat control to 1 and heat for 15 more minutes.



12. After a total of 30 minutes of heating, turn the heater off. Remove the cylinder/gas bubbler assembly as a unit from the J-tube connector.



13. Rinse the gas bubbler by moving it up and down in the Arsenic Absorber Solution.



14. Pour the reacted Arsenic Absorber Solution into a sample cell (the prepared sample). Stopper.



15. Fill a clean sample cell with unreacted Arsenic Absorber Solution to use as a blank. Proceed with colorimetric measurement according to instructions received with your colorimeter or spectrophotometer.

Preparing Arsenic Absorber Solution

- Weigh 1.00 g of silver diethyldithiocarbamate on an analytical balance.
- 2. Transfer the silver diethyldithiocarbamate to a 200-mL volumetric flask and dilute to volume with pyridine (use only in a well-ventilated hood).
- **3.** Mix well to dissolve and store the prepared Arsenic Absorber Solution in a dark amber bottle. The reagent is stable for one month if stored in this manner.

ACCURACY CHECK

1. Prepare a 10.0,g/L arsenic working standard by pipetting 1.00 mL of Arsenic Standard Solution, 1000 mg/L as As, into a 100-mL volumetric flask. Dilute to volume with demineralized water. Mix well.

2. Prepare a 0.12 mg/L arsenic standard by pipetting 3.0 mL of the working standard into 250-mL volumetric flask. Dilute to volume with demineralized water a mix.

Required Reagents

Description	Cat. No
Hydrochloric Acid, ACS, 500 mL	134-49
Lead Acetate Solution, 10%, 100 mL	14580-42
Potassium Iodide Solution, 20%, 100 mL	14568-42
Pyridine, 500 mL	14469-49
Silver Diethyldithiocarbamate, 25 g	14476-24
Stannous Chloride Solution, 100 mL	14569-42
Zinc, 20-mesh, ACS, 454 g	795-01

Required Apparatus

Description	Cat. No
Balance, analytical, 80 g capacity, Scien Tech, 0.1 mg	29367-01
Balls, cotton, 100/pkg	2572-01
Bottle, amber, 237 mL, 6/pkg	7144-41
Cap, polypropylene, 6/pkg	21667-06
Cylinder, graduated, 25 mL	508-40
Cylinder, graduated, 250 mL	508-46
Distillation Apparatus, Arsenic Accessories	22654-00
Flask, volumetric, 200 mL	14574-45
Paper, weighing, 100 x 100 mm, 500/pkg	14738-85
Pipet Filler, safety bulb	14561-00
Pipet, serological, 5 mL (2 required)	532-37
Stopper, hollow, poly, No. 0, 6/pkg	14480-00
Select one based on available voltage:	_
Distillation Apparatus Heater, 115 V, 60 Hz	22744-00
Distillation Apparatus Heater, 230 V, 60 Hz	22744-02

Description	Cat. No
Arsenic Standard Solution, 1000 mg/L As	14571-42
Flask, volumetric, 100 mL	14574-42
Flask, volumetric, 200 mL	14574-45
Flask, volumetric, 250 mL	14574-46
Hydrochloric Acid, ACS, 2.8 kg	134-06
Nitric Acid, ACS, 500 mL	152-49

Arsenic Distillation

Description	Cat. No
Nitric Acid Solution, 1:1, 500 mL	2540-49
pH Meter, HQ40d	HQ40d53000000
pH Paper, 1-11 pH units, 5 rolls/pkg	391-33
pH Probe, IntelliCAL, standard, 1 meter cable	PHC10101
Pipet, volumetric, 1.00 mL, Class A	14515-35
Pipet, volumetric, 3.00 mL, Class A	14515-03
Standard Methods for the Examination of Water and Wastewater, current edition	22708-00
Water, demineralized, 4 L	272-53
Zinc Acetate Solution, 2 N, 100 mL	14703-32

Cyanide Distillation¹



1. Set up the distillation apparatus for cyanide; do not connect the thistle tube yet. Refer to the assembly section preceding this section for proper assembly. Place a stirring bar in the distillation flask.



2. Turn on the water and make sure a steady flow is maintained through the condenser.



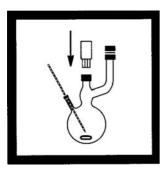
3. Remove and fill the distillation apparatus cylinder to the 50-mL mark with 0.25 N Sodium Hydroxide Standard Solution. Re-assemble the distillation cylinder to the assembly.



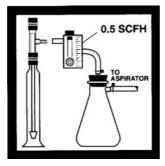
graduated cylinder to the 250-mL mark with sample and pour it into the distillation flask.

Note: For proof of accuracy, use a 0.10 mg/L cyanide standard solution (see ACCURACY CHECK) in place of the sample.

4. Fill a clean 250-mL

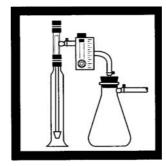


5. Attach the thistle tube to the flask.



6. Arrange the vacuum system as shown in the assembly drawing, but do not connect the vacuum tubing to the gas bubbler. Turn on the water to the aspirator to full flow and adjust the flow meter to 0.5 SCFH (standard cubic feet per hour).

Note: The tubing to flow meter connections should be secured tightly with cable ties.



7. Connect the vacuum system tubing to the gas bubbler, making certain that air flow is maintained (check flow meter) and that air is bubbling from the thistle tube and the gas bubbler.



8. Turn the power switch on and set the stir control to 5.

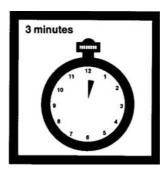
¹Adapted from Standard Methods for the Examination of Water and Wastewater.



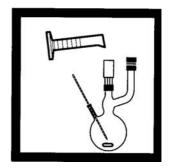
9. Using a 50-mL graduated cylinder, pour 50 mL of 19.2 N Sulfuric Acid Standard Solution into the distillation flask via the thistle tube.



10. Using a water bottle, rinse the thistle tube with a small amount of demineralized water.



11. Allow the solution to mix for 3 minutes.



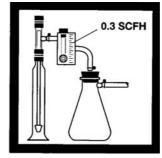
12. Add 20 mL of Magnesium Chloride Reagent into the flask via the thistle tube. Rinse the thistle tube again.



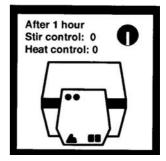
13. Allow the solution to mix for 3 more minutes.



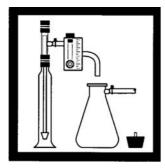
14. Verify there is a constant flow of water through the condenser, then turn the heat control to 10.



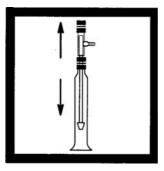
15. It is very important to monitor the distillation at this point. Once the sample boils, slowly lower the air flow to 0.3 SCFH. If the contents of the distillation flask begin to back up through the thistle tube, increase the air flow by adjusting the flow meter until the contents do not back up. Allow the sample to boil for one hour.



16. After one hour, turn the heater off but maintain air and water flow for 15 minutes. Maintain water flow through the condenser.



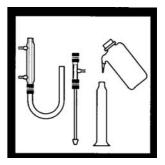
17. After 15 minutes, remove the rubber stopper on the 500-mL vacuum flask to break the vacuum. Turn off the water to the aspirator. Turn off the water to the condenser.



18. Remove the gas bubbler/cylinder assembly from the distillation apparatus. Separate the gas bubbler from the cylinder.



19. Pour the contents of the cylinder into a 250-mL erlenmeyer flask.



20. Rinse the gas bubbler, cylinder, and J-tube connector with demineralized water and add washings to the erlenmeyer flask.



21. Fill the flask to the 200-mL mark with demineralized water and gently swirl to mix thoroughly.



22. Add three drops of Phenolphthalein Indicator Solution. Adjust the pH of the sample with 4.0 N Hydrochloric Acid Solution. Using the plastic dropper, add the Hydrochloric Acid Solution drop-wise until the solution changes from pink to colorless.

Note: Do not use Sulfuric Acid to neutralize the sample.



23. Transfer the contents into a 250-mL volumetric flask. Dilute to volume with demineralized water. Stopper to mix. The sample is now ready for analysis by the pyridine-pyrazalone colorimetric method.

ACCURACY CHECK

Standard Solution Method

CAUTION

Cyanides and their solutions, and the hydrogen cyanide liberated by acids, are very poisonous. Both the gas and the solutions can be absorbed through the skin.

To assure the accuracy of the test, prepare the following standard solutions:

1. Prepare a 100 mg/L cyanide solution weekly by dissolving 0.2503 grams of potassium cyanide in demineralized water and diluting to 1000 mL.

2. Immediately before use, prepare a 0.10 mg/L cyanide working solution by diluting 1.00 mL of stock solution to 1000 mL with demineralized water.

Required Reagents

Description	Cat. No
Hydrochloric Acid Standard Solution, 2.5 N, 100-mL MDB	1418-32
Magnesium Chloride Solution, 1000 mL	14762-53
Sodium Hydroxide Standard Solution, 0.25 N, 1 L	14763-53
Sulfuric Acid Standard Solution, 19.2 N, 500 mL	2038-49
Water, demineralized, 4 L	272-56
Phenolphthalein Indicator Solution, 15 mL SCDB	1897-36

Required Apparatus

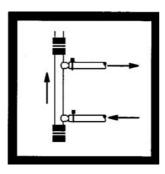
Description	Cat. No
Bottle, wash, 500 mL	620-11
Cylinder, graduated, 25 mL	508-40
Cylinder, graduated, 50 mL	508-41
Cylinder, graduated, 250 mL	508-46
Distillation Apparatus, cyanide accessories	22658-00
Distillation Apparatus Heater and Support Apparatus, 115 V, 60 Hz	22744-00
Distillation Apparatus Heater and Support Apparatus, 230 V, 50 Hz	22744-02
Flask, volumetric, Class A, 250 mL	14574-46
Flask, erlenmeyer, 250 mL	505-46

Description	Cat. No
Balance, analytical, 80 g capacity, ScienTech, 0.1 mg	29367-01
Flask, volumetric, 1000 mL (need two)	547-53
Pipet, volumetric, 1.00 mL	14515-35
Pipet Filler, safety bulb	14651-00
Potassium Cyanide, ACS, 125 g	767-14
Sodium Hydroxide Standard Solution, 5.0 N, 1 L	2450-53
Standard Methods for the Examination of Water and Wastewater, current edition	22708-00
Support Ring, 4-in.	580-01
Support Stand	563-00
Timer, 3-channel	23480-00

Fluoride Distillation¹



1. Set up the distillation apparatus for general purpose distillation. Refer to the assembly section preceding this section for proper assembly. Use a 300-mL erlenmeyer flask to collect the distillate. Place a stir bar in the distillation flask.

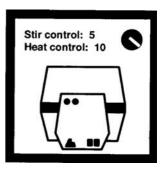


2. Turn on the water and make certain a steady flow is maintained through the condenser.



3. Measure 100 mL of sample into the distillation flask using a 100-mL graduated cylinder. Add a magnetic stir bar and 5 glass beads.

Note: For proof of accuracy, use a 1.0 mg/L Fluoride Standard Solution (listed under Optional Reagents) in place of the sample.



4. Turn the stirrer power switch on. Turn the stir control to 5.



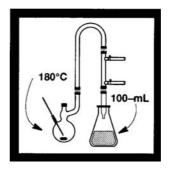
5. Using a 250-mL graduated cylinder, carefully add 150 mL of prepared Acid Distillation Solution into the flask. Cap the flask.

Note: See Preparing Acid Distillation Solution.

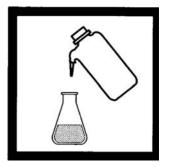
Note: When distilling samples with high amounts of chloride, add 5 mg (0.005 g) of Silver Sulfate to the sample for every mg/L of chloride in the sample.



6. With the thermometer in place, turn the heat control to 10. The yellow pilot lamp indicates the heater is on.



7. When the temperature reaches 180 °C or when 100 mL of distillate has been collected, turn the still off (requires about 1 hour).



8. Dilute the distillate to a volume of 100 mL, if necessary. The distillate may now be analyzed by the SPADNS or the fluoride ion-selective electrode method.

Preparing Acid Distillation Solution

- Using safety goggles, gloves and a 250 mL graduated cylinder, add 170 mL of demineralized water into a 1000 mL erlenmeyer flask.
- 2. Add a stir bar to the flask and place on a stir plate.

¹Adapted from Standard Methods for the Examination of Water and Wastewater.

3. Using a 500 mL graduated cylinder, carefully add 330 mL of conc. sulfuric acid to the flask while stirring.

CAUTION

The mixture will become hot. Cool the mixture to room temperature before using it in Step 5 of the distillation procedure.

Required Reagents

Description	Cat. No
Sulfuric Acid, ACS, 2.5 L	979-09

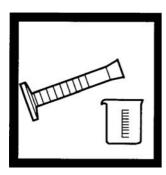
Required Apparatus

Description	Cat. No
Cylinder, graduated, 100 mL	508-42
Cylinder, graduated, 250 mL	508-46
Distillation Heater and Support Apparatus Set, 115 V, 50/60 Hz	22744-00
Distillation Heater and Support Apparatus Set, 230 V, 50/60 Hz	22744-02
Distillation Apparatus General Purpose Accessories	22653-00
Flask, erlenmeyer, 300 mL	14574-42
Flask, Erlenmeyer, 1000 mL	505-53
Glass Beads	2596-00
Gloves, Chemical Resistant, Size 10	24101-05
Goggles, Safety, Standard	29279-02
Stir Bar, Octagonal	20953-51
Stirrer, Magnetic, 4.25 x 4.25", 120 volts	28812-00
Thermometer, -20 to 260 °C (included in Support Apparatus set)	20959-26
Water, demineralized, 4 L	272-56

Description	Cat. No
Balance, Analytical 80 g capacity, ScienTech, 0.1 mg	29367-01
Fluoride ISE Analysis Package	51928-10
Fluoride Standard Solution, 1.0 mg/L F-, 500 mL	291-49
Fluoride Standard Solution, 10 mg/L F, 500 mL	359-49
pH/ISE Meter, sensION 2	51725-00
pH Probe, Platinum Series, 1 meter cable	5191000
Pipet Filler, safety bulb	14651-00
Silver Sulfate, ACS, 113 g	334-14
Standard Methods for the Examination of Water and Wastewater, current edition	22708-00

Nitrogen-Ammonia Distillation¹

For Use with Nessler and Salicylate Methods in colorimetric testing



1. Measure 250 mL of water sample into a clean 250-mL graduated cylinder. Pour into a 400-mL beaker.

Note: For Proof of Accuracy, use a 1.0 mg/L Ammonia Nitrogen Standard Solution (listed in Optional Reagents) in place of sample.

Note: If necessary, destroy residual chlorine by adding 2 drops of Sodium Arsenite Solution or 1.0 mL of Sodium Thiosulfate Solution for every mg/L of Cl₂.

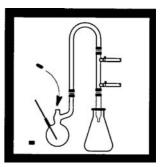
Note: To prepare Sodium Thiosulfate Solution, dissolve 3.5 g of sodium thiosulfate pentahydrate, in 1.0 liter of demineralized water. Prepare fresh weekly.Cl₂.



2. TMeasure 25 mL of Borate Buffer Solution in a 25-mL graduated cylinder. Add it to the beaker and mix.

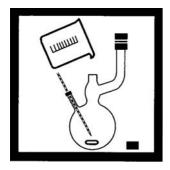


3. Measure the pH of the solution with a pH meter. Using a dropper, add 1 N Sodium Hydroxide Standard Solution drop-wise until the pH is about 9.5.



4. Set up the distillation apparatus by assembling the general purpose accessories as shown in Figure 2 in the Assembly section. Place a stir bar in the distillation flask.

¹Adapted from Standard Methods for the Examination of Water and Wastewater.



5. Pour the solution into the distillation flask. Cap the distillation flask.



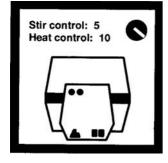
6. Nessler Method:
Using a 25-mL graduated cylinder, pour 25 mL of demineralized water into a clean, 300-mL erlenmeyer flask. Proceed to step Salicylate method: Add 25 mL of 0.04 N Sulfuric Acid Solution to a 300 mL erlenmeyer flask. Proceed to step 8. See Preparing 0.04 N Sulfuric Acid Solution.



7. Add the contents of one Boric Acid Powder Pillow to the erlenmeyer flask. Mix thoroughly by swirling.



8. Place the flask under drip tube. Elevate the flask so the end of the drip tube extends below the level of solution in the flask. This may require the use of a laboratory jack.



9. Turn on the stirrer power switch. Set the stir control to 5. Set the heater control to 10. Turn on the water and adjust it so a constant flow is maintained through the condenser.



10. Collect 150 mL of distillate, then turn the heater off. Immediately remove the erlenmeyer flask. Using a 250-mL graduated cylinder, measure the distillate to be sure that 150 mL has been collected (total volume should be 175 mL).

Note: The flask is removed immediately so the distillate is not drawn back into the distillation flask by vacuum. Or, remove the small glass stopper in the thermometer well to break the vacuum.



11. Return the distillate to the erlenmeyer flask.
Adjust the pH of the distillate to about 7 by adding 1N Sodium Hydroxide Standard Solution drop-wise.
Measure the pH with a pH meter.

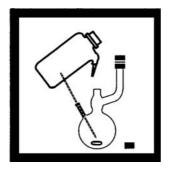


12. Pour the distillate into a 250-mL volumetric flask. Rinse the erlenmeyer flask with demineralized water and add the rinsings to the volumetric flask. Dilute to the mark. Stopper and mix thoroughly by inversion. The distillate is ready for analysis. If albuminoid nitrogen is not being determined, the remainder of the solution in the distillation flask may be discarded. If albuminoid nitrogen is being measured, continue with Step 13.

Albuminoid Nitrogen (Nessler Method Only)



13. Prepare the alkaline potassium permanganate reagent by following the instructions under Preparing Alkaline Potassium Permanganate following these steps.



14. Allow the distillation flask to cool, then add 125 mL of demineralized water.



15. Using a graduated cylinder, add 25 mL of alkaline potassium permanganate to the flask. Re-cap the flask.



16. Using a graduated cylinder, add 25 mL of demineralized water into a clean 300-mL erlenmeyer flask. Add the contents of one Boric Acid Powder Pillow. Mix thoroughly by swirling.



17. Place the flask under the drip tube. Elevate the flask so the end of the drip tube extends below the level of the solution in the flask.



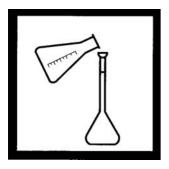
18. Turn on the stirrer power switch. Set the stir control to 5. Set the heat control to 10. Make sure a constant flow of water is maintained through the condenser.



19. Collect 125 mL of distillate, then turn the still off. Immediately remove the erlenmeyer flask. Using a graduated cylinder, measure the distillate to be sure that 125 mL has been collected. (Total volume should be 150 mL.)



20. Return the distillate to the erlenmeyer flask. Adjust the pH of the distillate to about 7 by adding 1N Sodium Hydroxide Standard Solution drop-wise. Measure the pH with a pH meter.



21. Pour the distillate into a 250-mL volumetric flask. Rinse the erlenmeyer flask with demineralized water and add the rinsings to the volumetric flask. Dilute to the mark with demineralized water. The distillate is ready for analysis for albuminoid nitrogen by the Nessler method.

Preparing Alkaline Potassium Permanganate

- 1. Weigh out 8 g of potassium permanganate and pour into a 2-liter beaker. Add about 500 mL of ammonia-free water. Mix until the crystals dissolve.
- 2. Weigh out 144 g of sodium hydroxide pellets and add to the beaker. Add ammonia-free water until the volume is about 1250 mL and mix.
- **3.** When the sodium hydroxide is dissolved, heat the solution to boiling on a hot plate and concentrate it to about 1 liter. Store the reagent in a glass amber bottle after it has cooled.
- 4. Repeat the albuminoid nitrogen procedure on 250 mL of demineralized water each time the reagent is prepared. Analyze the distillate by the Nessler method to determine the reagent blank value. Subtract this value from each sample measurement.

Preparing 0.04 N Sulfuric Acid Solution

- **1.** Dilute 1.0 mL of concentration Sulfuric Acid to one liter in a 1-liter volumetric flask.
- 2. Stop and invert to mix.

Required Reagents

Description	Cat. No
Borate Buffer Solution, 1000 mL	14709-53
Boric Acid Powder Pillows, 100/pkg	14817-99
-OR-	
Sulfuric Acid, 36 N, conc. 500 ml	979-49
Potassium Permanganate, ACS, 454 g	168-01
Sodium Hydroxide Solution, 1N, 100 mL	1045-32
Sodium Hydroxide Solution, 5 N, 100 mL	2450-32
Sodium Hydroxide Pellets, 500 g	187-34
Water, demineralized, 4 L	272-56

Required Apparatus

Description	Cat. No
Beaker, 400 mL	500-48
Bottle, glass, amber, 1000 mL, 6/pkg	7144-63
Cylinder, graduated, 25 mL	508-40
Cylinder, graduated, 250 mL	508-46
Distillation Apparatus, General Purpose Accessories	22653-00
Distillation Heater and Support Apparatus, 115 V	22744-00
Distillation Heater and Support Apparatus, 230 V	22744-02
Dropper, plastic, 0.5 and 1-mL marks, 20/pkg	21247-20
Flask, volumetric, Class A, 250 mL	14574-46
Flask, volumetric, Class A 1000 mL	14574-53
Flask, erlenmeyer, 300 mL	505-46
Hot Plate, 120 V, 50/60 Hz	12067-01
pH Meter, HQ40d	HQ40d53000000
pH Probe, IntelliCAL, standard, 1 meter cable	PHC10101
Pipet Filler, Safety Bulb	14651-00
Pipet, Volumetric, 1.0 mL	14515-35
Standard Methods for the Examination of Water and Wastewater, current edition	22708-00

Description	Cat. No
Jack, laboratory, platform	22743-00
Nitrogen Ammonia Standard Solution, 1.0 mg/L as N, 500 mL	1891-49
Sodium Arsenite Solution, 5 g/L, 100 mL MDB	1047-32
Sodium Thiosulfate, pentahydrate, ACS, 454 g	460-01

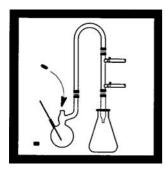
Nitrogen-Ammonia Distillation for ISE

For Use with Hach Ammonia Ion Selective Electrode

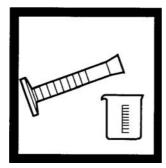
Distilling may be used instead of the Air Gap Accessory in samples that contain detergents or fatty acids.

This procedure is an adaptation of the distillation procedure written in the 17th edition of *Standard Methods for the Examination of Water and Wastewater.* The reagent volumes have been reduced by half in the adapted method. For the distillation, a borosilicate apparatus, such as the Hach Distillation Apparatus (Cat. No. 22653-00), must be used.

Nitrogen-Ammonia Distillation Procedure¹



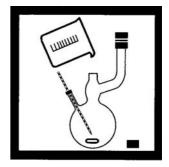
1. Set up the distillation apparatus by assembling the general purpose accessories as shown in Figure 2 in the Assembly section. Place a stir bar into the distillation flask.



2. Add 250 mL of ammonia-free water and 10 mL of borate buffer to a 400-mL beaker.



3. Measure the pH of the solution with a pH meter. Using a dropper, add 6 N sodium hydroxide (NaOH) drop-wise until the pH is 9.5.



4. Pour the solution into the distillation flask. Cap the distillation flask.

¹Adapted from Standard Methods for the Examination of Water and Wastewater, 17th edition.



5. Turn the heater on and set the control to 10. Heat the flask and steam out the apparatus until the distillate shows no traces of ammonia. Check with the ammonia ISE to make sure ammonia is absent.

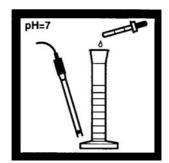


6. Turn the heater off when done steaming.



7. Collect 250 mL of the sample in a 500-mL graduated mixing cylinder. Add 0.5 mL dechlorinating agent for every mg/L Cl2 in 250 mL sample, if necessary. Stopper and invert to mix.

Note: For proof of accuracy, use a 1.0 mg/L Ammonia Nitrogen Standard (listed in Operational Reagents) in place of the sample.



Measure the pH of the

solution with a pH meter.

Using a dropper, add 1 N

sodium hydroxide or 1N

sulfuric acid drop-wise until the pH is 7.

Note: Periodically stopper and invert cylinder several times to insure mixing when adjusting pH.

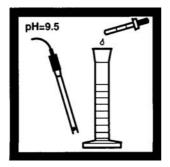
place of the sample.

Note: See Preparation of

Dechlorinating Agent.



9. Measure 12.5 mL of borate buffer in a graduated cylinder. Add the measured buffer to the solution in the cylinder. Stopper and invert to mix.



10. Measure the pH of the solution with a pH meter. Using a dropper, add 6 N sodium hydroxide drop-wise until the pH is 9.5.

Note: Periodically stopper and invert cylinder several times to insure mixing is complete when adjusting pH.



11. Transfer sample to a distillation flask using a funnel.

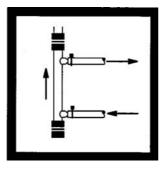


12. Measure 25 mL of 0.04 N sulfuric acid into a graduated cylinder. Pour this into a 300-mL erlenmeyer receiving flask (comes with the distillation apparatus).



13. Place the tip of the delivery tube below the surface of the 0.04 N sulfuric acid in the receiving flask.

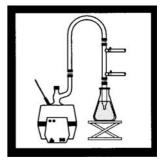
Note: A laboratory jack or similar apparatus may be used to elevate the flask.



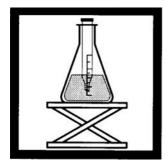
14. Turn on the water to the distillation column and adjust it so a constant flow through condenser is maintained.



15. Turn the heater on and set the heat control to 10. Be sure the stir control is still set on 5.



16. Distill the sample at a rate of 6-10 mL/minute. Be sure the tip of the delivery tube is below the 0.04 N sulfuric acid in the receiving flask.



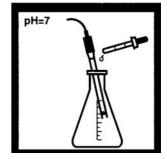
17. Distill until at least 100 mL of distillate is collected or until there is at least 125 mL total volume in the receiving flask..



18. Lower the receiving flask away from the delivery tube. Immediately cover the flask with parafilm or a stopper to prevent atmospheric contamination.



19. Place a 100-mL beaker under the delivery tube. Continue to distill for 1-2 minutes to clean the dispenser and delivery tube. Then turn the heater off.



20. Measure the pH of the solution with a pH meter. Using a dropper, add 1 N sodium hydroxide drop-wise until the pH is 7.



21. Transfer the distillate into a clean 250-mL volumetric flask. Rinse the erlenmeyer receiving flask with ammonia-free water and add rinsings to the volumetric flask. Dilute to the mark with ammonia-free water.



22. Proceed with the appropriate ammonia ISE analysis.

Preparation of Dechlorinating Agent

Dissolve 3.5 grams of sodium thiosulfate (pentahydrate) in one liter of ammonia-free water. Prepare fresh weekly.

Required Reagents

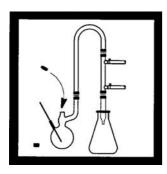
Description	Cat. No
Borate Buffer Solution, 1000 mL	14709-53
Sodium Hydroxide, 6 N, 1L	23324-53
Sodium Hydroxide, 1 N, 100 mL MDB	1045-32
Sodium Thiosulfate, pentahydrate, ACS, 454 g	460-01
Sulfuric Acid, 1 N, 1000 mL	1270-53
Sulfuric Acid, 0.04 N, 500 mL	23393-49
Water, demineralized, 4L	272-56

Required Apparatus

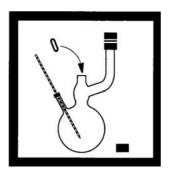
Description	Cat. No
Beaker, 100 mL	500-42
Beaker, 400 mL	500-48
Cylinder, graduated, 25 mL	508-40
Cylinder, graduated, mixing, 500 mL	26363-49
Distillation Apparatus, General Purpose	22653-00
Flask, volumetric, 250-mL, Nalge	14060-46
Funnel, 65 mm	1083-67

Description	Cat. No
Ammonia ISE Analysis Package	23487-00
Jack, laboratory, platform	22743-00
SensION 2 Portable pH/ISE Meter	5172511
Standard Methods for the Examination of Water and Wastewater, current edition	22708-00
Nitrogen Ammonia Standard Solution, 1.0 mg/L as N, 500 mL	1891-49

Phenols Ammonia Distillation¹



1. Set up the general purpose distillation apparatus as shown in the Assembly section. Use the 500-mL erlenmeyer flask to collect the distillate. It may be necessary to elevate the flask with a laboratory jack.

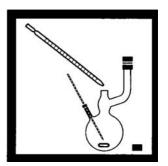


2. Place a stirring bar in the distillation flask.

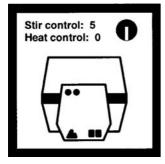


3. Measure 300 mL of water sample in a clean 500-mL graduated cylinder. Pour it into the distillation flask.

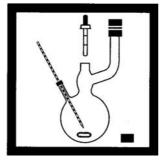
Note: For proof of accuracy, use a 0.10 mg/L Phenol Standard Solution (see ACCURACY CHECK) in place of the sample.



4. Using a serological pipet, add 1 mL of Methyl Orange Indicator Solution to the distillation flask.



5. Turn on the stirrer power switch. Set the stir control to 5.



6. Add 10% Phosphoric Acid Solution drop-wise until the indicator changes from yellow to orange.

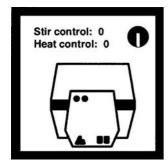


7. Add the contents of one Copper Sulfate Powder Pillow and allow to dissolve (omit this step if copper sulfate was used to preserve the sample). Cap the distillation flask.



8. Turn the water on and adjust it so a constant flow is maintained through the condenser. Set the heat control to 10.

¹Adapted from Standard Methods for the Examination of Water and Wastewater.



9. Collect 275 mL of distillate in the erlenmeyer flask, then turn the still off.



10. Fill a 25-mL graduated cylinder to the 25-mL mark with demineralized water. Add the water to the distillation flask.



11. Turn the still back on. Heat until another 25 mL of distillate is collected.



12. Using a clean graduated cylinder, re-measure the distillate to be sure that 300 mL has been collected. The distillate is now ready for analysis by the 4-Amino-antipyrine Method.

ACCURACY CHECK

Standard Solution Method

Verify the accuracy of the distillation by performing the distillation using a known phenol standard in place of the sample.

Prepare a 0.10 mg/L phenol solution as follows:

- 1. Weigh out 1.00 g of phenol, ACS. Transfer to a 1-liter volumetric flask. Dilute to the mark with freshly boiled and cooled demineralized water. This is a 1-g/L stock solution.
- 2. Transfer 1 mL of the 1-g/L stock solution to a 100-mL volumetric flask. Dilute to the mark with demineralized water. This is a 10-mg/L working solution.
- 3. Prepare a 0.10-mg/L standard solution by pipetting 5.0 mL of the 10-mg/L working solution into a 500-mL volumetric flask. Dilute to the mark with deionized water.

Required Reagents

Description	Cat. No
Copper Sulfate Powder Pillows, 50/pkg	14818-66
Methyl Orange Indicator Solution, 500 mL	148-49
Phosphoric Acid Solution, 10%, 100 mL	14769-32
Water, demineralized, 4 L	272-56

Required Apparatus

Description	Cat. No
Cylinder, graduated, 25 mL	508-40
Cylinder, graduated, 500 mL	508-49
Distillation Apparatus General Purpose Accessories	22653-00
Distillation Apparatus Heater, 115 V	22744-00
Distillation Apparatus Heater, 230 V	22744-02
Flask, 500 mL erlenmeyer	505-49
Pipet, serological, 1.0 mL	9190-02

Description	Cat. No
Balance, analytical, 80 g capacity, ScienTech, 0.1 mg	29367-01
Flask, volumetric, Class A, 100 mL	14574-42
Flask, volumetric, Class A, 500 mL	14574-49
Flask, volumetric, Class A, 1000 mL	14574-53
Jack, laboratory, platform	22743-00
Phenol, ACS, 113 g	758-14
Pipet, volumetric, Class A, 1.00 mL	14515-35
Pipet, volumetric, Class A, 5.00 mL	14515-37
Pipet Filler, safety bulb	14651-00
Standard Methods for the Examination of Water and Wastewater, current edition	22708-00

Selenium Distillation



1. Measure 500 mL of sample into a 1000-mL beaker.

Note: Perform this procedure in a fume hood.

Note: For proof of accuracy, use a 0.5 mg/L Selenium Standard Solution (see ACCURACY CHECK) in place of the sample.



2. Add 1 mL of Methyl Orange Indicator. Stir with a glass rod.



3. Using a dropper add 0.1 N Hydrochloric Acid Standard Solution drop-wise until the solution becomes pink. Then add an additional 2 mL.



4. Pipet 5.00 mL of Calcium Chloride Solution to the beaker. Mix well.



5. Using a dropper, add 1-g/L Potassium Permanganate Standard Solution drop-wise until the solution is purple.

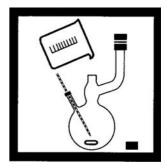


6. Place the beaker on a hot plate. Evaporate the solution to about 250 mL. Periodically add 1-g/L Potassium Permanganate Solution to keep the sample solution purple.

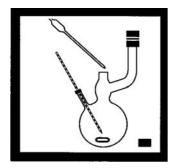
Note: Any precipitate formed at this point is manganese dioxide and may be ignored.



7. Allow the sample to cool. While cooling, set up the distillation apparatus by assembling the general purpose accessories as shown in Figure 2 of the Assembly section.



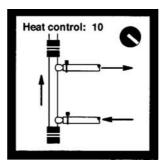
8. Pour the treated sample solution into the distillation flask. Place a stir bar into the distillation flask.



9. Pipet 5.00 mL of 0.1 N Sodium Hydroxide Standard Solution into the flask. Cap the distillation flask.



10. Turn the stirrer power switch to ON and set the stir control to 5.



11. Turn on the water and adjust so a constant flow is maintained through the condenser. Set the heat control to 10.



12. Allow the sample to distill until only a few mL are left in the distillation flask. Turn the power switch off. Discard the distillate in the erlenmeyer flask.



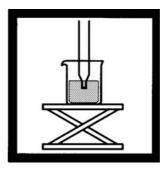
13. Perform this step and Step 14 under a hood. When the distillation flask is cool, add 50 mL of 19.2 N Sulfuric Acid Standard Solution to the flask using a 50-mL graduated cylinder.



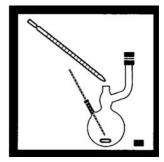
14. Add one 10-gram scoop of Potassium Bromide to the distillation flask.

flask.

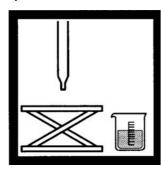
Note: Hach does not supply Potassium Bromide. Order directly from your local chemical supply vendor.



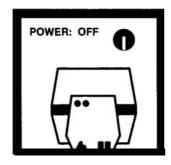
15. Fill a 250-mL beaker to the 75-mL mark with deionized water. Place it under the drip tube. Elevate the beaker so the tip of the tube extends below the surface of the water.



16. Add 1.00 mL of 30% Hydrogen Peroxide Solution to the distillation flask. Turn the stir control to 5 and the heat control to 10. Cap the distillation flask.



17. Heat the distillation flask until the yellow color is gone from the entire distillation apparatus, including the J-tube and condenser. Remove the beaker from under the drip tube.



18. Turn off the heater power switch.



19. When the J-tube and condenser have cooled, rinse them with demineralized water using a plastic wash bottle. Add the washings to the 250-ml beaker. The total volume in the beaker should be about 100 mL.



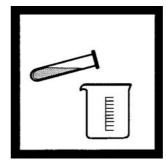
20. Add Phenol Solution, drop-wise, to the distilled sample to remove the yellow bromine color. A white precipitate of tribromophenol will form.



21. Allow the precipitate to settle. Use a dropper to collect about 5 mL of the clear, colorless distillate and transfer it to a test tube.



22. Test the solution for completeness of precipitation by adding 2 drops of Phenol Solution to the test tube. If the solution becomes cloudy, residual bromine is present (proceed to next step). If no cloudiness occurs, the sample is ready for analysis by the Diaminobenzidine Method.



23. If cloudiness occurs, return the 5 mL aliquot to the original sample and continue to add Phenol Solution until no turbidity is formed in subsequent 5-mL aliquots.



24. Transfer the sample solution into a 500 mL volumetric flask. Rinse the beaker with demineralized water and add to the flask. Dilute to volume with demineralized water, stopper and mix thoroughly. The sample is ready for analysis by the Diaminobenzidine Method.

ACCURACY CHECK

Standard Solution Method

Prepare a 100 mg/L selenium standard solution by pipetting 10.0 mL of 1000 mg/L Selenium Standard Solution, into a 100-mL volumetric flask. Dilute to volume with demineralized water. Prepare a 0.5 mg/L selenium standard by pipetting 5.00 mL of the prepared 100 mg/L selenium standard solution into a 1000 mL volumetric flask. Dilute to volume with demineralized water. Transfer 500 mL of the diluted standard into a 1000-mL beaker. Perform the test as described above.

Required Reagents

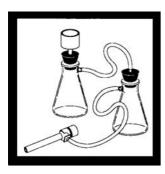
Description	Cat. No
Calcium Chloride Solution, 500 mL	428-49
Hydrochloric Acid Standard Solution, 0.1 N, 1000 mL	14812-53
Hydrogen Peroxide, 30%, 473 mL	144-11
Methyl Orange Indicator Solution, 0.1 N (0.05%), 500 mL	148-49
Phenol Solution, 30 g/L, 29 mL	2112-20
Potassium Bromide, ACS	Order locally
Potassium Permanganate Standard Solution, 100 mL	14164-42
Sodium Hydroxide Standard Solution, 0.1 N, 1000 mL	191-53
Sulfuric Acid Standard Solution, 19.2 N, 500 mL	2038-49
Water, demineralized, 4 L	272-56

Required Apparatus

Description	Cat. No
Beaker, 1000 mL	500-53
Beaker, 250 mL	500-46H
Bottle, wash, 500 mL	620-11
Cylinder, graduated, 50 mL	508-41
Cylinder, graduated, 500 mL	508-49
Distillation apparatus accessories for general purpose	22653-00
Distillation Apparatus Heater, 115 V	22744-00
Distillation Apparatus Heater, 230V	22744-02
Dropper, 1-mL mark, 6/pkg	23185-06
Hot Plate, 3.5" diameter, 120 V	12067-01
Jack, laboratory, platform	22743-00
Pipet Filler, safety bulb	14651-00
Pipet, serological, 10 mL	532-38
Pipet, volumetric, Class A, 1.00 mL	14515-35
Pipet, volumetric, Class A, 5.00 mL	14515-37
Rod, stirring, glass, 3/pkg	1770-01
Scoop, plastic, 10 g	26572-10
Test Tube, 10mL, 10/pkg	565-10

Description	Cat. No
Flask, volumetric, 1000 mL	14574-53
Selenium Standard Solution, 1000 mg/L, 100 mL	22407-42
Standard Methods for the Examination of Water and Wastewater, current edition	22708-00

Volatile Acid Distillation¹



1. Arrange the filtering apparatus as shown.



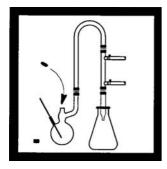
2. Using plastic tweezers, place a filter paper in the Buchner funnel. Use a wash bottle to moisten the paper with a small volume of demineralized water.



3. Fill a 250-mL graduated cylinder to the 200-mL mark with sample.



4. Turn the water on to the vacuum aspirator and pour the 200 mL of sample into the Buchner funnel. Filter the entire sample.

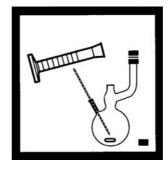


5. Set up the distillation apparatus by assembling the general purpose accessories as shown in Figure 2 in the Assembly section. Place a stir bar into the distillation flask.



6. Using a graduated cylinder, measure 100 mL of the filtrate from step 4. Pour it into the distillation flask.

Note: For proof of accuracy, use a 1000 mg/L Volatile Acids Standard Solution (listed in Optional Reagents) in place of the sample



7. Measure 100 mL of demineralized water with a 100-mL graduated cylinder. Add this to the distillation flask.



8. Turn on the stirrer power switch and set the stir control to 5.

¹Adapted from Standard Methods for the Examination of Water and Wastewater



9. Using a pipet bulb and a 5-mL serological pipet, add 5 mL of 19.2 N Sulfuric Acid Standard Solution to the flask. Cap the flask.



10. Set the heat control to 10. Ensure a constant flow of water is maintained through the condenser.



11. Collect 150 mL of distillate in the 300-mL erlenmeyer flask, then turn the heater off. Use a graduated cylinder to measure the distillate to be sure 150 mL has been collected. The distillate is ready for analysis by the Esterification or Titrimetric Method.

Required Reagents

Description	Cat. No
Sulfuric Acid Standard Solution, 19.2 N, 500 mL	2038-49
Water, demineralized, 4 L	272-56

Required Apparatus

Description	Cat. No
Bottle, wash, 500 mL	620-11
Cylinder, graduated, 100 mL	508-42
Cylinder, graduated, 250 mL	508-46
Distillation apparatus accessories for general purpose	22653-00
Distillation Apparatus Heater, 115 V	22744-00
Distillation Apparatus Heater, 230 V	22744-02
Filters, 9.0 cm, 100/pkg	506-55
Flask, filtering, 500 mL	546-49
Funnel, Buchner, 91 mm	550-87
Pipet Filler,	12189-00
Pipet, serological, 5 mL	532-37
Tubing, rubber, 3.6 m (12 ft)	560-18
Tweezers, plastic	14282-00
Vacuum, Aspirator	2131-00

Description	Cat. No
Standard Methods for the Examination of Water and Wastewater, current ed.	22708-00
Volatile Acids Standard Solution, 1000 mg/L as acetic acid, 100 mL	14205-42

Replacement Parts

Arsenic Distillation Apparatus

Description	Cat. No.
Arsenic Distillation Apparatus Set	22654-00
Set includes:	
Cylinder, 50 mL	22657-00
Gas Bubbler	22656-00
Powder Funnel	22644-67
Gas Scrubber	22655-00

Cyanide Distillation Apparatus

Description	Cat. No.
Cyanide Distillation Apparatus Set	22658-00
Set includes:	
Aspirator	2131-00
Cable Ties (12)	6790-45
Cylinder, 50 mL	22657-00
Flask, filter, 500-mL	546-49
Flow Meter	17875-00
Gas Bubbler	22656-00
Stopper Assembly, filter flask	17877-00
Thistle Tube	22652-00
Tubing, rubber, 0.6 m (2 ft), 3/16" ID	18106-02
Tubing, rubber, 3.6 m (12 ft), 1/4" ID	560-18

General Purpose Distillation

Description	Cat. No.
General Purpose Distillation Apparatus Set	22653-00
Set includes:	
Beaker, 400-mL	500-48
Cable Ties (12)	6790-45
Cap, large	22647-00
Cap, small	22739-00
Condenser	22650-00
Connector, Teflon, small (4)	22648-00
Connector, thermometer	22740-00
Connector	22741-00
Drip Tube	22651-00
Flask, distillation, 500 mL	22646-49
Flask, Erlenmeyer, 300 mL	505-47
J-tube	22649-00

Replacement Parts

General Purpose Distillation (continued)

Description	Cat. No.
Manual, Distillation Procedures	22653-08
Stir Bar	20953-51
Thermometer, -10 to 225 °C	26357-00
Tubing, Rubber, 3.6 m (12 ft) 1/4" ID	560-18

Heater and Support Apparatus

Description	Cat. No.
Heater and Support Apparatus, 115 Vac, 60 Hz	22744-00
Heater and Support Apparatus, 230 Vac, 50 Hz	22744-02
Set includes:	
Heater, 115 Vac, 60 Hz (included with 22744-00 only)	22737-00
Heater, 230 Vac, 60 Hz (included with 22744-02 only)	22737-02
Clamp, holder	326-00
Clamp, 3-prong, universal	422-00
Support Rod	22738-00

Optional Replacement Apparatus

Description	Cat. No.
Laboratory Jack	22743-00
Stir Bar Retriever, 45.7 cm (12 in. long)	15232-00

P.O. Box 389, Loveland, CO 80539-0389 U.S.A. Tel. (970) 669-3050 (800) 227-4224 (U.S.A. only) Fax (970) 669-2932 orders@hach.com www.hach.com

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