TECHNICAL REPORT NO. LWL-CR-03B69

IMPROVED WATER ANALYSIS KIT

Final Report

By

William H. Collins
The Franklin Institute Research Laboratories
Philadelphia, Pennsylvania 19103

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U. S. ARMY LAND WARFARE LABORATORY
Aberdeen Proving Ground, Maryland 21005

AD-756952

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ABSTRACT

An improved Water Test Kit which is smaller in size and simpler to use and stock than Water Quality Control Set, FSN 6630-262-7288, Standard B, has been developed under Work Assignments No. 2 and 3 of Contract DAAD05-72-C-0113 by The Franklin Institute Research Laboratories for the U. S. Army Land Warfare Laboratory.

The kit has a minimum of glassware, weighs 4 1/2 pounds, measures 9 x 6 x 6 inches, and uses techniques completely different than former methods. Eliminated is the need of preparing reagents, performing titrations and other tedious measurements. Most of the tests are conducted by dipping a paper or plastic strip into the water sample and reading the height of a column or comparing the color obtained to a chart. No complicated calculations are required.

Water samples can be examined for pH, acidity, alkalinity, chlorine residual, chlorine demand, chloride, sulfate, turbidity and coagulation characteristics. Each kit contains enough material to examine at least 50 water samples for each of the above characteristics before requiring refill. The simplicity of the kit allows personnel with little or no training to use it effectively.

FOREWORD

This report is submitted in compliance with contractual requirements as directed by the U. S. Army Land Warfare Laboratory, Aberdeen Proving Ground, Maryland, under Contract No. DAAD05-72-C-0113. Mr. Harold H. Rosen, Biological Sciences Branch, served as Technical Supervisor for the work, and we would like to acknowledge his insights and assistance during the project.

Principal investigator for the program at the Franklin Institute Research Laboratories was Mr. William H. Collins, Manager of the Multidisciplinary Projects Laboratory. Other FIRL personnel contributing significantly to the program were Dr. Peter Mitchell, Senior Staff Scientist and Mr. Francis Sweeney, Research Scientist of the Materials & Physical Sciences Department.

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I. INTRODUCTION

The purpose of Work Assignments Nos. 2 and 3 was to develop an improved water test kit for field use which is relatively simple, easy to use, rugged under field conditions, small, moderate in cost and possesses a long shelf life. Current water analysis kits for field use have the disadvantage that chemicals deteriorate rapidly requiring that fresh solutions be made up in the field. In addition, breakage of laboratory apparatus included in the kit is a problem and under the environments encountered in SEA, electronic gear such as photometers, rapidly become inoperable.

The scope of work for Work Assignments Nos. 2 and 3 specified that the simple water analysis kit should be capable of performing the following tests:

- Hardness
- Alkalinity
- Chloride
- Sulfate
- Residual Chlorine
- pH
- Turbidity
- Flocculation Test
- Chlorine Demand
- Acidity

II. CONCLUSIONS

A. An improved water analysis kit was designed to analyze water for the following constituents:

. Hardness	. Chlorine Demand
. pH	. Chlorine Residual
. Acidity	. Sulfate
. Alkalinity	. Flocculation
. Chloride	. Turbidity

B. The improved water analysis kit weighs less than five pounds; measures 9 x 6 x 6 inches; uses simplified techniques; and eliminates the need of preparing reagents, performing titrations and other tedious measurements.

C. The improved water analysis kit makes extensive use of test papers and prepelletized reagents to determine concentration of impurities. No complicated calculations are required.

D. Each kit contains sufficient material to examine at least 50 water samples for each of the above characteristics before requiring refill.

E. The simplicity of the kit allows personnel with little or no prior training to use the kit effectively.

III. TEST KIT DESIGN

A. Approach

The approach to the design of the new test kit was based on the observation that accuracies of one part per million were not actually required in the field and that, in fact, establishing a range covering 25 or 50 ppm for any given quantity except residual chlorine would be sufficient. The other guiding criterium was that simple test strips and papers were to be used wherever possible in testing the water sample. For tests where a simple test strip was not feasible, it was decided to develop a method of testing that would involve a minimum of manipulation by relatively untrained personnel. Other design goals included (1) elimination of all electronic apparatus, the need for preparing reagents, performing titrations and other tedious measurements; (2) fabrication of apparatus which would have a long service life; (3) a simple instruction manual with calculations held to an absolute minimum, and (4) design of a functional kit featuring minimum weight and size along with other standard characteristics including ruggedness and environmental compatability. Figure 1 is a photograph of the kit.

B. Specific Tests

1. Hardness

The ions which cause hardness in water are calcium and magnesium. Both of these ions form insoluble soaps which gave the basis for soap solution titration to determine hardness. The soap titration requires well standardized solutions, considerable glassware, and the test should be performed by an experienced operator.

More recently it has been found that certain dyes such as Eriochrome Black T form colored complexes with calcium and magnesium. Thus, in the presence of these ions, the normally blue dye solution is pink. The sodium salt of ethylene diamine tetra acetic acid (EDTA) has the capability of removing the ions from the complex quantitatively by forming a more stable colorless complex and when all of the complexed ions are released from the dye, the blue color of the dye is restored. If several tubes of water to be tested each receives a different number of drops of EDTA solution and then Eriochrome

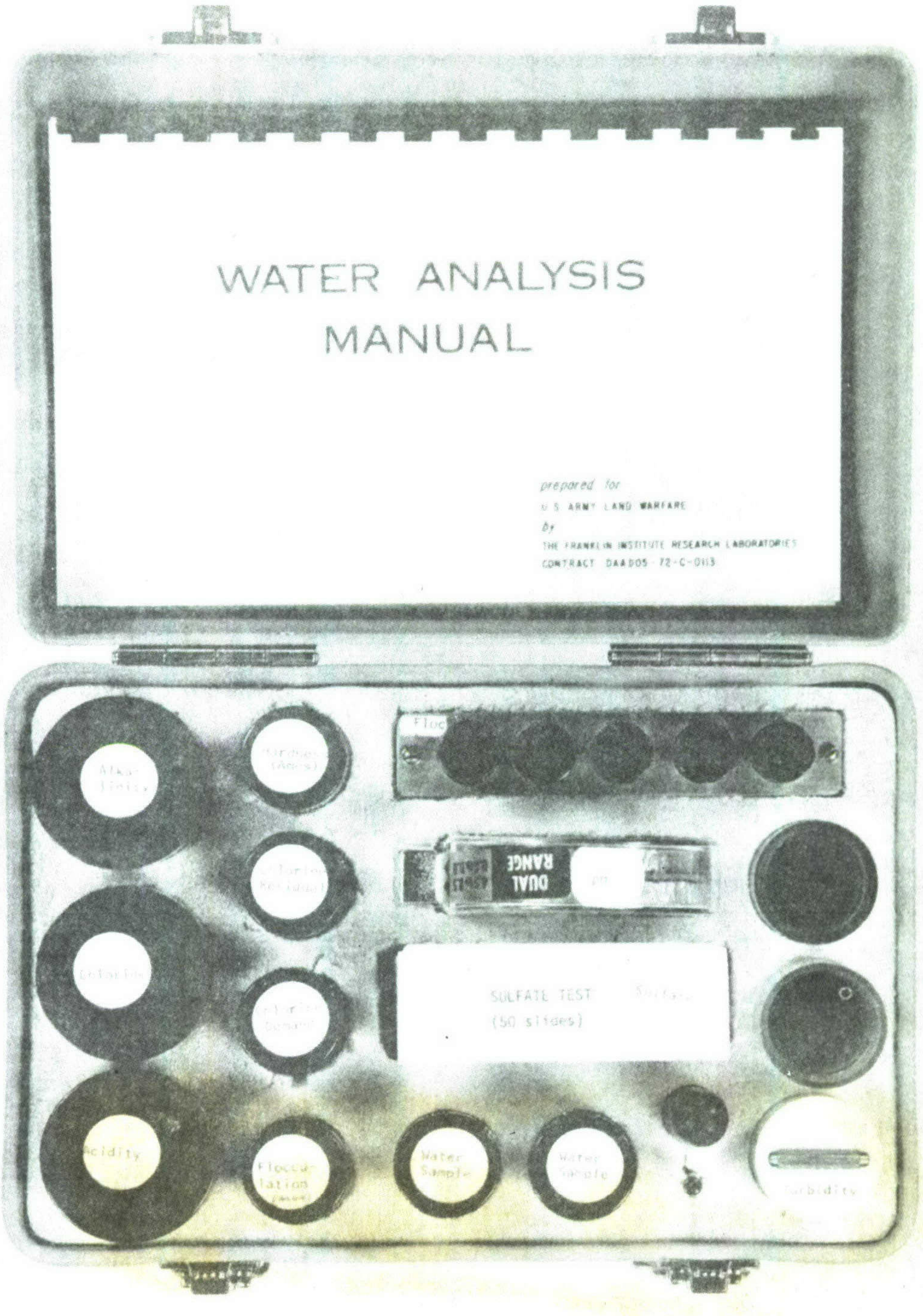


FIGURE 1. IMPROVED WATER ANALYSIS KIT

Black T is added, the blue dye remains blue in those tubes where sufficient EDTA is present to complex all of the calcium and magnesium, while the dye turns pink where EDTA concentrations are not sufficient and free calcium and magnesium are present.

When FIRL first approached the problem, no commercial product was available and so FIRL developed a test paper. The FIRL method consisted of impregnating standard chromatography paper with a 5% solution of Eriochrome Black T in water-ethanol (1:1). The strip is immersed into the test solution up to a prescribed mark. The chromatograph is allowed to develop by capillary action until the liquid interface reaches the top of the paper strip. The height of the color change is then measured against a comparator chart and ppm hardness determined.

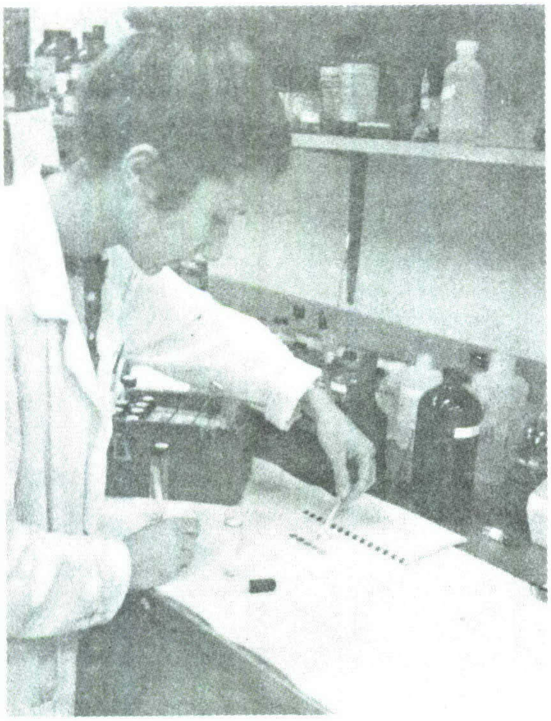
Two commercial test strips for the Hardness Test later appeared on the market and were evaluated. Both the Ames Company and the Macherey-Nagel Company manufacture a water hardness test strip. The Ames test strip required that the end of the strip be immersed in the water sample for fifteen seconds and after removal, the color of the strip is compared to a color chart to determine the hardness in parts per million. The range of the Ames test strip is 0 to 425 ppm as $CaCO_3$.

The Macherey-Nagel Company markets their test paper under the trade name "Aquadur." With this test strip, the hardness range is 0 to 450 ppm as $CaCO_3$. This strip is made in Germany and distributed in the United States by Gallard-Schlesinger Chemical Manufacturing Corporation.

Although the FIRL test strip was at least as accurate as the commercial products, it was decided to use the commercial products rather than expend additional funds on refinement of the FIRL strip. Of the two commercial test strips, the Ames strip was selected for the kit for several reasons including (a) American made and therefore more readily available, (b) more distinctive color change, and (c) easier to use. Figure 2 contains a photograph of the test and the instructions.

2. Chloride Test

A review of the literature indicated that the "Quantab" chloride test strips manufactured by the Ames Company of Elkhart, Indiana, might meet



1. Dip strip in water sample and remove immediately.
2. Wait 15 seconds.
3. Compare color of strip to color chart below.

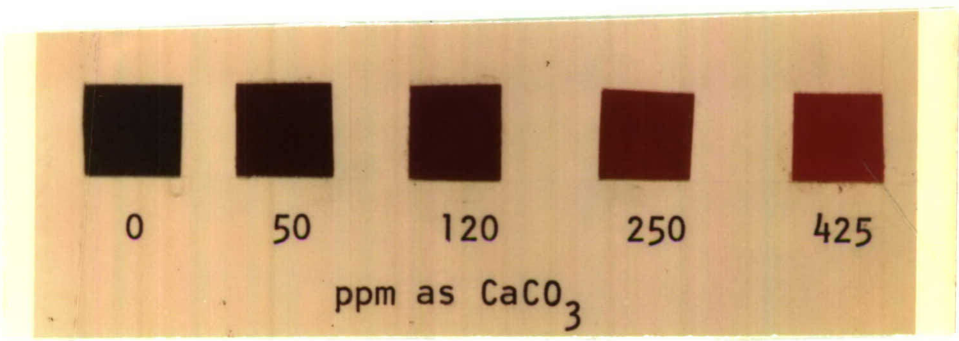


FIGURE 2. HARDNESS TEST PROCEDURES

the requirements. In the Quantab test strip, a column filled with silver chromate is enclosed in plastic. The tip of the column is dipped into the water containing chloride, and the water by capillary action will rise to the top of the column. The chloride will react as the water rises to form silver chloride. The height of white chloride column is a measure of the chloride content of the sample.

Ames makes the Quantab test strips in three different ranges. The range selected (Ames No. 1260) for the LWL kit measures chloride content from 30 to 1500 parts per million. The simple instructions for the chloride test strip is shown in Figure 3.

3. Alkalinity

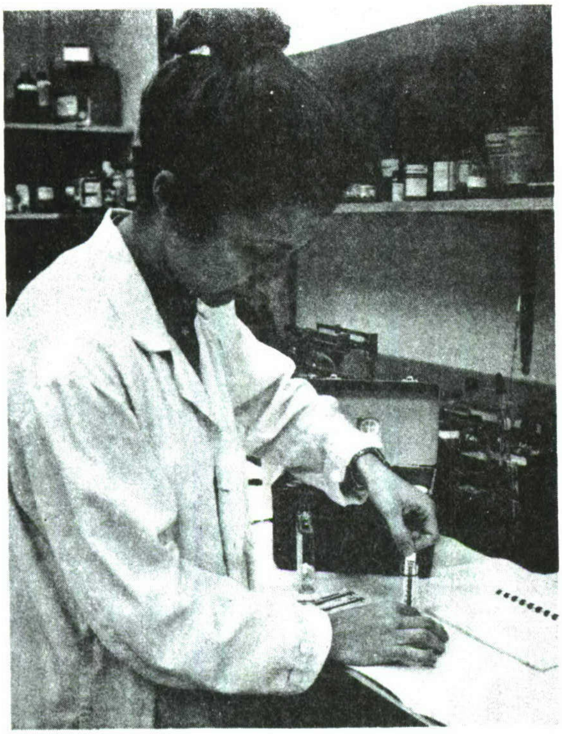
In the standard test kit, alkalinity is determined by titration to a given pH with a solution of a strong acid. The titration to a phenolphthalein end point (pH 9) and a methyl orange end point (pH 4) gives total carbonate, bicarbonate and hydroxide alkalinities. FIRL approached the problem from two directions. The first was the use of pelletized reagents and the second was the use of a test paper.

In the pelletized reagent approach, the strong acid solution was replaced by sulfonic acid, a strong acid which is in the form of powder. Suitably cut with soluble but inert material, this acid can be tableted, with each tablet being equivalent to a desired parts per million alkalinity. The "titration" then consists of adding tablets until the indicators change color.

A suitable test paper for alkalinity was secured from the Ames Company. Ames markets three different range test papers and the No. 1160 was found to fall into the desired range. It was decided that the Ames test strip was the more suitable approach and it was incorporated into the water test kit. Figure 4 is a photograph of the test along with the instructions.

4. pH

The decision with respect to measuring water sample pH was whether to use electronic pH meters, color comparators/indicator solutions or pH test strips. Since the accuracy of the pH test papers is at least ± .25 of a pH unit, it was decided to use the pH test papers.



Scale Reading	ppm
1.0	30
1.5	50
2.0	75
2.5	100
3.0	135
3.5	175
4.0	215
4.5	260
5.0	310
5.5	370
6.0	440
6.5	520
7.0	610
7.5	715
8.0	845
8.5	1020
9.0	1260
9.5	1575

1. Add about 1" of water sample to test vial.
2. Immerse lower end of test strip into water sample.
3. Allow test solution to saturate column (yellow indicator line at top of test strip will turn dark blue) - 12 minutes.
4. Remove strip and wait one minute.
5. Read height of reaction in the test strip to nearest 0.1 division.
6. Compare reading with chart for ppm Chloride.

FIGURE 3. CHLORIDE TEST PROCEDURES

Scale Reading	ppm
0.2	25
0.4	50
0.6	100
0.8	140
1.0	185
1.2	230
1.4	295
1.6	360
1.8	440
2.0	530
2.2	625
2.4	740
2.6	860
2.8	990
3.0	1140
3.2	1370
3.4	1500
3.6	1800
3.8	2000
4.0	2300
4.2	2750
4.4	3050
4.6	3550
4.8	4150
5.0	4850
5.2	5600
5.4	6500
5.6	7600
5.8	9000

1. Put about 1" of water sample in test vial.
2. Immerse lower end of test strip into water sample.
3. Allow test solution to completely saturate column (approximately 15 minutes). Progression of fluid from bottom to top is readily observed.
4. Remove test strip and wait one minute.
5. Read height of reaction in the test strip to nearest 0.2 division.
6. Compare reading with chart for ppm alkalinity.

FIGURE 4. ALKALINITY TEST PROCEDURE

Dual range Hydrion test papers, which are distributed by the Arthur H. Thomas Company, were selected for the kit. The dual range test papers consisted of one roll of wide range (pH 0 to 11.0) paper and one roll of short range (pH 4.5 to 8.5) paper. In operation the wide range paper is used initially to determine the approximate pH of the water and then the short range paper is used to measure the degree of acidity/aklalinity of the water. Refer to Figure 5.

5. Residual Chlorine

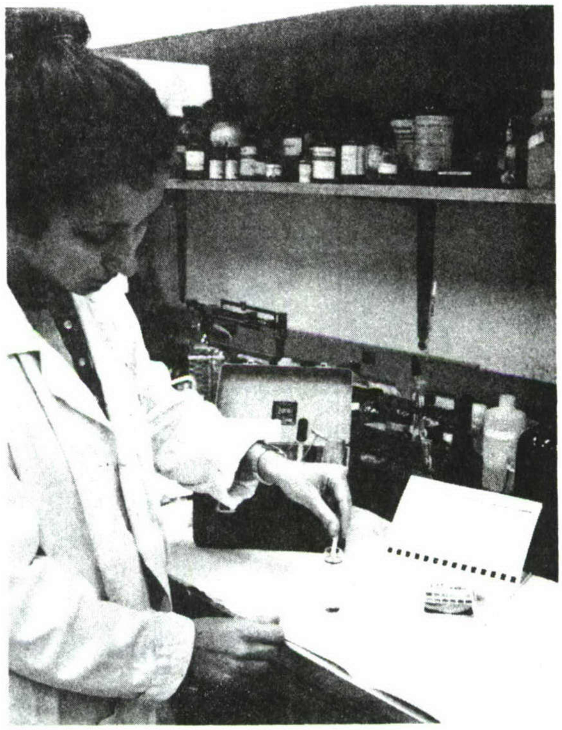
Originally, it was decided to use the standard residual chlorine kit developed by the U. S. Army in the FIRL developed test kit. The standard kit uses an optical comparator based on the *Orthotolidine* method. Next, FIRL developed a test paper which indicated residual chlorine. The test paper used an adaptation of the orthotolidine method.

Later, we discovered that the Ames Company was developing a chloride residual test for use in measuring anti-freeze solution life. After discussing the matter with Ames, they agreed to modify their test strip to measure residual chlorine in the desired range of 0 to 10 parts per million. The modified residual chlorine test strip was incorporated into the Improved Water Analysis Test Kit. See Figure 6 for instructions and photograph.

6. Sulfate

Initially, a pelletized reagent procedure was developed to measure sulfate. The procedure utilized the apparatus developed to measure turbidity (Figure 11) and a pellet containing barium chloride. A predetermined sample of water is placed into the turbidity tube and a pellet of $BaCl_2$ is added and dissolved. A fine precipitate of $BaSO_4$ is formed. By pulling the plunger of the apparatus down through the solution until the black tip disappears from view, an approximate sulfate concentration can be determined. Because of the relative inaccuracy of this method, it was decided to pursue another approach.

After considerable investigation, a technique was developed to measure sulfate concentration which utilized the reaction of a drop of sample water with a $BaCl_2$ impregnated gelatine film on a glass microscope slide. The sulfate ion in a drop of water reacts with the $BaCl_2$ to form $BaSO_4$. The amount



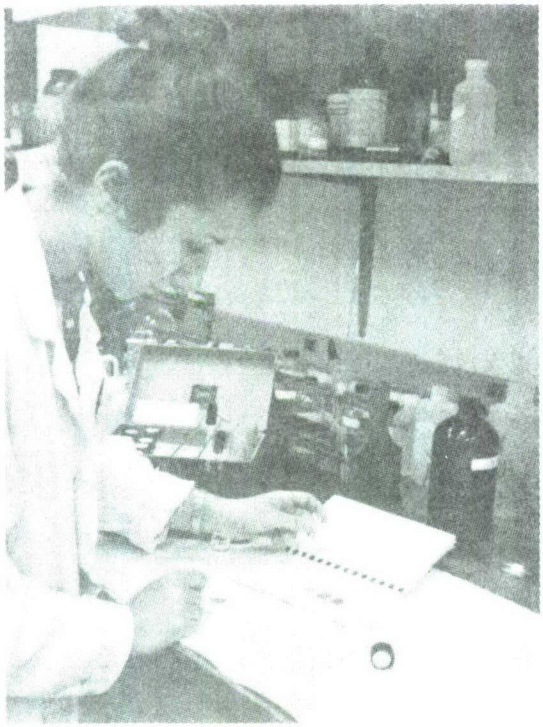
Wide Range

1. Remove approximately 1 1/2 inches of test paper from gold foil covered roll in dispenser.
2. Insert approximately 1 inch of test paper into water sample for 2-3 seconds.
3. Immediately compare color of test paper with color chart on dispenser.
4. Estimate pH within 0.50 of value.

Narrow Range

1. Remove approximately 1 1/2 inches of test paper from silver foil covered roll in dispenser.
2. Insert approximately 1 inch of test paper into water sample for 2-3 seconds.
3. Immediately compare color of test paper with color chart on dispenser.
4. Estimate pH within 0.25 of value.

FIGURE 5. pH TEST PROCEDURES



1. Dip test strip in water sample and swirl for 2-3 seconds.
2. Immediately compare with color chips below to estimate ppm residual chlorine (estimate within 1 ppm).

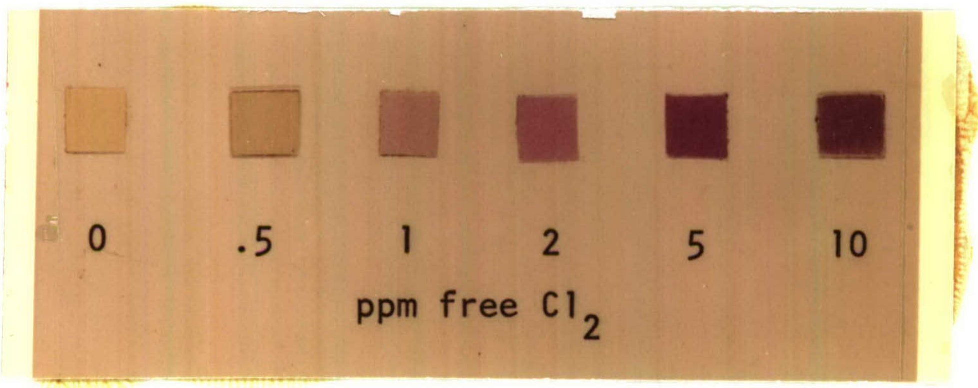


FIGURE 6. CHLORINE RESIDUAL TEST PROCEDURES

of sulfate present determines the opacity of the spot. The spot formed by the unknown water sample is then compared to a series of control standards. The range of control standards include 25 to 1,000 ppm of sulfate. See Figure 7.

7. Chlorine Demand

The chlorine demand test uses buffered pellets of chlorine dry bleach (calcium hypochlorite). Each pellet contains approximately 3 parts per million chlorine when used with a 20 ml sample of water. The pellets are added to the sample until the water indicates an excess of chlorine when measured by the residual chlorine test strips. By multiplying the number of pellets by three and then substrating the excess chlorine reading, the chlorine demand (in ppm) can be readily determined. See Figure 8.

8. Acidity

The Ames Company manufactures an acidity test strip which is suitable for both strong and weak acids. This test strip was incorporated into the kit. See Figure 9 for photograph and instructions.

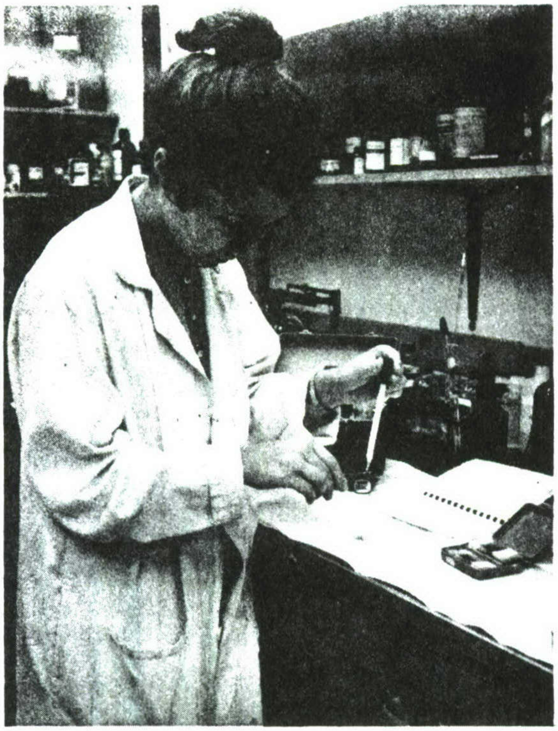
9. Flocculation Test

A miniaturized flocculation test was developed which utilized prepelletized reagents and a small test rack. Two different types of pellets were prepared--an alum pellet and an iron chloride pellet. Each pellet averaged 55 milligrams and when dissolved in 20 ml of water, yielded 2 grains of alum and ferric chloride respectively. The procedure is described in Figure 10 along with a photograph.

Formation of the pellets is as follows:

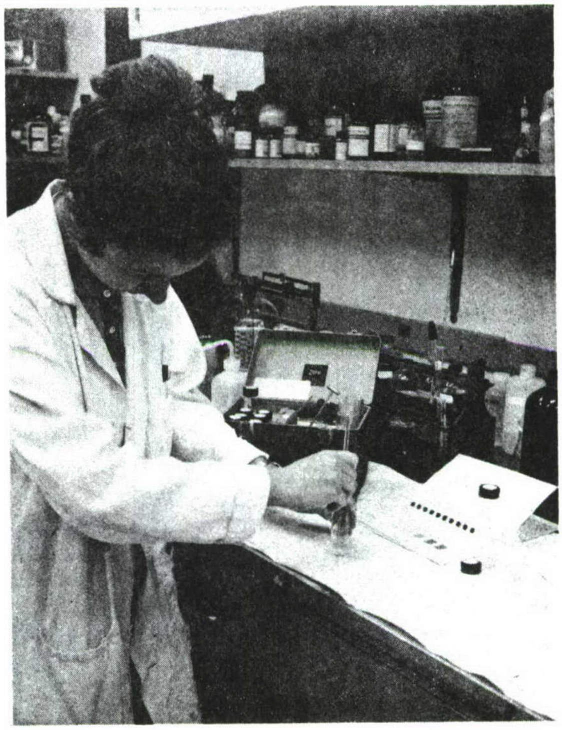
1. Alum Pellets
   - 1500 g Dextrose
   - 10 g Alum
   - 9 g Na_2CO_3
   - 1½g Zinc Stearate

2. Iron Chloride Pellets
   - 1500 g Dextrose
   - 18 g $FeCl_3$
   - 9 g Na_2CO_3
   - 1½g Zinc Stearate



1. Remove a slide from box and place on level surface.
2. Place one drop of water sample on the treated side of the slide.
3. After five minutes, blot excess water off slide.
4. Compare opacity of test spot with the comparative spots on the control slide.
5. Select matching opacity. If test spot is between two of the comparative spots, estimate the approximate ppm. For example, a test spot between 50 and 250 can be estimated at 150 ppm.

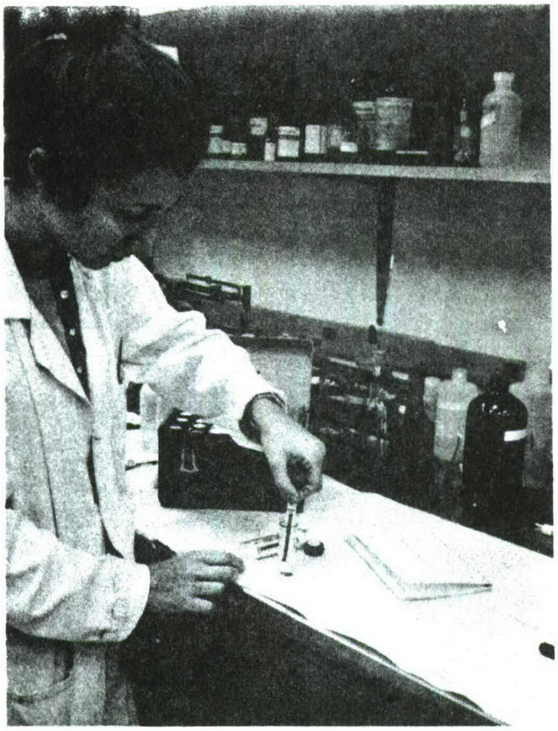
FIGURE 7. SULFATE TEST PROCEDURES



1. Place 2 chlorine demand pellets in glass vial.
2. Add 20 ml of water sample (to black line).
3. Crush pellets with glass rod and shake until pellets dissolve.
4. Allow vial to set for 15 minutes and then test with "Chlorine Residual" test strip. (See Chlorine Residual test.)
5. Subtract the reading from 6 to determine Chlorine Demand in ppm (see example).
6. If no color is formed in (4), repeat test using 4 pellets and subtract result from 12 to calculate chlorine demand.

Example: If 2 pellets resulted in a reading of 4 ppm on the Residual Chlorine Test paper, subtract '4' from '6' and the result '2' is the chlorine demand in ppm.

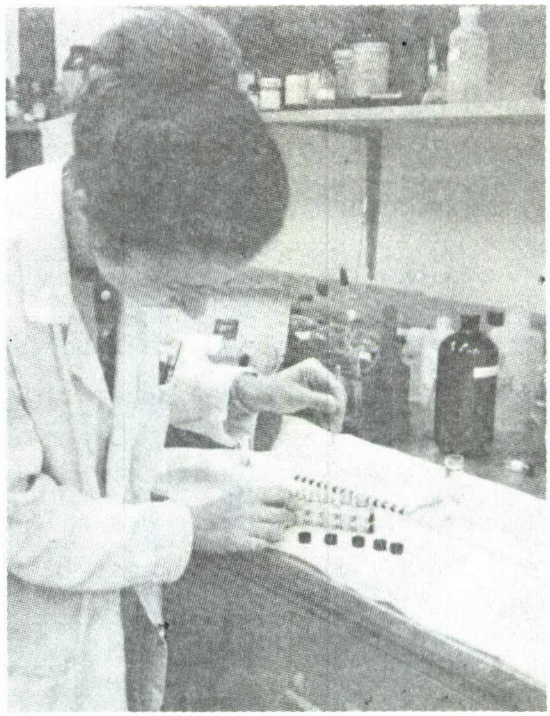
FIGURE 8. CHLORINE DEMAND TEST PROCEDURES



Scale Reading	% HCl
0.6	0.10
0.8	0.18
1.0	0.25
2.0	0.60
3.0	1.00
4.0	1.80
5.0	2.50
6.0	4.00
7.0	6.00
8.0	9.00

1. Put about 1" of water sample in test vial.
2. Immerse lower end of test strip into water sample.
3. Allow test solution to completely saturate column (approximately 15 minutes). Progression of fluid from bottom to top is readily observed.
4. Remove test strip and wait one minute.
5. Read height of reaction in the test strip to nearest 0.2 division.
6. Compare reading with chart for acidity in %.

FIGURE 9. ACIDITY TEST PROCEDURE



1. Remove caps from the 5 small test vials.
2. Place 1 Floc pellet in first tube; 2 Floc pellets in second tube; 3 in third, etc.
3. Crush pellets with glass rod.
4. Fill vials with water sample to black line.
5. Cap and shake all tubes at once until pellets are completely dissolved (approximately 2 minutes).
6. Allow tubes to settle for 15 minutes.
7. Select tube with best floc (coarsest precipitate).
8. Multiply by 5, the number of pellets used to find dosage in ppm.

FIGURE 10. FLOCCULATION TEST PROCEDURES

10. Turbidity

A special piece of apparatus was developed to measure turbidity. It consists basically of a one-inch diameter clear plastic tube fitted with an internal sliding (1/8" dia.) plunger. The sample of water is added to the tube and the plunger is lowered until the black end of the plunger cannot be seen due to the turbidity of the water. The plastic tube is calibrated in parts per million.

The apparatus is taken apart for storage in the kit box. The plastic tube disassembles into three sections and the brass plunger unscrews into two parts. Figure 11 is a photo of the apparatus and operation directions.

C. Special Apparatus

Two special pieces of apparatus were designed and fabricated for the water analysis test kit. The first was a collapsible turbidity measuring device and the second was a small test tube rack. Drawings of each of the pieces of equipment are included in Appendix A.

D. Kit Hardware and Assembly

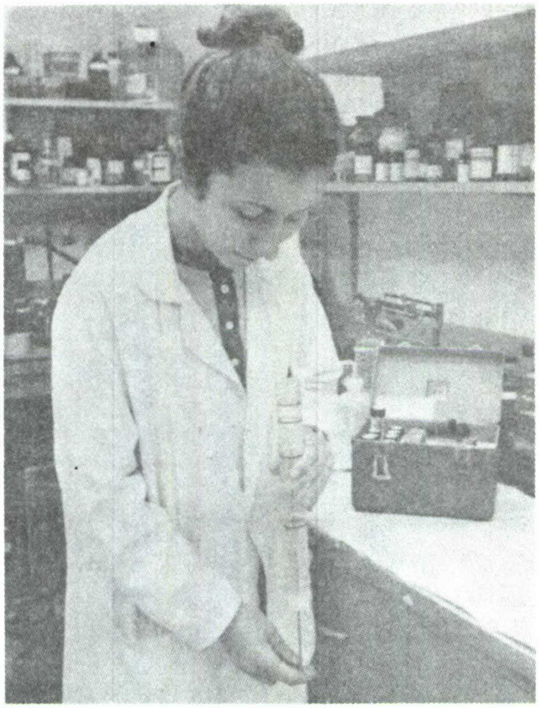
The Improved Water Analysis Kit is shown in Figure 1. In addition to the special apparatus, the following standard apparatus was included:

2 Stirring Rods

2 Eye Droppers

2 Sample Vials

The container was secured from Zero Manufacturing Corporation and is a standard item. To hold the components securely and to minimize the possibility of component breakage, a foam plastic insert was fabricated to hold the individual reagents, hardware and special apparatus. Figure 12 is a photograph of the container.



1. Assemble test fixture.
2. Add water sample up to Fill Mark.
3. Slowly pull rod through water sample until black top of rod is no longer visible.
4. Pour out water sample - do not disturb rod position.
5. Observe height of rod in relation to turbidity readings on side of plastic apparatus.
6. Estimate turbidity to nearest "hundred parts per million."

FIGURE 11. TURBIDITY TEST PROCEDURES

FIGURE 12. PHOTOGRAPH OF KIT

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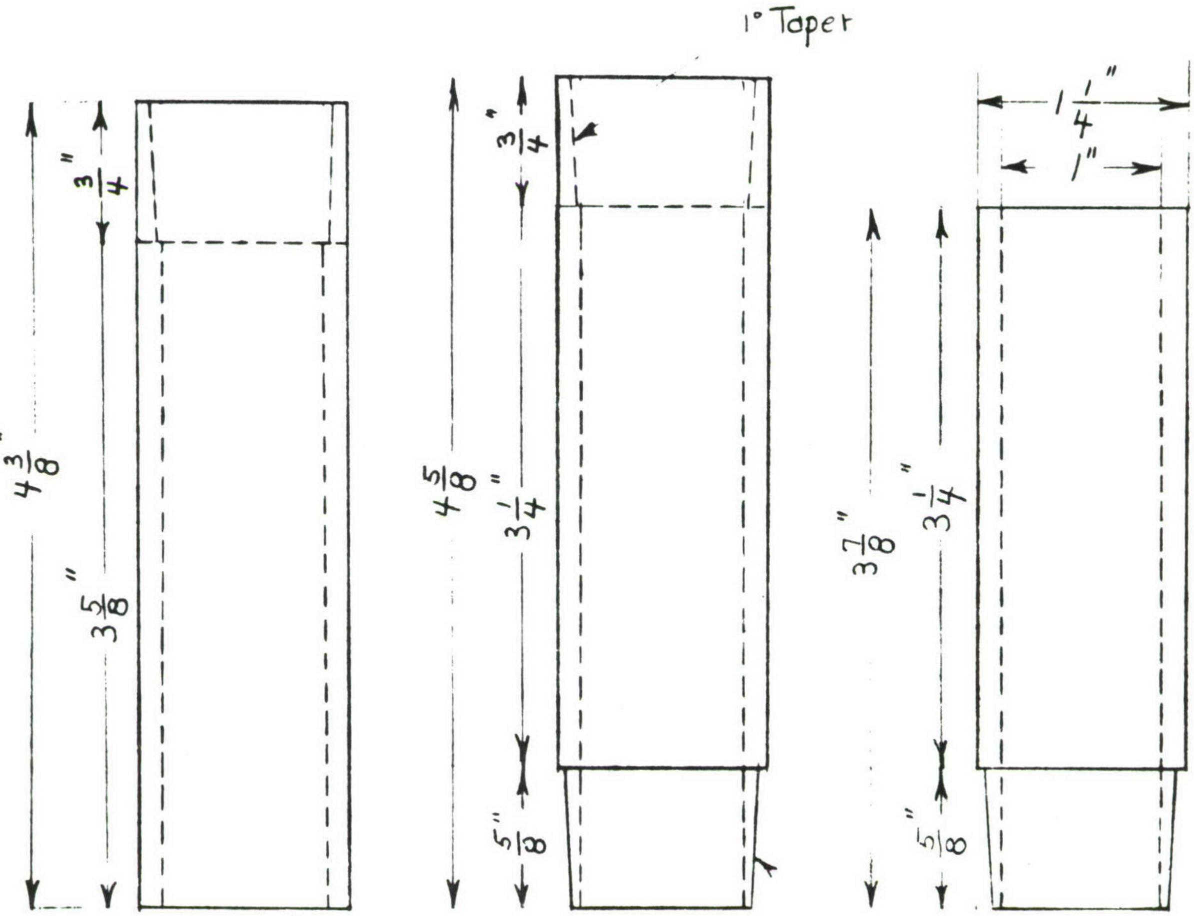
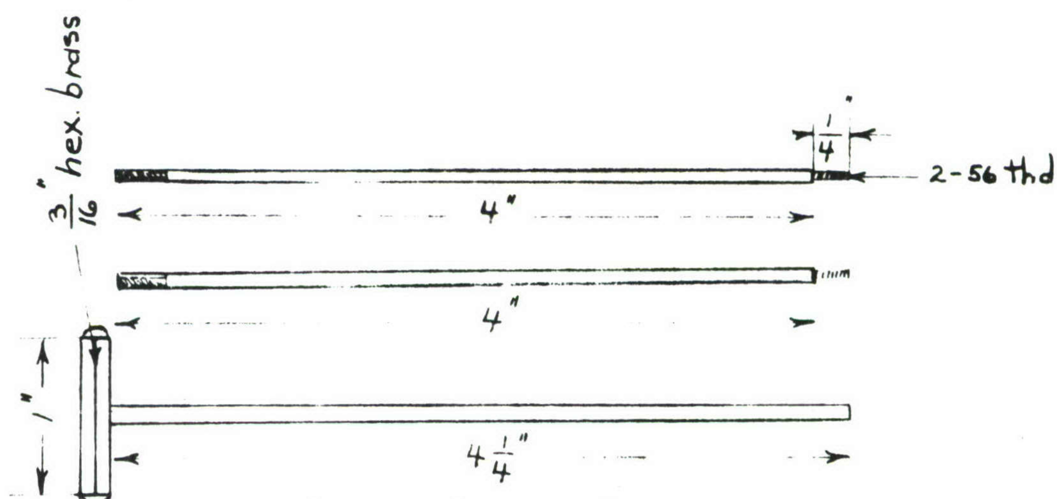
TITLE
Turbidity Apparatus

APPENDIX A



Mat: 1" I.D. × 1¼" O.D. Plexiglass 1° Taper



Mat: Stainless Steel rod.

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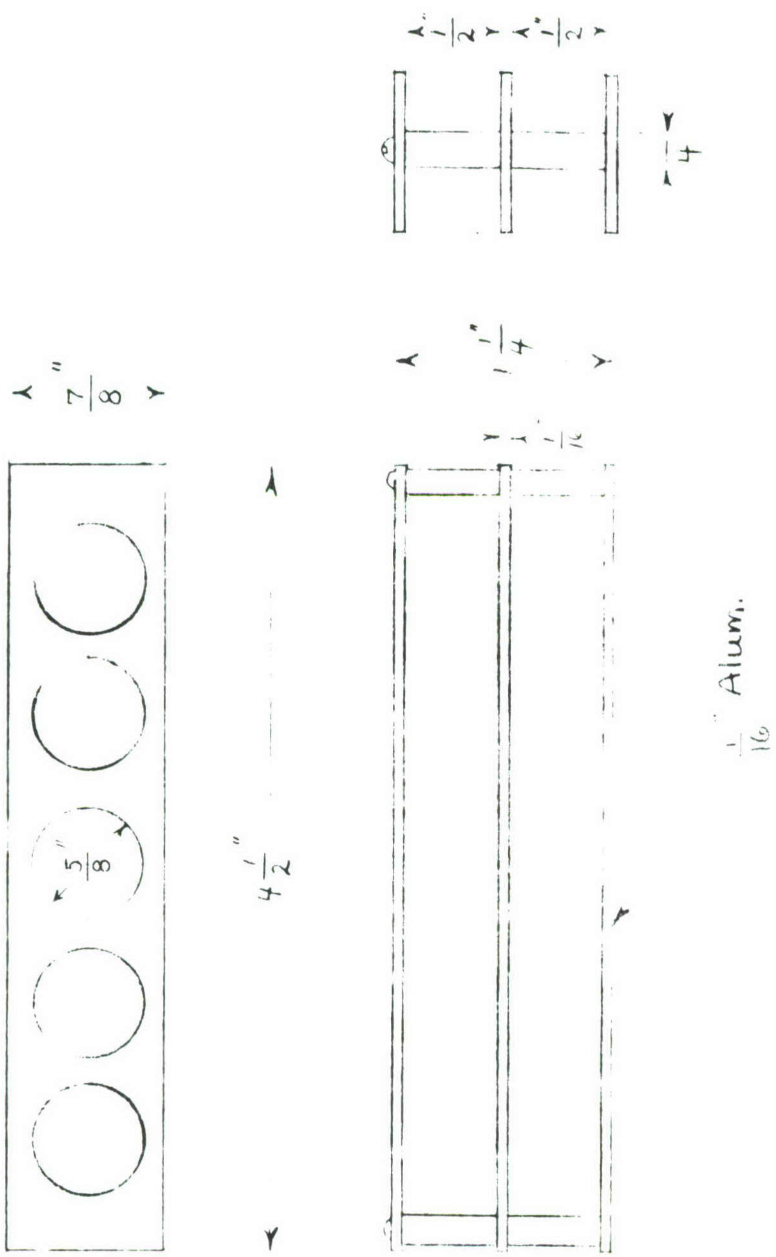
TITLE Minature Rack

APPENDIX A



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UNCLASSIFIED

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)	2a. REPORT SECURITY CLASSIFICATION
Franklin Institute Research Laboratories Benjamin Franklin Parkway Philadelphia, Pennsylvania 19103	Unclassified
	2b. GROUP

3. REPORT TITLE

Improved Water Analysis Kit

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Final Report

5. AUTHOR(S) (First name, middle initial, last name)

William H. Collins

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
December 1972	27	0

8a. CONTRACT OR GRANT NO.	9a. ORIGINATOR'S REPORT NUMBER(S)
DAAD05-72-C-0113	F3222-02/03
b. PROJECT NO. LWL Task 03-B-69	
c.	9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)
d.	

10. DISTRIBUTION STATEMENT

Approved for public release; distribution unlimited.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
	U. S. Army Land Warfare Laboratory Aberdeen Proving Ground, Md. 21005

13. ABSTRACT

An Improved Water Test Kit which is smaller in size and simpler to use and stock than Water Quality Control Set, FSN 6630-262-7288, Standard B, has been developed under Work Assignments No. 2 and 3 of Contract DAAD05-72-C-0113 by The Franklin Institute Research Laboratories for the U. S. Army Land Warfare Laboratory.

The kit has a minimum of glassware, weighs 4 1/2 pounds, measures 9 x 6 x 6 inches and uses techniques completely different than former methods. Eliminated is the need of preparing reagents, performing titrations and other tedious measurements. Most of the tests are conducted by dipping a paper or plastic strip into the water sample and reading the height of a column or comparing the color obtained to a chart. No complicated calculations are required.

Water samples can be examined for pH, acidity, alkalinity, chlorine residual, chlorine demand, chloride, sulfate, turbidity and coagulation characteristics. Each kit contains enough material to examine at least 50 water samples for each of the above characteristics before requiring refill. The simplicity of the kit allows personnel with little or no training to use it effectively.

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Water Analysis Kit						
Water Quality Determination						
pH						
Acidity Test						
Alkalinity Test						
Chlorine Residual Test						
Sulfate Test						
Chlorine Demand Test						
Acidity Test						
Flocculation Test						
Turbidity Test						