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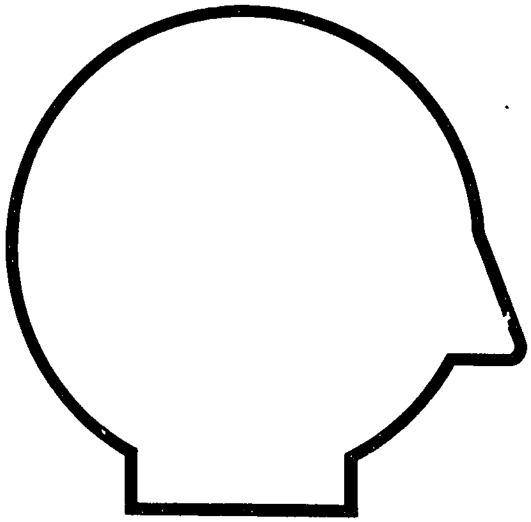
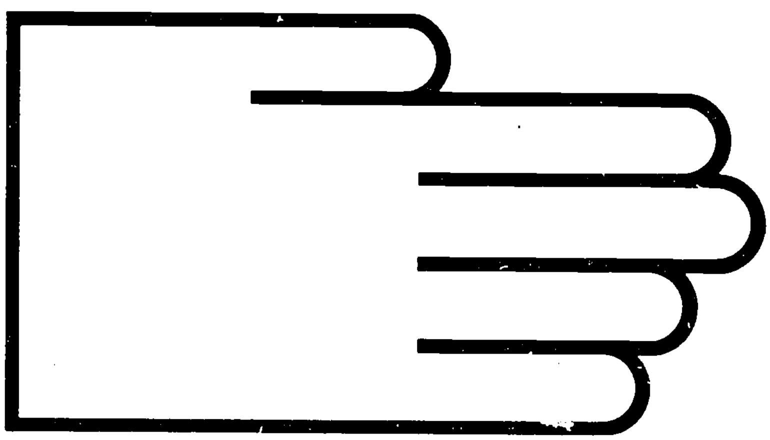
ABSTRACT

This module, one of 25 on vocational education training for careers in environmental health occupations, contains self-instructional materials on performing titration analysis for water quality. Following guidelines for students and instructors and an introduction that explains what the student will learn are three lessons: (1) naming each part of the titrating apparatus and telling how each works or is used; (2) getting the titrating apparatus ready for use by putting it together and by cleaning the buret; and (3) reaching the endpoint of a titration to standardize the titrating solution, sodium thiosulfate. Each lesson contains objectives, recommended methods and locations for practice, performance criteria, equipment and supplies to perform a task, detailed step-by-step instructions for learning a task, and performance exercises. Two performance tests cover preparing the titration apparatus and doing a titration to standardize the titrant.



Performing Titration Analyses for Water Quality



Module 17

U.S. DEPARTMENT OF EDUCATION, Office of Vocational and Adult Education

FOREWORD

The Curriculum and Instruction Branch of the Office of Vocational and Adult Education, U.S. Department of Education, identified a need to improve the training opportunities for vocational education students interested in pursuing careers in environmental health. To fulfill that need, Consumer Dynamics, Inc., a Rockville, Maryland, based company, was awarded the contract to develop performance-oriented, competency-based modules in the environmental health sciences.

PERFORMING TITRATION ANALYSES FOR WATER QUALITY is one of the modules in the series, "Vocational Education Training in Environmental Health Sciences." The module content is based on selected texts and other materials in the environmental health field. The module is intended to supplement existing course materials.

CONTENTS

USING THESE SELF-INSTRUCTION MATERIALS

This self-instruction learning package or module is designed to allow both students and instructors flexibility of use. Although primarily intended for use in existing training programs, the module can be used by anyone interested in learning new skills or brushing up old ones. Therefore, two sets of guidelines are presented--one set addressed to students and the other set addressed to instructors. First, find out how you, the student, should use the materials in this book.

GUIDELINES FOR STUDENTS

Take the Performance Test as a pretest.

When you pick up this book and work through it, your goal will not be a letter grade or a high score on an exam. Instead, you will work to develop skills that you can measure. You will not have to worry about how well someone else is doing. Before you start work on this book, you should, first, find out if you have sufficient skills to start training by reading through the section called PERFORMANCE TEST. If you think you can do all or most of the items in this test, ask your instructor to help you set up the equipment and obtain reagents you will need to perform a titration. One word of caution--you should have had a high school course in chemistry or learned the equivalent on the job. Before doing a titration, there are a number of chemistry techniques you should already know how to apply. Some of them include knowing how to handle acids and bases and how to prepare chemical reagents, using pipets and other volumetric measuring glassware and equipment. If you are sure you meet these criteria, demonstrate to your instructor how well you can do a titration.

Work on parts you need to practice.

If you do everything well, according to the criteria in the Performance Test guidelines, you will not need to spend time working on this module. If after taking the Performance Test you discover there are parts you need to practice, follow the key to each item in FOR FURTHER STUDY. To successfully complete a titration, however, you should be able to complete items 6, 7, 9, 10, and 11 of the Performance Test.

USING THESE SELF-INSTRUCTION MATERIALS

Work straight through each lesson in the order presented.

Should you decide to completely work through this book, begin with the INTRODUCTION and go straight through each of the three lessons. The lesson begins with the OBJECTIVE of the training. Follow the instruction for each part in the order presented. Practice each step in a lesson until you can do it according to the criteria stated for the step. At the end of a lesson, do the EXERCISES. To test your skills in following titration procedures, the exercises at the end of Lesson Three include steps for doing the dissolved oxygen test. When there are audiovisuals listed at the end of a lesson, ask your instructor for help in obtaining them.

Take the Performance Test as a posttest.

Finally, after you have mastered the exercises, ask your instructor to watch you perform a titration. The guidelines in the Performance Test can be used as a posttest to evaluate the quality of your performance. Turn now to the Performance Test.

GUIDELINES FOR INSTRUCTORS

Approach

The approach of these materials is to provide the student with the techniques for preparation of the equipment and for standardizing the reagents against a "known." In the exercises for Lesson Three, the student is required to apply all of the techniques presented in the module for determining the dissolved oxygen content in an unknown sample.

Preparation of Chemistry

This module can be used as a laboratory orientation to performing titrations. Whether students should standardize titrants and fix water samples is a decision you should make.

Independent Study

Students can work independently and at their own pace. Depending on the time frame you set for completing each lesson, you may want to start a group off in each lesson with a demonstration and informal presentation.

As a Laboratory Workbook

Alternatively, you may choose to use this module as a laboratory workbook in a structured laboratory session. With this option, you may allow students greater access to your assistance, especially in watching them perform the pre- and posttest portions of the training.

General Instructions

Read through each lesson to anticipate what equipment, reagents, and supplies you will need to make available for students to use. Also, order any audiovisuals or reading materials you think may present a complementary perspective to the training in this module. Use the guidelines presented in the Performance Test as the minimum requirements for gauging successful completion of the training.

BACKGROUND

Performing chemical tests on water or wastewater is a necessary part of determining water quality. The U.S. Environmental Protection Agency has determined that certain amounts of some chemicals in the Nation's waterways are harmful to people, animals, and plants. State and Federal laws have been passed to protect the environment. Those laws limit the amount of harmful substances that cities, factories, and people can dump into lakes, ponds, streams, or rivers.

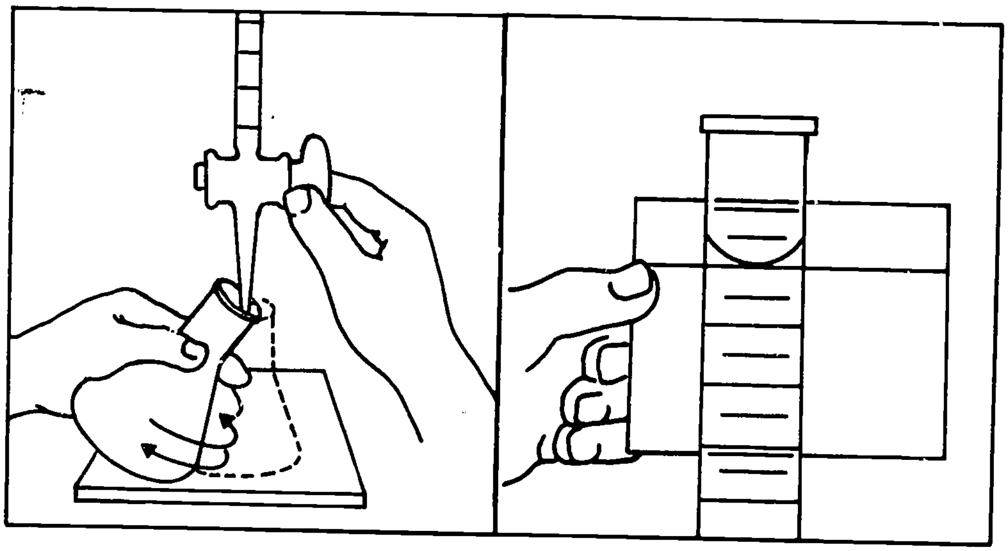
By chemically testing the water, it is possible to determine the degree of pollution. Public health officials review the results of water quality tests to determine if local or Federal laws have been broken. They also decide what action may be necessary to decrease or stop any more of the harmful substances from entering water supplies.

Most routine chemistry tests for water quality are currently being performed using field test kits. Although very sophisticated field test kits are now available, they are not yet widely used. Readily available field test kits provide results that are only close to the real value. Such results signal changes in conditions. The kits cannot be relied upon to yield results that will in turn be used to calculate fine adjustments to a pollution control system. Even if you will use more sophisticated equipment, manual titrations are useful as a backup.

You may be required to perform laboratory tests for water quality if you are employed by an electric utility company, a chemical testing laboratory of a manufacturing company, or a food processing company, or if you are involved in several other related jobs. The chemical testing techniques you will learn in this book will help you to perform several specific tests that require chemical testing by titration.

WHAT YOU WILL LEARN

When you finish working through the steps and exercises in this book, you will be able to perform a chemical test for water quality using the techniques of titration. To help you learn how to titrate or add small amounts of chemicals to a water sample, you will learn how to do a test for dissolved oxygen (DO) using titrating apparatus, reagents, and other laboratory supplies.



You will learn how to perform a titration in three lessons:

- <u>Lesson One</u>

  You will be able to name each part of the titrating apparatus and tell how each works or is used.

- <u>Lesson Two</u>

  You will be able to get the titrating apparatus ready for use by putting it together and by cleaning the buret.

- <u>Lesson Three</u>

  You will be able to reach the endpoint of a titration to standardize the titrating solution, sodium thiosulfate.

OBJECTIVE

You will be able to name each part of the titrating apparatus and to tell how each works or is used.

WHERE AND HOW TO PRACTICE

Because titration requires laboratory equipment and supplies, you should practice this lesson in a laboratory setting. Practice identifying the parts by indicating the names for the labeled parts. You may wish to make notes on your work in the space provided or in the extra spaces on each page.

HOW WELL YOU MUST DO

You must be able to name all parts of the apparatus and describe how they work or are used in doing the titration. You should be able to do this in less than 10 minutes.

THINGS YOU NEED

You will need the following equipment:

- buret stand
- buret holder or clamps
- buret, 100-ml with ground glass stopcock
- buret, 100-ml with Teflon stopcock
- stopcock grease

<u>Instructions</u>: Now turn to the next page and begin work on Lesson One, "Getting There--Steps."

GETTING THERE--STEPS

STEP 1

Notice how the buret stand is constructed. It has a heavy base made of metal or ceramic material. The shaft is threaded on one end and is secured by a nut or metal thread in the stand.

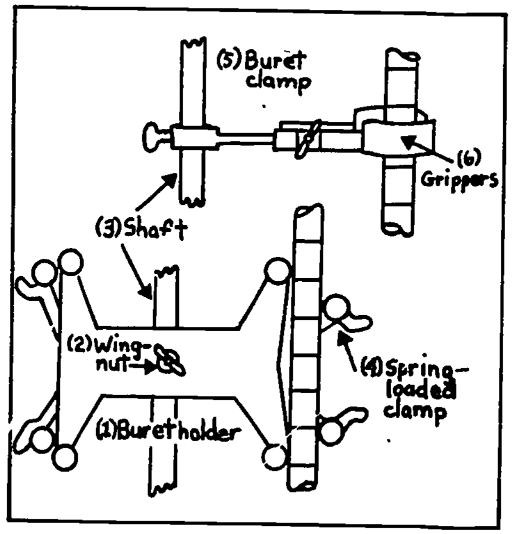
KEY POINT 1

Make sure the shaft is securely fastened to the base.

STEP 2

Pick up the buret holder or clamp. The buret holder (1) has a wingnut (2) that tightens the holder to the shaft (3). The holder has spring-loaded clamps (4) to hold the buret. If a buret holder is not available, use a clamp (5) with protected expandable grippers (6) to hold the buret.

KEY POINT 2

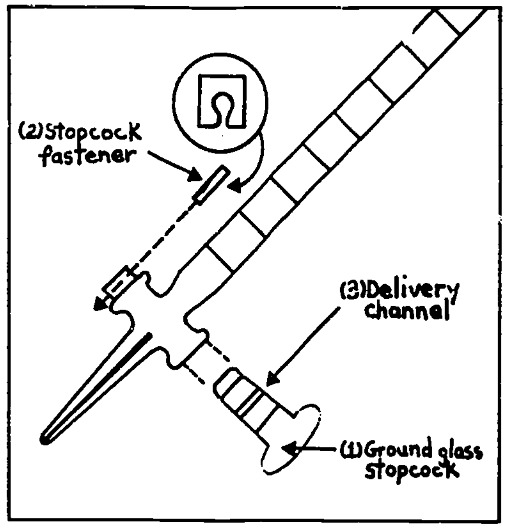


Make sure the wingnuts are tight. The expandable grippers must not be too tight on the buret.

STEP 3

Pick up the 100-ml buret with a tapered ground glass stopcock (1) and stopcock grease. Spread a very thin layer of grease on the ground glass portion of the stopcock. Insert the stopcock into the buret and secure with a stopcock fastener (2). Turn the stopcock to spread the grease.

KEY POINT 3

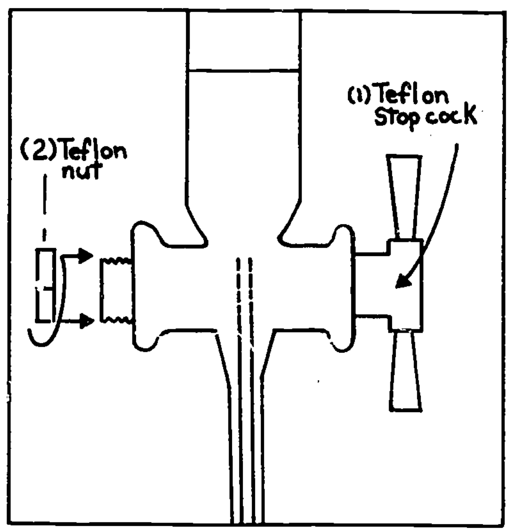


Do not add too much stopcock grease because it could clog the delivery channel (3).

STEP 4

Pick up the 100-ml buret with Teflon stopcock (1). The stopcock has a threaded end for a Teflon nut (2). Tighten the nut so there is no movement of the stopcock from side to side.

KEY POINT 4

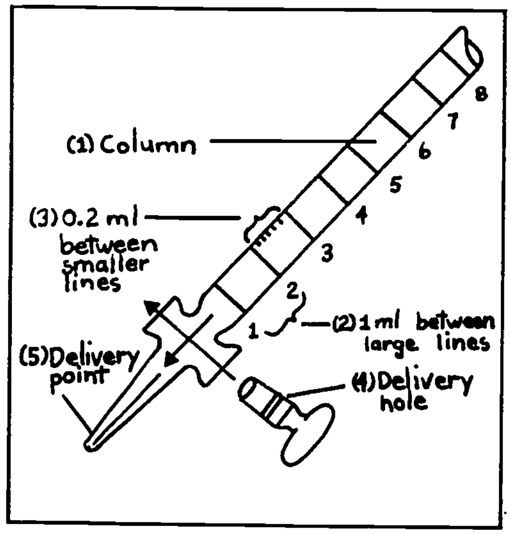


Tighten the Teflon nut to prevent side-to-side movement but not too tightly to keep it from turning easily.

STEP 5

The buret column (1) is graduated in milliliters (ml). The volume the column can hold between each number printed on the column is 1 full ml (2). On the 100-ml buret, the individual lines in between the units indicate 0.2 ml (3). Titrating solution is stored in the column until the stopcock is turned so that the fluid can run through the delivery hole (4) and then through the delivery point (5) to the sample.

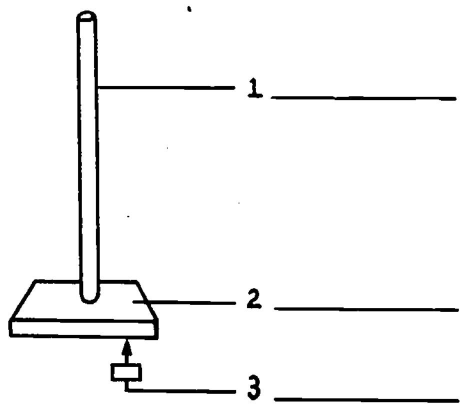
KEY POINT 5

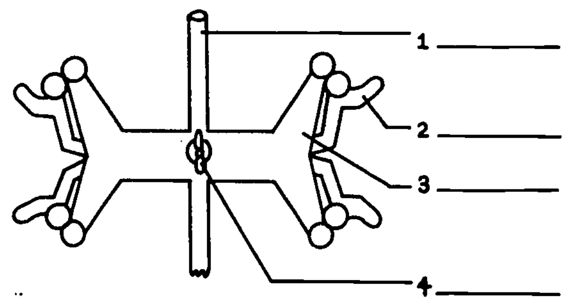


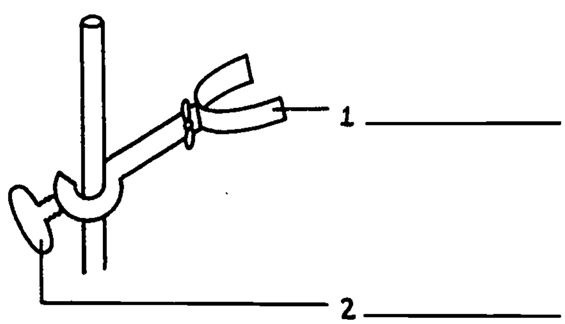
Titrating solution can be released from the column drop by drop or in a steady stream.

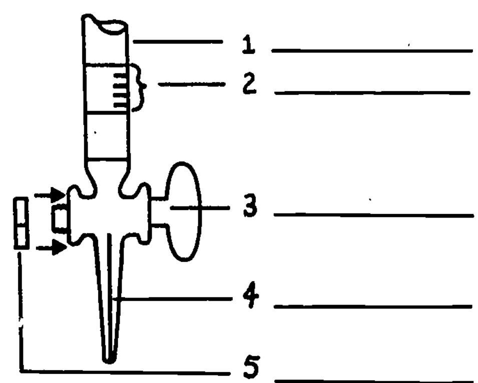
EXERCISES

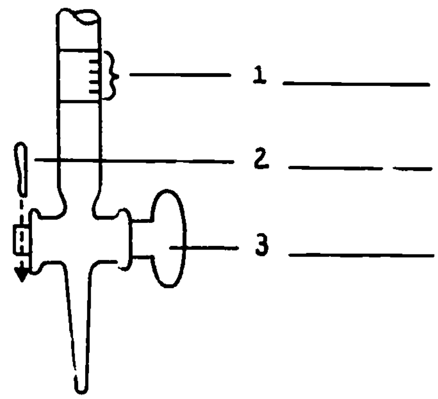
Instruction 1: Referring to the equipment and/or drawings in the lesson, label the following drawings. You must be able to name each part in the drawing and write a short paragraph on how it functions or is used.











Instruction 2: When you can correctly label each part in this exercise and tell what its use is, you are ready to begin work on Lesson Two.

OBJECTIVE

You will be able to get the titrating apparatus ready for use by putting it together and by cleaning the buret.

WHERE AND HOW TO PRACTICE

As in Lesson One, you should practice the steps in Lesson Two in a laboratory. Because you will be handling caustic materials used for cleaning, exercise extreme caution. Should the caustic materials contact your skin, wash them off with plenty of water. Inform your instructor or laboratory supervisor as soon as possible. Never work in the laboratory alone. Follow each step in "Getting There" and go over each one as many times as it takes to do the step correctly.

HOW WELL YOU MUST DO

Assemble the titrating apparatus so that the buret is held firmly but not so tightly that the glass sides of the buret will crack when a titration is performed. Clean the buret column so that distilled water flows evenly over the buret walls without beading or forming bubbles. Clean the delivery point and delivery channel so that they are completely free of grease, other kinds of dirt, or chemical residues. Lubricate and/or tighten the stopcock so that it turns freely but does not leak.

THINGS YOU NEED

In addition to the equipment you used in Lesson One, you will need the following:

- o acetone
- o beaker (250-ml)
- o buret brush
- o chromic acid cleaning solution in squeeze bottle[1]
- o copper wire (thin)
- o distilled water
- o full-face shield
- o laboratory apron (acid-resistant)
- o paper towels
- o phosphate-free liquid detergent
- o rubber gloves (acid-resistant)
- o safety glasses with side shields
- o small funnel
- o waste container (noncorrosive)

Caution: When working with acids, bases, or other caustic and corrosive chemicals, wear a full-face shield made of corrosion-resistant plastic. You should wear safety glasses with side shields at all other times when working in a laboratory.

Instructions: Now turn to the next page and begin work on Lesson Two, "Getting There--Steps."

[1]Prepare acid cleaning solution according to "Standard Methods," 14th ed., p. 336.

GETTING THERE--STEPS

STEP 1

Place the buret holder or adjustable clamp so that it is about one-third of the way down the shaft. Fasten the holder securely by turning in the wingnut or thumbscrew.

KEY POINT 1

Fasten the buret holder securely.

STEP 2

Adjust the buret so that the delivery point is approximately 4 to 6 inches above the buret stand base.

KEY POINT 2

When using adjustable clamps instead of a holder, do not tighten too much or you will crack the glass column.

STEP 3

Locate the squeeze bottle of chromic acid cleaning solution. Pick up the buret and close the stopcock. Pour about 5 ml of chromic acid solution into the buret.

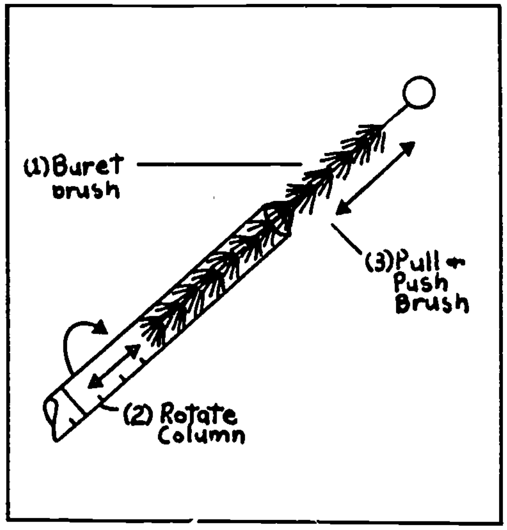
KEY POINT 3

Wear gloves, apron, and fullface shield when handling caustic or corrosive materials over a laboratory sink.

STEP 4

Insert the buret brush (1) carefully into the column. With one hand slowly rotate the column (2). With the other hand slowly pull and push (3) the brush up and down the column.

KEY POINT 4



Perform operation over a laboratory sink. Be careful not to splash the acid cleaning solution.

STEP 5

Remove the buret brush and place in sink. Pour the chromic acid solution into a noncorrosive waste container, using a funnel to prevent splashing. Open the stopcock. Fill a beaker with tapwater and flush the buret with a beakerful of water at least five times.

KEY POINT 5

Pour used chromic acid into a waste container rather than pouring it down the sink.

STEP 6

Close the stopcock. Pour a 10-ml portion of liquid detergent into the buret. Repeat Step 4 after carefully rinsing the buret brush. Open the stopcock to allow detergent to drain through the delivery point.

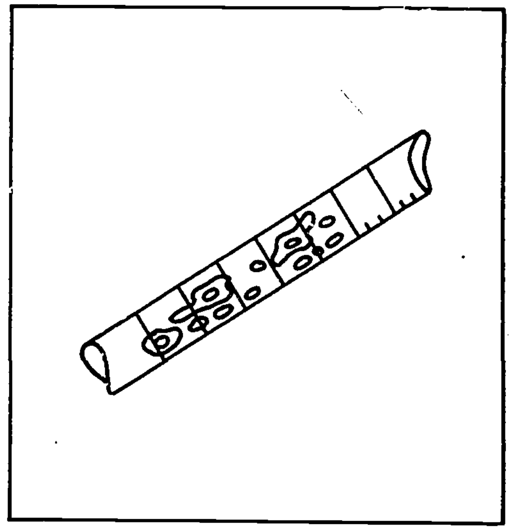
KEY POINT 6

Be sure to close the stopcock before adding the detergent.

STEP 7

Rinse the buret at least five times with tapwater and five times with distilled water. If the buret is clean, the distilled water will flow evenly over the surfaces of the column without beading or forming bubbles. Drain the column and close the stopcock.

KEY POINT 7



Bubbles form when a dirty buret is filled with a liquid.

STEP 8

Pour a small portion of acetone into the column. Hold the buret at an angle and slowly rotate it to allow the acetone to wet all surfaces. Open the stopcock and allow the column to drain into the sink.

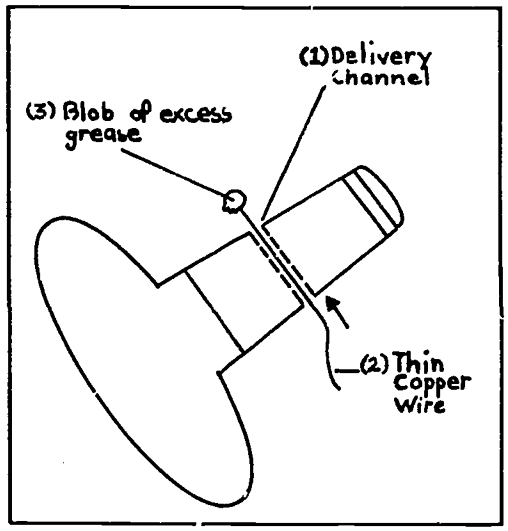
KEY POINT 8

Use acetone to help dry the buret after you see that distilled water wets all surfaces evenly.

STEP 9

Turn the ground glass stopcock to see if it turns easily. Spread a thin layer of grease on the ground glass portion as you did in Step 3 of Lesson One. Replace the stopcock in the buret and turn it a few times. Check the delivery channel (1) to make sure it is not blocked. If grease is blocking the hole, use the thin copper wire (2) to push the grease out (3).

KEY POINT 9



Push a thin copper wire through the delivery channel to clear it.

STEP 10

Turn the Teflon stopcock to check its adjustment as in Step 4 of Lesson One. If the hole is blocked use a thin copper wire to clear it.

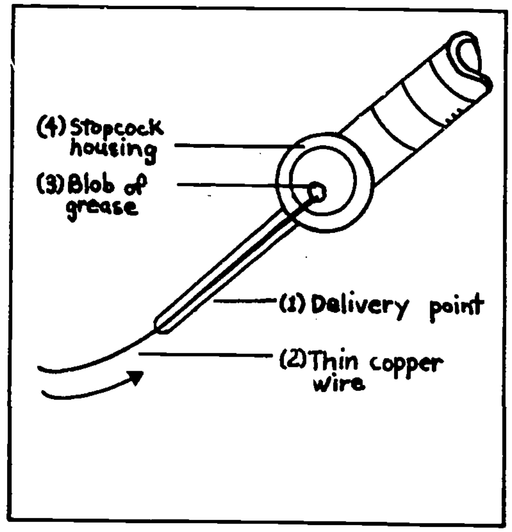
KEY POINT 10

Do not grease a Teflon stopcock.

STEP 11

Check the delivery point (1) to see that it is not blocked. Run a thin copper wire (2) into the point and remove the grease or dirt (3) from the inside of the stopcock housing (4).

KEY POINT 11



Push a thin copper wire through the point to clear it.

STEP 12

Place the cleaned buret upside down on the buret stand until ready to use. Open the stopcock.

KEY POINT 12

Open the stopcock to allow for complete draining.

EXERCISES

Instruction 1: Practice each step in Lesson Two until you can do the following:

1. Use the chromic acid cleaning solution and liquid detergent to clean the buret so that distilled water flows evenly over the buret walls without beading or forming bubbles.

2. Lubricate a ground glass stopcock so that it turns freely and the delivery channel does not become blocked.

3. Adjust and clean a Teflon stopcock so it turns freely, does not leak, and fluid passes freely through the delivery channel.

4. Clean the delivery point so that it is free from all grease and dirt.

5. Adjust the buret holder so it is one-third of the way down the shaft of the buret stand.

6. Place the buret in the holder so that it is 4 to 6 inches from the buret stand base, and secured so the column does not crack.

Instruction 2: When you have mastered each of the above, you are ready to begin work on Lesson Three.

OTHER READING

E.I. DuPont de Nemours and Company, Inc., "Titration and Its Glassware" (no date).

This manual provides an introduction to common types of laboratory glassware and units of volumetric measurement, and the steps in titrating a sample. If the manual is used as the basis of instruction, an average of 3 hours is required for presentation.

It is available for $7.40 from E.I. DuPont, Education and Applied Technology Division, Brandywine Building, Wilmington, DE 19898.

LESSON THREE

OBJECTIVE

You will be able to reach the endpoint of a titration to standardize the titrating solution, sodium thiosulfate.

WHERE AND HOW TO PRACTICE

In this lesson you will again do all your work in the laboratory. As in Lesson Two you should work with at least one other person; then, if an accident occurs you will have help. Wear personal protective equipment, including a lab apron and safety glasses. When handling acids wear the fullface shield. Practice standardizing the 0.0375 N sodium thiosulfate.[1] When you have mastered this part of the dissolved oxygen (DO) test,[2] practice the DO test, using a water sample containing an unknown amount of DO.

HOW WELL YOU MUST DO

You must be able to read the volume in a buret to within plus or minus (±) 0.05 ml. When standardizing or determining the actual normality of the sodium thiosulfate titrating solution, you must be able to do duplicate determinations that agree within ± 0.2 ml[3] and within ± 0.0004 N of the normality of the standardized titrant.

[1]To find the exact normality or N of the titrating solution, you must prepare a sample containing a known amount of a chemical that will react completely with the titrant and show a color change.

[2]Modified Winkler test, using full-bottle technique.

[3]0.2 ml at 7.5 mg/l DO is the limit of reproducibility (USEPA "Methods for Chemical Analysis of Water and Wastes").

THINGS YOU NEED

In addition to the equipment and supplies you used in the previous two lessons, you will need the following:

- distilled water
- two 500-ml Erlenmeyer flasks
- 10-ml graduated pipet
- 20-ml volumetric pipet
- pipet bulb
- potassium biniodate standard titrant, 0.0375 N[1]
- potassium iodide (KI), 2 g[1]
- sodium thiosulfate standard titrant, 0.0375 N[1-3]
- starch solution[1]
- 10% sulfuric acid (H_2SO_4)[1]

Instructions: Step 5 requires the use of graduated and volumetric pipets. Before attempting to do this step, practice using the pipets so you can deliver fluids to within 0.05 ml of their capacities. Now turn to the next page and begin work on Lesson Three, "Getting There--Steps."

[1]These materials should be prepared by your instructor, or your instructor can provide separate instructions for fixing samples. The titrants should already be standardized so you can test the accuracy of your titrations. (See USEPA Methods for Chemical Analysis of Water and Wastes, 1974.)

[2]Titrations may be made with 0.025 N $Na_2S_2O_3$; use 200-203 ml of sample to obtain 1 mg titrant to 1 mg DO/l.

[3]Phenylarsine oxide (PAO) is more stable than $Na_2S_2O_3$ and is the preferred titrant; $Na_2S_2O_3$ is used here because it is still commonly known and used.

GETTING THERE--STEPS

STEP 1

Prepare a buret for use. Locate the standard sodium thiosulfate titrating solution (titrant), a clean small funnel, and a clean 100-ml buret. Close the stopcock. Pour a 10- to 20-ml portion of the titrant from the stock bottle into a clean beaker. From the beaker pour the titrant through the funnel into the buret. Take the buret out of the holder and, holding the buret at an angle, rotate it as you did in Step 8, Lesson Two. Open the stopcock and drain the buret.

KEY POINT 1

All glassware must be thoroughly cleaned. Always pour stock solutions into another well-cleaned container to prevent contamination of the stock solution. Do not pour excess liquids back into the stock solution bottle.

STEP 2

Replace the buret in the holder. Close the stopcock. Pour more titrant into the beaker used in Step 1 and slowly fill the buret nearly to the top of the column. If bubbles are present, take the buret out of the holder and rotate it as you did previously. If bubbles remain, drain all of the titrant into a waste container and clean the buret following the steps in Lesson Two.

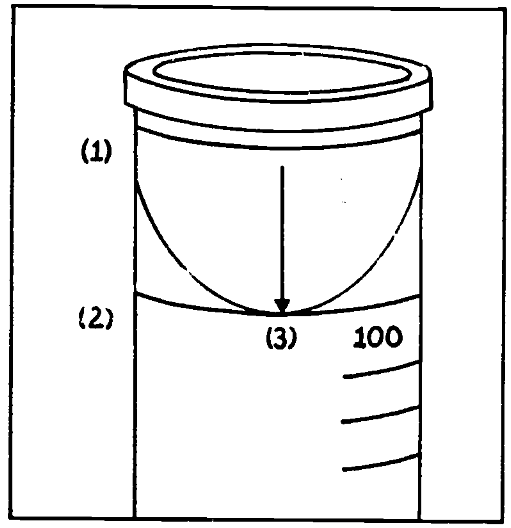
KEY POINT 2

Remove bubbles by rotating the buret. If bubbles remain, the buret may be dirty.

STEP 3

Adjust the level of titrant in the column. Place a beaker for waste titrant under the delivery point. Open the stopcock just enough to allow drops to form on the point and then slowly fall into the beaker. Watch the level of the titrant drop from (1) toward (2), the 100-ml line. Close the stopcock slowly as it reaches the line. Close the stopcock completely when the curved bottom of the fluid meniscus just touches the line at (3).

KEY POINT 3

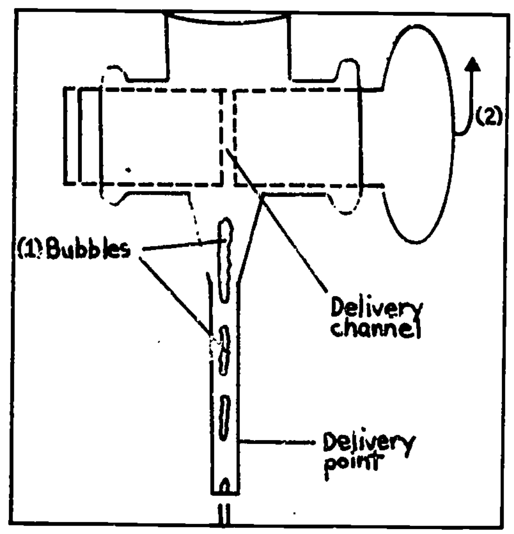


Close the stopcock completely when the meniscus (3) touches the 100-ml line.

STEP 4

Check for bubbles (1) in the delivery point. If there are bubbles, open the stopcock (2) until the titrant streams out. The bubbles will flow out of the point when the stopcock is open completely. Add more titrant and repeat Step 3.

KEY POINT 4

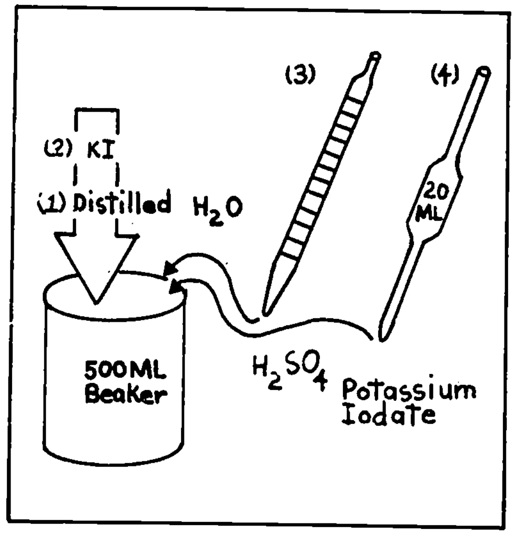


Open the stopcock completely to remove bubbles from the delivery point.

STEP 5

Prepare a sample containing a known amount of potassium biniodate. Into a 500-ml beaker put 150 ml distilled water (1). Add 2 g potassium iodide (2) and dissolve. Using a 10-ml graduated pipet, add 10 ml of 10% H_2SO_4 (3). Using a 20-ml volumetric pipet, add 20 ml potassium iodate solution (4).

KEY POINT 5



When adding (3) and (4), do not pipet by mouth. Always use a pipet bulb.

STEP 6

Place beaker with contents added in Step 5 into the dark for 5 minutes. After that time carefully pour the sample into a 500-ml graduated cylinder. Add enough distilled water with a squeeze bottle to bring the volume up to 300 ml. The sample should be a deep golden yellow.

KEY POINT 6

Transfer the sample to a 500-ml graduated cylinder and add distilled water.

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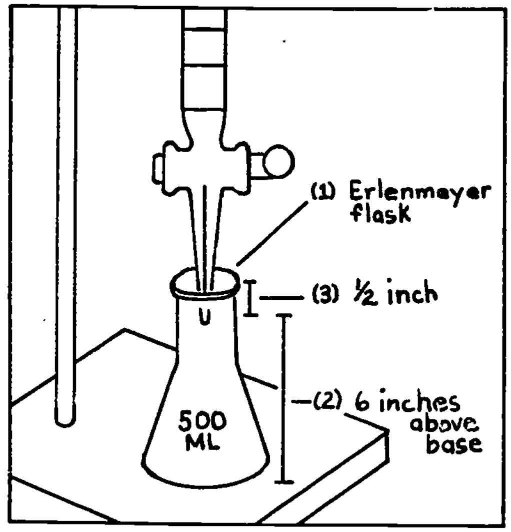
STEP 7

Transfer the sample from the cylinder to a 500-ml Erlenmeyer flask (1). Adjust the height of the buret so the delivery point is about 6 inches above the buret stand base (2). The point should be about one-half inch inside the flask (3).

KEY POINT 7

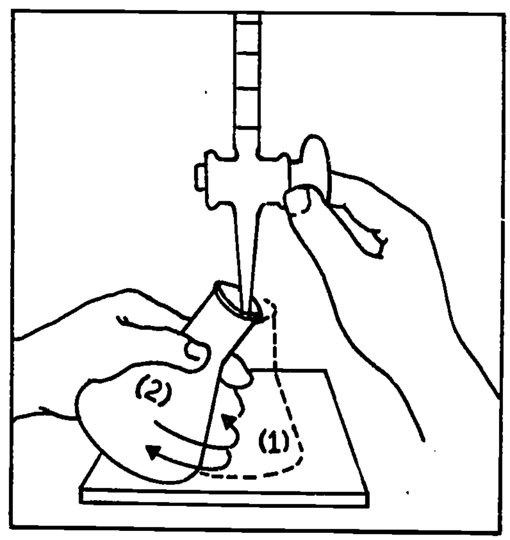


The delivery point should be about one-half inch inside the flask.

STEP 8

Add titrant to the sample. Center the flask directly under the delivery point (1). Open the stopcock slowly and add a few drops of titrant. Close the stopcock. Swirl the flask several times (2). Open the stopcock and add a few more drops. Repeat this procedure until the golden yellow color changes to a pale straw yellow.

KEY POINT 8



Add the titrant directly into the flask so it doesn't run down the sides.

STEP 9

Titrate to the color change. First, add 1 to 2 ml of starch solution. Swirl the flask until the blue color is evenly distributed through the sample. Add titrant drop by drop. As you get close to the endpoint, a single drop may make the blue color just about vanish. When you get to this point, give the stopcock a quick turn to deliver less than a full drop. The titration is completed when one drop or less permanently changes the sample color from blue to colorless. The change signals the endpoint. The blue color may return if the sample sits for 10 to 15 minutes.

KEY POINT 9

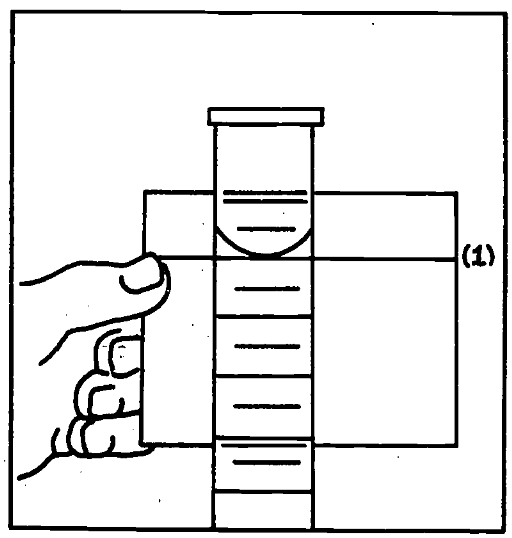
It can take as little as one drop to turn the sample from blue to colorless. Add the titrant very slowly as you get closer to the endpoint.

STEP 10

Read the amount of titrant used to reach the endpoint. Place a card (1) with a black line behind the buret at the meniscus. Record the amount used here:
_____________ ml

Repeat Steps 1-10 to do a duplicate sample. Record the amount of titrant used for the second sample here: _____________ ml

KEY POINT 10



Read the meniscus when the endpoint is reached.

STEP 11

Calculate the exact normality (N) of the standard sodium thiosulfate titrant. Use the simple mathematical expression: normality (1) of sodium thiosulfate (2) times (3) milliliters of it titrated (4) equals the normality (5) of potassium biniodate (6) times (7) the milliliters of it in the sample (8).

KEY POINT 11

(1) N	(2) $Na_2S_2O_3$	(3) X	(4) ml Titrant	=
	(?)		(20.2)	
(5) N	(6) $KHIO_3$	(7) X	(8) ml in sample	
(0.0375)			(20.0)	

STEP 12

This is the information you need to calculate the normality of the titrant:

(1) ? N (4) 20.2 ml[1]

(5) 0.0375 N (8) 20.8 ml

Determine the normality of the sodium thiosulfate you used with this mathematical expression:

$$(1) = \frac{(5) X (8)}{(4)}$$

KEY POINT 12

$$(1) = \frac{\overset{(5)}{0.0375} \times \overset{(8)}{20.8}}{\underset{20.2}{(4)}}$$

(1) = 0.0371 N

Instructions: In the exercises you will be titrating a water sample for unknown dissolved oxygen content. Use a portion of the titrant for which you found the exact normality. Turn to the next page and work through the exercises.

[1]This may not be the number of milliliters of titrant you used. If the normality of the standardized titrant is 0.0375 N, then your result may vary only ±0.2 ml.

EXERCISES

Instruction 1: You should practice all the steps in Lesson Three until you can duplicate determinations that agree within ±0.05 ml. When you can do this, you are ready to test water samples containing unknown amounts of dissolved oxygen.

Instruction 2: When testing for DO, EPA recommends using the Modified Winkler with Full-Bottle Technique, presented in "Methods for Chemical Analysis of Water and Wastes." To do this test you will need the following equipment and supplies in addition to those you used in Lesson Three:

- buret, 100-ml
- water sample in a 300-ml BOD bottle fixed with alkaline
- iodide-azide solution, manganous sulfate, and sulfuric acid[1]

You may find differences among DO procedures that are referenced in this book. By following whichever procedure you prefer, you will be able to develop the same level of skill knowledge.

Follow the steps starting on the next page. Doing a titration to determine an unknown amount of dissolved oxygen in a water sample should take less than 10 minutes.

[1]Fixed samples should be prepared by your instructor, or your instructor can provide separate instructions for fixing samples.

STEP 1

Clean two 100-ml burets and place them in the buret holder. Fill one buret with the sodium thiosulfate ($Na_2S_2O_3$) titrant that you standardized in Lesson Three. Fill the other buret with standard potassium biniodate [$KH(IO_3)_2$] titrant.

KEY POINT 1

Fill the burets and adjust the fluid levels as you did in Steps 1-4 in Lesson Three.

STEP 2

Transfer the fixed sample from the BOD bottle to a cleaned Erlenmeyer flask. To titrate, turn back to Steps 7, 8, and 9 in Lesson Three. Record your result here:

____

KEY POINT 2

Slowly pour the fixed sample from the BOD bottle into the flask so as not to lose any of the sample.

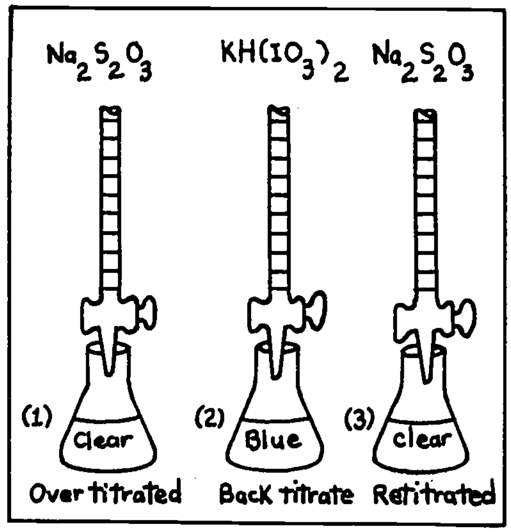
STEP 3

If you add too much $Na_2S_2O_3$ (1), you can still make an accurate determination using the $KH(IO_3)_2$ titrant in the other buret. Record the amount of $Na_2S_2O_3$ used here: (a)________.

Add the $KH(IO_3)_2$ titrant drop by drop until the blue color returns (2). Record the amount used here: (b)________________.

Retitrate using $Na_2S_2O_3$ (3). Record the amount used here: (c)________________.

KEY POINT 3



By backtitrating with $KH(IO_3)_2$ you will be able to continue the determination of unknown DO.

STEP 4.

Calculate the DO content of your sample. Each ml of 0.0375 N $Na_2S_2O_3$ is equal to 1 mg DO per liter when a 300-ml sample is used.

KEY POINT 4

For a 300-ml Sample:

X ml of 0.0375 N $Na_2S_2O_3$ titrated is equivalent to 1 mg/l DO

STEP 5

Calculate DO when you have to backtitrate. Transfer your results from Step 3:

(a) $Na_2S_2O_3$ ____________ ml

(b) $KH(IO_3)_2$ ____________ ml

(c) $Na_2S_2O_3$ ____________ ml

1. Subtract the ml of $KH(IO_3)2$ in (b) from the ml of $Na_2S_2O_3$ in (a).

2. To the resulting number, add the ml of $Na_2S_2O_3$ in (c). The result, ml (2.), is equal to mg/l DO in a 300-ml sample.

KEY POINT 5

1. $Na_2S_2O_3$ (a) ________ ml

minus $KH(IO_3)_2$ (b) ________ ml

equals ________ ml $Na_2S_2O_3$.

2. ________ ml (1.) plus

$Na_2S_2O_3$ (c) ________ ml

equals ________ ml (2.).

Instruction 3: To test your accuracy in determining the unknown DO in a sample, have your instructor make up a 1000-ml fixed sample. This can then be divided into three 300-ml portions. Do titrations on each sample. Your results should vary no more than 0.2 mg/l DO among all three samples.

Instruction 4: You should now be ready to take the Performance Test. Your instructor will give you a fixed water sample and watch how you do each step.

FILMS AND SLIDE/TAPE PROGRAMS

Feldmann, C.R. "Dissolved Oxygen Determination," April 1976.

This slide/tape program, consisting of a 15-minute tape, 72 slides, a script, quiz, and key, is designed for beginning laboratory personnel in chemistry. It covers sampling techniques, basic chemistry of the Winkler dissolved oxygen determination, methods for the addition of reagents, sample preservation, and the basic calculations used in the determination.

Available on loan from the National Training and Operational Technology Center (NTOTC), 26 W. St. Clair, Cincinnati, OH 45268.

Institute for Environmental Education. "Testing for Dissolved Oxygen Using the Modified Winkler Method," 1977.

This 65-frame filmstrip with tape cassette, text, reagents, and procedures manual is designed for secondary schools and colleges in monitoring programs. It introduces the significance of dissolved oxygen as an indicator of water quality and demonstrates field sampling and laboratory procedures using the modified Winkler method.

Available for $33.50 from the Institute for Environmental Education, 8911 Euclid Avenue, Cleveland, OH 44106.

PERFORMANCE TEST

Instructions: Check your skill level or progress by working through each of the following items. If you can perform the item as well as required, place a check in the space provided. When all of the items are checked, you are ready to demonstrate your skills to your instructor. You will be considered trained in those skills after your instructor approves your performance of each item.

(Instructors: You may use this checklist in guiding your evaluation.)

PREPARING THE TITRATION APPARATUS

No. 1 _____ Clean the buret column so that fluids wet the buret walls without beading or forming bubbles.

No. 2 _____ Dispose of used chromic acid cleaning solution in a waste container rather than down the sink.

No. 3 _____ Clean and lubricate a ground glass stopcock so that it turns freely without clogging.

No. 4 _____ Clean and adjust a Teflon stopcock so it turns freely without leaking.

No. 5 _____ Adjust the apparatus by securing the buret stand shaft, by positioning the buret holder or clamp, and by securing the buret column with holder or clamp so the column does not crack.

FOR FURTHER STUDY

If you could not perform one or more of the five items above, review and practice the following lesson steps.

No. 1

Lesson Two, Steps 3 to 8

No. 2

Lesson Two, Step 5

No. 3

Lesson One, Step 3; Lesson Two, Step 9

No. 4

Lesson One, Step 4; Lesson Two, Step 10

No. 5

Lesson One, Steps 1 and 2; Lesson Two, Steps 1 and 2

DOING A TITRATION TO STANDARDIZE THE TITRANT

No. 1 _____ Add titrant to the buret column and adjust the fluid level by reading the meniscus to within 0.05 ml of the actual fluid level.

No. 2 _____ Prepare a sample containing a known amount of standard potassium biniodate titrant, using a volumetric pipet for adding the standard and graduated pipets for adding reagents.

No. 3 _____ Add indicator solution only after titrating the sample to a pale yellow color.

No. 4 _____ Add only enough titrant to produce a permanent color change from blue to colorless.

No. 5 ____ Read the meniscus to within 0.05 ml of the actual fluid level.

No. 6 ____ Calculate the normality of the sodium thiosulfate titrant to within 0.0004 N of the standardized value.

FOR FURTHER STUDY

If you could not perform one or more of the six items above, review and practice the following lesson steps.

No. 1

Lesson Three, Steps 1-4; Lesson Two

No. 2

Lesson Three, Steps 5, 6, and 7

No. 3

Lesson Three, Steps 8 and 9

No. 4

Lesson Three, Step 9

No. 5

Lesson Three, Steps 3 and 10

No. 6

Lesson Three, Steps 11 and 12

REFERENCES

U.S. Army, Field Manual, FM 8-91S1/2, Soldier's Manual, MOS 91S, Environmental Health Specialist, Washington, DC, August 1977.

U.S. EPA, Learner's Guide, "Water Quality Monitoring--An Instructional Guide for the Two-Year Quality Monitoring Curriculum," Washington, DC, 1978.

Standard Methods for the Examination of Water and Wastewater, 14th ed., American Public Health Association, 1975, pp. 443-47.