This two-part instructor's guide was designed for a five-day course for chemists and technicians with little or no experience in inorganic analyses. Part I provides information on course planning and management including course description, staff responsibilities, suggested course plan and agenda, timeline for planning and conducting the course, equipment list, description of nature, and sources and availability of instructional resources. Part II contains instructional package worksheets which provide a perspective of each analytical procedure, the learning achievement levels the students should attain, available audiovisual and other instructional resources, and an example course of action in pre-course preparation and classroom/laboratory instruction. Topics included within the course are: acidity, alkalinity, hardness, chlorine, total phosphorus, fluoride, nitrate and nitrite nitrogen, total and suspended solids, turbidity and specific conductance, sample handling, compliance methodology, accuracy, precision and error of data, laboratory safety practices, and elements of quality assurance programs. (Author/DC)
Inorganic Analyses in Water Quality Control Programs

Instructor's Guide
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Introduction

This is one of several courses offered by the National Training and Operational Technology Center (NTOTC) which have been prepared in packaged form for use by the States in their training programs, and by other organizations having a need to present this type of training. Each course package consists of:

1. An instructor's guide containing material related to course planning and conduct,
2. A training manual, for use by the participants, containing the course subject matter and
3. Supportive visual and audiovisual training aids used by NTOTC in presenting the course. The training aids are available on loan to those offering the course. The content of each slide is reproduced in this manual.

This guide reflects the manner in which the course has been offered by NTOTC. It is intended to assist the organization offering the training, and should not be considered as an inflexible method of presenting the course. Some may want to follow the format exactly as presented; others may not. In either case, this guide should prove helpful in reducing the amount of original developmental work required, and in suggesting methods and approaches when modifications of the course plan presented herein are being considered.

Organization of the Guide

This instructor's guide consists of two major parts. Part I contains information required for course planning and management. Part II consists of a series of Instructional Package Worksheets (IPW's) which set forth learning objectives and the instructional approach used by NTOTC for each topic included in the training manual.

Analytical Methodology for Regulatory Programs

It is essential that analytical procedures taught in the course conform to those prescribed for use in USEPA regulatory programs. These procedures are identified in regulations which appear first in the Federal Register, and which are later codified in Title 40 of the annual edition of the Code of Federal Regulations.

For the National Pollutant Discharge Elimination System (NPDES), Part 136 of Title 40, "Guidelines Establishing Test Procedures for the Analysis of Pollutants", specifies methods to be used for the measurement of contaminants in wastewater.

For drinking water, Part 141 of Title 40, "National interim Primary Drinking Water Regulations" (NIPDWR), specifies methods to be used in determining the level of contaminants in finished water.
The Instructional Package Worksheet on Compliance Methodology in Part II of this guide provides details concerning EPA regulations for these programs.

For additional information concerning this course, or other packaged courses, contact:

Director, NTOTC
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268
PART I COURSE PLANNING AND MANAGEMENT

This section of the manual is concerned with the administrative aspects of planning, preparing, and conducting the course.

A. COURSE DESCRIPTION

This description was prepared for course presentation at the NTOTC and may be useful to others in course-related publicity.

INORGANIC ANALYSES IN WATER QUALITY CONTROL PROGRAMS
5 Days

This course is for chemists and technicians with little or no experience in inorganic analyses commonly required for the National Pollutant Discharge Elimination System (NPDES), the National Interim Primary Drinking Water Regulations (NIPDR), and for other water quality programs. Applicants should have one year of college level inorganic chemistry and one semester of college level quantitative analysis (or equivalent). They should have basic laboratory skills including use of analytical balances, volumetric glassware and titration assemblies. They should be actively engaged in a water quality control program.

After successfully completing the course, the student will know the general classes of methods listed as approved in the Federal Register for analysis of inorganic pollutants and will be able to use the methods to measure each parameter selected for the course. He will also know how to apply quality assurance techniques to his work and how to validate his analytical accuracy and precision.

The training is a five-day course which includes classroom instruction, student performance of laboratory procedures, and discussion of each laboratory assignment and reported results.

The student will perform the test procedures for acidity and alkalinity (titrations to pH end points), hardness (EDTA titration), chlorine (amperometric back titration), total phosphorus (persulfate digestion, ascorbic acid reduction, colorimetry), fluoride (SPADNS colorimetry and ion electrode), nitrate and nitrite (cadmium reduction, colorimetry), total and suspended solids (gravimetric), turbidity (nephelometer), and specific conductance (Wheatstone bridge conductivity meter). Other topics are sample handling; compliance methodology; accuracy, precision and error of data; elements of quality assurance programs.
B. PERSONNEL

Some of the sections in Part I of this manual refer to various personnel associated with this training course when it is presented at the National Training and Operational Technology Center (NTOTC). Their course-related activities are listed below.

1. Training Supervisor -
   Has overall responsibility for the NTOTC training program.

2. Course Coordinator -
   Is responsible for all elements involved in planning and conducting a specific course.

3. Course Secretary -
   Performs all course-related clerical duties.

4. Instructor -
   For assigned topics, is responsible for planning instructional approach, developing instructional materials, and delivering the instruction. During laboratory sessions, another instructor may be designated to assist the (primary) instructor so that participants can be provided as much individualized attention as possible.

5. Laboratory Assistant -
   Assists in the preparation of laboratory reagents, assembles equipment and is available as required during laboratory exercises.
C. SUMMARY PLAN FOR THE COURSE AND COURSE SCHEDULE

A convenient format to use in the early stages of devising a course plan is a day-to-day assignment of time blocks based on estimates by instructors of the training time required for each parameter. (An example is on the next page.) Using available time as a first criterion will allow a variety of possible sequences. One such sequence, which has been successfully used by NTOTC to conduct this course in the past, begins on page 2-3. Examples of other considerations are:

1. If some equipment must be used in more than one test, schedule another topic between the two tests to allow time for the required clean-up.

2. Schedule the topics so each instructor alternates between prime and assistant responsibilities to allow time for preparations which must be done right before training sessions.

3. If one procedure requires skills taught in another procedure, order the presentations accordingly.

Although NTOTC presents all of these topics in a five day course, there is nothing to prevent your choosing individual topics for instruction. The Instructional Package Worksheet for each topic (in Part II of this Guide) represents an independent module. If any other topic is directly related, this will be stated in the "Entry Level Behavior" section (V) of the IPW.

Likewise, you can develop any time schedule convenient to your staff and students, e.g., weekly presentations of individual topics. The time block approach is very useful for planning such a series, and also the IPW section (VIII B) on "Sequencing" which includes a time breakdown for classroom and laboratory activities. The time allotments for each subject are very feasible, providing ample time for instruction in essentials. Thorough preparation by the instructor is imperative to make each instructional minute count.
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COURSE SCHEDULE:

INORGANIC ANALYSES IN WATER QUALITY CONTROL PROGRAMS (100.4)
(Location)
(Date)

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<tr>
<td></td>
<td>Group B - Break</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>- Weigh Total Solids</td>
<td>26</td>
<td>Instructor #1</td>
</tr>
<tr>
<td>9:45 - 10:30</td>
<td>Specific Conductance</td>
<td>16</td>
<td>Instructor #3</td>
</tr>
<tr>
<td>10:30 - 12:15</td>
<td>Laboratory</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10:30 - 11:20</td>
<td>Group A - Turbidity</td>
<td>28</td>
<td>Instructor #1</td>
</tr>
<tr>
<td></td>
<td>Second Weighing of Solids</td>
<td>26, 27</td>
<td>Instructor #1</td>
</tr>
<tr>
<td></td>
<td>Group B - Specific Conductance</td>
<td>29</td>
<td>Instructor #3</td>
</tr>
<tr>
<td>11:25 - 12:15</td>
<td>Group A - Specific Conductance</td>
<td>29</td>
<td>Instructor #3</td>
</tr>
<tr>
<td></td>
<td>Group B - Turbidity</td>
<td>28</td>
<td>Instructor #1</td>
</tr>
<tr>
<td></td>
<td>Set Weighing of Solids</td>
<td>26, 27</td>
<td>Instructor #1</td>
</tr>
<tr>
<td>12:15 - 1:00</td>
<td>Lunch</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1:00 - 1:30</td>
<td>Calculations and Class Discussion: Specific Conductance</td>
<td></td>
<td>Instructor #3</td>
</tr>
<tr>
<td></td>
<td>Solids Data</td>
<td></td>
<td>Instructor #1</td>
</tr>
<tr>
<td></td>
<td>Turbidity Data</td>
<td></td>
<td>Instructor #1</td>
</tr>
<tr>
<td>1:30 - 2:15</td>
<td>Post-Course Evaluation</td>
<td></td>
<td>Course Coordinator</td>
</tr>
<tr>
<td>2:15 - 2:30</td>
<td>Course Closing</td>
<td></td>
<td>Course Coordinator</td>
</tr>
</tbody>
</table>

*Note. For actual course presentation, insert the name of the primary instructor opposite the title of his/her assignment. Assistant instructors are designated by ( ).
D. MILESTONES IN COURSE PLANNING AND PREPARATION

The following pages list major areas of course responsibilities in a chronological order to facilitate orderly and timely accomplishment. The table also serves as an example for assignment of these responsibilities to various staff members. It has been successfully used by NTOTC to conduct this course in the past.

The table headings are job titles associated with the listed tasks. A suggested staff is cited, including a laboratory assistant. It is recognized, however, that staff is often limited and one individual may serve in several of the defined roles. Having this summary according to an ideal situation should facilitate an equitable division of the required tasks among fewer persons.

Before using the milestones table, decisions must be made about the course content. It may be desirable to teach the approved test procedures for parameters that are not included in this package, but that are required locally to meet regulatory requirements. In that case, the table must be changed. Delete items identified for topics you omit and add the items needed for the topics you want to add.
<table>
<thead>
<tr>
<th>5 TO 6 MONTHS BEFORE COURSE</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Determination of the need and decision to have course.</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Designation of Course Director and Course Secretary.</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Review responsibilities.</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Review responsibilities.</td>
<td></td>
<td></td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>Commit classroom and laboratory facilities.</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Develop and release Course Announcement including location, date, general statement of course content and training objectives.</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Prepare all forms and information sheets related to student registration procedures.</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Decide on staff members.</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>4 TO 5 MONTHS BEFORE COURSE</th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Receive, review, act upon Course Applications, continuing until course begins.</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maintain records on deposition of each application, continuing through course.</td>
<td></td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inventory Instructor's Guides. Order needs.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>3 MONTHS BEFORE COURSE</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>------------------------</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Commit all staff members who will participate in Course.</td>
<td>x x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Develop Milestone Checklist for Course.</td>
<td>x x</td>
<td></td>
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</tr>
<tr>
<td>Distribute copies to staff of Milestones, Instructor's Guide, Student Manual and any pertinent training resources.</td>
<td>x x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Review responsibilities.</td>
<td>x x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Assign topics to Primary (P) and Assistant (A) Instructors: Sample Handling</td>
<td>x x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Compliance Methodology</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Laboratory Safety</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Analytical Techniques</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volumetric Analysis</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>pH</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acidity and Alkalinity</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Precision</td>
<td>P P</td>
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<td></td>
<td></td>
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<tr>
<td>Hardness</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Chlorine</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Phosphorus</td>
<td>P P</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Spectrophotometer</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Accuracy</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fluoride</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nitrate and Nitrite</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Solids</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Quality Assurance</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Turbidity</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specific Conductance</td>
<td>P P</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Develop summary plan for course.</td>
<td>x x x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inventory chemicals and laboratory equipment/supplies. List and commit lending sources. Order rest of needs.</td>
<td>x x x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inventory classroom equipment/supplies. List and commit lending sources. Order rest of needs.</td>
<td>x x x</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>(Continued)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Training Supervisor</td>
<td>Course Coordinator</td>
<td>Course Secretary</td>
<td>Instructor #1</td>
<td>Instructor #2</td>
</tr>
<tr>
<td>---------------------</td>
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<td>------------------</td>
<td>---------------</td>
<td>---------------</td>
</tr>
<tr>
<td>Inventory student training manuals. Order needs.</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**2 MONTHS BEFORE COURSE**

- Finalize Course Schedule (Agenda).
  - x
  - x
  - x
  - x
  - x
  - x
- Request laboratory/classroom needs from lending sources.
  - x
  - x
  - x
  - x
  - x
  - x
- Request training aids from lending sources.
  - x
  - x
  - x
  - x
  - x
  - x

**6 WEEKS BEFORE COURSE**

- Check out operation of all major pieces of equipment.
  - x
  - x
  - x
  - x
  - x
  - x

**Primary and Assistant Instructors** go through laboratory procedures in student training manual, using IPWs to standardize instructions for students.

**1 MONTH BEFORE COURSE**

- Summary (to date) to staff of registered students, continuing to course beginning.
  - x
  - x
- Check on progress of staff preparations for instruction, continuing through course.
  - x
  - x
  - x
  - x
  - x
- Prepare all administrative forms and materials needed for course presentation.
  - x
  - x
- Plan and rehearse classroom presentations using all required training aids.
  - x
  - x
  - x
  - x
  - x

(Continued)
Obtain any duplicated instructional materials (data sheets, etc.).

Review summary of laboratory equipment and supply needs for expected number of students doing the selected procedures.

Clean all glassware required by students. Note any special requirements.

Reserve all specially-cleaned glassware or apparatus.

Assemble other student equipment and supplies.

**2 WEEKS BEFORE COURSE**

Arrange for security of classroom and laboratory.

Make reagents required by students EXCEPT those with specified, limited stability.

Make final arrangements to obtain required samples. Plan for any "synthetics."

Determine range of concentration of desired constituent(s) in samples for the course.

Arrange for disposal of special test wastes.

(Continued).
<table>
<thead>
<tr>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Course Secretary</th>
<th>Instructor 1</th>
<th>Instructor 2</th>
<th>Instructor 3</th>
<th>Lab Assistant</th>
</tr>
</thead>
</table>

Give Laboratory Assistant final list of equipment and supplies to be at each laboratory position. Discuss arrangement of shared equipment.

**1 WEEK BEFORE COURSE**

Inform building food service of number of expected students and course lunch times (as appropriate).

**3 DAYS BEFORE COURSE**

Finalize seating arrangement for classroom.

Assemble course materials in classroom (student manuals, administrative materials, etc.). Distribute as appropriate.

Ready classroom instructional aids (boards, erasers, etc.).

Check out all classroom equipment (electrical systems, PA, projection equipment) and obtain back-up accessories (bulbs, etc.).

**COURSE OPENING**

Conduct opening exercises. Participate in course opening.

Complete any required student records, including roster.

(Continued)
<table>
<thead>
<tr>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Course Secretary</th>
<th>Instructor #1</th>
<th>Instructor #2</th>
<th>Instructor #3</th>
<th>Lab Assistant</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prepare course certificates if needed at end of week.</td>
<td>x</td>
<td></td>
<td>x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>EVERY DAY OF COURSE</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maintain general supervision of course.</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Prepare unstable reagents and/or samples on day of test.</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
<td>x</td>
</tr>
<tr>
<td>Obtain samples for each test on day of test.</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>When assistant instructor, make any student evaluation records requested by the lead instructor.</td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>When primary instructor, compile evaluation record for each student.</td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Keep any general records (e.g. attendance) as required to document successful course completion.</td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Oversee disposal of special test wastes.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>x</td>
</tr>
<tr>
<td>SECOND-LAST DAY OF COURSE</td>
<td>Training Supervisor</td>
<td>Course Coordinator</td>
<td>Instructor #1</td>
<td>Instructor #2</td>
<td>Lab Assistant</td>
<td></td>
</tr>
<tr>
<td>---------------------------</td>
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</tr>
<tr>
<td>Distribute course critique sheet to students.</td>
<td>x</td>
<td></td>
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</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>LAST DAY OF COURSE</th>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Instructor #1</th>
<th>Instructor #2</th>
<th>Lab Assistant</th>
</tr>
</thead>
<tbody>
<tr>
<td>Check and sign course certificates if applicable</td>
<td>x</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Collect students' critique sheets.</td>
<td></td>
<td>x</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Conduct closing exercises.</td>
<td></td>
<td></td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Participate in course closing.</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>Clean up classroom and laboratory.</td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>WITHIN THO WEEKS OF COURSE PRESENTATION</th>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Instructor #1</th>
<th>Instructor #2</th>
<th>Lab Assistant</th>
</tr>
</thead>
<tbody>
<tr>
<td>Return or replace any borrowed classroom equipment/supplies.</td>
<td></td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Return or replace any borrowed laboratory equipment/supplies.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>Return or replace any borrowed training aids.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>Order repairs or replacements of own equipment, used in course.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>Complete and file evaluation records on all students in predetermined area.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
<tr>
<td>See that all students have been appropriately notified of their degree of success in completing the course.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
<td></td>
</tr>
<tr>
<td>Task</td>
<td>Trainer</td>
<td>Course Coordinator</td>
<td>Course Secretary</td>
<td>Instructor #1</td>
<td>Instructor #2</td>
</tr>
<tr>
<td>----------------------------------------------------------------------</td>
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<td>------------------</td>
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<td>---------------</td>
</tr>
<tr>
<td>Participate in staff session on evaluation of course and recommendations for future offerings. Complete and file entire course records in mutually determined area. Prepare course summary/evaluation report.</td>
<td>x</td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>
E. INSTRUCTIONAL RESOURCES

Most training institutions will make the fullest possible use of available instructional resources. The purpose of this section is to describe the nature, sources and availability of instructional resources suggested for use with this course.

1. Student Training Manual and Instructor's Guide for the course, "Inorganic Analyses in Water Quality Control Programs." Ordering information may be obtained from:

   U.S. Environmental Protection Agency
   National Training and Operational Technology Center
   Cincinnati, Ohio 45228
   (513) 684-7501

2. Slides, Slide/Tape Units, Overhead Transparencies and Videocassette (See IPW Sections VIII B and XI for a description). According to topic, these instructional training aids are:

   a. Acidity and Alkalinity
      1) X-20: "Alkalinity", 28 assembled slides
   b. Calcium and Magnesium Hardness
      1) X-22: "Hardness", 23 assembled slides
   c. Chlorination and Chlorine Determinations
      1) X-21: "Chlorine", 36 assembled slides
      2) KEH-70: "Amperometric Determination of Total Chlorine in Wastewater"
         It is available for review and purchase from the College, 6301 Kirkwood Blvd., Cedar Rapids, Iowa 52405.
   d. Determinate Error-Accuracy
      1) X-25: "Accuracy", 14 assembled slides
   e. Elements of a Quality Assurance Program
      1) X-26: "Quality Assurance", 5 assembled slides
   f. Fluoride
      1) XT-80: "Fluoride Analytical Procedures", 46 slides, 16 minute tape and script
g. Indeterminate Error-Precision
   1) X-24: "Precision", 20 assembled slides

h. Laboratory Safety Practices
   1) X-27: "Laboratory Safety", 40 assembled slides

i. Nitrate and Nitrite Nitrogen
   1) X-23: "Nitrate and Nitrite Nitrogen", 20 assembled slides

j. pH
   1) OT-11: "pH", 7 overhead transparencies

k. Phosphorus
   1) XT-44: "The Determination of Phosphorus", 53 slides, 14 minute tape and script

l. Sample Handling-Field Through Laboratory
   1) X-17: "Sample Handling", 8 assembled slides

m. Solids
   1) X-28: "Solids", 32 assembled slides

n. Specific Conductance
   1) X-29: "Specific Conductance", 15 assembled slides

o. Turbidity
   1) X-30: "Turbidity", 10 assembled slides

p. Use of a Spectrophotometer and Calibration Graphs
   1) X-8: "Use of a Spectrophotometer", 16 assembled slides
   2) X-9: "Calibration Graphs", 6 assembled slides
   3) XT-51: "Use of the Spectronic 20 Spectrophotometer", 35 slides, 8 minute tape and script

q. Volumetric Analysis
   1) X-18: "Volumetric Analysis", 30 assembled slides
All units described in 2. above are available on scheduled loan from NTOTC to institutions conducting this course. Requests should contain the information items on the "Request for Loan" form at the end of this section. Send requests to the National Training and Operational Technology Center at the address on page 4-1.

It is urged that materials desired from NTOTC for a specific course offering be requested in a single, consolidated communication. This will give greatest assurance of a well-coordinated response. Because these requests ordinarily will cover a number of different items, telephonic requests should not be made.

Requests should be timely. To assure effective delivery in time for use in the course, requests should be received at NTOTC at least 45 days prior to the course date. NTOTC will, in turn, make every effort to assure that the requested materials are delivered to the requesting institution several days prior to the start of the course in which they are to be used. This will permit review and practice by the instructional staff for the most effective use of such resources.

It is expected that all borrowed resources be returned to NTOTC within two weeks after completion of the course in which they are used.

With returned borrowed training resources, it is requested that the user provide NTOTC with an evaluation of the training resource(s) used. In this manner, the experience of users can be a factor in continuous improvements and responses to problems in using the resources. All reports on the use of such resources should include the number of students with whom the material was used.

3. Supportive References

a. Manual: EPA-EMSL, "Methods for Chemical Analysis of Water and Wastes". This is the reference source for many of the methods presented in this course. Address requests for a copy to:

U.S. Environmental Protection Agency
Environmental Monitoring and Support Laboratory (EMSL)
Cincinnati, Ohio 45268


Note: Items 3. a., b., and c. are those currently cited by the Federal Registers for National Pollutant Discharge Elimination System (NPDES) and National Interim Primary Drinking Water Regulations (NIPDWR) (copies are in the Instructional Package Worksheet on Compliance Methodology). Contact your U.S. EPA Regional Quality Assurance Coordinator for pertinent Federal Registers issued after 8/80. (Call U.S. EPA, EMSL, at 513/684-7301, for the name and phone number of your Coordinator).

Catalog: "Water Quality Control Instructional Materials." This includes visual instructional units developed by NTOTC. Although not developed specifically for this course, several of the units are on course topics and might be useful supplementary material. Address requests for a copy to NTOTC at the address given on page 4-1.

4. Instructional Resources Already in Possession of the Training Institution

a) Many training organizations prefer to develop, or have developed, their own texts and audiovisual training resources.
REQUEST FOR LOAN
AUDIOVISUAL INSTRUCTIONAL UNIT

Title and Catalog No. __________________________

Intended Use: __________________________

Preferred Date of Use: __________________________

Alternate Date: __________________________

BORROWER'S NAME __________________________

Title __________________________

Organization __________________________

Address __________________________

(First Initial) __________________________ (Zip)

Phone Number (include Area Code). __________________________

There is no charge for use of the Audiovisual Instructional Units. However, the BORROWER assumes financial responsibility for the value of all loaned equipment and instructional materials.

Unless special arrangements are made with the loaning office, units should be returned within two weeks. Return the unit by REGISTERED, CERTIFIED or INSURED MAIL IMMEDIATELY after use.

EPA-171 (Cm) (8-74)
LABORATORY EQUIPMENT AND SUPPLY REQUIREMENTS

The consolidated list in this section is for overall planning purposes. It was compiled from the Instructional Package Worksheet (IPW) sections (part II of this manual) IX, "IPW Equipment and Supply Requirements" and X, "IPW Reagent Requirements." Sections IX and X can also be used on a day-to-day basis during the course to prepare for the laboratory exercises.

The first column of this consolidated list contains a description of the item as found in commercial catalogs.

The second column identifies the tests utilizing the equipment. At the beginning of the table, there is a key to the abbreviations used in this column.

The third column lists the minimum quantity per laboratory station (no more than two persons) required for each test. In many cases, multiple use of the item is required to carry out the procedure. The quantities are based on the assignment stated in section IX of the related IPW.

For convenience, a list of the assignments for each topic can be found as part of the key for abbreviations at the beginning of the table. If an Instructor chooses a different assignment for a topic, some quantities must be changed accordingly. As noted, numbers represent minimum quantities. It is strongly recommended that Instructors provide surplus equipment and additional supplies ready for use in case of need. Many instructors plan for a margin of at least 10% of extra supplies to provide for student errors, planning miscalculations, or other unforeseen events.

The fourth and final column contains remarks that may be useful when deciding on class needs or when ordering equipment.

Before using the list, decisions must be made about the course content. It may be desirable to teach the approved test procedures for parameters that are not included in this package, but that are required locally to meet regulatory requirements. In that case, the list must be changed. Delete items identified for topics you omit and add the items needed for the topics you want to add.

This list can be of great value in pre-course planning, to determine the availability of needed equipment and supplies, and to take action to provide needed resources. Further, this list can be of vital importance when planning for courses to be conducted in field locations. Copies of the list in the hands of the Course Coordinator and a representative of the host organization can be used to determine who will provide needed resources on an item-by-item basis. When the responsibility is assigned/accepted, this can be annotated in the "remarks" column on the copy in the hands of the Course Coordinator and the copy of the representative of the host organization. Each can then use the annotated equipment and supply list as a checklist for carrying out his own agreed-upon responsibilities in preparing for the course.
Following is the key for abbreviations used for course topics in the table. The second column lists the laboratory assignment for each station as given in the corresponding IPW in Part II of this guide. (There should be no more than two persons at a station.) The quantity given for each laboratory item in the table was calculated according to the IPW assignment. If you change the assignment, adjust the quantities accordingly.

<table>
<thead>
<tr>
<th>ABBREVIATIONS FOR TESTS</th>
<th>IPW ASSIGNMENTS (per station)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A/A Acidity and Alkalinity, Titration</td>
<td>1 sample for acidity 2 samples for alkalinity</td>
</tr>
<tr>
<td>Cl Chlorine, Perometric (Back) Titration</td>
<td>1 sample for two titrations</td>
</tr>
<tr>
<td>F Fluoride, SRADNS and Electrode with Specific Ion Meter</td>
<td>4 samples by each method</td>
</tr>
<tr>
<td>H Hardness, Titration</td>
<td>tap water 1 standard 1 effluent sample (or synthetic)</td>
</tr>
<tr>
<td>N/N Nitrate and/or Nitrite, Cadmium Reduction</td>
<td>Prepare and Activate a column Zero standard plus 1 standard or sample for nitrate. Same for nitrite.</td>
</tr>
<tr>
<td>P Total Phosphorus, Digestion and Reduction</td>
<td>2 standards (zero + 1 other) 1 sample</td>
</tr>
<tr>
<td>S Solids Total and Suspended, Gravimetric</td>
<td>1 influent sample for total solids 1 influent and 1 effluent sample for suspended solids</td>
</tr>
<tr>
<td>SC Specific Conductance, Meter</td>
<td>Determine cell constant for 3 ranges 3 samples</td>
</tr>
<tr>
<td>I Turbidity, Nephelometer</td>
<td>Dilute a stock solution to make 3 or 4 standards 2 samples (tap water + 1 synthetic)</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>TEST(S)</td>
</tr>
<tr>
<td>-------------</td>
<td>---------</td>
</tr>
<tr>
<td>APRONS, laboratory (plastic acceptable)</td>
<td>All</td>
</tr>
<tr>
<td>BALANCES, analytical, 0.1 mg sensitivity at a load of 200 g, with Instruction Manuals</td>
<td>S</td>
</tr>
<tr>
<td>BEAKERS, glass, 50 ml</td>
<td>A/A</td>
</tr>
<tr>
<td>BEAKERS, glass, 100 ml</td>
<td>A/A</td>
</tr>
<tr>
<td>BEAKERS, glass, 150 ml</td>
<td>A/A</td>
</tr>
<tr>
<td></td>
<td>H</td>
</tr>
<tr>
<td></td>
<td>N/N</td>
</tr>
<tr>
<td>BEAKERS, glass, 250 ml</td>
<td>H</td>
</tr>
<tr>
<td></td>
<td>N/N</td>
</tr>
<tr>
<td>BEAKERS, glass, 400 ml</td>
<td>N/N</td>
</tr>
<tr>
<td></td>
<td>SC</td>
</tr>
<tr>
<td>BEAKERS, glass, 600 ml</td>
<td>SC</td>
</tr>
<tr>
<td>BOILING BEADS, glass, 5mm</td>
<td>A/A</td>
</tr>
<tr>
<td></td>
<td>P</td>
</tr>
<tr>
<td>BOTTLES, glass or plastic, about 2.5 liter for Cd rinsings. Used acid bottles serve well.</td>
<td>N/N</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>TEST(S)</td>
</tr>
<tr>
<td>------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>BRUSHES, balance</td>
<td>S</td>
</tr>
<tr>
<td>BURETS, 25 ml, 0.1 ml graduations; teflon</td>
<td>A/A</td>
</tr>
<tr>
<td>stopcock plug preferred</td>
<td>H</td>
</tr>
<tr>
<td>CLAMPS, buret for titration stand; to hold</td>
<td>A/A</td>
</tr>
<tr>
<td>two BURETS</td>
<td>H</td>
</tr>
<tr>
<td>CLAMPS, screw to regulate flow through 4</td>
<td>N/N</td>
</tr>
<tr>
<td>cm ID rubber TUBING</td>
<td>H/N</td>
</tr>
<tr>
<td>CLAMP, pinchcock for 4 cm ID rubber TUBING</td>
<td>N/N</td>
</tr>
<tr>
<td>CLAMP, screw to regulate flow through 4</td>
<td>N/N</td>
</tr>
<tr>
<td>cm ID rubber TUBING</td>
<td>H/N</td>
</tr>
<tr>
<td>CONDUCTIVITY METERS, Wheatstone bridge with</td>
<td>SC</td>
</tr>
<tr>
<td>cell(s) to measure conductance ranging from 15 to 13,000 μ mhos/cm @ 25°C. Each should have an accessory or means to check the resistance.</td>
<td></td>
</tr>
<tr>
<td>CONDUCTIVITY METERS, Wheatstone bridge with</td>
<td></td>
</tr>
<tr>
<td>cell(s) to measure conductance ranging from 15 to 13,000 μ mhos/cm @ 25°C. Each should have an accessory or means to check the resistance.</td>
<td></td>
</tr>
<tr>
<td>CONTAINERS for instrument cell wastes, about 2 liters</td>
<td>C1 N/N</td>
</tr>
<tr>
<td>CYLINDERS, graduated, 10 ml</td>
<td>H</td>
</tr>
<tr>
<td>CYLINDERS, graduated, 25 ml</td>
<td>H</td>
</tr>
<tr>
<td>CYLINDERS, graduated 50 ml</td>
<td>N/N</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>TEST(S)</td>
</tr>
<tr>
<td>-------------</td>
<td>---------</td>
</tr>
<tr>
<td>CYLINDERS, graduated, 100 ml</td>
<td>N/N</td>
</tr>
<tr>
<td>CYLINDERS, graduated, 200 ml</td>
<td>C1</td>
</tr>
<tr>
<td>DESICCATORS, with active desiccant</td>
<td>S</td>
</tr>
<tr>
<td>EVAPORATING DISHES, 150 ml, porcelain</td>
<td>S</td>
</tr>
<tr>
<td>FILTERING ASSEMBLIES for 10 ml, 50 ml, and 100 ml volumes.</td>
<td>P</td>
</tr>
<tr>
<td>FILTER DISCS, phosphorus-free, 0.45 micron pore size, Gelman GA-6 or equivalent, to fit FILTERING ASSEMBLIES</td>
<td>P</td>
</tr>
<tr>
<td>FILTER DISCS, glass fiber, no organic binder, Reeve Angel 934AH, Gelman type A/E, Millipore AP-40, Whatman GF/C or equivalent. Diameter ~ 5cm for FILTERING ASSEMBLY and also ~ 2.1 cm for CRUCIBLE</td>
<td>S</td>
</tr>
<tr>
<td>FILTER PAPER, fluted, about No. 12 to fit 14 cm FUNNEL for Cd rinsings</td>
<td>N/N</td>
</tr>
<tr>
<td>FLASKS, Erlenmeyer, wide mouth, 125 ml</td>
<td>F</td>
</tr>
<tr>
<td>FLASKS, Erlenmeyer, wide mouth, 250 ml</td>
<td>N/H</td>
</tr>
<tr>
<td>FLASKS, volumetric, 50 ml, with glass stoppers</td>
<td>P</td>
</tr>
<tr>
<td>FLASKS, volumetric, 100 ml with glass stoppers</td>
<td>N/H</td>
</tr>
<tr>
<td>FORCEPS, pairs, blunt-tipped, to manipulate FILTER DISCS</td>
<td>P</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>TEST(S)</td>
</tr>
<tr>
<td>-----------------------------------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>FUNNELS, short stem, diameter about 5 cm to fit neck of 50 ml VOLUMETRIC</td>
<td>A/A</td>
</tr>
<tr>
<td>FLASK and 25 or 50 ml BURET</td>
<td>H</td>
</tr>
<tr>
<td>P</td>
<td>1</td>
</tr>
<tr>
<td>FUNNELS, medium stem, diameter about 14 cm to fit neck of old BOTTLES for</td>
<td>N/N</td>
</tr>
<tr>
<td>Cd rinsings</td>
<td></td>
</tr>
<tr>
<td>GLASSES, pairs of safety</td>
<td>AJ1</td>
</tr>
<tr>
<td>GLASS WOOL, small wad for end of 100 ml cut-off PIPET</td>
<td>N/N</td>
</tr>
<tr>
<td>HOT PLATÉS with continuous settings, not just &quot;low&quot;, &quot;medium&quot;, and &quot;high&quot;.</td>
<td>A/A</td>
</tr>
<tr>
<td></td>
<td>P</td>
</tr>
<tr>
<td>OVEN, hot air, to give uniform temperatures and with thermometer to</td>
<td>S</td>
</tr>
<tr>
<td>register accurately in range of 103°-105°C.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>OVEN, a second oven as described above or a steam bath for use at 98°C</td>
<td>S</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>PAPER CLIPS</td>
<td>N/N</td>
</tr>
<tr>
<td>PENCILS, WAX MARKING</td>
<td>A11</td>
</tr>
<tr>
<td>pH METERS, ELECTROMETRIC, accurate to at least 0.1 pH unit, each with</td>
<td>A/A</td>
</tr>
<tr>
<td>electrode(s)</td>
<td>H</td>
</tr>
<tr>
<td></td>
<td>N/N</td>
</tr>
<tr>
<td></td>
<td>P</td>
</tr>
<tr>
<td>PIPETS, dropping (medicine droppers) with bulb about 1 ml</td>
<td>A/A</td>
</tr>
<tr>
<td>PIPETS, measuring, glass, 1 ml graduated at 0.1 ml</td>
<td>C1</td>
</tr>
<tr>
<td>PIPETS, measuring (Mohr), glass, 10 ml</td>
<td>N/N</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>TEST(S)</td>
</tr>
<tr>
<td>------------------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 1 ml</td>
<td>N/N</td>
</tr>
<tr>
<td></td>
<td>P</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 2 ml</td>
<td>N/N</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 3 ml</td>
<td>P</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 5 ml</td>
<td>C/H</td>
</tr>
<tr>
<td></td>
<td>N/N</td>
</tr>
<tr>
<td></td>
<td>P</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 10 ml</td>
<td>F</td>
</tr>
<tr>
<td></td>
<td>N/N</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 20 ml</td>
<td>P</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 25 ml</td>
<td>H</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 50 ml</td>
<td>A/A</td>
</tr>
<tr>
<td></td>
<td>F</td>
</tr>
<tr>
<td></td>
<td>N/N</td>
</tr>
<tr>
<td></td>
<td>P</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 100 ml</td>
<td>T</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 100 ml with top and tip cut off for reduction column</td>
<td>N/N</td>
</tr>
<tr>
<td>PIPETS BULBS, large with opening shaped to fit variety of PIPET sizes</td>
<td>A/A</td>
</tr>
<tr>
<td>BULBS, aluminum or stainless steel, 6 cm diameter</td>
<td>F</td>
</tr>
<tr>
<td>PROP PIPE BULBS</td>
<td>S</td>
</tr>
<tr>
<td></td>
<td>C/H</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>TEST(S)</td>
</tr>
<tr>
<td>-------------------------------------</td>
<td>---------</td>
</tr>
<tr>
<td>REAGENT SPOONS, 0.4 gram</td>
<td>P</td>
</tr>
<tr>
<td>REDUCTION COLUMNS</td>
<td>N/N</td>
</tr>
<tr>
<td>SAFETY GLASSES</td>
<td>All</td>
</tr>
<tr>
<td>SIEVES, 60 mesh</td>
<td>N/N</td>
</tr>
<tr>
<td>SPATULAS</td>
<td>A/A</td>
</tr>
<tr>
<td></td>
<td>Cl</td>
</tr>
<tr>
<td></td>
<td>P</td>
</tr>
<tr>
<td>SPECIFIC ION METERS, each with a fluoride ion activity electrode or with a combination fluoride electrode</td>
<td>F</td>
</tr>
<tr>
<td>SPECTROPHOTOMETERS, for use at 570, 540, 880 (or 650) nm, providing a light path of at least 1 cm</td>
<td>F</td>
</tr>
<tr>
<td>SPECTROPHOTOMETER CELLS, 1 cm for instruments on hand</td>
<td>F</td>
</tr>
<tr>
<td>STANDS, titration with porcelain bases</td>
<td>A/A</td>
</tr>
<tr>
<td>STIRRING APPARATUS, MAGNETIC</td>
<td>Cl</td>
</tr>
<tr>
<td>STOP WATCHES, or timing devices</td>
<td>A/A</td>
</tr>
<tr>
<td>THERMOMETERS</td>
<td>A/A</td>
</tr>
<tr>
<td></td>
<td>F</td>
</tr>
<tr>
<td>TISSUES, small, soft, in boxes</td>
<td>Cl</td>
</tr>
<tr>
<td></td>
<td>S</td>
</tr>
<tr>
<td></td>
<td>SC</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>TEST(S)</td>
</tr>
<tr>
<td>-------------</td>
<td>---------</td>
</tr>
<tr>
<td>TITRATORS, amperometric with cell and any accessories, e.g., pipets.</td>
<td>Cl</td>
</tr>
<tr>
<td>TONGS, crucible; pairs</td>
<td>S A/A</td>
</tr>
<tr>
<td>TUBING, soft rubber, 4 cm ID, 7.5 cm length</td>
<td>N/N</td>
</tr>
<tr>
<td>TURBIDIMETERS, Nephelometers that measure light scattered at right angles to the path of the incident light, with cell and any standard accessories.</td>
<td>T</td>
</tr>
<tr>
<td>VACUUM SOURCES: preferably a central service. An electric vacuum pump assembly with suitable hoses, water traps and shut-off valves and with capability of drawing 15 inches of mercury can be used.</td>
<td>S</td>
</tr>
<tr>
<td>WASH BOTTLES, plastic squeeze type, 500 ml</td>
<td>All</td>
</tr>
<tr>
<td>WATCH GLASSES, about 6 cm diameter</td>
<td>S</td>
</tr>
<tr>
<td>WATER-STILL to produce water free of the constituent sought in each test.</td>
<td>All</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY PER STATION</td>
</tr>
<tr>
<td>-----------------------------------</td>
<td>----------------------</td>
</tr>
<tr>
<td><strong>ACIDITY and ALKALINITY</strong></td>
<td>See Remarks</td>
</tr>
<tr>
<td>Buffer, pH 4.X - 1000 ml</td>
<td>75 ml</td>
</tr>
<tr>
<td>10.12 g potassium biphthalate</td>
<td></td>
</tr>
<tr>
<td>Buffer, pH 6.X - 1000 ml</td>
<td>150 ml</td>
</tr>
<tr>
<td>3.388 g potassium dihydrogen phosphate</td>
<td></td>
</tr>
<tr>
<td>3.533 g disodium hydrogen phosphate</td>
<td></td>
</tr>
<tr>
<td>Buffer, pH 9.X - 1000 ml</td>
<td>75 ml</td>
</tr>
<tr>
<td>3.80 g sodium borate decahydrate (borax)</td>
<td></td>
</tr>
<tr>
<td>Hydrogen peroxide, 30 % solution</td>
<td>5 drops</td>
</tr>
<tr>
<td>Potassium biphthalate - 1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>10.0 g</td>
<td></td>
</tr>
<tr>
<td>Sodium carbonate - 1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>2.5 g</td>
<td></td>
</tr>
<tr>
<td>Sodium hydroxide, 0.1N-1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>11 g</td>
<td></td>
</tr>
<tr>
<td>Sodium hydroxide, 0.020 N - 1000 ml</td>
<td>15 ml</td>
</tr>
<tr>
<td>200 ml 0.1 N sodium hydroxide</td>
<td></td>
</tr>
<tr>
<td>Sulfuric acid, 0.1 N - 1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>3.0 ml concentrated sulfuric acid</td>
<td></td>
</tr>
<tr>
<td>Sulfuric acid, 0.020 N - 1000 ml</td>
<td>25 ml</td>
</tr>
<tr>
<td>200 ml 0.1 N sulfuric acid</td>
<td></td>
</tr>
<tr>
<td>Sample for acidity - 1000 ml</td>
<td>50 ml</td>
</tr>
<tr>
<td>300 ml 0.020 N sulfuric or hydrochloric acid</td>
<td></td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY PER STATION</td>
</tr>
<tr>
<td>---------------------------------------------------------</td>
<td>----------------------</td>
</tr>
<tr>
<td>Sample for alkalinity - 1000 ml</td>
<td>50 ml</td>
</tr>
<tr>
<td>300 ml 0.020 N sodium carbonate</td>
<td></td>
</tr>
<tr>
<td>Sample for alkalinity - 1000 ml</td>
<td>50 ml</td>
</tr>
<tr>
<td>200 ml 0.020 N sodium hydroxide</td>
<td></td>
</tr>
<tr>
<td>CHLORINE, Amperometric (Back) Titration</td>
<td>See Remarks</td>
</tr>
<tr>
<td>Arsenite solution, 0.1 N - 1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>4.95 g arsenic trioxide</td>
<td></td>
</tr>
<tr>
<td>15 g sodium hydroxide</td>
<td></td>
</tr>
<tr>
<td>carbon dioxide to saturate 250 ml sol.</td>
<td></td>
</tr>
<tr>
<td>Buffer, pH 4.0 - 1000 ml</td>
<td>8 ml</td>
</tr>
<tr>
<td>243 g sodium acetate trihydrate</td>
<td></td>
</tr>
<tr>
<td>480 g glacial acetic acid</td>
<td></td>
</tr>
<tr>
<td>Hydrochloric acid - 5 to 10 drops</td>
<td>See Remarks</td>
</tr>
<tr>
<td>Iodine solution, 0.1 N - 1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>40 g potassium iodide</td>
<td></td>
</tr>
<tr>
<td>13 g resublimed iodine</td>
<td></td>
</tr>
<tr>
<td>Iodine solution, 0.0282 N - 1000 ml</td>
<td>2 ml</td>
</tr>
<tr>
<td>25 g potassium iodide</td>
<td></td>
</tr>
<tr>
<td>282 ml of solution prepared to be 0.1 N iodine</td>
<td></td>
</tr>
<tr>
<td>Phenylarsine oxide, 0.00564 N - 1000 ml</td>
<td>10 ml</td>
</tr>
<tr>
<td>0.8 g phenylarsine oxide</td>
<td></td>
</tr>
<tr>
<td>1.8 g sodium hydroxide</td>
<td></td>
</tr>
<tr>
<td>1 + 1 hydrochloric acid (to adjust pH)</td>
<td></td>
</tr>
<tr>
<td>0.05 g potassium iodide</td>
<td></td>
</tr>
<tr>
<td>Hydrochloric acid - 5 to 10 drops</td>
<td>See Remarks</td>
</tr>
<tr>
<td>Iodine solution, 0.0282 N - 1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>25 g potassium iodide</td>
<td></td>
</tr>
<tr>
<td>282 ml of solution prepared to be 0.1 N iodine</td>
<td></td>
</tr>
<tr>
<td>Phenylarsine oxide, 0.00564 N - 1000 ml</td>
<td></td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY PER STATION</td>
</tr>
<tr>
<td>-------------</td>
<td>---------------------</td>
</tr>
<tr>
<td>Potassium iodide crystals</td>
<td>2 g</td>
</tr>
<tr>
<td>Sodium chloride, U.S.P., pellets</td>
<td>20 g</td>
</tr>
<tr>
<td>Starch indicator</td>
<td>See Remarks</td>
</tr>
<tr>
<td>Sample</td>
<td>400 ml</td>
</tr>
<tr>
<td>FLUORIDE, SPADNS and Electrode</td>
<td>See Remarks</td>
</tr>
<tr>
<td>Acid Zirconyl - SPADNS reagent - 1000 ml</td>
<td>75 ml</td>
</tr>
<tr>
<td>500 ml SPADNS solution</td>
<td></td>
</tr>
<tr>
<td>500 ml Zirconyl - acid reagent</td>
<td></td>
</tr>
<tr>
<td>Fluoride stock solution - 1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>0.2210 g anhydrous sodium fluoride</td>
<td></td>
</tr>
<tr>
<td>Fluoride standard, 1 mg - 1000 ml</td>
<td>100 ml</td>
</tr>
<tr>
<td>20 ml stock solution</td>
<td></td>
</tr>
<tr>
<td>Fluoride standard, 2 mg - 1000 ml</td>
<td>100 ml</td>
</tr>
<tr>
<td>20 ml stock solution</td>
<td></td>
</tr>
<tr>
<td>Fluoride standard, 10 mg - 1000 ml</td>
<td>60 ml</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY PER STATION</td>
</tr>
<tr>
<td>-------------</td>
<td>----------------------</td>
</tr>
<tr>
<td>TISAB - 1000 ml</td>
<td>300 ml*</td>
</tr>
<tr>
<td>57 ml glacial acetic acid</td>
<td></td>
</tr>
<tr>
<td>58 g sodium chloride</td>
<td></td>
</tr>
<tr>
<td>4.0 g 1,2 cyclohexylene dinitrilo tetra-acetic acid (CDTA)</td>
<td></td>
</tr>
<tr>
<td>~ 30 g sodium hydroxide</td>
<td></td>
</tr>
<tr>
<td>Žirconyl - acid reagent - 1000 ml</td>
<td>See Remarks</td>
</tr>
<tr>
<td>0.266 g zirconyl chloride octahydrate</td>
<td></td>
</tr>
<tr>
<td>700 ml concentrated hydrochloric acid</td>
<td></td>
</tr>
<tr>
<td>See Remarks</td>
<td></td>
</tr>
<tr>
<td>Sample #1 - 1000 ml</td>
<td>100 ml</td>
</tr>
<tr>
<td>5.5 ml fluoride stock solution</td>
<td></td>
</tr>
<tr>
<td>Sample #2 - 1000 ml</td>
<td>100 ml</td>
</tr>
<tr>
<td>10.5 ml fluoride stock solution</td>
<td></td>
</tr>
<tr>
<td>Sample #3 - 1000 ml</td>
<td>100 ml</td>
</tr>
<tr>
<td>7.5 ml fluoride stock solution</td>
<td></td>
</tr>
<tr>
<td>0.4437 g anhydrous sodium sulfate</td>
<td></td>
</tr>
<tr>
<td>Sample #4 - 1000 ml</td>
<td>100 ml</td>
</tr>
<tr>
<td>9.5 ml fluoride stock solution</td>
<td></td>
</tr>
<tr>
<td>52.752 mg aluminum potassium sulfate</td>
<td></td>
</tr>
<tr>
<td>Buffer, pH 6.X</td>
<td>100 ml</td>
</tr>
<tr>
<td>Buffer, pH 9.X</td>
<td>100 ml</td>
</tr>
</tbody>
</table>
| Buffer, test reagent - 1000 ml | 6 ml | Store in tightly-stoppered plastic or resistant-glass container. Do not store more than a month's supply in a frequently opened container. See "Standard Methods" regarding alternate "odorless buffers,"
| 67.6 g ammonium chloride | | |
| 572 ml concentrated ammonium hydroxide | | |
| 4.716 g EDTA (also listed below) | | |
| 3.120 g magnesium sulfate · 7 H2O OR | | |
| 2.576 g magnesium chloride · 6 H2O | | |
| Calcium carbonate - 1000 ml | 25 ml | Also used to standardize EDTA. |
| 1.000 g anhydrous calcium carbonate | | |
| 1+1 hydrochloric acid to dissolve calcium carbonate and adjust pH | | |
| 3 N(1+4) ammonium hydroxide for pH | | |
| 3 drops methyl red indicator | | |

---

52
<table>
<thead>
<tr>
<th>DESCRIPTION</th>
<th>QUANTITY PER STATION</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>EDTA, 0.01 M - 1000 ml</td>
<td>75 ml</td>
<td>See &quot;Standard Methods&quot; for commercial equivalents.</td>
</tr>
<tr>
<td>3.723 g disodium ethylenediamine tetra-acetate dihydrate</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Indicator</td>
<td>0.6 g</td>
<td>This is usually not required. See &quot;Standard Methods&quot; for discussion and choices.</td>
</tr>
<tr>
<td>0.5 g Erlochrome Black T</td>
<td></td>
<td></td>
</tr>
<tr>
<td>100 g sodium chloride</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inhibitor</td>
<td>See Remarks</td>
<td>See Remarks</td>
</tr>
<tr>
<td>See Remarks</td>
<td></td>
<td>For DETAILS OF REAGENT PREPARATIONS, see 1974 EPA, &quot;Methods for Chemical Analysis&quot;, page 202 (or the method cited if a later edition is approved)</td>
</tr>
<tr>
<td>Sample - 1000 ml</td>
<td>25 ml</td>
<td>A wastewater treatment plant sample may be used if it will give positive hardness results.</td>
</tr>
<tr>
<td>10 ml calcium carbonate solution</td>
<td></td>
<td></td>
</tr>
<tr>
<td>NITRATE/NITRITE, Cadmium Reduction</td>
<td>See Remarks</td>
<td>See Remarks</td>
</tr>
<tr>
<td>Ammonium chloride - EDTA, conc. - 1000 ml</td>
<td>610 ml</td>
<td>400 ml is used for the testing of the assigned (4) solutions. The additional 210 ml is used to prepare 350 ml dilute solution for preparing one column.</td>
</tr>
<tr>
<td>1.7 g disodium ethylenediamine tetra-acetate</td>
<td></td>
<td></td>
</tr>
<tr>
<td>13 g ammonium chloride</td>
<td></td>
<td></td>
</tr>
<tr>
<td>conc. ammonium hydroxide to adjust pH</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ammonium chloride - EDTA, dilute - 1000 ml</td>
<td>350 ml</td>
<td>For pH adjustment of sample</td>
</tr>
<tr>
<td>600 ml concentrated solution</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ammonium hydroxide, concentrated</td>
<td>~ 1 ml</td>
<td>See this item in ACIDITY and ALKALINITY section.</td>
</tr>
<tr>
<td>Buffer, pH 4.0</td>
<td>100 ml</td>
<td>See this item in ACIDITY and ALKALINITY section.</td>
</tr>
<tr>
<td>Buffer, pH 6.0</td>
<td>100 ml</td>
<td>See this item in ACIDITY and ALKALINITY section.</td>
</tr>
<tr>
<td>Cadmium, granulated 40-60 mesh</td>
<td>~ 20 g</td>
<td>Can be purchased</td>
</tr>
<tr>
<td>Color reagent - 1000 ml</td>
<td>8 ml</td>
<td></td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY PER STATION</td>
<td>REMARKS</td>
</tr>
<tr>
<td>-------------</td>
<td>----------------------</td>
<td>---------</td>
</tr>
<tr>
<td>Copper sulfate, 2% - 1000 ml 20 g copper sulfate pentahydrate</td>
<td>200 ml</td>
<td></td>
</tr>
<tr>
<td>Hydrochloric acid, concentrated</td>
<td>~1 ml</td>
<td>For pH adjustment</td>
</tr>
<tr>
<td>Hydrochloric acid, 6N-1000 ml 500 ml concentrated hydrochloric acid</td>
<td>60 ml</td>
<td></td>
</tr>
<tr>
<td>Potassium nitrate stock - 1000 ml 7.218 g potassium nitrate 2 ml chloroform</td>
<td>See Remarks</td>
<td>Used to prepare activation and standard solutions. This solution is stable for at least six months, kept under refrigeration.</td>
</tr>
<tr>
<td>Potassium nitrate activation solution - 1000 ml 1 ml stock solution</td>
<td>25 ml</td>
<td>UNSTABLE - prepare just before use.</td>
</tr>
<tr>
<td>Potassium nitrate standard - 1000 ml 10 ml stock solution (for 1 cm cells)</td>
<td>15 ml</td>
<td>Also used to prepare sample UNSTABLE - prepare just use.</td>
</tr>
<tr>
<td>Potassium nitrite stock - 1000 ml 6.072 g potassium nitrite 2 ml chloroform</td>
<td>See Remarks</td>
<td>Used to prepare standard solution. This solution is stable for about three months, kept under refrigeration.</td>
</tr>
<tr>
<td>Potassium nitrite standard - 1000 ml 10 ml stock solution (for 1 cm cells)</td>
<td>15 ml</td>
<td>Also used to prepare sample. UNSTABLE - prepare just before use.</td>
</tr>
<tr>
<td>Sample - 1000 ml 40 ml nitrate standard 40 ml nitrite standard</td>
<td>50 ml</td>
<td>UNSTABLE - prepare just before use. A wastewater treatment plant sample can be used. This should contain both nitrate and nitrite.</td>
</tr>
<tr>
<td>PHOSPHORUS, Digestion &amp; Reduction</td>
<td>See Remarks</td>
<td>For DETAILS OF REAGENT PREPARATIONS, see 1974 EPA, &quot;Methods for Chemical Analysis&quot;, page 252 (or the method cited if a later edition is approved).</td>
</tr>
<tr>
<td>Ammonium persulfate</td>
<td>1.2 g</td>
<td></td>
</tr>
<tr>
<td>Buffer, pH 4.1</td>
<td>100 ml</td>
<td>See this item in ACIDITY and ALKALINITY section</td>
</tr>
<tr>
<td>Buffer, pH 6.1</td>
<td>100 ml</td>
<td>See this item in ACIDITY and ALKALINITY section</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY PER STATION</td>
<td>REMARKS</td>
</tr>
<tr>
<td>-------------</td>
<td>----------------------</td>
<td>---------</td>
</tr>
<tr>
<td>Combined Reagent - 1000 ml</td>
<td>24 ml</td>
<td>LIMITED STABILITY - Combine the four solutions just before use. Follow all preparation instructions carefully.</td>
</tr>
<tr>
<td>Potassium acid phosphate stock - 1000 ml</td>
<td>See Remarks</td>
<td>Used to prepare standard solution and sample.</td>
</tr>
<tr>
<td>Potassium acid phosphate standard - 1000 ml</td>
<td>70 ml</td>
<td>UNSTABLE - Prepare just before use.</td>
</tr>
<tr>
<td>Sodium hydroxide, 10N-1000 ml</td>
<td>~ 10 ml</td>
<td>For pH adjustment</td>
</tr>
<tr>
<td>Sodium hydroxide 0.1N-1000 ml</td>
<td>~ 5 ml</td>
<td>For pH adjustment</td>
</tr>
<tr>
<td>Sulfuric acid, 11N-1000 ml</td>
<td>~ 4 ml</td>
<td>For digestion and for treating sample</td>
</tr>
<tr>
<td>Sulfuric acid, 0.11N-1000 ml</td>
<td>~ 5 ml</td>
<td>For pH adjustment</td>
</tr>
<tr>
<td>Sample - 1000 ml</td>
<td>50 ml</td>
<td>UNSTABLE - Prepare just before use. A wastewater sample containing phosphorus may be used.</td>
</tr>
<tr>
<td>SOLIDS, Total and Suspended</td>
<td>See Remarks</td>
<td>No reagents are needed for these gravimetric tests.</td>
</tr>
<tr>
<td>Sample, plant influent</td>
<td>~ 200 ml</td>
<td>If a treatment plant sample is not available, a quality control sample of suspended solids could be used for both tests. EPA-EMSL, Cincinnati, Ohio 45268 is one supplier.</td>
</tr>
<tr>
<td>Sample, plant effluent</td>
<td>~ 100 ml</td>
<td>See above Remarks for alternative.</td>
</tr>
<tr>
<td>SPECIFIC CONDUCTANCE, meter</td>
<td>See Remarks</td>
<td>For DETAILS OF REAGENT PREPARATIONS, see 14th edition, &quot;Standard-Methods&quot;, page 74 (or the method cited if a later edition is approved).</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY PER STATION</td>
<td>REMARKS</td>
</tr>
<tr>
<td>-------------</td>
<td>----------------------</td>
<td>---------</td>
</tr>
<tr>
<td>Potassium chloride stock, 1N-1000 ml 74.56 g potassium chloride</td>
<td>See Remarks</td>
<td>Used to prepare two standards and Sample A</td>
</tr>
<tr>
<td>Potassium chloride, 0.1N-1000 ml 100 ml 1N solution</td>
<td>400 ml</td>
<td>Also used to prepare 0.001 N solution and Sample B.</td>
</tr>
<tr>
<td>Potassium chloride, 0.01N-1000 ml 10 ml 1N solution</td>
<td>400 ml</td>
<td>Also used to prepare 0.0001N solution and Sample C.</td>
</tr>
<tr>
<td>Potassium chloride, 0.001N-1000 ml 10 ml 0.1N solution</td>
<td>400 ml</td>
<td></td>
</tr>
<tr>
<td>Potassium chloride, 0.0001N-1000 ml 10 ml 0.01N solution</td>
<td>400 ml</td>
<td></td>
</tr>
<tr>
<td>Sample A - 1000 ml 10 to 100 ml 1N solution</td>
<td>400 ml</td>
<td>A water sample with measurable conductivity may be used.</td>
</tr>
<tr>
<td>Sample B - 1000 ml 10-100 ml 0.1N solution</td>
<td>400 ml</td>
<td>A water sample with measurable conductivity may be used.</td>
</tr>
<tr>
<td>Sample C - 1000 ml 10-100 ml 0.01N solution</td>
<td>400 ml</td>
<td>A water sample with measurable conductivity may be used.</td>
</tr>
<tr>
<td>TURBIDITY, Nephelometer</td>
<td>See Remarks</td>
<td>For DETAILS OF REAGENT PREPARATIONS, see 1974 EPA, &quot;Methods for Chemical Analysis&quot;, page 296 (or the method cited if a later edition is approved).</td>
</tr>
<tr>
<td>Formazin stock, 400 NTU-1000 ml 1 g hydrazine sulfate 10 g hexamethylenetetramine</td>
<td>10 ml</td>
<td>The stock is a mixture of solutions of the two chemicals listed. Also used to prepare Formazin standard</td>
</tr>
<tr>
<td>Formazin standard, 40 NTU-1000 ml 100 ml Formazin stock</td>
<td>See Remarks</td>
<td>Used to make sample.</td>
</tr>
<tr>
<td>Manufacturer's standards</td>
<td>See Remarks</td>
<td>As supplied to standardize the meter.</td>
</tr>
<tr>
<td>Sample One - 1000 ml 5 ml 40 NTU standard</td>
<td>50 ml</td>
<td>A water sample with measurable turbidity may be used.</td>
</tr>
<tr>
<td>Sample Two - Tap Water</td>
<td>50 ml</td>
<td>Another water sample with measurable turbidity may be used.</td>
</tr>
</tbody>
</table>
PART II - INSTRUCTIONAL PACKAGE WORKSHEETS

The Worksheets are for guidance to the Instructor who develops the subject matter covered in the course. These Worksheets are not scripts. The Instructor will need to make extensive and detailed preparation in order to perform the assigned tasks effectively and efficiently. The Instructional Package Worksheets do provide a perspective of each analytical procedure, the learning achievement levels the students should attain, available audiovisual and other instructional resources, and an example course of action in pre-course preparation and classroom/laboratory instruction. The Instructor is free to modify the Worksheets to meet individual needs. It should be noted however, that associated additions and deletions will then be required in Sections V through XI of the Worksheet, and in Sections E and F of Part I.

Application of these Instructional Packages will help the Instructor to reduce the time required for planning and organizing a strategy of preparation and instruction. However, time and effort are required for physical preparations for classroom and laboratory instruction; time and effort are required for rehearsals of Instructor performance in classroom and laboratory. These requirements never can be met by such an Instructor’s Guide as this, ultimately the Instructor is the key person in assuring that the student acquires the needed knowledge and skills.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT/MATTER: Course Objectives - Introductions

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 15 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: To give overview of course and allow class to begin to know each other.

V. ENTRY LEVEL BEHAVIOR:
   A. Application and acceptance as a participant in the course,
   B. Registration forms and details have been completed.

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will have an overview of the course schedule, be able to use the training manual and correlate it to the agenda, and will know the requirements for successful completion of the course as stipulated for earning continuing education units. He/she will also know more about the other members of the class.
   B. Conditions: He/she will be provided with a course schedule, a copy of the training manual, and a short discussion by the Course Director.
   C. Accepted Performance: Attention during the discussion and use of the provided materials as directed.

VII. INSTRUCTIONAL RESOURCES:
   A. Available Media:
      1. The course schedule
      2. The training manual
   B. Suggested Media:
      1. None

VIII. INSTRUCTIONAL APPROACH:
   A. Preparation for Instruction:
      1. Prepare a summary of the employers of the participants.
      2. Review the requirements you have established for award of continuing education units for this course, if applicable. At NTOTC, we awarded 2.5 CEU's (1500 minutes contact time) with the following requirements:
a. 95% attendance
b. Satisfactory participation in class and laboratory sessions as judged by the course instructors
c. A score of 70% or more on the post-course evaluation if the pre-course score on the same test was less than 50%. Pre-course scores of 50% or higher are to improve by at least 20% to a maximum requirement of 90%. Any exceptions to this standard must be documented in the course file.

3. Duplicate copies of the course schedule for each student.

4. Secure a copy of the training manual for each student.

B. Sequencing

1. Welcome

2. Summary of employers of class participants. Special welcome to people from other countries.

   a. Use page with course description. Restate the course objectives including the list of topics in the final paragraph.
   b. Contents page - location of lecture and laboratory outlines.

4. Agenda
   a. Note topics and manual outline numbers.
   b. Note breaks and lunch times. They schedule own break during laboratory sessions.
   c. Note time course ends.
   d. General format is lecture, laboratory, follow-up discussion for each topic.

5. Scan outlines before class, especially laboratory procedures.

6. Explain requirements for earning continuing education units for course (if applicable).

7. Have each person rise and give name, employer, and principle job responsibilities.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. For each student:
   1. One copy of the training manual.
   2. One copy of the course schedule.
X. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. None
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Pre-Course - Post-Course Evaluation

II. UNIT OF INSTRUCTION: Summary of NTOTC Use of the Test

III. ESTIMATED TIME: 75 minutes (30 + 45)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: There is an "Accepted Performance" standard stated in the "Instructional Objective" for each subject in this course (VI C. in each IPW). It states the mode of evaluation used at NTOTC for that subject and includes the requirement that the participant must correctly answer 70% of the items on the subject in the post-course evaluation. Thus a minimum final score of 70% on the post-test is required. To earn continuing education units for the entire course (2.5 CEU's), NTOTC also requires evidence of increased knowledge by an increase of 20 points or more (to a maximum requirement of 90%) in the score for the same test taken before and after participating in the course.

The test contains items for every topic in the course. You may find these items useful even if you prefer to administer a written test for each topic as it is completed. You can compile topical tests using these items plus your own questions to obtain a more comprehensive evaluation of achievement regarding the individual subjects. (You may need to increase the time allotments given in the NTOTC course schedule for this additional evaluation activity).

V. ENTRY LEVEL BEHAVIOR:

A. Pre-Course Evaluation
   I. Is a chemist or chemistry technician
   2. Has the prerequisites to participate in the course

B. Post-Course Evaluation
   1. Attended 95% of the course

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will answer the same set of test questions before and after participating in the course to fulfill the requirement for award of 2.5 continuing education units for the course.

B. Conditions: He/she will be given the test and 30 minutes.

C. Accepted Performance: An initial grade of less than 50 should increase to 70 or more. An initial grade of 50 or more should increase 20 points with 90 as a maximum required score.
VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. A set of questions on main points presented for each topic on the NTOTC course schedule, an answer sheet, scoring information and a key are at the end of this IPW.

2. An example letter sent to participants who attended 95% of the course but are not awarded CEU's is at the end of this IPW.

3. An example letter sent to participants who did not attend 95% of the course so are not awarded CEU's or a course certificate is at the end of this IPW.

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Duplicate one copy of the questions to be used and two copies of the answer sheet for each student.

B. Sequencing:

1. Before the course topics are presented - 30 minutes
   a. If the "Instructional Objective" (VI above) has not been presented to the participants (in the preceding "Course Objectives" IPW), explain it now.
   b. Distribute one copy of questions and one answer sheet to each participant.
   c. Go over the directions. Emphasize that one should not write anything on the question sheets.
   d. Allow the rest of 30 minutes for each to complete the answer sheet.
   e. Collect all distributed materials. Make sure that each answer sheet has the name and date recorded.

2. Outside of class:
   a. Check all question sheets to be sure none have been marked in any way. Reserve for future use.
   b. Correct the answer sheets and assign scores.
   c. The results for groups of items are indicators of each participant's entry level knowledge about each topic.

3. After the course topics are presented - 30 minutes
   a. Distribute the same question sheets and one blank answer sheet to each participant.
b. Repeat the procedures in 1. c. through e. above

4. After collecting completed surveys - 15 minutes
   a. Use acetates of the question sheets with circled answers to go through the test questions as a summary of course content.

5. Outside of class:
   a. Check all question sheets to be sure none have been marked in any way. Reserve the clean copies for future use.
   b. Correct the answer sheets and assign scores.
   c. For each participant, record the pre-course and post-course scores.
   d. Use the "Accepted Performance" standard to determine which participants have earned the CEU's for the course.
   e. Take appropriate action for each student regarding this requirement for award of continuing education units. At NTOIC, the action for those earning the CEU's is to make arrangements for recording the award on the person's course certificate. Those who attended the entire course, but who did not earn CEU's, receive a course certificate and a letter documenting why the CEU's are not recorded on the certificate. (An example of the letter is at the end of this IPW).
   f. Inspect the answer sheets, item-by-item. A tally of the errors per item can serve as an evaluation of the instruction given on the topic involved, as well as of the participants' mastery of the topic of the item (or group of items). Make a note if any changes in the instructional approach for topics should be planned for future courses.
   g. File the scored answer sheets with any other evaluation information about the students.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. For Each Student:
   1. One copy of the test questions and two copies of the answer sheet for each.

X. IPW REAGENT REQUIREMENTS:

A. None
1. A "representative" water sample represents
   a. the best water quality area of the source at the time of sampling.
   b. the conditions existing in the water quality of the source at the time of sampling.
   c. the worst water quality area of the source at the time of sampling.

2. Written procedures to provide an accurate record for the chain-of-custody of monitoring samples should be developed for
   a. sample collection and transport.
   b. sample collection, transport and delivery to a laboratory.
   c. sample collection, transport, delivery to a laboratory and distribution to analyst(s).
   d. sample collection, transport, delivery to a laboratory, distribution to analyst(s) and storage or disposal.

3. For NPDES samples, the first source of information about preservation measures to be applied are
   a. those developed by the responsible laboratory.
   b. those published in the source of the analytical method to be used.
   c. those published in the EPA "Methods for Chemical Analysis of Water and Wastes."
   d. those published in the Federal Register.

4. The first consideration when drawing up a time schedule for analyzing samples is
   a. the preservative used.
   b. the number of analysts available.
   c. the holding time guidelines for the constituent of interest.
   d. the quality control checks to be used.
   e. the number of samples to be processed.
Some items have more than one correct choice.

5. Which of the following have the right to apply for permission to use a procedure different from those published in the Federal Register for NPDES or drinking water compliance analyses?
   a. A permit holder
   b. A representative of a monitoring agency
   c. A representative of a certified laboratory
   d. A representative of a non-certified laboratory
   e. A representative of an instrument manufacturer

6. According to Federal Register regulations, which of the following apply/applies to field kits for NPDES compliance monitoring?
   a. Field kits may never be used.
   b. If a kit contains all the reagents and equipment listed in a referenced method, it may be used.
   c. Field kits with non-referenced reagents and/or equipment are considered an alternative procedure and require specific permission for use.

7. Which of the following is/are precision statistics?
   a. % relative error
   b. % relative standard deviation
   c. % bias
   d. % recovery
   e. range
   f. standard deviation

8. Which of the following is/are related to control of accuracy?
   a. Analysis of reagent blanks
   b. Analysis of duplicate samples
   c. Analysis of spiked samples
   d. Analysis of standards to check reproducibility of a curve
   e. Analysis using standard addition techniques

9. When an analyst begins a titration, which of the following is/are unknown?
   a. The concentration in the sample.
   b. The concentration in the titrant (standard solution)
   c. The volume of sample
   d. The volume of titrant required

10. What is another name for a substance which is a proton acceptor?
    a. acid
    b. base
    c. salt
    d. isomer
    e. polymer
11. If a volume requirement is stated as "50.0" ml, which one of the following should be used to measure and deliver it?

a. A graduated 50 ml beaker
b. A graduated 50 ml erlenmeyer flask
c. A 50 ml graduated cylinder
d. A 50 ml volumetric pipet

12. In reference to volumetric analyses, which of the following is/are true?

a. Volumetric analysis requires a completed chemical reaction.
b. Volumetric analysis requires an indicator of when the correct volume of titrant has been used.
c. Calculations for volumetric analyses represent the molarity relationships of the solutions involved in a chemical reaction.
d. Volumetric analysis cannot be used to determine the weight of a constituent in a volume of sample.

13. The end point for an alkalinity determination on an NPDES sample is

a. the neutralization product pH for organic acids.
b. the neutralization product pH for mineral acids.
c. the neutralization product pH for alkali metal ions.
d. the neutralization product pH for hydroxide ions.
e. the neutralization product pH for bicarbonate ions.

14. Which of the following describe(s) every buffer?

a. It keeps the pH at 7.0
b. It resists changes in pH.
c. It controls volatile interferences.
d. It keeps an acid pH.
e. It keeps a basic pH.

15. Reaction completion in a total hardness titration produces a

a. blue color.
b. green color.
c. red color.
d. pH 4.5
e. pH 8.2
f. change in amperage.

16. What is the titrant (in the buret) for a total hardness determination?

a. EDTA
b. Combined reagent
  0.02 N hydrochloric acid
c. Phenylarsine oxide
d. SPADNS reagent
Some items have more than one correct choice.

17. One indicator which is approved for use in a total hardness titration is
   a. phenolphthalein.
   b. starch
   c. methyl red.
   d. Eriochrome Black-T (EBT).
   e. Formazin.

18. The end point in one of the total residual chlorine titration tests produces a
   a. blue-green color.
   b. green color.
   c. red color.
   d. pH 4.5
   e. pH 8.2
   f. change in amperage.

19. A back-titration must be used to accurately determine total residual chlorine in
   a. samples held beyond 6 hours.
   b. samples preserved with inorganic acids.
   c. samples requiring pH adjustments.
   d. samples with dissolved inorganic materials.
   e. samples with dissolved organic materials.

20. The recognized method(s) to determine total residual chlorine for NPDES purposes is/are:
   a. Back-titration with phenylarsine oxide titrant
   b. Back-titration with iodine titrant
   c. Direct titration with ferrous ammonium sulfate titrant
   d. Color comparison using a DPD kit

21. The analytical reaction used to determine total phosphorus produces a
   a. blue color.
   b. green color.
   c. red color.
   d. pH 4.5
   e. pH 8.2
   f. change in amperage.

22. The total phosphorus determination includes
   a. sulfuric acid hydrolysis.
   b. combined reagent addition.
   c. persulfate oxidation.
   d. pH control.
   e. developing a standard curve.
Some items have more than one correct choice.

23. To determine total phosphorus for NPDES requirements, all phosphorus in the sample must either already be in, or be converted to, which of the following forms?
   a. P
   b. P₂O₅
   c. H₃PO₄
   d. PO₄³⁻

24. The analytical reaction used in the SPADNS determination of fluoride involves a
   a. blue color.
   b. green color.
   c. red color.
   d. pH 4.5
   e. pH 8.2
   f. change in amperage.

25. The distillation procedure for fluoride
   a. distills over interferences leaving F⁻ behind
   b. distills over F⁻ leaving interferences behind
   c. requires temperature measurements
   d. produces the color used for subsequent measurement
   e. requires fresh sulfuric acid for each sample

26. The fluoride electrode can be used with
   a. an expanded scale pH meter
   b. any pH meter
   c. a conductivity meter
   d. a specific ion meter
   e. a spectrophotometer with adapted connectors

27. The analytical reaction used in the cadmium reduction determination of nitrate and nitrite produces a
   a. blue color.
   b. green color.
   c. red color.
   d. pH 4.5
   e. pH 8.2
   f. change in amperage.

28. Interferences in the cadmium reduction method include
   a. temperature change
   b. grease clogging of the column
   c. suspended solids in the column
   d. the short-term usability of the cadmium
   e. nitrate color interferences
Some items have more than one correct choice.

29. In the cadmium reduction method, the analysis depends on a test for:
   a. ammonium ions.
   b. copper ions.
   c. cadmium ions.
   d. nitrate ions.
   e. nitrite ions.

30. For NPDES purposes, which of the following filters is/are approved for the determination of non-filterable (suspended) and filterable (dissolved) residues?
   a. asbestos
   b. paper fibers
   c. glass fibers
   d. membrane
   e. paper mat

31. The drying temperature specified for NPDES determinations of total and non-filterable (suspended) residues is
   a. 103-105°C
   b. 180°C
   c. 550°C

32. The NPDES methods for residue (solids) determinations would be classed as
   a. automated.
   b. gravimetric analysis
   c. meter determinations
   d. photometric colorimetry.

33. Before the final weight is calculated for suspended solids,
   a. only one weighing must be carried out after heating.
   b. only two weighings must be carried out after heating.
   c. a constant weight must be obtained after heating.
   d. agreement on results must be obtained with a turbidimeter.
   e. agreement on results must be obtained with a conductivity meter.

34. For NPDES or drinking water, turbidity is measured with
   a. an instrument containing a Wheatstone bridge.
   b. a reference solution to determine potential differences.
   c. a colorimeter.
   d. a nephelometer.
   e. a spectrophotometer.
35. Turbidity is a parameter of water quality interest because
   a. results are an accurate estimate of mg/L suspended solids.
   b. results are an accurate estimate of mg/L dissolved solids.
   c. results indicate possibility of interference for chlorine disinfection process.
   d. results are an index to the aesthetic quality of water.
   e. results indicate the efficiency of treatment processes.

36. Standardization of the instrument used to measure turbidity for NPDES or drinking water data involves the use of
   a. Formazin.
   b. Fuller's earth.
   c. a curve supplied by the manufacturer.
   d. a Jackson candle turbidimeter.
   e. Kaolin.

37. For NPDES, specific conductivity is measured with
   a. an instrument containing a Wheatstone bridge.
   b. a reference solution to determine potential differences.
   c. a colorimeter.
   d. a nephelometer.
   e. a spectrophotometer.

38. Specific conductivity is a parameter of water quality interest because
   a. results are an estimate of mg/L suspended solids.
   b. results are an estimate of mg/L dissolved solids.
   c. results are used to determine flow patterns.
   d. results are used to signal "dumps" into a plant influent.
   e. results are used to check the purity of distilled and deionized water.

39. Which of the following affect(s) the results for specific conductivity measurements?
   a. the cell constant
   b. the color of the sample
   c. the temperature
   d. the number and kind of ions in a sample
   e. the resistivity of sample components

40. ON THE ANSWER SHEET, list four titles (or the publishers) of books of chemical procedures which are recognized in the Federal Register as sources of nationally-tested analytical methods applicable to water samples.
<p>| | | | | | |</p>
<table>
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INORGANIC ANALYSIS IN WATER QUALITY PROGRAMS
EVALUATION

SCORING INFORMATION

There are 40 items on the test. Each item was scored as an entity and had a maximum value of 2½ points. Within each item, any wrong* answers were subtracted from the correct answers, and the remainder (zero was the minimum used) was given the following credit for the item:

1. For items requiring only one correct answer:
   a. a remainder of 1, 2½ points

2. For items requiring two correct answers:
   a. a remainder of 1, 1¼ points

3. For items requiring three correct answers:
   a. a remainder of 2 or 3, 2½ points
   b. a remainder of 1, 1¼ points

4. For items requiring four correct answers:
   a. a remainder of 3 or 4, 2¼ points
   b. a remainder of 2, 1¾ points
   c. a remainder of 1, no credit

5. For items requiring five correct answers:
   a. a remainder of 4 or 5, 2¼ points
   b. a remainder of 2 or 3, 1½ points
   c. a remainder of 1, no credit

*NOTE: Missing answers were not specifically counted. The scoring system is based on the total number of answers that should be given and thereby accounts for any missing answers.
1. a b c
2. a b c d (Must have d circled. If d plus others are circled, that's OK).
3. a b c d e (11/80, b is answer. Check if proposed d has been finalized).
4. a b c d e
5. a b c d e
6. a b c
d
7. a b c d e f
8. a b c d e
9. a b c d e
10. a b c d e
11. a b c d e
12. a b c d e
13. a b c d e
14. a b c d e
15. a b c d e f
16. a b c d e
17. a b c d e
18. a b c d e f
19. a b c d e
20. a b c d e
21. a b c d e f
22. a b c d e
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24. a b c d e f
25. a b c d e f
26. a b c d e f g
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39. a b c d e f g
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41. a b c d e f g
42. a b c d e f g
43. a b c d e f g
44. a b c d e f g
45. a b c d e f g
46. a b c d e f g
47. a b c d e f g
48. a b c d e f g
49. a b c d e f g
50. a b c d e f g
Methods for Chemical Analysis of Water and Wastes
or EPA

b. Standard Methods for the Examination of Water and Wastewater
or Standard Methods
or APHA-AWWA-WPCF

c. Annual Book of ASTM Standards (Part 31)
or ASTM

d. U.S. Geological Survey Techniques of Water-Resources Investigations

or Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases

or USGS
Our current policy is not to award continuing education units for the course, Inorganic Analyses in Water Quality Control Programs, January 9-13, 1978 unless a score of 50 or above on the pre-course evaluation is increased by 20 points (or more to a maximum required final score of 90) on the post-course evaluation. Your scores showed an increase of 17 points, so the units are not recorded on your certificate.

The course evaluation is our objective basis for awarding continuing education units for the course. However, we realize that each person who attends has an individual background of interests and individual goals to be achieved. The evaluation items do not necessarily measure the accomplishment of these individual goals nor all the improvements of knowledge and skills derived by each person in the class.

Your effort and interest in all the course activities indicated that you must have been successful in achieving your personal goals. It was really a pleasure to have you in the class and we hope you will let us know if we can be of further service to you.

Sincerely yours,

Name
Course Coordinator
National Training and Operational Technology Center

Enclosure - Certificate
Dear [Name],

Our current policy is not to award continuing education units or course certificates to those who do not attend at least 95% of the lecture/laboratory sessions.

This policy will apply to you, since your illness prevented your participation in the Tuesday and Wednesday activities of the course, Inorganic Analyses in Water Quality Control Programs, January 9-13, 1978.

I would, however, like to acknowledge your attendance and full participation in the other three days of the course. It was really a pleasure to have you in the class and we hope you will let us know if we can be of further service to you.

Sincerely yours,

[Name]

Course Coordinator
National Training and Operational Technology Center
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Sample Handling - Field Through Laboratory

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 30 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: A basic element of quality assurance for chemical analytical data is a reliable and valid sample, properly preserved, stored and documented. An analyst must be knowledgeable about sample handling to carry out his/her responsibilities.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of inorganic chemistry

B. Though not essential, it would aid comprehension/retention of information if the participant has some experience in chemical laboratory work involving water samples.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will have an overview of all the elements of a program to assure a valid, properly preserved sample, whose integrity is documented from collection to storage.

B. Conditions: He/she will be given the training manual, handouts (VII. A. 4. and 5.) regarding sample preservation techniques, and a 30 minute discussion of the elements involved.

C. Accepted Performance: He/she must participate in the discussion and correctly answer 70% of the items on this topic in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the training manual, "Sample Handling - Field Through Laboratory".

2. (Optional) Copies for display of the standard reference books of methods listed in the training manual outline section, "VI. Methods of Analysis".

3. Eight slides, X-17: Sample Handling, listing items from divisions of the outline in the training manual. (See XI Description of Visual Materials).

4. Handout: The preservation procedures for samples collected for National Pollutant Discharge Elimination System or State...
Certification report purposes are to be finalized in the Federal Register sometime after January, 1981. To ensure that you distribute current information, contact your EPA Regional Quality Assurance Coordinator for copies of the appropriate information.

5. Handout: The preservation procedures specifically designed for drinking water samples have been published in the U.S. EPA Report No. EPA 600/8-78-008, May, 1978. A copy of the applicable information is at the end of this SPW.

B. Suggested-Media:
1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:
1. Review visual materials and available handouts.
2. Prepare the lesson using the Sequencing below or your own organization of the material.
3. Duplicate copies of any handout materials.

B. Sequencing:
1. Follow the outline in the training manual for discussion. Participants should use the outline, too. The slides, X-17, Sample Handling, can be used to guide the discussion according to the divisions in the training manual outline. The divisions are referenced in the following:
   a. I Planning a Sample Program
      1) A. Discuss the factors to consider - slide 1
         Define grab and composite samples - slide 2
         B. Decisive Criteria for making decisions should be woven into the discussion.
      b. II Representative Samples - Associated factors - slide 3
      c. III Sample Identification - Topics for discussion - slide 4
      d. IV Containers - Considerations for discussion - slide 5
   e. V Preservation, A. Functions - slide 6
      B. General Methods - slide 7
      C. Specific Methods - Note sources of information
      D. Federal Register Methods - Distribute handouts and discuss
f. VI Names of Standard References for Methods - Display a copy as each title is given.
   E. Federal Register Methodology - Note that there are regulations for NPDES, State Certifications and Drinking Water. This topic is covered in detail in a separate lesson, Compliance Methodology.

   g. VII Order of Analyses - Based on holding times which depend on factors listed on slide 8.
      A. Give examples of different holding times
      B. Give sources of information

   h. VIII Record Keeping
      A. Field - kind of information
      B. Lab - kind of information

   i. IX Summary (as in outline)

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

   A. For each student:
      1. One copy of handout material

X. IPW REAGENT REQUIREMENTS:

   A. None

XI. DESCRIPTION OF VISUAL MATERIALS:

   A. Eight slides, X-17: Sample Handling - See next page.
COLLECTION OF SAMPLES
- SAMPLING SITE
- EQUIPMENT
- QUANTITY
- TYPE
- PRESERVATION.

TYPES OF SAMPLES
- GRAB SAMPLE - INDICATES CONDITION AT TIME OF SAMPLING
- COMPOSITE SAMPLE - INDICATES AVERAGE CONDITION OVER COMPOSITING PERIOD

REPRESENTATIVE SAMPLES
- CLEAN CONTAINER
- COMPOSITING SAMPLES
- PROPER EQUIPMENT
- SLUDGE OR MUD

SAMPLE IDENTIFICATION
- PROPER TAG
- USEFUL IDENTIFICATION INFORMATION
- SAMPLE CUSTODY

SAMPLE CONTAINERS
- AVAILABLE MATERIALS
- CHEMICAL AND PHYSICAL INTERACTIONS
- MAILING REQUIREMENTS
- PRELIMINARY CHECK
- CLEANING
- STORAGE

SAMPLE PRESERVATION
- RETARD BIOLOGICAL ACTION
- RETARD PRECIPITATION OR HYDROLYSIS
- REDUCE VOLATILITY

GENERAL METHODS OF SAMPLE PRESERVATION
- PH CONTROL
- CHEMICAL ADDITION
- REFRIGERATION OR FREEZING

TIME INTERVAL BETWEEN COLLECTION AND ANALYSIS
- DEPENDS ON SAMPLE TYPE
- STABILITY
- STORAGE CONDITIONS
### Table IV.2 — Sample collecting, handling, and preservation

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Preservative</th>
<th>Container</th>
<th>Maximum holding time</th>
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<tr>
<td>Arsenic</td>
<td>Conc HNO₃ to pH&lt;2</td>
<td>P or G</td>
<td>6 months</td>
</tr>
<tr>
<td>Barium</td>
<td>Conc HNO₃ to pH&lt;2</td>
<td>P or G</td>
<td>6 months</td>
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<tr>
<td>Cadmium</td>
<td>Conc HNO₃ to pH&lt;2</td>
<td>P or G</td>
<td>6 months</td>
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<tr>
<td>Chromium</td>
<td>Conc HNO₃ to pH&lt;2</td>
<td>P or G</td>
<td>6 months</td>
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<td>Lead</td>
<td>Conc HNO₃ to pH&lt;2</td>
<td>P or G</td>
<td>6 months</td>
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<td>Mercury</td>
<td>Conc HNO₃ to pH&lt;2</td>
<td>P</td>
<td>38 days</td>
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<tr>
<td>Nitrate</td>
<td>Conc H₂SO₄ to pH&lt;2</td>
<td>P or G</td>
<td>14 days</td>
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<td>Selenium</td>
<td>Conc HNO₃ to pH&lt;2</td>
<td>P or G</td>
<td>6 months</td>
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<td>Silver</td>
<td>Conc HNO₃ to pH&lt;2</td>
<td>P or G</td>
<td>6 months</td>
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<tr>
<td>Fluoride</td>
<td>None</td>
<td>P or G</td>
<td>1 month</td>
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<tr>
<td>Chlornated hydrocarbons</td>
<td>Refrigerate at 4° C as soon as possible</td>
<td>G with foil- or Teflon-lined cap</td>
<td>14 days⁵</td>
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<tr>
<td>Chlorophenoxys</td>
<td>Refrigerate at 4° C as soon as possible</td>
<td>G with foil- or Teflon-lined cap</td>
<td>7 days⁵</td>
</tr>
</tbody>
</table>

¹If a laboratory has no control over these factors, the laboratory director must reject any samples not meeting these criteria and notify the authority requesting the analyses.

²If HNO₃ cannot be used because of shipping restrictions, sample may be initially preserved by icing and immediately shipping it to the laboratory. Upon receipt in the laboratory, the sample must be acidified with conc HNO₃ to pH<2. At time of analysis, sample containers should be thoroughly rinsed with 1:1 HNO₃, washings should be added to sample.

³P = Plastic, hard or soft; G = Glass, hard or soft

⁴In all cases, samples should be analyzed as soon after collection as possible.

⁵Well-stoppered and refrigerated extracts can be held up to 30 days.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Compliance Methodology

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 70 minutes (60 + 10)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE. A basic element of quality assurance for chemical analytical data is use of a recognized analytical procedure. In addition, federal regulations affect the final choice of methodology for compliance samples. An analyst must know about these regulations to fulfill the attendant responsibilities.

V. ENTRY LEVEL BEHAVIOR:

A: Familiarity with fundamentals of chemical analytical methods

B. Though not essential, it is orderly to have completed the lesson (or equivalent):

1. Sample Handling

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior. The participant will be able to use Federal Registers and pertinent EPA explanatory memos to choose approved analytical methodology for compliance with the National Pollutant Discharge Elimination System and the National Interim Primary Drinking Water Regulations.

B. Conditions: He/she will be given the training manual, copies of the pertinent information (See V A), a 60 minute explanation of their applicability and format and 10 minutes at a later time to resolve any questions.

C. Accepted Performance: Satisfactory according to an Instructor rating of attention and use of the materials as directed during the discussion. He/she must also correctly answer 70% of the items on this topic in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in the training manual:

   a. "Methodology for Chemical Analysis of Water and Wastewater" is a glossary of the methods promulgated for obtaining compliance data.
2. Handouts:

a. National Pollutant Discharge Elimination System - NPDES:

1) Guidelines Establishing Test Procedures. 40 CFR 136, Federal Register, 12/1/76, pp 52780-52786. A copy is at the end of this IPW, and also an errata sheet. Changes were proposed 12/18/79, pp 75028-75052 and should be finalized after January, 1981. Check with your EPA Regional Quality Assurance Coordinator to see if the finalized Guidelines have been published. (Name and number is available from EMSL-Cincinnati, 513/684-7301). The Coordinator can supply copies to you.

2) Summary of protocols to apply for approval of an alternative test procedure for regulatory purposes. A copy is at the end of this IPW.

3) Federal Register notices of alternative methods approved to date (8/80) are at the end of this IPW. Contact your EPA Regional Quality Assurance Coordinator for copies of notices published since then.

4) EPA-EMSL memo on "Use of Chemical Test Kits for Compliance Monitoring." A copy is at the end of this IPW.

5) EPA-EMSL memo on "Use of "Prepared" Reagents in NPDES Compliance." A copy is at the end of this IPW.

b. National Interim Primary Drinking Water Regulations - NIPDWR:

1) NIPDWR, 40 CFR 141, Federal Register, 12/24/75, pp 59566-59574. A copy is at the end of this IPW.

2) Amendments to NIPDWR were proposed 7/19/79, 40 CFR 141. The amendments were finalized 40 CFR Part 141, Federal Register, 8/27/80, pp 57332-57346. Copies of pp 57344-57346 are at the end of this IPW. The pages contain the current (8/80), approved analytical requirements for drinking water analysis. These include an update of the approved methods presented in the 12/24/75 NIPDWR and also alternate analytical techniques approved by EPA since that date. Check with your EPA Regional Quality Assurance Coordinator to get copies of any additional pertinent Federal Register notices published after 8/80.

c. National Secondary Drinking Water Regulations - NSDWR:

1) NSDWR, 40 CFR 143, Federal Register, pp 42196-42199. A copy is at the end of this IPW.

B. Suggested Media:

1. OPTIONAL: Copies of the methods manuals cited for NPDES and NIPDWR could be available for display/inspection.
VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Contact your EPA Regional Quality Assurance Coordinator to check if any Federal Registers pertaining to NPDES or NIPDWR have been issued since the ones referenced in VII. A. above (8/80). If so, obtain copies from the Coordinator for your class.

2. Thoroughly review all pertinent Federal Register information and handout materials. Since the regulations are legally binding, it is imperative to impart correct and current information to the participants.

3. Prepare the lesson using the Sequencing below or your own organization of the materials.

4. Duplicate copies of all the handout materials. Most can be copied back-to-back, but be careful to keep each handout as an entity so participants can use each independent of the other. Collate the handouts into a packet for each participant to minimize distribution time.

B. Sequencing:

1. Hand out the packets of information.

2. Introduction
   a. Choice of methodology affects validity and reliability of data.
   b. Analytical programs affected by federal legislation include those involving point source discharges (NPDES) and drinking water (NIPDWR).


4. Overview of Table 1
   1) Column 1: Alphabetical listing of parameters and units. Note subcategories.
   2) Column 2: Method listings and possibly specification of a pretreatment and/or method choice, e.g. acidity end point is to be pH of 8.2. A summary of the principles involved in the listed methodology can be found in the training manual outline, "Methodology for Chemical Analysis of Water and Wastewater." You can use the outline to list the types of methodology found in the Table. Note that many automated methods are approved.
3) Remaining columns list sources (with page numbers) of approved methods. You can display copies of the cited manuals at this time and leave them to be available for inspection by participants.

4) Note the location (by number) of the parameters to be taught during the course. Also note the method to be used for course laboratory sessions.

5) Note any errata (handout).

c. Go over highlights of Applying for Approval of Alternative Test Procedures (handout).

1) Limited use
2) Nationwide use
d. Go through the Federal Register notices of alternative methods approved for nationwide use, briefly noting the contents.
e. Go over highlights of the memo on "Use of Chemical Test Kits for Compliance Monitoring" (handout).
f. Note highlights of the memo on "Use of "Prepared" Reagents in NPDES Compliance" (Handout).

4. Drinking Water Regulations. CAUTION: Use the current Federal Registers. The 12/24/75 Federal Register is the current (8/80) National Interim Primary Drinking Water Regulation (NIPDWR), along with the Amendments to NIPDWR which were finalized 8/27/80.

b. Maximum Contaminant Levels are listed in 12/24/75 for Chemicals on page 59570 and for Turbidity on page 59571.
c. The analytical methodology in the 12/24/75 regulation was updated and expanded in the 8/27/80 regulation. Therefore, use pp. 57344-57346 from the 8/27/80 Federal Register to cite the location of the listings of approved methods: 141.22 Turbidity, 141.23 Inorganics, 141.24 Organics, 141.41 Sodium, 141.42 Corrosivity Characteristics. Also highlight section 141.28, Approved Laboratories, and section 141.27, Alternate Analytical Techniques. (Alternate methods approved after the original NIPDWR (12/24/75) are included in the sections just cited).
d. There are "National Secondary Drinking Water Regulations" which were finalized in the 7/19/79 Federal Register on pp. 42198-42199. These represent reasonable goals for the aesthetic quality of drinking water but are not to be federally enforceable. Suggested Maximum Contaminant Levels and Analytical Methodology are presented on these pages. 
5. A 10 minute discussion period should be scheduled later to answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:
   A. For each student:
      1. Copies of handout materials

X. IPW REAGENT REQUIREMENTS:
   A. None
Guidelines Establishing Test Procedures for the Analysis of Pollutants

PART II:

ENVIRONMENTAL PROTECTION AGENCY

WATER PROGRAMS

WEDNESDAY, DECEMBER 1, 1976
### RULES AND REGULATIONS

#### Parameter and units

<table>
<thead>
<tr>
<th>Parameter and units</th>
<th>Method</th>
<th>EPA methods referred to</th>
<th>Other methods referred to</th>
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<td>1. Chlorinated organic compounds (except pesticides), milligrams per liter</td>
<td>Gas chromatography</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2. Chlorine—Total residual, milligrams per liter</td>
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<td></td>
<td></td>
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<tr>
<td>3. Chromate, total, milligrams per liter</td>
<td></td>
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<td></td>
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<tr>
<td>4. Cyanide, total, milligrams per liter</td>
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<tr>
<td>5. Fluoride, milligrams per liter</td>
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<tr>
<td>6. Hydrogen ion (pH), pH units, milligrams per liter</td>
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<tr>
<td>7. Sulfate, total, milligrams per liter</td>
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<tr>
<td>8. Ammoniacal nitrogen, milligrams per liter</td>
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<tr>
<td>9. Ammonium, dissolved, milligrams per liter</td>
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<tr>
<td>10. Bicarbonate, dissolved, milligrams per liter</td>
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<tr>
<td>11. Carbon dioxide, total, milligrams per liter</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>12. Calcium, total, milligrams per liter</td>
<td></td>
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<tr>
<td>13. Carbon steel, total, milligrams per liter</td>
<td></td>
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<tr>
<td>14. Chemical oxygen demand, milligrams per liter</td>
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<tr>
<td>15. Chemical oxygen demand, biological, milligrams per liter</td>
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<tr>
<td>16. Chemical oxygen demand, nitrogen, milligrams per liter</td>
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<tr>
<td>17. Chemical oxygen demand, phosphorus, milligrams per liter</td>
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</table>

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**FEDERAL REGISTER, VOL. 41, NO. 233—WEDNESDAY, DECEMBER 1, 1976**

10-9
<table>
<thead>
<tr>
<th>Substance</th>
<th>Concentration Unit</th>
<th>Method of Analysis</th>
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<tbody>
<tr>
<td>Calcium</td>
<td>Milligrams per Liter</td>
<td>Calcium absorption</td>
</tr>
<tr>
<td>Sodium</td>
<td>Milligrams per Liter</td>
<td>Sodium absorption</td>
</tr>
<tr>
<td>Chloride</td>
<td>Milligrams per Liter</td>
<td>Chloride absorption</td>
</tr>
<tr>
<td>Magnesium</td>
<td>Milligrams per Liter</td>
<td>Magnesium absorption</td>
</tr>
<tr>
<td>Potassium</td>
<td>Milligrams per Liter</td>
<td>Potassium absorption</td>
</tr>
<tr>
<td>Copper</td>
<td>Milligrams per Liter</td>
<td>Copper absorption</td>
</tr>
<tr>
<td>Zinc</td>
<td>Milligrams per Liter</td>
<td>Zinc absorption</td>
</tr>
<tr>
<td>Iron</td>
<td>Milligrams per Liter</td>
<td>Iron absorption</td>
</tr>
<tr>
<td>Lead</td>
<td>Milligrams per Liter</td>
<td>Lead absorption</td>
</tr>
<tr>
<td>Mercury</td>
<td>Milligrams per Liter</td>
<td>Mercury absorption</td>
</tr>
<tr>
<td>Arsenic</td>
<td>Milligrams per Liter</td>
<td>Arsenic absorption</td>
</tr>
</tbody>
</table>

Methodology for total analysis:
- Calcium: Dissolved, milligram total.
- Sodium: Dissolved, milligram total.
- Chloride: Dissolved, milligram total.
- Magnesium: Dissolved, milligram total.
- Potassium: Dissolved, milligram total.
- Copper: Dissolved, milligram total.
- Zinc: Dissolved, milligram total.
- Iron: Dissolved, milligram total.
- Lead: Dissolved, milligram total.
- Mercury: Dissolved, milligram total.
- Arsenic: Dissolved, milligram total.

Other analytical methods followed by referenced methods.
RULES AND REGULATIONS

Parameter and units

<table>
<thead>
<tr>
<th>Parameter and units</th>
<th>Method</th>
<th>Reference</th>
<th>Other suggested methods</th>
</tr>
</thead>
<tbody>
<tr>
<td>78. Thallium—Total, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
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<tr>
<td>79. Thallium—Dissolved, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
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<tr>
<td>80. Total, milligrams per liter</td>
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<tr>
<td>81. Thallium—Dissolved, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
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<tr>
<td>82. Total, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
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<tr>
<td>83. Vanadium—Total, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
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<tr>
<td>84. Vanadium—Dissolved, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
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<tr>
<td>85. Vanadium—Total, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
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<tr>
<td>86. Vanadium—Dissolved, milligrams per liter</td>
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<td>87. Zinc—Dissolved, milligrams per liter</td>
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<tr>
<td>88. Zinc—Total, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
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<tr>
<td>89. Nitrate, milligrams per liter</td>
<td>Reduction, hydride generation and atomic absorption</td>
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<tr>
<td>90. Arsenic, milligrams per liter</td>
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<tr>
<td>91. Uranium, total (PU), milligrams per liter</td>
<td>Reduction—indirect</td>
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<tr>
<td>92. Uranium, total (UE), milligrams per liter</td>
<td>Reduction—direct</td>
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<td>93. Phosphorus, total, milligrams per liter</td>
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<td>94. Carbon, total, milligrams per liter</td>
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<td>95. Chloride, milligrams per liter</td>
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<td>96. Fluoride, milligrams per liter</td>
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<td>97. Phosphorus, elemental, milligrams per liter</td>
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<td>98. Phosphorus total as P</td>
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<td>99. Alkalinity, milligrams per liter</td>
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<td>100. Alumina, milligrams per liter</td>
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<td>101. Calcium, milligrams per liter</td>
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<td>102. Chloride, milligrams per liter</td>
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<td>103. Copper, milligrams per liter</td>
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<td>104. Fluoride, milligrams per liter</td>
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<td>105. Iron, milligrams per liter</td>
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<tr>
<td>106. Lead, milligrams per liter</td>
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<tr>
<td>107. Magnesium, milligrams per liter</td>
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<td>108. Manganese, milligrams per liter</td>
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<td>109. Nickel, milligrams per liter</td>
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<td>110. Nitrate, milligrams per liter</td>
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<tr>
<td>111. Oxygen, milligrams per liter</td>
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<td>112. Phosphorus, milligrams per liter</td>
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<td>113. Potassium, milligrams per liter</td>
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<tr>
<td>114. Sodium, milligrams per liter</td>
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<tr>
<td>115. Turbidity, NTU</td>
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</tbody>
</table>

References

*Recommended for sampling and preservation of samples according to parameter measured may be found in 'Methods for Chemical Analysis of Water and Waste 1971' U.S. Environmental Protection Agency, Table 2, pp. 44-111.
§ 1365 [Amended]

5 In § 1365, paragraph (a) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "test procedure" and before the period that ends the paragraph.

6 In § 1365, paragraph (b) is amended by deleting the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "such application" and immediately before the comma.

The second sentence of paragraph (b) is amended by deleting the phrase "Methods Development and Quality Assurance Research Laboratory" immediately after the phrase "for the "State Permit Program" and to the Director of the " at the end of the sentence, and inserting in its place the phrase "Environmental Monitoring and Support Laboratory, Cincinnati."

7 In § 1365, paragraph (c) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "such application for an alternate test procedure and immediately before the comma and by deleting the phrase "Methods Development and Quality Assurance Laboratory" immediately after the phrase "for review and recommendation" and inserting in its place the phrase "for review and recommendation and immediately before the phrase for the "Environmental Monitoring and Support Laboratory, Cincinnati."

8. In § 1365, the first sentence of paragraph (d) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge," immediately after the phrase, "application for an alternate test procedure, and immediately before the comma.

The second sentence of paragraph (d) is amended by deleting the phrase "Methods Development and Quality Assurance Research Laboratory, immediately after the phrase, "to the Regional Administrator by the Director of the," and immediately preceding the period ending the sentence and inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."

The third sentence of paragraph (d) is amended by deleting the phrase "Methods Development and Quality Assurance Research Laboratory, immediately after the phrase, "forwarded to the Director," and immediately before the second comma and by inserting in its place the phrase, "Environmental Monitoring and Support Laboratory, Cincinnati."
§ 136.5 Approval of alternate test procedures.

(e) Within ninety days of the receipt by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati of an application for an alternate test procedure for nationwide use, the Director of the Environmental Monitoring and Support Laboratory, Cincinnati shall notify the applicant of his recommendation to the Administrator to approve or reject the application, or shall specify additional information which is required to determine whether to approve the proposed test procedure. After such notification, an alternate method determined by the Administrator to satisfy the applicable requirements of this part shall be approved for nationwide use to satisfy the requirements of this subchapter; alternate test procedures determined by the Administrator not to meet the applicable requirements of this part shall be rejected. Notice of these determinations shall be submitted for publication in the Federal Register not later than 15 days after such notification and determination is made.

[FR Doc 76-35082 Filed 11-30-76; 8:45 am]
PART 136--GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Amendment of Regulations; Corrections*

<table>
<thead>
<tr>
<th>Page</th>
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<th>Correction or Addition</th>
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<tbody>
<tr>
<td>52784</td>
<td>89</td>
<td>Parameter and units, change &quot;Nitrate&quot; to &quot;Nitrite&quot;</td>
</tr>
<tr>
<td>52784</td>
<td>96, Phenols</td>
<td>Delete the present method designation, &quot;Colorimetric, (4 AAP),&quot; and replace it with the method designation, &quot;Distillation followed by colorimetric, (4 AAP).&quot;</td>
</tr>
<tr>
<td>52784</td>
<td>96, Phenols</td>
<td>14th ed. Standard Methods change &quot;582&quot; to &quot;574&quot;</td>
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Amendment; Correction**

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<thead>
<tr>
<th>Page</th>
<th>Parameter Number</th>
<th>Correction or Addition</th>
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</thead>
</table>

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*Federal Register, Volume 42, No. 12, Tuesday, January 18, 1977, pp. 3306-3307

**Federal Register, Volume 42, No. 139, Wednesday, July 20, 1977, p. 37205
NPDES/CERTIFICATIONS
ALTERNATIVE TEST PROCEDURES FOR LIMITED USE

TOPIC: Application and approval for individual permit-holders to use alternate test procedures (alternate to those published in the Federal Register for NPDES/certification purposes) for specific discharges.

SOURCE: Federal Register, Title 40, Chapter I, Subchapter D, Part 136:
- Vol 38, No. 199, October 16, 1973
- Vol 41, No. 232, December 1, 1976

A. Application for Alternate Test Procedures

The responsible person or firm making the discharge applies to the EPA Regional Administrator (RA) in the Region where the discharge occurs, through the Director of the State agency having permit-issuing authority. If the state does not issue permits, the application is sent directly to the EPA RA. One must:

1. Provide identifying information; i.e., name, address, permit number, etc.
2. Identify the pollutant or parameter involved.
4. Provide a detailed description of the proposed procedure with references regarding the applicability to the effluents in question.

B. Approval of Alternate Test Procedures

An EPA Regional Administrator (RA) has the final responsibility for approval.

1. The State Director conducts a technical and administrative review and forwards the application and his recommendation to the RA.
2. The RA conducts a technical and administrative review.
   a. If the State Director recommended rejection for scientific and technical reasons, the RA denies the application and sends a copy of the rejected application and his decision to the applicant, the State Director and to the Director of the Environmental Monitoring and Support Laboratory (EMSL).
   b. Before approving any application, the RA sends a copy of the application to the Director of EMSL for review and recommendation.
3. Prior to 90 days of receipt of the application by the RA, the Director of EMSL forwards to the RA a recommendation providing the scientific and other-technical basis for acceptance or rejection of the application.
4. Within 90 days of receipt, the RA notifies the applicant and the appropriate State agency of approval or rejection, or else specifies additional information required for the decision.
5. A copy of all approval and rejection notifications are sent to EMSL for purposes of national coordination.
NPDES/CERTIFICATIONS
ALTERNATIVE TEST PROCEDURES FOR NATIONWIDE USE

TOPIC: Application and approval by any person, laboratory, manufacturer, etc., for nationwide use of an alternate test procedure (alternate to those published in the Federal Register for NPDES/Certification purposes).


A. Application for Alternate Test Procedures

Any interested person, laboratory, manufacturer, etc., applies to the Director, Environmental Monitoring and Support Laboratory (EMSL), Cincinnati, Ohio 45268. One must:

1. Provide identifying information, i.e., name and address of the responsible person or firm.
2. Identify the pollutant(s) or parameter(s) involved.
3. Provide a detailed description of the proposed procedure with references regarding its applicability.
4. Provide comparability data (proposed procedure compared to procedure published in the Federal Register).

B. Approval of Alternate Test Procedures

The Administrator of the U.S. Environmental Protection Agency has the final responsibility for approval.

1. The Director of EMSL conducts a technical review.
2. Within 90 days of receipt, the Director of EMSL notifies the Administrator of his recommendation to approve or reject the application, or else returns the application for additional information required for the decision.
3. After notification of the EMSL recommendations, the Administrator determines whether or not the alternate test procedures meet the requirements set forth in the Federal Register, i.e., whether the procedures are to be approved or rejected.
4. Within fifteen days of the notification and determination, notice of the final decision is submitted to the Federal Register for publication.
In accordance with section 136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (Federal Register, Vol. 41, No. 233, Wednesday, December 1, 1976, pp. 52760-52786), the Oceanography International Corp. is approved for approval of a new test procedure for the measurement of COD. The new procedure incorporates a modification of the digestion technique of the approved procedure in that it uses a semimicro volume of sample and reagents, sealed in a glass ampule, with reflux digestion performed in an oven.

As an approved alternate COD procedure, the Oceanography International COD procedure is acceptable for use by any person required to use approved procedures under §136.3 of the Federal Water Pollution Control Act Amendments of 1972. For such use, the procedure must be used in strict accordance with the methods descriptions. The approved methods descriptions are available for the standard, low value, and high chloride ampule COD procedures from Oceanography International Corp., P.O. Box 2060, 612 West Loop, College Station, Tex. 77840.

Additional information concerning this action may be obtained by writing to the Director, Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45268.


Stephen J. Gage, Acting Assistant Administrator for Research and Development.
Notice Approval of Alternate Water Pollutant Testing Procedure; Nitrite/Nitrogen

In accordance with §136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (Federal Register, Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780-52788), the Hach Chemical Company applied for approval of a new test procedure for the measurement of nitrite/nitrogen. The new Hach procedure is a diazotization method using NitriVer III nitrite reagent.

After a thorough review and evaluation by the Environmental Protection Agency (EPA) of the results of a comparability testing study and other information submitted by the applicant in accordance with §136.5, the EPA has designated the Hach procedure as an approved alternate procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection to the extent consistent with 40 CFR Part 2 (EPA's regulations implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 28 West St Clair Street, Cincinnati, Ohio 45288.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under §304(h) of the Clean Water Act Amendments of 1977. For such use, the procedure must be used in strict accordance with the method descriptions. The approved method description and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: April 24, 1979.

Robert L. Cook.
Assistant Administrator for Research and Development.

FOR: 136.5-4

FR Doc. 79-1352 Filed 4-20-79 8:45 am]
BILLING CODE 6560-51-M

Federal Register / Vol. 44, No. 85

Tuesday, May 1, 1979

Approval of Alternate Water Pollutant Testing Procedure; Total Manganese

In accordance with §136.5, 40 CFR Part 136, "Guidelines for Test Procedures for the Analysis of Pollutants" (Federal Register, Vol. 41, No. 232, Wednesday, December 1, 1976, pp. 52780-52788), the Hach Chemical Company applied for approval of a new test procedure for the measurement of manganese. The new Hach procedure is a colorimetric procedure using prepackaged reagents based upon the same chemical reactions involved in the approved colorimetric procedure referenced in 40 CFR Part 136.

After a thorough review and evaluation by the Environmental Protection Agency (EPA) of the results of a comparability testing study and other information submitted by the applicant in accordance with §136.5, the EPA has designated the Hach procedure as an approved alternate procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection to the extent consistent with 40 CFR Part 2 (EPA's regulations implementing the Freedom of Information Act), at the Environmental Monitoring and Support Laboratory, 28 West St Clair Street, Cincinnati, Ohio 45288.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under §304(h) of the Clean Water Act Amendments of 1977. For such use, the procedure must be used in strict accordance with the method descriptions for manganese, periodate oxidation method, "Wastewater Analysis Handbook," Hach Chemical Company, p. 275 and p. 281.

For the analysis of total manganese, addition of acid and digestion as indicated in Footnote 15 of Federal Register, Vol. 41, No. 232, Wednesday, December 1, 1976, p. 52785, and §4.1.4, p. 83 of "Methods for Chemical Analysis of Water and Waste," USEPA, 1974, is the required sample pretreatment. This approved alternate test procedure for total manganese is approved also for the determination of dissolved manganese after sample filtration through a 0.45 micron filter.

The approved method description and prepackaged reagents are available from the Director of Technical Information, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

Dated: June 4, 1979.

Tom Murphy,
Acting Assistant Administrator for Research and Development.

[FR Doc. 79-1352 Filed 4-20-79 8:45 am]
BILLING CODE 6560-51-M

Federal Register / Vol. 44, No. 118

Thursday, June 14, 1979
Approval of Alternate Water Pollutant Testing Procedure; Chemical Oxygen Demand (COD)

In accordance with § 136.5, 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants," the Hach Chemical Company applied for approval of an alternate test procedure for the measurement of COD. The new procedure incorporates a modification of the digestion technique of the approved procedure in that it uses a semi-micro volume of sample and reagents in a capped vial with reflux digestion performed in an oven.

After a thorough review and evaluation by the U.S. Environmental Protection Agency (EPA) of the results of comparability testing studies and other information submitted by the applicant, in accordance with § 136.5, the EPA has designated the Hach procedure as an approved alternate test procedure for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act) at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45288.

As an approved alternate test procedure, the Hach procedure is acceptable for use by any person required to use approved procedures under § 304[h] of the Clean Water Act Amendments of 1977. For such use, the procedures must be used in strict accordance with the method description: Oxygen Demand, Chemical Reaction Digestion Method, Water Analysis Handbook, pp. 2-168-2-170, 1979 edition.

The approved method descriptions and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: April 14, 1980.
Stephen J. Gage,
Assistant Administrator for Research and Development.

ENVIRONMENTAL PROTECTION AGENCY

Approval of Alternate Water Pollutant Testing Procedures; Total and Dissolved Zinc and Copper

In accordance with § 136.5, 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants," the Hach Chemical Company applied for approval of alternate test procedures for the measurement of total and dissolved zinc and copper.

After a thorough review and evaluation by the United States Environmental Protection Agency (EPA) of the results of comparability testing studies and other information submitted by the applicant, in accordance with § 136.5, the EPA has designated the Hach procedures as approved alternate test procedures for nationwide use. All information submitted by the applicant is on file and available for public inspection, to the extent consistent with 40 CFR Part 2 (EPA's regulation implementing the Freedom of Information Act) at the Environmental Monitoring and Support Laboratory, 26 West St. Clair Street, Cincinnati, Ohio 45288.

As approved alternate test procedures, the Hach procedures are acceptable for use by any person required to use approved procedures under § 304[h] of the Clean Water Act Amendments of 1977. For such use, the procedures must be used in strict accordance with the method descriptions for copper: Bicinchoninate Method, Method 8508; and for zinc: Zincon Method, Method 8009. In making this approval, the EPA disassociates itself from any statements in the approved Hach Chemical Company procedures which do not specifically pertain to the determination of analytical equivalency.

The approved method descriptions and prepackaged reagents are available from the Hach Chemical Company, Post Office Box 389, Loveland, Colorado 80537.

Dated: May 21, 1980.
Stephen J. Gage,
Assistant Administrator for Research and Development.

Federal Register / Vol. 45, No. 105
Thursday, May 29, 1980
DATE: September 20, 1978

SUBJECT: Use of Chemical Test Kits for Compliance Monitoring

FROM: Dwight G. Ballinger, Director
Environmental Monitoring and Support Laboratory - Cincinnati

TO: Regional Quality Assurance Coordinators

A number of Regional Coordinators and Permit staff have received inquiries concerning the use of test kits for the parameters required by the Effluent Guidelines and Compliance Monitoring Sections of PL 92-500. This memorandum defines the position of EMSL on these test methods and contains recommendations for regional and state response to inquiries concerning acceptance.

Our interpretation of Section 304(g), implemented as "Guidelines Establishing Test Procedures for Analysis of Pollutants," in Federal Register, Dec. 1, 1976, is that the test kits are not equivalent to the procedures promulgated in the Federal Register and therefore are subject to the requirements governing alternate procedures. Therefore, it is necessary for the applicant to request the use of these test kits from the appropriate Regional Administrator as presented in the regulations. This recommendation is based upon the following factors:

SAMPLE PREPARATION

Most of the procedures selected for implementation of Section 304(g) are designed to measure the total constituent or element present in the sample or are specifically modified to determine a precisely defined form of the substance. The test kits considered herein generally do not utilize the necessary digestion or pretreatment required to measure the total constituent. In many cases, such as preliminary digestion with acids or distillations, these pretreatments cannot be performed satisfactorily under field conditions. Thus, the final result reported, when a kit is used, is nearly always less than the true value and acceptance of such data will lead to wrong interpretation of effluent loadings.
INTERFERENCES

It should be recognized that a majority of these test kit measurement techniques were developed for field use on domestic water supplies or relatively clean ambient streams and are not designed to provide for removal of interferences often encountered in municipal and industrial waste effluents.

MEASUREMENT SYSTEMS

In some cases the physical and chemical measurement principles differ from those employed in the reference methods. In addition, proprietary reagents of unknown composition are often provided with little or no information available on the reactions involved. Even though some of the tests are "based on Standard Methods," a number of factors rule out the acceptance of the test procedures as equivalent.

REAGENTS

In most cases the reagent concentrations are not the same as those described in the reference method. Volumes are generally pre-measured under unknown conditions and addition to the sample is often by means of inaccurate droppers or pipets. In short, the quality and quantity of reagents is not under the direct control of the analyst, as required by good analytical technique.

CALIBRATION AND COLOR MEASUREMENT

In most test kits a comparatively simple photometer is provided or available light is used to measure color intensity. Calibration scales are supplied by the manufacturer based upon factors developed under ideal conditions in his laboratory, and recalibration is difficult or not recommended. No provision is made for changes in reagent composition due to inadequate quality control in manufacturing or adverse storage conditions. The photometers usually available will not accept cells of sufficient path length to achieve the required sensitivity, and sealed standards incorporated in the kits are subject to changes in color with time. Finally, the band pass of these photometers is generally too wide for accurate measurement of the appropriate wavelength.

DATA REQUIREMENTS FOR ALTERNATE TEST PROCEDURES

When consideration of the above factors are not sufficient to reject an application for use of test kits, a request should be made for
comparative data upon which to judge the applicability of the alternative procedures. The attached protocol is recommended in developing the necessary information. The regulations require that the alternate procedures be used on the waste being monitored and that application be made to the Regional Administrator or State Director having jurisdiction over the permit issuance.

It is recognized that under unusual conditions the applicant may have difficulty obtaining comparative data because of lack of laboratory facilities at remote locations. Even in these cases, the applicant should be urged to obtain adequate data by having the necessary work done at a base laboratory or on contract. The acceptance of alternate procedures without supporting laboratory results may significantly weaken the pollution control efforts intended by PL 92-500.

The use of field test kits, as with other alternate procedures, should be considered on a case-by-case basis, with judgment based on all of the factors involved. No blanket acceptance of test methods will be recommended unless a large volume of data have been accumulated clearly showing that the use of the specific alternate procedure on a wide variety of sample types will provide test results equivalent in precision and accuracy to the reference methods. When such data are available, the method will probably be incorporated in amendments to the listing of Dec. 1, 1976, making further substantiation unnecessary.

Attachment

cc: Walter G. Gilbert, Director
    National Training & Operational Technology Center
DATA REQUIREMENTS
NATIONWIDE APPROVAL OF ALTERNATE TEST PROCEDURES

1) Five industrial (discharge) sources identified by Standard Industrial Classification (SIC) code or five drinking water sources.
2) Six samples from each source.
3) Four replicate analyses each by the proposed and approved method.

<table>
<thead>
<tr>
<th>Sources</th>
<th>Samples</th>
<th>Replicates</th>
<th>Methods</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>6 x</td>
<td>4 x 2</td>
<td></td>
<td>240</td>
</tr>
</tbody>
</table>

DATA REQUIREMENTS
LIMITED USE APPROVAL OF ALTERNATE TEST PROCEDURE
STATE OR REGIONAL USE

1) Five sources.
2) Three samples from each source.
3) Four replicate analyses each by the proposed and approved method.

<table>
<thead>
<tr>
<th>Sources</th>
<th>Samples</th>
<th>Replicates</th>
<th>Methods</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>3 x</td>
<td>4 x 2</td>
<td></td>
<td>120</td>
</tr>
</tbody>
</table>
DATA REQUIREMENTS
LIMITED - USE APPROVAL OF ALTERNATE TEST PROCEDURE
PERMIT HOLDER OR DRINKING WATER SYSTEM

1) Three samples from each source.
2) Four replicate analyses each by the proposed and approved method.

<table>
<thead>
<tr>
<th>Sources</th>
<th>Samples</th>
<th>Replicates</th>
<th>Methods</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>X 3</td>
<td>X 4</td>
<td>X 2</td>
<td>= 24</td>
</tr>
</tbody>
</table>

STATISTICAL PROTOCOL
APPROVAL OF ALTERNATE TEST PROCEDURES

1) Calculate basic statistics of mean and standard deviation.
2) Test for outliers.
3) Frequency counts and histogram to check distribution.
4) Cochran's test for equality among-within-sample standard deviation.
5) F-test for equality of pooled within-sample variances.
6) T-test for equality of method means.
DATE: September 20, 1978

SUBJECT: Use of "Prepared" Reagents in NPDES Compliance

FROM: Dwight G. Ballinger, Director
Environmental Monitoring and Support Laboratory - Cincinnati

TO: Regional Quality Assurance Coordinators

We encourage the use of pre-prepared solutions and standards by sewage treatment plant operators and others for their required compliance monitoring activities, providing such solutions have been prepared according to the reagents section of the approved methods cited in the Federal Register of December 1, 1976. We would, however, be opposed to the use of commercial reagents that are of unknown composition.

In allowing the use of known pre-prepared solutions and standards, we strongly recommend that the following quality control checks be observed to insure their validity:

1. Date all solutions upon receipt of shipment, store in separate, appropriate areas, and observe stated shelf life.

2. Verify that solutions and standards are valid by initially checking them against a quality control check sample available from EMSL through the appropriate Regional QA Coordinator. These check samples are available for most of the common measurements required in the NPDES. They are shipped in a sealed vial as a sample concentrate with the actual composition being provided in a separately sealed envelope.

3. Verify that these solutions are stable on a routine basis by periodically comparing them against a quality control check sample or a standard from another source.

cc: Walter G. Gilbert, Director
National Training & Operational Technology Center
WEDNESDAY, DECEMBER 24, 1975

PART IV:

ENVIRONMENTAL PROTECTION AGENCY

WATER PROGRAMS

National Interim Primary Drinking Water Regulations
Title 40—Protection of Environment
CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY
SUBCHAPTER D—WATER PROGRAMS
§ 141.467
PART 141—NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS

On March 14, 1975 the Environmental Protection Agency (EPA) proposed National Interim Primary Drinking Water Regulations pursuant to sections 1412, 1414, 1415, and 1450 of the Public Health Service Act ("the Act") as amended by the Safe Drinking Water Act ("SDWA", Pub L. 93-523) 40 FR 11990 EPA held public hearings on the proposed regulations in Boston, Chicago, San Francisco, and Washington during the month of April. Several thousand pages of comments on the proposed regulations were received and Water Resources. In addition, EPA has received comments and information from the proposed regulations from the National Drinking Water Advisory Council, the Secretary of Health, Education and Welfare, and numerous others during meetings with representatives of State agencies, public interest groups and others. The regulations only with the basic legal requirements Descriptive material will be provided in a guidance manual for use by public water systems and States.

The purpose of this preamble is to summarize the significant changes made in the proposed regulations as a result of comments received and the further consideration of available information. A more detailed discussion of the comments and discussion changes in the proposed regulations is attached as Appendix A.

WATER SYSTEMS COVERED.

The Safe Drinking Water Act applies to so-called "public water systems," which is defined in Section 1401(4) of the Act as "a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or serves at least twenty-five individuals." Privately owned as well as publicly owned systems are covered. Service to "the public" is interpreted by EPA to include factories and private housing developments (See generally, House Report, pp. 15-17).

The definition of "public water system" proposed in the Interim Primary Drinking Water Regulations sought to explain the meaning of the statutory reference to "regular" service. It was proposed to interpret this term as including water systems by which 59 percent or greater of the community's residents would be provided service for at least twenty-five persons at least twenty-five individuals at least 60 days out of the year. The use of a minimum number of days rather than months also makes clear that a system may qualify as a public water system even if it is not open every day during a given month.

Once "public water system" has been defined, it is necessary to define the two major types of systems: those serving residents and those serving transients or intermittent users. The possible health effects of a contaminant in drinking water are quite different for a person drinking the water for a long period of time than for a person drinking the water only briefly or intermittently. Different regulatory considerations may vary in some cases apply to systems which serve residents as opposed to systems which serve transients or intermittent users. Accordingly, § 141.2(e) makes clear that all "public water systems" fall within either the category of "community water systems" or the category of "non-community water systems.

To make clear which regulatory requirements apply to which type of system, the category covered is specifically indicated throughout the regulations.

The proposed regulations defined a "community water system" as a "public water system which serves a population of which 75 percent or greater are residents of that system. It has been defined that the percentage of water system users who are residents would result in treating some fairly large resort communities with many year-round residents as non-community systems. Therefore, the definition of "community water system" has been changed to cover any system which serves at least 15 service connections in which more than 50 percent of the system's users are residents or which at least 25 year-round residents serve.

SMALL COMMUNITY WATER SYSTEMS

Many community water systems in the country are quite small. Since it is the intention of the Act to provide basically the same level of health protection to individuals served by small systems as to residents of large cities and since a number of advanced water treatment techniques are made feasible only by economies of scale, the costs of compliance with the requirements of the Act may pose a serious problem for many small communities. The regulations seek to recognize the financial problems of small communities by requiring a more realistic monitoring for systems serving fewer than 1,000 persons. Variance applications authorized by the Act can also be obtained or with respect to the small community systems in appropriate cases, at least temporarily, EPA will provide technical assistance on effective treatment processes which can be used by small systems.

These methods of dealing with the financial problems of some small community systems may not be sufficient in specific instances to comply with all applicable regulatory requirements. E.g. EPA is commencing a study of potential problems faced by small community systems in meeting applicable requirements under the Act and these regulations, and, if necessary, will make additional adjustments in the interim Primary Drinking Water Regulations prior to their effective date.

NON-COMMUNITY SYSTEMS

"Non-community systems" are basically those systems which serve transients or intermittent users who are not residents of the communities in which the systems are located. Examples include campgrounds, service stations, and other public accommodations which have their own water system and which have at least 15 service connections or serve water to a daily average of at least 25 persons. Some schools, factories and churches are also included in this category. It is conservatively estimated that there are over 200,000 non-community water systems in the country. However, it should be recognized that while their number is large, they normally are not the principal source of water for the people they serve.

The regulations as proposed would have applied all maximum contaminant levels to non-community systems as well as to community systems. However, community systems could be excused from having to take into account the fact that the proposed maximum contaminant levels for organic chemicals and most inorganic chemicals are based on the potential health effects of long-term exposure. Those levels are not necessary to protect transients or intermittent users. Therefore the final regulations provide that maximum contaminant levels for organic chemicals, and for inorganic chemicals other than nitrates, are not applicable to non-community systems. An exception was made for nitrates because they can have an adverse health effect on susceptible infants in a short period of time.

Even without monitoring for organic chemicals or most inorganic chemicals, in the initial stages of implementation of the drinking water regulations, monitoring results from tens of thousands of non-community systems could overstate the numbers they serve.

This could delay effective implementation of the regulations with respect to the community systems which are the most important. Therefore, the regulations provide that non-community systems be given two years after the effective date of the regulations. In the meantime, non-community systems which already monitor their water are encouraged to continue to do so, and States are also encouraged to take appropriate measures to test or require monitoring for non-community systems that serve large numbers of people.

Of course, non-community systems which pose a threat to health would be dealt with as quickly as possible. The maximum contaminant levels applicable to non-community water systems are 15 service connections and are expected to be set as soon as possible after promulgation. At the same time, they are not applicable to community systems. Inspection and enforcement authority will apply to non-community systems at the same time as to community systems.

SANITARY SURVEYS

EPA encourages the States to conduct sanitary surveys on a systematic basis.
RULES AND REGULATIONS

The maximum contaminant level (MCL) is the highest contaminant level allowed in drinking water under federal law. The MCL is based on the best available science and takes into account the potential health effects of consuming the contaminant. The MCL is set by the Environmental Protection Agency (EPA) and is enforced by state and local governments.

MCLs are established for various contaminants, including mercury, lead, copper, and iron. The MCL for a contaminant is the maximum concentration that is safe for human consumption. If the concentration of a contaminant in a water supply exceeds the MCL, the EPA and state and local governments are required to take action to reduce the concentration to a level that is safe for human consumption.

MCLs are set through a process that involves scientific and public input. The EPA conducts risk assessments and reviews scientific literature to determine the maximum concentration of a contaminant that is safe for human consumption. The EPA then sets the MCL based on this information.

MCLs are reviewed periodically to ensure that they continue to be based on the best available science. If new scientific information becomes available, the MCL may be revised to reflect this information.

In summary, the MCL is a critical component of federal, state, and local regulations to ensure the safety of drinking water for all Americans. By setting and enforcing MCLs, the EPA and state and local governments are able to protect public health and ensure that drinking water is safe for human consumption.
RULES AND REGULATIONS

The promulgated definition of maximum contaminant level (MCL) still retains the requirement that the maximum contaminant level be measured at the intake of water, which should be measured at the point of source or the point of entry to the distribution system, however, the definition has been expanded to include contaminants added to the water by circumstances under the control of the consumer, or are the responsibility of the supplier of water to include the contaminants result from various piping and plumbing materials.

The concept of the MCL is complex and involves many factors. The MCL is calculated based on the premise that the contaminant should not cause any adverse health effects. The MCL is used to set limits on the amount of a contaminant that can be present in drinking water to ensure public health.

The promulgated MCLs for organic chemicals obtained by the carbon chloroform extract (CCE) method in the Revised Primary Drinking Water Regulations will be dealt with primarily in the revised primary drinking water regulations. The CCE method is widely used in the United States to determine the presence of organic chemicals in drinking water. The CCE standard might provide an appropriate means of dealing with the organic chemistry problem in drinking water.

The promulgated MCLs were promulgated in the Revised Primary Drinking Water Regulations to set limits on the amount of organic chemicals that can be present in drinking water to ensure public health. The CCE standard is a test for undesirable tastes and odors in drinking water. As concern developed over specific health effects of organic chemicals, the CCE standard was considered.

First EPA is embarking on an intensive research program to find answers to the following four questions:

1. What are the effects of common contaminants in human health?
2. What analytical procedures should be used to measure finished drinking water?
3. What changes in treatment practices are required to minimize the formation of these compounds in treated water?
4. What treatment technology must be applied to reduce contaminant levels to concentrations that may be specified in the Revised Primary Drinking Water Regulations?
A key element of quality control for public water systems is accurate laboratory analysis. Section 141.26 of the regulations provides that analyses conducted for the purpose of determining compliance with maximum contaminant levels must be conducted by a laboratory approved by the state. Primary enforcement responsibility will be assigned as possible, in cooperation with the States and other interested parties, criteria, and procedures for laboratory approval. A State with primary enforcement responsibility will maintain a list of laboratories approved by EPA pursuant to the criteria and procedures, and in turn will certify laboratories within the State.

Record-keeping requirements and reports to the State also will aid in quality control efforts.

ECONOMIC AND COST ANALYSIS

A comprehensive economic study has been made of the Interim Primary Drinking Water Regulations. This study estimates the costs of the regulations, evaluates the potential economic impact, and considers possible material and labor shortages. The results of this analysis are summarized here.

Total investment costs to community water systems to achieve compliance with these new regulations are estimated to range from $50 to $300 million. It is estimated that non-community systems will invest an additional $24 million. The range of the estimate is due to uncertainty as to the design flow that will be used in installing treatment facilities. Systems not in compliance will have to consider sizing their new components to accommodate average daily flow conditions, or maximum daily flow conditions in cases where system storage is not adequate.

This investment will be spread over several years. Investor-owned systems will bear about one-fourth of these costs, and publicly-owned systems the remainder. It is not anticipated that any of these facilities will result in an increase in costs to consumers. Because many of these systems are expected to be within the following ranges.

| Capital costs | $100-247 |
| Operations and maintenance | 3-9 |
| Monitoring (routine only) | 14.35 |

Although these aggregate figures are significant for many systems, they will be much lower for smaller systems. For those users in systems serving 10,000 persons or more, the average annual treatment cost per capita is estimated to range from $0.75 to $2.50. For smaller systems, the cost will be greater, but still significant. For example, a system serving 500 persons will have an average annual treatment cost per capita of $1.50 to $4.50.
RULES AND REGULATIONS

Sec. 141.2 Turbidity sampling and analytical requirements
141.23 Monochromatic colorimetric or turbidimetric analytical requirements
141.24 Organic chemical sampling and analytical requirements
141.27 Alternative analytical techniques
141.28 Approved laboratories
141.29 Monitoring of public water systems

Subpart D—Reporting, Public Notification, and Record keeping
141.41 Reporting requirements
141.42 Preparation of reports, exceptions and non-compliance with regulations
141.43 Record maintenance

Authority: Secs. 1412, 1414, 1415, and 1416 of the Public Health Service Act (42 U.S.C. 300g-1 to 300g-6 and 300g-9)

Subpart A—General
§141.11 Applicability.

This part establishes primary drinking water regulations pursuant to section 1112 of the Public Health Service Act (as amended by the Safe Drinking Water Act Pub L 93-523), and related regulations applicable to public water systems

§111.2 Definitions

As used in this part the term "Act" means the Public Health Service Act as amended by the Safe Drinking Water Act Pub L 93-523
"Contaminant" means any physical, chemical, biological or radiological substance or matter in water
"Maximum contaminant level" means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of a public water system, except in the case of turbidity where the maximum permissible level is measured at the point of entry to the distribution system. Contaminants added to the water under circumstances controlled by the user, except in the case of fluoridation and in the event of piping and plumbing causes by water quality, are excluded from this definition
"Person" means an individual, corporation, association, partnership, or Federal agency
"Public water system" means a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily, at least 60 days out of the year. Such a system includes (a) any collection, treatment, storage and distribution facilities under control of the operator of such system and used primarily in connection with such system and (b) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A "water system is either a "community water system" or a "non-community water system"

(a) "Non-community water system" means a public water system which serves at least 15 service connections used by year-round residents or regularly serves a year-round resident population of at least 25 year-round residents

(b) "Community water system" means a public water system that is not a non-community water system

"Sanitary survey" means an on-site review of the water source, facilities, equipment and maintenance of public water system for the purpose of evaluating the adequacy of such source, facilities, equipment, operation and maintenance for producing and distributing safe drinking water

"Standard sample" means the aliquot of finished drinking water that is examined for the presence of coliform bacteria

"State" means the agency of the State government which has jurisdiction over public water systems. During any period when a State does not have primary enforcement responsibility pursuant to Section 1413 of the Act, the term "State" means the Regional Administrator, U.S. Environmental Protection Agency

"Supplier of water" means any person who owns or operates a public water system

§141.3 Coverage.

This part shall apply to each public water system except public water system meets all of the following conditions

(a) Meets all of the necessary collection, storage, and treatment facilities

(b) Obtains all of its water from, but is not owned or operated by a public water system to which such regulations apply

(c) Does not sell water to any person

(d) Is not a carrier which conveys passengers in interstate commerce

Variances or exemptions from certain provisions of these regulations may be granted hereunder pursuant to Sections 1415 and 1416 of the Act by the entity with primary enforcement responsibility provisions under Part 142, National Interim Primary Drinking Water Regulations. Implementations. Subpart E (Variance and Exemptions) applies where EPA has primary enforcement responsibility

§141.5 Siting requirements.

Before a person may enter into a financial commitment for or initiate construction of any new public water system or increase the capacity of an existing public water system, he shall notify the State and, to the extent practicable, avoid locating part or all of the new or expanded facility at a site which

(a) Is subject to a significant risk from earthquakes, floods, fires or other disasters which could cause a breakdown of the public water system or a portion thereof, or

(b) Except for intake structures, is within the floodplain of a 100-year flood or is lower than any recorded high tide where appropriate records exist

The U.S. Environmental Protection Agency will not seek to override land use decisions affecting public water systems giving which are made at the State or local government levels

§141.6 Effective date.

The regulations set forth in this part shall take effect 18 months after the date of promulgation

Subpart B—Maximum Contaminant Levels
§141.11 Maximum contaminant levels for inorganic chemicals.

(a) The maximum contaminant level for fluoride in the community water system is applicable to both community and non-community water systems. The levels for the other inorganic chemicals apply only to community water systems. Compliance with the maximum contaminant levels for inorganic chemicals is calculated pursuant to §141.23.

(b) The following are the maximum contaminant levels for inorganic chemicals other than fluoride.

<table>
<thead>
<tr>
<th>Contaminant</th>
<th>Level, milligrams per liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Arsenic</td>
<td>0. 50</td>
</tr>
<tr>
<td>Barium</td>
<td>10.00</td>
</tr>
<tr>
<td>Chromium</td>
<td>0.05</td>
</tr>
<tr>
<td>Lead</td>
<td>0.05</td>
</tr>
<tr>
<td>Mercury</td>
<td>0.002</td>
</tr>
<tr>
<td>Selenium</td>
<td>0.01</td>
</tr>
<tr>
<td>Silver</td>
<td>0.05</td>
</tr>
</tbody>
</table>

(1) When the annual average of the maximum daily air temperatures for the location in which the community water system is situated is the following, the maximum contaminant levels for fluoride are

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Level, milligrams per liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>25°F and below</td>
<td>0.005</td>
</tr>
<tr>
<td>40°F to 70°F</td>
<td>0.005</td>
</tr>
</tbody>
</table>

§141.12 Maximum contaminant levels for organic chemicals.

The following are the maximum contaminant levels for organic chemicals. They apply only to community water systems. Compliance with maximum contaminant levels for organic chemicals is calculated pursuant to §141.24

<table>
<thead>
<tr>
<th>Contaminant</th>
<th>Level, milligrams per liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chlorinated hydrocarbons</td>
<td>0.0002</td>
</tr>
<tr>
<td>Endrin (1,3,4,10-tetrachloro</td>
<td>0.0002</td>
</tr>
<tr>
<td>1,4-epoxy-1, 4, 5, 6-</td>
<td>0.0002</td>
</tr>
</tbody>
</table>
| endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-endo-eno...
### RULES AND REGULATIONS

**§ 111.13 Maximum contaminant levels for turbidity**

The maximum contaminant levels for turbidity are applicable to both community water systems and non-community water systems (including surface water sources in whole or in part). The maximum contaminant levels for turbidity in drinking water, measured at a representative entry point, is as follows:

1. One turbidity unit (TU), as determined by a monthly average pursuant to § 1141.22, except that five or fewer turbidity units may be allowed if the supplier of water can demonstrate to the State that the higher turbidity does not exceed any of the following:
   - Interference with disinfection.
   - Maintenance of an effective disinfectant agent throughout the distribution system.
   - Interference with microbiological determinations.

(b) Five turbidity units based on an average for two consecutive days pursuant to § 1141.22.

**§ 1141.21 Maximum microbiological contaminant levels.**

The maximum contaminant levels for coliform bacteria, applicable to community water systems and non-community water systems, are as follows:

1. One coliform per 100 milliliters as the arithmetic mean of all samples examined per month pursuant to § 1141.21(b) or (c).

2. Four per 100 milliliters in more than one sample when less than 20 are examined per month, or four per 100 milliliters in more than five percent of the samples when 20 or more are examined per month.

(b) When the membrane filter technique pursuant to § 1141.21(a) is used, coliform bacteria shall not be present in any of the following:

1. More than 10 percent of the samples in any month pursuant to § 1141.21(b) or (c).

2. Three or more portions in more than one sample when less than 20 samples are examined per month; or

3. Three or more portions in more than five percent of the samples when 20 or more samples are examined per month.

(c) When the membrane filter method and 10 milliliter standard portions pursuant to § 1141.21(a) are used, coliform bacteria shall not be present in any of the following:

1. More than 60 percent of the portions in any month pursuant to § 1141.21(b) or (c).

2. Five portions in more than one sample when less than five samples are examined per month or

3. Five portions in more than one sample when five or more samples are examined per month.

### Microbiological contaminant sampling and analytical requirements

(a) Suppliers of water for community water systems and non-community water systems shall analyze for coliform bacteria for the purpose of determining compliance with § 1141.14 Analyses shall be conducted in accordance with the analytical recommendations set forth in "Standard Methods for the Examination of Water and Wastewater," American Public Health Association, 13th Edition, pp. 662-685, except that a standard sample size shall be 100 milliliters. The standard sample used in the membrane filter procedure shall be 100 milliliters. The standard sample used in the 5 tube most probable number (MPN) procedure (fermentation method) shall be 5 times the standard portion.

(b) The supplier of water for a community water system shall take coliform density samples at regular time intervals, and in number proportionate to the population served by the system. In no event shall the frequency be less than as set forth below:

<table>
<thead>
<tr>
<th>Population served</th>
<th>Minimum number of samples per month</th>
</tr>
</thead>
<tbody>
<tr>
<td>25 to 1,000</td>
<td>1</td>
</tr>
<tr>
<td>1,001 to 2,500</td>
<td>2</td>
</tr>
<tr>
<td>2,501 to 5,000</td>
<td>3</td>
</tr>
<tr>
<td>5,001 to 10,000</td>
<td>4</td>
</tr>
<tr>
<td>10,001 to 25,000</td>
<td>5</td>
</tr>
</tbody>
</table>

Based on a history of no coliform bacteriological contamination and on a sanitary survey by the State showing the water system to be supplied solely by a protected ground water source and free of sanitary defects, a community water system serving 25 to 1,000 persons, with the written permission of the State, may reduce this sampling frequency, except that in no case shall it be reduced to less than one per quarter.

(c) The supplier of water for a non-community water system shall sample for coliform bacteria in each calendar quarter during which the system provides water to the public. Such sampling shall begin within two years after the effective date of this part. If the State, on the basis of a sanitary survey, determines that some other frequency is more appropriate, that frequency shall be the frequency required under these regulations. Such frequency shall be confirmed and changed on the basis of subsequent surveys.

(d) When the coliform bacteria in a single sample exceed four per 100 milliliters (§ 1141.14(a)), at least one consecutive daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency estab-
RULING AND REGULATIONS

listed by the State until the results obtained from at least two, consecutive check samples taken on successive days is less than one coliform bacterium per 100 milliliters.

(b) When coliform bacteria occur in three or more 10 ml portions of a single sample (§ 141.14(b)(1)), at least two consecutive check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency established by the State, until the results obtained from at least two consecutive check samples show no positive tubes.

(c) When coliform bacteria occur in all five of the 100 ml portions of a single sample (§ 141.14(b)(2)), at least two daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily or at a frequency established by the State, until the results obtained from at least two consecutive check samples show no positive tubes.

(d) The location at which the check samples shall be collected pursuant to paragraphs (d)(1), (2), or (3) of this section shall not be eliminated from future sampling without approval of the State. The responsibility for sampling back-up samples performed pursuant to this subsection except those obtained from check samples and special purpose samples shall be to determine compliance with the maximum contaminant level for coliform bacteria as established in § 141.14. Check samples shall not be included in calculating the total number of samples taken every four months to determine compliance with § 141.21(b) or (c).

(e) When the presence of coliform bacteria in water taken from a particular sampling point has been confirmed by the check samples examined as directed in paragraphs (d)(1), (2), or (3) of this section, the supplier of water shall report to the State within 48 hours.

(f) Any sample showing the maximum contaminant level set forth in paragraphs (a), (b), or (c) of § 141.14 is exceeded, the supplier of water shall notify the State and notify the public as prescribed in § 141.31.

(g) Special purpose samples, such as those taken to determine whether disinfection practices following pipe placement, replacement, or repair have been sufficient, shall not be used to determine compliance with § 141.14 or § 141.21(b) or (c).

(h) A supplier of water of a community water system or of a non-community water system may, with the approval of the State and based upon a sanitary survey, substitute the use of chlorine residual monitoring for not more than 25 percent of the samples required to be taken by paragraph (b) of this section. Provided, That the supplier of water takes chlorine residual samples at points which are representative of the conditions within the distribution system at the frequency of at least four for each substituted microbiological sample. There shall be at least daily determination of chlorine residual. When the supplier of water elects the option provided in this paragraph (h) of this section, he shall maintain no less than 0.2 mg/l free chlorine throughout the public water distribution system. When a particular sampling point has been shown to have a free chlorine residual of less than 0.2 mg/l, the water at that location shall be tested as soon as practicable and in any event within one hour of the determination. This fact shall be reported to the State within 48 hours. Also if the analyzes is confirmed, a sample for coliform bacterial analysis must be collected from the same sampling point as soon as practicable and preferably within one hour, and the results of such analyzes, reported to the State within 48 hours after the results are known to the supplier of water. Analyzes for residual chlorine shall be made in accordance with "Standard Methods for the Examination of Water and Wastewater," 13th Ed., pp 129-132. Compliance with the maximum contaminant levels for coliform bacteria shall be determined on the monthly mean values only as specified in § 141.14 including those samples taken as a result of failure to maintain the required chlorine residual level. The State may waive its approval of the use of chlorine residual substitution at any time.

§ 141.22 Turbidity sampling and analysis requirements.

(a) Samples shall be taken by supplies of water for both community water systems and non-community water systems to a representative entry point to the water distribution system at least once per day, for the purpose of making turbidity measurements to determine compliance with § 141.1. The measurements shall be made by the nephelometric method in accordance with the recommendations set forth in "Standard Methods for Chemical Analysis of Water and Wastewater," 13th Ed., pp 298-299. Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(b) If the result of a turbidity analysis indicates that the maximum allowable limit has been exceeded, the supplier shall notify the State within 48 hours. The repeat sample shall be the sample used for the purpose of calculating the monthly average. If the monthly average of the daily samples exceeds the maximum allowable limit or if the average of two samples taken on consecutive days exceeds 5 FTU, the supplier of water shall report to the State and notify the public as directed in § 141.31 and § 141.32.

(c) Sampling for non-community water systems shall begin within two years after the effective date of this part.

(d) The requirements of this § 141.22 shall apply only to public water systems which use water obtained in whole or in part from surface sources.
RULES AND REGULATIONS

mental Protection Agency Office of Technology Transfer Washington DC 20460 1974

§ 111.21 Organic chemical sampling and analytical requirements

1. An analysis of substances for the purpose of determining compliance with § 111.12.b, shall be conducted in accordance with the method prescribed in this part.
2. The frequency of sampling and analysis shall be determined by the State or the owner or operator of the systems.
3. The results of the analysis shall be reported to the State within 30 days after the date of the analysis.
4. The results of the analysis shall be used to determine compliance with § 111.12.b.

§ 111.22 Organic chemical monitoring requirements

When a public water system supplies water to one or more other public water systems the State may require monitoring of the public water system supplying water to the other systems. The State shall notify the public water system supplying water to the other systems that it is required to monitor for certain organic chemicals. The public water system shall notify the State of the results of the monitoring on a regular basis.
RULES AND REGULATIONS

and in any event by written notice within three months. Such notice shall be repeated at least once every three months so long as the system's failure continues or the variance or exemption remains in effect. By the system's failure Is not served by a daily newspaper of general circulation in the area, the system failure shall be completed within fourteen days after the supplier of water of the failure.

(2) By furnishing a copy of the notice to the radio and television stations serving the area served by the system. Such notice shall be furnished within seven days after the supplier of water learns of the failure.

(c) If the area served by a community water system is not served by a daily newspaper of general circulation, notification by newspaper required by paragraph (a) of this section shall be given by posting the notice in post offices within the area served by the system.

(d) If a non-community water system fails to comply with an applicable maximum contaminant level established in Subpart B of this part, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable maximum contaminant level, fails to comply with the requirement of any schedule prescribed pursuant to a variance or exemption or fails to perform monitoring or sampling pursuant to Section 1415(a) of the Act, the supplier of water shall give notice of such failure or grant to the persons served by the system. The form and manner of such notice shall be prescribed by the State, and shall insure that the public using the system is adequately informed of the failure or grant.

(e) Notices given pursuant to this section shall be written in a manner reasonably designed to inform fully the users of the system. The notice shall be conspicuous and shall not use undue technical language, unduly small print or other methods which would frustrate the purpose of the notice. The notice shall disclose all material facts regarding the subject including the nature of the problem, and the corrective action to be taken.

(1) A fair explanation of the significance or seriousness to the public health of the failure.

(f) Notice to the public required by this section may be given by the State or the supplier of water.

(g) In any instance in which notification by mail is required by paragraph (a) of this section but notification by newspaper or to radio or television stations is not required by paragraph (b) of this section, the State may order the supplier of water to provide notification by newspaper and to radio and television stations when circumstances make more immediate or broader notice appropriate to protect the public health.

§ 141.33 Record maintenance.

Any owner or operator of a public water system subject to the provisions of this part shall retain on its premises or at a convenient location near its premises the following records:

(a) Records of bacteriological analyses made pursuant to this part shall be kept for not less than 5 years. Records of chemical analyses made pursuant to this part shall be kept for not less than 10 years. Actual laboratory reports may be kept, or data may be transferred to tabular summaries. provided that the following information is included:

(1) The date, place, age, time, and name of the person who collected the sample;

(2) Identification of the sample as to whether it was a routine distribution system sample, check sample, raw or process water sample or other special purpose sample;

(3) Date of analysis;

(4) Laboratory and person responsible for performing analysis;

(5) The analytical technique/method used; and

(6) The results of the analysis.

(b) Records of action taken by the system to correct violations of primary drinking water regulations shall be kept for a period not less than 3 years after the last action taken with respect to the particular violation involved.

(c) Copies of any written reports, summaries or communications relating to sanitary surveys of the system conducted by the system itself, by a private consultant, or by any local, State or Federal agency, shall be kept for a period not less than 10 years after completion of the sanitary survey involved.

(d) Records concerning a variance or exemption granted to the system shall be kept for a period ending not less than 5 years following the expiration of such variance or exemption.

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FROM p. 57332:

ENVIRONMENTAL PROTECTION AGENCY
40 CFR Part 141
IFRL 1525-71
Interim Primary Drinking Water Regulations; Amendments
AGENCY: Environmental Protection Agency (EPA).
ACTION: Final rule.

FROM p. 57332:

EFFECTIVE DATE: These amendments to the regulations will be effective August 27, 1980 except that sodium monitoring and reporting, determination of the types of materials in distribution systems, and monitoring and reporting corrosion characteristics will be effective 18 months following the date of promulgation. The sodium and corrosion requirements must be completed within 12 months following the effective date.

FROM p. 57344:

9. Amending § 141.22(a) to read as follows and adding (e):

§ 141.22 Turbidity sampling and analytical requirements.
 (a) Samples shall be taken by suppliers of water for both community and non-community water systems at a representative entry point(s) to the water distribution system at least once per day, for the purpose of making turbidity measurements to determine compliance with § 141.13. If the State determines that a reduced sampling frequency in a non-community system will not pose a risk to public health, it can reduce the required sampling frequency. The option of reducing the turbidity frequency shall be permitted only in those public water systems that practice disinfection and which maintain an active residual disinfectant in the distribution system, and in those cases where the State has indicated in writing that no unreasonable risk to health existed under the circumstances of this option. The turbidity measurements shall be made by the Nephelometric Method in accordance with the recommendations set forth in "Standards and Methods for Examination of Water and Wastewater," American Public Health Association, 14th Edition, pp. 132-134; or Method 180.1-1. Nephelometric Method.
 (b) The State has the authority to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies. (c) The State may require the submission of raw data, general summary reports, and other information as may be necessary to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies.
 (d) The State has the authority to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies.
 (4) Amending § 141.23(a) to read as follows and adding (e) through (i):

§ 141.23 Inorganic chemical sampling and analytical requirements.
 (a) . . .
 (b) . . .
 (c) . . .
 (d) . . .
 (e) . . .
 (f) . . .
 (g) . . .
 (h) . . .
 (i) . . .

(from page 57344 continued)
and Wastewater Industrial Method
\#129-71 W. "Technicon Industrial
Systems, Tarrytown, New York 10591.
December 1972, or Fluoride. Total.
Colorimetric. Zirconium—Eriochrome
Cyanine R Method 4-1-3325-75, pp.
365-369.

11. Amending \$141.24(a)(3), (e) and (f)
to read as follows:

§ 141.24 Organic chemical sampling and
analytical requirements.
(a) Analysis made to determine
compliance with § 141.12(a) shall be
made in accordance with "Methods for
Organochlorine Pesticides and
Chlorophenoxy Acid Herbicides in
Drinking Water and Raw Source
Water," available from ORD
Publications. CERI, EPA, Cincinnati.
Ohio 45268, or "Organochlorine
Pesticides in Water," 1977 Annual Book
of ASTM Standards, part 31, Water.
Method D3088, or Method 509-A, pp.
555-556. \( ^* \) or Gas Chromatographic
Methods for Analysis of Organic
Substances in Water. USGS, Book S.
(f) Analysis made to determine
compliance with § 141.12(b) shall be
conducted in accordance with "Methods
for Organochlorine Pesticides and
Chlorophenoxy Acid Herbicides in
Drinking Water and Raw Source
Water," available from ORD
Publications. CERI, EPA, Cincinnati.
Ohio 45268, or "Chlorinated Phenoxy-
Acid Herbicides in Water," 1977 Annual
Method D3478, or Method 509-B, pp.
555-562. \( ^* \) or Gas Chromatographic
Methods for Analysis of Organic
Substances in Water. USGS, Book S.

§ 141.25 [Amended]
12. Amending \$ 141.25 to add [e]
(e) The State has the authority to
determine compliance or initiate
enforcement action based upon
analytical results or other information
compiled by their sanctioned
representatives and agencies.
13. Amending \$ 141.25(a)(5) to read as
follows:

§ 141.27 Alternate analytical techniques.
(a) With the written permission of the
State, concurrent in by the Administrator
of the U.S. EPA, an alternate analytical
technique may be employed. An
alternate technique shall be accepted
only if it is substantially equivalent to
the prescribed test in both precision and
accuracy as it relates to the
determination of compliance with any
MCL. The use of the alternate analytical
technique shall not decrease the
frequency of monitoring required by this
Part.
14. Amending \$ 141.28 to read as
follows:

§ 141.28 Approved laboratories.
(a) For the purpose of determining
compliance with \$ 141.23 through
\$ 141.27, samples may be considered
only if they have been analyzed by a
laboratory approved by the State except
that measurements for turbidity, free
chlorine residual, temperature and pH
may be performed by any person
acceptable to the State.
(b) Nothing in this Part shall be
construed to preclude the State or any
duly designated representative of the
State from taking samples or from using
the results from such samples to
determine compliance by a supplier of
water with the applicable requirements
of this Part.

15. Amending \$ 141.30(a) and (c) and
adding paragraphs (d) and (e) to read as
follows:

§ 141.31 Reporting requirements.
(a) Except where a shorter period is
specified in this Part, the supplier of
water shall report to the State the
results of any test measurement or
analysis required by this part within (A)
the first ten days following the month in
which the results are received or (B) the
first ten days following the end of the
required monitoring period as stipulated
by the State, whichever is shorter.
(d) The water supply system, within
ten days of completion of each public
notification required pursuant to
\$ 141.32, shall submit to the State a
representative copy of each type of
notice distributed, published, posted,
and/or made available to the persons
served by the system and/or to the
media.
(e) The water supply system shall
submit to the State within the time
stated in the request copies of any
records required to be maintained under
\$ 141.32 hereof or copies of any
documents received in interstate which
the State or the Administrator is entitled
to inspect pursuant to the authority of

§ 1445 of the Safe Drinking Water Act or
the equivalent provisions of State law.
16. Amending \$ 141.33(b)(1)(d)(3), (c)
and (d) to read as follows:

§ 141.33 Public notification.

(b) The water supply, system shall


...
the State; one during mid-winter and wholly or in part or more if required by the State, one during mid-winter and one during mid-summer. The supplier of the water shall collect one sample per plant for analysis for each plant using ground water sources or more if required by the State. The minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples.

(2) Determination of the corrosivity characteristics of the water shall include measurement of pH, calcium hardness, alkalinity, temperature, total dissolved solids (total filterable residue), and calculation of the Langelier Index in accordance with paragraph (c) below. The determination of corrosivity characteristics shall only include one round of sampling (two samples per plant for surface water and one sample per plant for ground water sources). However, States may require more frequent monitoring as appropriate. In addition, States have the discretion to require monitoring for additional parameters which may indicate corrosivity characteristics, such as sulfates and chlorides. In certain cases, the Aggressive Index, as described in paragraph (c), can be used instead of the Langelier Index. The supplier shall request in writing to the State and the State will make this determination.

(b) The supplier of water shall report to EPA and/or the State the results of the analyses for the corrosivity characteristics within the first 10 days of the month following the month in which the sample results were received. If more frequent sampling is required by the State, the supplier can accumulate the data and shall report each value within 10 days of the month following the month in which the analytical results of the last sample was received. The supplier of water shall not be required to report the results to EPA where the State has adopted this regulation and results are reported to the State. The supplier shall identify whether the following construction materials are present in their distribution system and report to the State:

- Lead from piping, solder, caulking, interior lining of distribution mains, alloys and home plumbing
- Copper from piping and alloys, service lines, and home plumbing
- Galvanized piping, service lines, and home plumbing
- Ferrous piping materials such as cast iron and steel
- Asbestos cement pipe
- Asbestos cement pipe in drinking water systems
- Copper piping and alloys in drinking water systems
- Galvanized piping in drinking water systems
- Cast iron and steel pipes and tanks


Adding a § 141.42 to read as follows:

§ 141.42 Special monitoring for corrosivity characteristics.

(a) Suppliers of water for community public water systems shall collect samples from a representative entry point to the water distribution system for the purpose of analysis to determine the corrosivity characteristics of the water.

(1) The supplier shall collect two samples per plant for analysis for each plant using surface water sources wholly or in part or more if required by the State, one during mid-winter and one during mid-summer. The supplier of
§ 143.3 Secondary Maximum Contaminant Levels. The Secondary Maximum Contaminant Levels for public water systems are as follows:

<table>
<thead>
<tr>
<th>Contaminant</th>
<th>Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Color</td>
<td>15 color units</td>
</tr>
<tr>
<td>Copper</td>
<td>1 part in 1,000,000</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.05 mg/l</td>
</tr>
<tr>
<td>Odor</td>
<td>3 thousand odor units</td>
</tr>
<tr>
<td>Ph</td>
<td>6.5-8.5</td>
</tr>
<tr>
<td>Sulfate</td>
<td>250 mg/l</td>
</tr>
<tr>
<td>Turbidity</td>
<td>50 mg/L</td>
</tr>
</tbody>
</table>

These levels represent reasonable goals for drinking water quality. The States may establish higher or (lower levels which may be appropriate) dependent upon local conditions such as unavailability of alternate source waters or other compelling factors, provided that public health and welfare are not adversely affected.

§ 143.4 Monitoring.

(a) It is recommended that the parameters in these regulations should be monitored at intervals no less frequent than those performed for inorganic chemical contaminants listed in the National Interim Primary Drinking Water Regulations as applicable to community water systems. More frequent monitoring would be appropriate for specific parameters such as pH, color, odor or others under certain circumstances as directed by the State.

(b) Analyses conducted to determine compliance with § 143.3 should be made in accordance with the following methods:


GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Laboratory Safety Practices

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 35 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participants must use correct laboratory safety practices during the laboratory sessions in this course and in their daily work situation.

V. ENTRY LEVEL BEHAVIOR:

A. At least two months experience performing any kind of chemical analyses in a laboratory.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participants will use proper safety procedures in carrying out all laboratory work.

B. Conditions. He/she will be given the training manual, a 30 minute discussion illustrating both equipment and practices required for safe conduct of laboratory procedures, and a 5 minute orientation to the location of safety devices in the student laboratory. Any safety equipment or facilities and any very specific, specialized safety precautions required for subsequent course laboratory assignments will be supplied.

C. Accepted Performance: Practice of the safety procedures during subsequent laboratory sessions.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in training manual, "Laboratory Safety Practices"

2. Forty slides, X-27: Laboratory Safety (See XI. Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the outline in the training manual and also the visual materials.
2. Prepare the lesson which includes an orientation to your laboratory facilities. Use the Sequencing below or your own organization of the material. Be prepared to give additional information such as examples, case histories, specific facts, etc. Encourage the participants to share their experiences, too.

B. Sequencing:

Slides are Series X-27: Laboratory Safety.

1. Classroom
   a. Lab safety generally falls under the jurisdiction of one of these two; X-27, 1.
   b. At one time, USEPA considered withholding certification of drinking water analytical labs which had persistent, major safety problems; X-27, 2.
   c. A good lab safety program requires three elements; X-27, 3, 4, and 5.
   d. Example chain of command through which safety regulations are promulgated. Chance for confusion is great because of all the levels; X-27, 6.
   e. Introducing the next six slides which show possible lab accidents; X-27, 7.
   f. Possible lab accidents; X-27, 8 through 12, and 13 (the unforeseen hazard).
   g. Introducing the next eleven slides which are photos of common lab safety errors; X-27, 14.
   h. Common lab safety errors; X-27, 15 through 22, 23 (possible back siphon, especially if there is a cross-connection with wastewater), 24 (can is overflowing and is not marked glass or paper), and 25 (no emergency phone numbers).
   i. Introducing the next thirteen slides which show common lab safety equipment; X-27, 26.
   j. Common lab safety equipment; X-27, 27 through 29 and 30 (other types of glasses/goggles are available), 31, 32 (first aid kits are not a substitute for professional help), 33 (Types A-Paper, B-Chemical, C-Electrical), 34 (siphon breaker) and 35 through 39.
   k. Purpose of a safety program is accident prevention, not just a program to assure prompt summons of an ambulance to carry someone out after an accident occurs; X-27, 40.

2. Laboratory-. Point out the location of such safety devices as the fume hoods, eye-wash fountains, fire extinguishers, first aid kit, etc.
3. Laboratory - For each laboratory exercise, point out any special safety problems, e.g., use of toxic chemicals, handling of acid solutions, etc.

4. Subsequent laboratory sessions
   a. Point out any special safety precautions specific to the laboratory assignment.
   b. Observe individual performances regarding practice of the safety procedures.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:
   A. Visuals for classroom session
   B. Safety equipment in laboratory for orientation
   C. Specialized equipment for subsequent course laboratory sessions as required.

X. IPW REAGENT REQUIREMENTS:
   A. None

XI. DESCRIPTION OF VISUAL MATERIALS:
   A. Forty slides, X-27: Laboratory Safety - See next pages.
XI. DESCRIPTION OF VISUAL MATERIALS

SERIES X-27, LABORATORY SAFETY

SLIDE 1
PRINTING:
STATE/SAFETY HEALTH PROGRAM
OCCUPATIONAL SAFETY & HEALTH ACT

SLIDE 2
CARTOON OF SAFETY CERTIFICATE
BEING DENIED BECAUSE OF HAZARDOUS LABORATORY PRACTICE

SLIDE 3
PRINTING:
SAFETY REQUIRES COMMON SENSE

SLIDE 4
PRINTING:
SAFETY COSTS MONEY

SLIDE 5
PRINTING:
SAFETY REQUIRES CHANGES IN ATTITUDE

SLIDE 6
PRINTING:
TOP MANAGEMENT - MIDDLE MANAGEMENT - PLANT SUPERVISOR - FOREMAN - PLANT WORKER

SLIDE 7
PRINTING:
LIFE-HEALTH ENDANGERING LABORATORY ACCIDENTS

SLIDE 8
CARTOON OF DEAD LAB WORKER
WITH SANDWICH IN HAND AND A BOTTLE MARKED "TOXIC" NEARBY

SLIDE 9
CARTOON OF UNCONSCIOUS LAB WORKER WITH UNSTOPPERED BOTTLE MARKED "TOXIC-VOLATILE" NEARBY
SERIES X-27, LABORATORY SAFETY

SLIDE 10

cartoon of open flames on a lab table with other items (including a worker's lab coat) on, fire

SLIDE 11

drawing of acid dripping from a tipped flask onto a lab worker's clothing and the floor

SLIDE 12

drawing of a lab worker with lighted cigarette working next to a bottle of liquid marked "flammable"

SLIDE 13

cartoon depicting hazards of public relations - lab worker being hit with a baseball bat by a woman who is saying, "my water tastes terrible."

SLIDE 14

printing: laboratory boos-boos

SLIDE 15

photo of cluttered work area

SLIDE 16

photo of lab worker mouth-pipetting

SLIDE 17

photo of lab worker pouring sulfuric acid with none of the usual precautions

SLIDE 18

photo of lab worker carrying bottle of sulfuric acid without a safety carrier, etc.
PHOTO OF LAB WORKER SMOKING A CIGARETTE WHILE TAKING A READING FROM A BURET

PHOTO OF A BOTTLE OF "HEONIS" WITH CAP OFF, OUTSIDE A HOOD

PHOTO OF PHONE IN A LAB BUT NO EMERGENCY NUMBERS ARE POSTED

PHOTO OF LAB WORKER EATING AN APPLE WHILE WATCHING A BURET DURING A TITRATION

PHOTO OF HOSE FROM A SINK FAUCET IMMERSED IN THE SINK.

PHOTO OF A FILLED WASTE CAN WITH NO MARKING ABOUT GLASS OR PAPER, ETC.

PHOTO OF A BEAKER AT A LAB BENCH

PHOTO OF LAB WORKER DRINKING FROM A BEAKER AT A LAB BENCH

PHOTO OF A FILLED WASTE CAN WITH NO MARKING ABOUT GLASS OR PAPER, ETC.

PHOTO OF PIPE BULBS
PHOTO OF TWO PILOTORS

PHOTO OF LAB WORKER WEARING PLASTIC APRON

PHOTO OF A CRYSTAL CLEAR CHEMICAL PROPERLY STORED IN A HOOD

PHOTO OF A FIRST AID KIT

PHOTO OF A FIRE EXTINGUISHER WITH AN INSPECTION TAG

PHOTO OF SINK AREA WITH NO GLASSWARE STANDING IN OR AROUND IT. THE NOSE FROM THE FAUCET COMES JUST TO THE TOP OF THE SINK.

PHOTO OF ACID BOTTLES IN TWO TYPES OF SAFETY CONTAINERS

PHOTO OF AN EXPLOSION-PROOF REFRIGERATOR
PHOTO TAKEN CLOSE-UP OF THE EXPLOSION-PROOF LABEL ON A REFRIGERATOR

SLIDE 37

PHOTO OF SIGN ON A REFRIGERATOR, "DO NOT STORE HAZARDOUS MATERIALS IN THIS UNIT."

SLIDE 38

PHOTO OF SIGNS ON A REFRIGERATOR, "DO NOT STORE FLAMMABLE MATERIALS IN THIS UNIT."

SLIDE 39

DRAWING OF AMBULANCE LEAVING TREATMENT PLANT

SLIDE 40 (LAST SLIDE)
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Analytical Techniques

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 20 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: To carry out the volumetric analyses in the course, participants must correctly use volumetric glassware and titration assemblies.

V. ENTRY LEVEL BEHAVIOR:
   A. Ability to use volumetric glassware
   B. Ability to use titration assemblies

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will have reviewed correct techniques involved in basic laboratory operations and in using volumetric glassware and titration equipment.
   B. Conditions: He/she will see silent films and be given numbers about correct techniques.
   C. Accepted Performance: Subsequent satisfactory performance of the volumetric analyses included in the course.

VII. INSTRUCTIONAL RESOURCES:
   A. Available Media:
      1. Four segments of silent film showing an analyst demonstrating safety practices, general laboratory operations (like pouring chemicals from containers), techniques involved in cleaning and using pipets and a 3-valve bulb, and techniques involved in cleaning burets and carrying out a titration. These are available from Kalmia Co., Dept. C 1, Concord, Massachusetts 01742. Equivalent visual material is available from several other companies. Also, demonstration of the techniques is very effective.
   
   B. Suggested Media:
      1. None

VIII. INSTRUCTIONAL APPROACH:
   A. Preparation for Instruction:
      1. Review films and/or equivalent visual materials and prepare commentary on techniques to review with participants. Personal experiences of Instructor and participants add interest and emphasis.
B. Sequencing:
   1. Introduction - purpose of session
   2. Show films and give commentary.
   3. Encourage class' sharing of pertinent experiences.

IX. IPW Equipment and Supply Requirements:
   A. Films or equivalent visual materials.

X. IPW Reagent Requirements:
   A. None
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Volumetric Analysis

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 55 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Participants should understand the principles of quantitation utilized to determine concentrations in water samples using volumetric analytical methodology as a prerequisite to the lessons on acidity, alkalinity, chlorine and hardness.

V. ENTRY LEVEL BEHAVIOR:

A. Fundamental knowledge of quantitative analysis.
B. Ability to perform multiplication and division operations.
C. Laboratory experience in using volumetric glassware and titration assemblies.

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will have reviewed the principles of quantitation involved in a volumetric analysis: analytical chemical reactions, indicators, volumes, standard solution-concentrations, calculations.

B. Conditions: He/she will be given the training manual and a classroom discussion of the elements involved in this analytical method and in the calculation formulas utilized to obtain results.

C. Accepted Performance. Subsequent satisfactory performance of volumetric analyses of samples and ability to correctly use the appropriate calculation formulas for acidity, alkalinity, hardness, and chlorine. He/she must also correctly answer 70% of the items on this topic in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in training manual, "Volumetric Analysis of Water Quality"
2. Thirty slides, X-18: Volumetric Analysis (See XI. Description of Visual Materials).

B. Suggested Media:

1. None
VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the topic outline in the training manual and the visual materials. See XI below.

2. Prepare the lesson using the sequencing below or your own organization of the material.

B. Sequencing:

Slides are series X-18: Volumetric Analysis
Participants can use the training outline as a reference for information on the slides.

1. Introduction - examples of water quality parameters that can be determined by volumetric analysis.

2. Titration Assembly, can use to give overview of topic - Slide 1

3. Titrations: definition, elements involved - Slides 2, 3, 4

4. Reactions involving solutions - Slides 5, 6, 7
   (Slide 5 - can define acid and base with this)

5. Completed reactions, items for discussion - Slide 8

6. Indicators - Slides 9, 10

7. Volume measurements for titrations - Slide 11

8. Standard Solutions: definition, terms - Slides 12, 13

9. Molar Solutions - Slides 14, 15, 16

10. Molecules react in definite ratio - Slide 17

11. Equivalent weights and normal solutions - Slides 18, 19, 20, 21

12. Volume relationship of normal solutions - Slide 22

13. Relating normality and grams - Slide 23

14. Finding normality - Slide 24

15. Finding grams - Slide 25

16. Application of formulas on slides 24 and 25 to acidity/alkalinity formula (Slide 26)

17. Primary standards - Slides 27, 28, 29

18. Preparation and storage of any standard solution - Slide 30

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:
A. Visual Materials

X. IPW REAGENT REQUIREMENTS:
   A. None

XI. DESCRIPTION OF VISUAL MATERIALS:
   A. Thirty slides, X-18: Volumetric Analysis - See next pages.
VOLUMETRIC ANALYSIS

STANDARD SOLUTION

SAMPLE

SLIDE 1

TITRATION

PROCESS OF DETERMINING THE STRENGTH OR CONCENTRATION OF AN ACCURATELY MEASURED VOLUME OF A SAMPLE SOLUTION BY REACTING IT WITH AN ACCURATELY MEASURED VOLUME OF A STANDARD SOLUTION (ONE WHOSE STRENGTH OR CONCENTRATION IS KNOWN TO A HIGH DEGREE OF ACCURACY)

SLIDE 2

EQUATION VOLUME OF ADDITION

KNOW CONCENTRATION OF SOLUTE CHEMICAL REACTS

WITH ALL OF THE SAMPLE COMPONENT THE UNKNOWN VOLUME OF SAMPLE CAN BE CALCULATED UNKOWN QUANTITY OF SAMPLE COMPONENT

SLIDE 3

1 REACTIONS

2 COMPLETED REACTIONS

3 VOLUMES

4 CONCENTRATIONS

5 CALCULATIONS

SLIDE 4

NEUTRALIZATION

ACID + BASE = SALT + WATER

+ DONOR (OR ACCEPTOR)

SLIDE 5

OXIDATION - REDUCTION

OXIDATION - INCREASE CHARGE

REDUCTION - DECREASE CHARGE

SLIDE 6

COMPLETION - ALL SAMPLE COMPONENT REACTED

PRODUCTS - GAS OR PRECIPITATE OR WATER

END POINT - ALL REACTANTS CHEMICALLY CHANGED

INDICATORS - SOME OBSERVABLE CHANGE AT THE END POINT

SLIDE 7

VISUAL INDICATORS

1 REAGENT

2 ACID-BASE INDICATORS

3 REDOX INDICATORS

4 PRODUCT

5 OTHERS

SLIDE 8

VALLEYS

HOO:CH₂ - H₂O
HOO:CH₂ - H₂O

M° - CHROME BLACK T (CHROME BLACK T)
M° - EDTA (EDTA) COMPLEX

SLIDE 9
## Electrical Indicators
1. Potential
2. Conductivity
3. Amperage
4. Dielectric Properties
5. Coulomb Measure

## Volumes
Sample - Use Pipet if 1.0L
Use Graduate if > 1L

Standard Solution - Use Burette

## Standard Solution - One Whose Composition and Concentration Are Known to a High Degree

---

### Important Terms
1. Mole
2. Equivalent Weight
3. Normality

---

### Molecular Weights

<table>
<thead>
<tr>
<th>Atom</th>
<th>Number</th>
<th>Weights</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>C_5</td>
<td>3</td>
<td>60</td>
<td>180</td>
</tr>
<tr>
<td>P</td>
<td>2</td>
<td>31.05</td>
<td>62</td>
</tr>
<tr>
<td>O</td>
<td>3</td>
<td>16</td>
<td>48</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>310</strong></td>
<td><strong>310</strong></td>
<td></td>
</tr>
</tbody>
</table>

- Molecular Weight = Sum of atomic weights
- $\text{Molecular Weight of } C_5H_4PO_3 = 310 \text{ g/mol}$

- One mole of $C_5H_4PO_3$ weighs 310 grams
- One mole of $C_5H_4PO_3$ contains 6.02 x 10$^{23}$ molecules

---

### Molar Solutions

- Design: Mole of parts of a solution
- Normality = Mole of solute
- Molarity = Number of moles solvent
- Mole (Moles) = 1 mole
- Liters (Molar) = 1 mole
- Liters (0.5 Molar) = 0.5 moles
- Liters (1.5 Molar) = 1.5 moles

---

### Equivalent Weights

- Equivalent Weight = Ions or Total = Ions
- Equivalent Weight = Weight of a substance which will furnish, react with, or be equivalent to one proton (H+) in the reaction which occurs.
SERIES X-18: VOLUMETRIC ANALYSIS

NORMAL SOLUTIONS

WEIGHT OUT EQUIVALENT WEIGHTS OR PARTS OF EQUIVALENT WEIGHTS, AND DILUTE AS PER LITERS OF SOLUTION.
NORMALITY = NUMBER OF EQUIVALENT WEIGHTS / LITER OF SOLUTION.

EQUIVALENT WEIGHTS - ACIDS

<table>
<thead>
<tr>
<th>rxn</th>
<th>at wt</th>
<th>eq wt</th>
<th>total wt</th>
</tr>
</thead>
<tbody>
<tr>
<td>HCl</td>
<td>36.46</td>
<td>36.46</td>
<td>36.46</td>
</tr>
<tr>
<td>HNO₃</td>
<td>63.01</td>
<td>63.01</td>
<td>63.01</td>
</tr>
<tr>
<td>H₂SO₄</td>
<td>98.00</td>
<td>98.00</td>
<td>98.00</td>
</tr>
</tbody>
</table>

EQUIVALENT WEIGHTS - SALTS

<table>
<thead>
<tr>
<th>oxn</th>
<th>eq wt</th>
<th>g wt</th>
<th>change in charge</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe²⁺</td>
<td>55.85</td>
<td>1.01</td>
<td>-0.35</td>
</tr>
<tr>
<td>Fe³⁺</td>
<td>55.85</td>
<td>3.01</td>
<td>+0.35</td>
</tr>
</tbody>
</table>

Oxidation: Fe²⁺ → Fe³⁺
Reduction: Fe³⁺ → Fe²⁺

TOTAL CHANGE IN CHARGE: 0.35 + 0.35 = 0.70

NORMALITY (%) SOLUTIONS

1. normality = no. eq. wts. / ltr. of soln.
2. (g) (vol %) = no. eq. wts. / (g) eq. wts.
3. (g) (vol %) = grams / eq. wt.
4. (g) (vol %) = (eq. wt.) / grams

FINDING GRAMS

grams = (sample) / (std) / (std)

ACIDITY OR ALKALINITY

mg/l CO₃⁻ = (V₁ - V₂) (Mₚ) / (1000)

mg/l CO₃⁻ = (Vₐ) (Mₚ) / (1000)

mg/l CO₃⁻ = (g eq. wt.) / (eq. wt.)

Thus:
mg/l CO₃⁻ = (g eq. wt.) / (eq. wt.)
**SERIES #18 VOLUMETRIC ANALYSIS**

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**SLIDE 28**

**Requirements for Primary Standards**

- Purity
- Quantitative reaction
- Heat stable
- Non reactive to air
- High equivalent weight available
- Reasonable cost

---

**SLIDE 29**

**Primary Standards**

1. For a base - Potassium acid phthalate
   - Sulfuric acid
   - Benzoic acid
2. For an acid - Sodium carbonate
   - Calcium carbonate
3. For salts - Pure form of substance
   - Salt will react with in analysis

---

**SLIDE 30**

**Preparation and Storage**

- Volumetric flasks
- High quality distilled water
- Clean glassware
- Dark glass bottles
- Tight cap
- Others specific for standard

---

13-7

142
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: pH

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 45 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Participants need to know how to correctly standardize and use a pH meter to determine acidity and alkalinity, and as part of the analytical procedures for hardness, phosphorus, and nitrate and nitrite nitrogen.

V. ENTRY LEVEL BEHAVIOR:
A. Understanding of terms:
   1. logarithm
   2. acid
   3. base
   4. ion

VI. INSTRUCTIONAL OBJECTIVE:
A. Terminal Behavior: The participant will know the meaning of pH scale numbers, how to check pH equipment before use, how to standardize a pH meter using two buffer solutions and how to use the calibrated meter to determine pH.

B. Conditions: He/she will be given the training manual and 45 minutes of instruction. Later in the course, he/she will be given time and the necessary equipment to calibrate and use a pH meter.

C. Accepted Performance. Subsequent use of pH meter to correctly determine acidity and alkalinity in samples, according to an Instructor's rating. He/she must also correctly answer 70% of the items on this topic in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:
A. Available Media:
   1. One outline in the training manual, "Acidity, Alkalinity, pH and Buffers"
   2. Seven overhead transparencies, OT-11:pH (See XI. Description of Visual Materials).

B. Suggested Media:
   1. None
VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the topic outline in the training manual and the visual materials. See XI below.

2. Prepare the lesson using Sequencing below or your own organization of the material.

3. Assemble meter, electrode(s), buffers, thermometer, beakers, distilled water, tissues, etc., to demonstrate use of a pH meter.

B. Sequencing:

Overheads are series OT-11:pH The training outline has reference information.

1. Overheads - The depth of this portion depends on the need and backgrounds of the participants. OT 4, 5, 6 and 7 topics may not be appropriate.

   a. Introduction - See IV above, Justification
   b. Basis and definition of pH scale - OT 1, 2
   c. Theory of operation utilized in pH meter - OT 3
      NOTE: A combination electrode has both functions.
   d. Construction and functions of electrodes - OT 4, 5, 6, 7
      NOTE: A combination electrode utilizes a specific metal chloride such as AgCl₂ along with the KCl.

2. Explanation with demonstration of:

   a. Checking a meter and electrode(s) for defects before use.
   b. Standardizing a meter using one buffer to set the meter and a second buffer to check for accurate read-out. Rinse between readings.
      NOTE: The first buffer should have a pH approaching that of the sample or of the end point desired in a titration procedure. The second buffer should have a pH about 4 units different from the first.
   c. Using the meter and electrode(s) to find pH values. Also rinsing between readings.

3. Note conditions for storing a meter and electrode(s).

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visual materials

B. Equipment to demonstrate use of a pH meter.
X. IPW REAGENT REQUIREMENTS:

A. None (Using a pH meter is utilized in subsequent laboratory assignments).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Seven overhead transparencies, OT-11:pH - See next page.
SERIES 0T-11. pH

IONIZATION CONSTANT FOR WATER

- Water dissociates to yield a concentration of hydrogen ions equal to 10^-7 molar:
  \[ H_2O \rightleftharpoons H^+ + OH^- \]
- Since water dissociates to produce one hydrogen ion for each hydrogen ion it is obvious that 10^-7 mole of hydrogen ion is produced simultaneously.

\[ [H^+] = 10^{-7} \times 10^{-7} \times 10^{-14} \]

SLIDE 1

pH CONCEPT

- Expression of hydrogen-ion concentration in terms of molar concentrations rather cumbersome.
- Sorensen (1909) proposed to express such values in terms of their negative logarithms.

\[ pH = -\log[H^+] \quad \text{or} \quad [H^+] = 10^{-\text{pH}} \]

SLIDE 2

pH METERS THEORY OF OPERATION

- Measure electrical potential between two suitable electrodes.
- Reference electrode assumes a constant potential (0.044 volt).
- Indicating electrode assumes a potential dependent on the pH of the solution.

SLIDE 3

INDICATING ELECTRODE

- Hydrogen ion selective glass electrode.
- Not affected by oxidizing or reducing substances.
- Basic design is a silver-silver chloride or mercury-mercurous chloride electrode contained in a solution of known pH and sealed in glass.

SLIDE 4

INDICATING ELECTRODE MECHANISM OF OPERATION

- The mechanism by which the glass membrane responds to hydrogen ion activity involves absorption of hydrogen ions on both sides of the membrane proportionately to the activity of the hydrogen ions in solution.

SLIDE 5

INDICATING ELECTRODE MECHANISM OF OPERATION

- The voltage of the glass electrode is a logarithmic function of the difference in hydrogen ion activity of the solutions on either side of the glass membrane.

SLIDE 6

\[ [H^+] = [H^+] \text{activity} \]

SLIDE 7

14c
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Acidity and Alkalinity

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 125 minutes (30+75+20)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:
   A. Fundamental knowledge of inorganic chemistry
   B. Ability to correctly use volumetric glassware and titration assemblies
   C. Completion of the lessons (or equivalent):
      1. pH
      2. Volumetric Analysis
      3. Laboratory Safety Practices
   D. Ability to perform calculations involving decimals

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will know pertinent facts about acidity and alkalinity (see VIII B.) and will determine acidity in one sample and alkalinity in two samples using the procedures in the training manual (see VII A.1, c. and d.). He/she will calculate and report results and participate in a class discussion of the topics and laboratory procedures.
   B. Conditions: He/she will be given the training manual, 30 minutes of instruction, 75 minutes laboratory time, all necessary equipment, reagents and samples, and 20 minutes for class discussion.
   C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an Instructor, and obtain results for each sample within ± 10% of the class average (unless the reason for a lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:
   A. Available Media:
      1. Four outlines in training manual:
         a. "Acidity, Alkalinity, pH and Buffers"
b. "Alkalinity and Relationships Among the Various Types of Alkalinites"


d. "Laboratory Procedure for Total Alkalinity" according to 1974 EPA Methods for Chemical Analysis, p. 3

2. Twenty-eight slides, X-20: Alkalinity (See XI. Description of Visual Materials.)

B. Suggested Media: None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies: See IX below.

2. Prepare all necessary reagents and samples as in X below.

3. Review the laboratory procedures by performing the student assignment using the outlines in the training manual.

4. Review the topic outlines in the training manual and the visual materials. See XI below.

5. Prepare the lesson using the Sequencing below or your own organization of the material.

6. Check pH meters for proper operation.

7. Distribute supplies and reagents to laboratory stations.

8. Assemble demonstration items: buret, pipet, pipet bulb and tissues.

B. Sequencing: Slides are series X-20: Alkalinity

1. Classroom Instruction on Alkalinity:

   a. Types of water samples of interest - Slide 1

   b. Definitions - Slides 2, 3

   c. Sources - Slides 4, 5

   d. Applications of data - Slides 6, 7, 8

   e. Sample handling - Slides 9, 10, 11, 12
      Note: Do not open bottle before analysis (Slide 10)
      Note: Do not dilute samples (Slide 12)

   f. Procedure - Slides 13, 14, 15, 16, 17
      Note: 4.5 is the specified endpoint for NPDES data (Slide 13)
      Note: For alkalinity greater than 1000 mg, use 0.1N titrant. (Slide 15)
2. Classroom Briefing on Alkalinity Laboratory - Use training manual outline, "Laboratory Procedure for Total Alkalinity."
   
a. Calibrate pH meter. Set with pH 4.0 buffer; check with pH 6.0 buffer. (Briefly recall the steps which were demonstrated in the lesson on pH).
   
b. Note section V with steps of alkalinity procedure. The acid has been standardized.
   
c. When titrating to 4.5, record pH when a rapid change in pH occurs. Continue drop-by-drop titration to pH 4.5.
   
d. Test two samples.
   
e. Calculation formula is in procedure write-up.
   
3. Classroom Instruction on Acidity
   
a. Sources - Strong mineral acids, weak acids, hydrolyzing salts
   
b. Applications of data - Corrosivity of water; affects certain chemical and biological processes, can reflect changes in water quality.
   
c. Sample handling - Same as for alkalinity
   
d. Procedure Overview - Reason for 8.2 end point which represents neutralization of carbonic acid to the bicarbonate ion. Note: 8.2 is the specified end point for NPDES data.
   
4. Classroom Briefing on Acidity Laboratory - Use training manual outline, "Laboratory Procedure for Acidity."
   
a. Section IV has steps of acidity procedure. The base has been standardized. For IV.D, caution about handling of hydrogen peroxide. Test one sample.
   
b. Do alkalinity procedure while acidity sample is cooling.
   
c. Calibrate pH meter. Set with pH 9.0 buffer; check with pH 6.0 buffer. (The steps involved were demonstrated in the lesson on pH).
   
d. Calculation formula is in procedure write-up.
   
5. Summarize order of laboratory work.
6. Laboratory-Student performance of the assigned determinations (75 minutes)

7. Class Discussion (20 minutes)
   a. Board-Calculation formula reduced to factor of 20
   b. Discuss significant figures to record for buret readings.
   c. Students calculate and post results. Also post ml of titrant used for each of the three samples.
   d. Find range of results.
   e. Announce theoretical results. See X. below.
   
   f. Demonstrate two corrections of technique error: removing air bubble in tip of buret and wiping excess off outside of pipet tip.
   g. Stress reason for 4.5 and 8.2 end points. Slide 27 has pH values associated with neutralizations. It can be used to discuss both endpoints. Stress that for acidity determinations for NPDES, the endpoint is 8.2, just below the 8.3 shown for bicarbonate ions in solution.
   h. Students report observations about rate of pH changes when titrating alkalinity samples.
   i. Relate observations to titration curves; rapid changes in pH during titration occur at the equivalence points shown by steeper curves on slide 28. Discuss applications shown in Table 4 of outline, "Alkalinity and Relationships Among the Various Types of Alkalinitites." Slide 28 can be used to demonstrate the presence of contributors of alkalinity at the pH values used for the Table and which account for the titration results shown there.
   j. Answer any questions.

IX. NEW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 acidity sample and 2 alkalinity samples

A. For each station (no more than two persons per station):
   1. Three 100 ml beakers (for buffers)
   2. One 100 ml beaker (for titrants)
   3. One 150 ml beaker (for acid sample)
   4. One 150 ml beaker (for alkalinity samples)
   5. One 150 ml or larger beaker (for buret wastes)
   6. One large pipet bulb
7. One 25 ml buret mounted on a stand
8. One small funnel to fit the buret
9. One pH meter with electrode(s) in a 100 ml beaker with enough 6.X buffer or distilled water to cover the tip(s)
10. Magnetic stirring apparatus
11. One thermometer
12. Tongs (to transport 150 ml beaker)
13. One squeeze bottle of distilled water
14. One marking pencil
15. One apron for each student
16. One pair safety glasses for each student

B. Shared
1. X bottles for samples (3 samples, 1 of each per 3 stations)
2. X 50.0 ml volumetric pipets (one in each bottle of sample)
3. Two 50 ml beakers (in hood for H₂O₂)
4. Two medicine droppers (in hood for H₂O₂)
5. Supply of acid-cleaned glass boiling beads, 3 each acid sample (in hood)
6. Scoop to transfer boiling beads
7. Hot plates to accommodate the total number of 150 ml beakers containing acidity samples


A. Minimum amounts per station (no more than two persons per station):
1. Seventy-five ml pH 4.X buffer
2. One hundred-fifty ml pH 6.X buffer
3. Seventy-five ml pH 9.X buffer
4. Five drops hydrogen peroxide, 30% solution
5. Twenty-five ml 0.020 N HCl or H₂SO₄ titrant
6. Fifteen ml 0.020 N NaOH titrant
7. Fifty ml acidity sample *(Dilute 300 ml 0.020 N HCl or H₂SO₄ to one liter. Fifty ml of this sample requires 15 ml 0.020 N NaOH to reach pH 8.2 and has 300 mg/L CaCO₃ acidity).

8. Fifty ml alkalinity sample *(Dilute 300 ml 0.020 N Na₂CO₃ solution to 1 liter. Fifty ml of this sample requires 15 ml 0.020 N HCl or H₂SO₄ to reach pH 4.5 and has 300 mg/L CaCO₃ alkalinity).

9. Fifty ml alkalinity sample *(Dilute 200 ml 0.020 N NaOH to 1 liter. Fifty ml of this sample requires 10 ml 0.020 N HCl or H₂SO₄ to reach pH 4.5 and has 200 mg/L CaCO₃ alkalinity).

10. Wastewater treatment plant influents or effluents may be used as samples if they are known to contain acidity/alkalinity.

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of distilled water should be available during laboratory sessions.

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Twenty-eight slides, X-20: Alkalinity - See next pages.
ALKALINITY MEASUREMENTS
NATURAL WATERS
WASTEWATERS
DIGESTING SLUDGES

SLIDE 1

ALKALINITY
CAPACITY TO ACCEPT
HYDROGEN IONS (PROTONS)

SLIDE 2

ALKALINITY
CAPACITY TO
NEUTRALIZE ACIDS

SLIDE 3

HYDROXIDE (OH)
CARBONATE (CO3)
BICARBONATE (HCO3)

SLIDE 4

Salts of Weak Acids
acetates phosphates
borates
sulfides silicates

SLIDE 5

TREATMENT PLANTS
AMENABILITY (to treatment)
BUFFER CAPACITY
ANAEROBIC DIGESTION
COAGULATION
INDUSTRIAL DISCHARGES

SLIDE 6

INDUSTRY
- BOILERS
- CORROSION CONTROL
- WATER SOFTENING

SLIDE 7

REGULATORY AGENCIES
- TREATED WATER
- ALKALINE DISCHARGES

SLIDE 8

PHOTO OF SAMPLE BOTTLES

SLIDE 9
**SERIES X-20: ALKALINITY**

**SLIDE 10:**

\[ CO_2 \uparrow \]

Do alkalinity determination immediately.

**SLIDE 11:**

- 24 hours
- 4°C

**SLIDE 12:**

Do not treat samples.

**SLIDE 13:**

Titrate samples to pH 4.5.

**SLIDE 14:**

Photo of adding titrant.

**SLIDE 15:**

Photo of two acids.

**SLIDE 16:**

Titration rapid / minimal agitation.

**SLIDE 17:**

Approaching pH 4.5 drop-by-drop additions.

**SLIDE 18:**

Results: equivalent calcium carbonate milligrams/liter.
Neutralization of Carbonate

\[ \text{H}^+ + \text{CO}_3^- \rightarrow \text{HCO}_3^- \]

\[ \text{H}^+ + \text{HCO}_3^- \rightarrow \text{H}_2\text{CO}_3 \]

Neutralization of Bicarbonate

\[ \text{H}^+ + \text{HCO}_3^- \rightarrow \text{H}_2\text{CO}_3 \]

Neutralization of Hydroxide

\[ \text{H}^+ + \text{OH}^- \rightarrow \text{H}_2\text{O} \]
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Indeterminate Error - Precision

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 60 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: An analyst must know how to check laboratory results for reproducibility (precision) to substantiate the reliability of his/her data.

V. ENTRY LEVEL BEHAVIOR:

A. Understanding of how to use formulas involving squares and square roots.

B. Though not essential, it is orderly to have completed the lessons (or equivalent):
   1. Sample Handling
   2. Compliance Methodology

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know the difference between accuracy and precision, the cause and synonyms for indeterminate error, the role of the normal distribution curve, where to find formulas and examples of each for variance, standard deviation, % relative standard deviation and range, how to establish one's precision in performing an analysis and how to continue to monitor precision on a daily basis. Each will complete an assignment to calculate three precision statistics.

B. Conditions: He/she will be given the training manual and 60 minutes of instruction.

C. Accepted Performance: He/she must correctly identify at least 70% of the items pertaining to the objective in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:
   1. Three outlines in training manual:
      a. "Statistics for Chemists"
      b. "Accuracy - Precision Error"
      c. "Elements of a Quality Assurance Program"
2. Twenty slides, X-24: Precision (See XI Description of Visual Materials).


4. HANDOUT: Assignment to calculate three statistics. A copy is at the end of this IPW.

5. HANDOUT: Key to assignment. A copy is at the end of this IPW.


B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review visual materials and available handouts.

2. Prepare the lesson using the Sequencing below or your own organization of the material.

3. Duplicate copies of any handout materials.

B. Sequencing: Slides are X-24: Precision (Optional—slides X-26: Quality Assurance)

1. Introduction:

   a. Before considering precision of results, one must have a valid sample and a skilled analyst using recognized analytical methodology.

   b. Overview: Consideration of precision, i.e. the ability to reproduce results of chemical analyses, causes of error, and how to quantitate the effect of errors we cannot control. Statistics presented will be those most commonly used in recognized method references: variance for calculating standard deviation, % relative standard deviation, and range because it is an estimate of standard deviation.

   c. Outlines: Formulas and examples of the calculations involved are in outline, "Statistics for Chemists" (S.C.). Discussion items from the outline "Accuracy - Precision - Error" are marked (A.P.E.).

2. Precision and Accuracy:
a. Analytical results are imperfect (A.P.E.) - Slide 1

b. Define/exemplify accuracy (A.P.E.) - Results are close to the true value-standard. Accuracy can also be checked by recovery of a constituent - spike. - Slide 2.

c. Define/exemplify precision and contrast it to accuracy (A.P.E.). Stress that both accuracy and precision are requirements for reliable data. - Slide 3.

3. Error

a. Overview: Classification and causes of errors (A.P.E.) - Slide 4

b. Synonyms for indeterminate error - Use (A.P.E.).

c. Normal Distribution (S.C.) - Slide 5

4. Statistics

a. Normal distribution is a population characteristic. An analyst deals with a sample of a population. Statistics are derived from samples. - Slide 6

b. Define "statistic". - Slide 7

c. Mean - Note location of aids to calculate in (S.C.) - Slide 8

d. Variance and standard deviation - Slide 9

e. Calculating variance - Slide 10, 11, 12

f. Calculating standard deviation - Slide 13

1) Note location of calculator formula (S.C.) - Slide 14

Also note location of aids to manually calculate (S.C)

2) Note variations of formula in (A.P.E.).

3) Note EPA manual often uses standard deviation for precision statement.

g. Relationship of standard deviation and normal distribution - Slide 15

h. Normal distribution requirement and checking with probability paper - Slide 16

i. Checking own precision (Elements of a Quality Assurance Program) - Slide 17

j. Coefficient of variation (% relative standard deviation) (S.C.) - Slide 18 CONTINUED
1) Note concentration affects precision and accuracy.

2) Note Standard Methods often uses % relative standard deviation for precision statement.

k. Range - Slide 19

1) Note application in (A.P.E.) as estimate of standard deviation - Slide 20.

5. Daily Application

a. Suggested (EPA Handbook for AQC) - 10% of tests be duplicates to check precision

b. Can construct control charts with these statistics to monitor daily precision performance. (This topic is in Quality Assurance Program lesson. You may want to use Slides 1, 2, and 3 from X-26, Quality Assurance, now to demonstrate precision control charts).

6. Summary

a. Again stress valid sample and skilled analyst using recognized analytical methodology are prior to checking precision.

b. Summarize presentation.

7. Assignment

a. Hand out assignment to calculate three statistics outside of class time. Give a due time.

b. When due, collect the assignment and check the work of each to identify any questions.

c. Return assignment with a copy of the key for participant's reference.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visual materials

X. IPW REAGENT REQUIREMENTS:

A. None

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Twenty slides, X-24: Precision - see next pages
ANALYTICAL RESULT

An Imperfect Estimate of Some Desired Real Value

SLIDE 1

DETERMINATE ERROR

- Method
- Operator
- Instrument

INDETERMINATE ERROR

- Random
- Uncontrollable

SLIDE 4

NORMAL DISTRIBUTION

POPULATION
- Any finite or infinite collection of individual objects
- Completeness
- Greek letters represent parameters, e.g., $\sigma$ represents variance

SAMPLE
- A portion of a population
- Incompleteness
- Roman letters represent statistics, e.g., $s$ represents variance

SLIDE 5

STATISTIC

- A Single Value
- Chosen or Computed from a Series of Sample Data

SLIDE 7

MEAN

$\bar{x} = \frac{1}{n} \sum x_i$

ARITHMETIC AVERAGE OF ALL THE VALUES IN A SAMPLE DISTRIBUTION

SLIDE 8

VARIANCE formula

ST'D DEVIATION formula

SLIDE 9
The table represents the data values and their mean deviations. The formula for calculating the standard deviation is shown, and the graph illustrates a normal distribution. The information suggests a precision analysis involving 4 concentration levels, 7 replicates, and 2 hour exposures, comparing to methods.
RANGE (R)
MAXIMUM VALUE
MINUS
MINIMUM VALUE

R as ESTIMATE of s
\( R \sim s \)
Given this set of 7 data items:

\[ x_1, 3, 3, 5, 5, 7, 7, 12 \]

What is the standard deviation, \( S \)?

\[
S = \sqrt{\frac{\sum_{i=1}^{n} x_i^2 - (\frac{\sum_{i=1}^{n} x_i}{n})^2}{n-1}}
\]

What is the % relative standard deviation, \( \frac{S}{\bar{x}} \times 100 \)?

What is the range, \( R \)?

16-0

164
Given this set of 7 data items:

\[ \begin{align*}
X_i & \quad X_i^2 \\
3 & \quad 9 \\
3 & \quad 9 \\
5 & \quad 25 \\
5 & \quad 25 \\
7 & \quad 49 \\
7 & \quad 49 \\
12 & \quad 144 \\
\end{align*} \]

What is the standard deviation, \( S \)?

\[
S = \sqrt{\frac{\sum_{i=1}^{n} X_i^2 - (\sum_{i=1}^{n} X_i)^2}{n-1}}
\]

\[
= \sqrt{\frac{\sum_{i=1}^{7} X_i^2 - (\sum_{i=1}^{7} X_i)^2}{6}}
\]

\[
= \sqrt{\frac{310 - 252}{6}}
\]

\[
= \sqrt{\frac{58}{6}}
\]

\[
= \pm 3.11
\]

What is the % relative standard deviation, \( \frac{S}{100} \)?

\[
\frac{S}{100} = \frac{3.11}{6.00} \times 100 = 51.83\%
\]

What is the range, \( R \)?

\[ 12 - 3 = 9 \]
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Calcium and Magnesium Hardness

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 92 minutes (40+45+7)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:
A. Fundamental knowledge of inorganic chemistry
B. Ability to correctly use volumetric glassware and titration assemblies
C. Ability to perform calculations involving decimals
D. Completion of the lessons (or equivalent):
   1. Volumetric Analysis
   2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:
A. Terminal Behavior. The participant will learn pertinent facts about hardness (see VIII. B.) and will determine total hardness in a municipal wastewater treatment plant effluent using the procedure in the training manual (see VII. A. 1. b.). He/she will calculate and report results and participate in a class discussion of the topics and laboratory procedures.
B. Conditions: He/she will be given the training manual, 40 minutes of instruction, 45 minutes laboratory time, all necessary equipment and reagents, a sample, and 7 minutes for class discussion.
C. Accepted Performance. He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedure to the satisfaction of an Instructor and obtain a result for the sample within ± 10% of the class average (unless the reason for the lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:
A. Available Media:
   1. Two outlines in training manual:
      a. "Determination of Calcium and Magnesium Hardness"
      b. "Laboratory Procedure for Total Hardness" according to 14th ed. Standard Methods, p 202
   2. Twenty-three slides, X-22: Hardness (See XI. Description of Visual Materials)
B. **Suggested Media:**

1. None

**VIII. INSTRUCTIONAL APPROACH:**

A. **Preparation for Instruction:**

1. Check equipment and supplies. See IX below.
2. Prepare all necessary reagents and sample as in X below.
3. Review the laboratory procedure by performing the student assignment using the outline in the training manual.
4. Review the topic outline in the training manual and the visual materials. See XI below.
5. Prepare the lesson using the Sequencing below or your own organization of the material.
6. Distribute supplies and reagents to laboratory stations.

B. **Sequencing:** Slides are Series X-22: Hardness

1. **Classroom Instruction - 40 minutes**
   a. Name and units of the parameter as mentioned in the Federal Register - Slide 1.
   b. Partial definition of hardness, excerpted from old US PHS literature - Slide 2.
   c. This slide is an extension of Slide 2 - Slide 3.
   d. Other hardness-causing cations - Slide 4.
   e. Anions associated with the hardness-causing cations - Slide 5.
   g. Ranges of hardness, according to Sawyer - Slide 7.
   h. Processes for removing hardness - Slide 8.
   i. Ionization/dissociation of inorganic and organic acids - Slides 9 and 10. The purpose of these slides is to show, by analogy to simple inorganic and organic acid molecules, how a soap molecule is formed via neutralization.
   j. How calcium forms an insoluble precipitate with a soap molecule - Slide 11.
   k. Federal Register methods for determining surfactants - Slide 12.

m. Steps in the EDTA titration method - Slide 14.
   The metal in step 1 is that which is part of the indicator; that in step 2 is what is in the sample.

n. Indicators used in the EDTA titration procedure - Slide 15.

o. Two-dimensional representation of the Calcium - EDTA complex - Slide 16.

p. Problems in the EDTA titration procedure - Slide 17.

q. Interferences in the EDTA procedure - Slide 18.

r. Two forms of calcium and magnesium which may be determined - Slide 19.

s. Indicators used in the EDTA titration of calcium - Slide 20.


2. Laboratory - 45 minutes - Use training manual

a. Briefing in laboratory
   1) Calibrate pH meter. Set with pH 9.0 buffer; check with pH 6.0 buffer.
   2) Do Section II of the laboratory outline using 25 ml tap water instead of the 25 ml of CaCO₃ indicated in II.A. This allows participants to become accustomed to the color changes.
   3) Repeat Section II, this time using CaCO₃.
   4) Calculate the "B" factor according to Section IV.A.
   5) Titrate the sample according to Section III.
   6) Calculate total hardness for the sample according to Section IV.B.

b. Student performance of the assignment
3. Class Discussion - 7 minutes
   a. Students post results.
   b. Discuss results, exclude outliers, calculate the average.
   c. Cover any items from laboratory session.
   d. Answer any questions.

IX. EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Three titrations - tap water, CaCO₃ standard solution, municipal wastewater treatment plant sample

A: For each station (no more than two persons per station):
   1. One 100 or 150 ml beaker (for buffer)
   2. One small beaker (for buret, wastes)
   3. One 25 ml buret mounted on a stand
   4. One small funnel to fit the buret
   5. One 10 ml graduated cylinder
   6. One 25 ml graduated cylinder
   7. One 25 ml volumetric pipet
   8. One pipet bulb
   9. One small spatula or measuring scoop (for 0.2 g of indicator)
   10. One pH meter with electrode(s) in a beaker with enough 6.0 buffer or distilled water to cover the tip(s). Also, one thermometer and stirring mechanism.
   11. One flask, 125 ml Erlenmeyer if a combination pH electrode is used. Otherwise, a 150 ml beaker.
   12. One squeeze bottle of distilled water
   13. One marking pencil
   14. One apron for each student
   15. One pair of safety glasses for each student

B. Shared:
   1. None
X. IPW REAGENT REQUIREMENTS. For details of reagent preparations, see 14th edition, "Standard Methods", page 203.

A. Minimum amounts per station (no more than two persons per station):

1. Twenty-five ml calcium carbonate standard, 1 mg CaCO₃/ml
2. Six ml buffer solution (in a pipettor; otherwise; use dropper bottles or a graduated pipet)
3. Seventy-five ml disodium ethylenediamine tetraacetic acid dihydrate
4. One hundred ml pH 6.X buffer
5. One hundred ml pH 9.X buffer
7. Twenty-five one hundredths g inhibitor. This is usually not required. (See Standard Methods for discussion and choices).
8. Twenty-five ml municipal wastewater treatment plant effluent for sample (If not available, use a calcium carbonate solution with concentration withing 15 ml titrant volume limit).

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Twenty-three slides, X-22, Hardness - See next pages.
TOTAL HARDNESS
mg CaCO₃/1

HARDNESS DEFINITION
the total concentration of just the calcium and magnesium ions...

HARD WATER - high in calcium, magnesium, and iron ions
Relative to the amount of sodium and potassium ions
SOFT WATER - low in calcium.

OTHER CONTRIBUTORS TO HARDNESS
- ALUMINUM
- IRON
- MANGANESE
- STRONTIUM
- HYDROGEN IONS

ASSOCIATED ANIONS
- HCO₃⁻
- SO₄⁻²
- CI⁻
- NO₃⁻
- SiO₄⁻²

VARIATIONS IN HARDNESS
- LEACHING OF DIFFERENT SOIL AND ROCK
- WASTE DISCHARGE
- USAGE OF THE WATER
  - IRRIGATION
  - SOFTENING

HARDNESS CLASSIFICATION
<table>
<thead>
<tr>
<th>Type</th>
<th>MG/L</th>
</tr>
</thead>
<tbody>
<tr>
<td>SOFT</td>
<td>0-75</td>
</tr>
<tr>
<td>MODERATELY HARD</td>
<td>75-150</td>
</tr>
<tr>
<td>HARD</td>
<td>150-300</td>
</tr>
<tr>
<td>VERY HARD</td>
<td>&gt;300</td>
</tr>
</tbody>
</table>

HARDNESS REMOVAL
- ZEOLITES
- LIME SODA
- HOT PHOSPHATE
- COMPLEX FORMATION
- DISTILLATION

Inorganic (Mineral) Acid

\[ HCl \rightarrow H^+ + Cl^- \]

cation hydrogen anion chloride proton ion
Organic (Cotbac) Acid

\[
\text{CH}_3\text{C}=\text{O} - \text{H} + \text{CH}_3\text{C} = \text{O} \\
\text{cotton anion}
\]

**Objectives to Hardness**
1. Soap destroying properties
2. Ca, HCO\(_3\), Na
3. Ca\(^{++}\) soluble
4. (Ca, HCO\(_3\), Ca)\(^{++}\) insoluble
5. 2 formation of deposits

**Total Hardness as CaCO\(_3\) mg/l**
1. EDTA titration
2. Technicon - E.B.T.
3. Calculation - Ca and Mg by Atomic Absorption

**HARDNESS**
- Application: Drinking and Surface Waters Domestic and Industrial Wastes
- Range: All Concentrations

**EDTA Hardness Titration**
1. Indicator - Metal \(\rightarrow\) Rose Complex
2. EDTA - Metal \(\rightarrow\) Colorless Complex
3. EDTA - Rose Complex \(\rightarrow\) Blue Color

**EDTA Titratation Problems**
- Poor lighting
- Old buffer

**Interferences**
- Metals (Al, Ba, Cd, Ca, Cu, Fe, Pb, Mn\(^{++}\), Ni, Sr, Zn)
- Polynphosphates
- Organics
CALCIUM AND MAGNESIUM
Total (digestion)
Dissolved
(0.45 µm filtration)

CALCIUM HARDNESS INDICATORS
- Murexide
- Eriochrome blue black R
- Others

DETERMINATION OF MAGNESIUM HARDNESS
- By difference total - calcium
- Gravimetric
- Colorimetric

PYROPHOSPHATE METHOD

\[ \text{REACTIONS} \]

\[ \text{CO}_3^{2-} \text{HARDNESS OF OTHER IONS} \]

1. Find concentration of other ion.
2. Use appropriate factor.
3. Ion hardness as calcium carbonate.

SLIDE 19

SLIDE 20

SLIDE 21

SLIDE 22

SLIDE 23
I. SUBJECT MATTER: Chlorination and Chlorine Determinations

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 98 minutes (45 + 45 + 8)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:
   A. Fundamental knowledge of inorganic chemistry
   B. Ability to correctly use volumetric glassware and titration assemblies
   C. Ability to perform calculations involving decimals
   D. Completion of the lessons (or equivalent):
      1. Volumetric Analysis
      2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will learn about chlorination products and terminology, and the chemistry involved in chlorine determinations by iodometric analyses and OPD methodology. He/she will twice determine total chlorine residual in a sample by back titration using amperometric apparatus (See VII A.1.b.), calculate and report results from the second determination and participate in a class discussion of the results.
   B. Conditions: He/she will be given the training manual, 45 minutes of instruction, 45 minutes laboratory time and all necessary equipment, and 8 minutes for class discussion.
   C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedure to the satisfaction of an Instructor and obtain a result for the sample within ± 10% of the class average (unless the reason for the lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:
   A. Available Media:
      1. Two outlines in training manual:
         a. "Chlorination and Chlorine Determinations"
2. Thirty-six slides, X-21: Chlorine (See XI. Description of Visual Materials).

3. OPTIONAL: KEH-70: 11 minute CCTV cassette on "Amperometric Determination of Total Chlorine in Wastewater" presents the stepwise procedure for the back titration using a Wallace and Tiernan titrator. Produced by Kirkwood Community College, it is available for review and purchase (6301 Kirkwood Blvd., Cedar Rapids, Iowa 52406).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.

2. Prepare all necessary reagents and samples as in X below.

3. Review the laboratory procedure by performing the student assignment using the outline in the training manual.

4. Review the topic outline in the training manual and the visual materials. See XI below. If you plan to use VC-116 rather than demonstrate the laboratory procedure, order and review the tape.

5. Prepare the lesson using the Sequencing below or your own organization of the material:

6. Check amperometric titrators for accurate operation. Clean and condition cells if necessary.

7. Distribute supplies and reagents to laboratory stations.

8. If you plan to demonstrate the back titration procedure with an amperometric titrator and/or starch-iodide titration apparatus, assemble the equipment and reagents.

9. If you plan to demonstrate the DPD titration and/or spectrophotometer method(s), assemble the equipment and reagents.

B. Sequencing:

Slides are Series X-21: Chlorine

Participants should use the training outline, "Chlorination and Chlorine Determinations" as a reference for this information.

1. Classroom instruction on chlorination, using slides to discuss the first 3 Sections of the outline in the training manual.

   a. Introduction about bactericidal use of chlorine - Slide 1

   b. Means of chlorination - Slide 2
c. Effects of applying chlorine gas - Slide 3, 4

d. Effects of applying calcium hypochlorite - Slide 5, 6

e. Ammonia reactions with hypochlorous acid produce chloramines - Slide 7, 8

f. Side reactions reduce availability of chlorine for disinfecting purposes - Slide 9

g. Chlorination products that have disinfection powers - Slide 10

h. Stress the meaning of the terms related to chlorine residuals - Slide 11, 12, 13

i. Factors affecting disinfection - Slide 14

j. It is convenient to use a blank slide (15) here.

2. Classroom overview of recognized chlorine determinations, using headings in training manual sections on "Iodometric Analyses" and "DPD Methodology", then the summary of "Compliance Methodology".

3. Classroom instruction on the chemistry involved in direct iodometric titrations to determine free, combined or total chlorine. This series of slides presents facts about the chemistry of the method with pictures of an analyst doing each step using an amperometric titrator. Presentation of the direct method gives an opportunity to teach the chemistry involved and also to re-enforce mastery of the meaning of the terms "free," "combined", and "total" residual chlorine. However, for wastewaters the indirect or back titration is to be used. If participants will routinely analyze wastewater, you can still use these slides to teach the chemistry involved and the meaning of the above terms, but spend more time on the actual steps to do a back titration (next topic).

A summary of Iodometric Titration Analyses is in Section IV of the outline in the training manual. Participants can use this as a reference for the following information:

a. Types of chlorine - Slide 16

b. Role of phenylarsine oxide - Slide 17

c. Picture of titration equipment - Slide 18

d. pH for types - Slide 19

e. Principle for free chlorine - Slide 20

f. 1 ml pH 7 buffer has been added and current flows, pointer to right - Slide 21
g. PAO reduces, current reduced - Slide 22
h. Adjust pointer to right - Slide 23
i. More PAO, current reduced - Slide 24
j. Needle stops, take reading - Slide 25
k. More PAO. When needle stays stopped, it's end. (Slide 26 is same as slide 25 to show needle staying same) - Slide 26
l. 0.00564 N PAO related to chlorine for a 200 ml sample - Slide 27
m. PAO related to free chlorine at pH 6.5 - 7.5 - Slide 28
n. Combined chlorine may be present - Slide 29
o. Utilize chlorine - iodide reaction - Slide 30
p. Need pH 4 and potassium iodide - Slide 31
q. Combined chlorine releases iodine - Slide 32
r. Add KI solution (crystals may be used instead of a solution) - Slide 33
s. Add pH 4 buffer - Slide 34
t. Repeat titration procedure as for free chlorine; when needle stays at one place, it is the end point - Slide 35
u. Add results for free and for combined chlorine to give total. Stress: can do total in one step by adding KI and pH 4 buffer directly to sample - Slide 36

4. Classroom instruction/demonstration on indirect (back titration) iodometric procedures to determine total chlorine. Reference information is in the outline in the training manual, Section IV:
   a. The indirect or back titration is required for wastewaters because constituents in the sample can react with liberated iodine during titration and cause low results for the PAO reaction with liberated iodine.
   b. Utilize same principles as the direct method. Stress additional step using iodine titrant and that the end point signal is reversed.
   c. CHOICE of instructional approach:
      1) Eleven minute CCTV cassette showing the back titration using an amperometric titrator.
      2) Demonstrate a back titration using an amperometric titrator and/or the starch-iodide colorimetric procedure. The latter requires addition of starch for end point detection.
5. Classroom instruction on the DPD Method to determine free or total chlorine. Section V of the outline in the training manual has the following information:

a. Chemistry involved is similar to iodometric method, depending on liberation of iodine from potassium iodide.

b. Role of DPD to produce color.

c. Ferrous ammonium sulfate is used as titrant. Note end point signal.

d. Spectrophotometric method

e. OPTIONAL: Demonstrate one or both DPD methods.

6. A summary of methodology recognized for compliance monitoring is in Section VI.

7. In laboratory - 45 minutes - Briefing which includes equipment preparation information and instrument controls, then student performance of two determinations of total chlorine in a sample by back titration using amperometric apparatus. Use training manual outline, "Amperometric Determination of Total Residual Chlorine."

8. Class Discussion - 8 minutes

a. Students calculate and post results.

b. Announce result obtained by Instructor.

c. Discuss problems responsible for wrong results.

d. Note sample collection and analysis cautions.

e. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: 1 total chlorine sample. Run one determination for practice, run second determination to report results.

To minimize the number of titrators required, the laboratory session could be conducted twice with half the class doing another procedure requiring about the same time, e.g., Hardness.

A. For each station (no more than two persons per station):

1. One 1 ml pipet graduated in tenths

2. One 5 ml volumetric pipet (Use a graduated pipet if using the delivery assembly for PAO on a titrator).

3. One 200 ml graduated cylinder

4. One prop pipet bulb
5. One scoop for KI crystals
6. One box tissues
7. One container for waste solutions
8. One amperometric titrator with cell containing electrolyte and conditioned for use
9. One squeeze bottle chlorine-free distilled water
10. One marking pencil
11. One apron for each student
12. One pair safety glasses for each student

B. Shared:
1. X bottles for sample (1 per 2 stations)


A. Minimum amounts per station (no more than two persons per station).
1. Ten ml 0.00564N phenylarsine oxide solution
2. Two g potassium iodide crystals (or one dropper full of KI solution)
3. Eight ml pH 4 acetate buffer (in dropper bottle)
4. Two ml 0.0282N iodine titrant (in dark bottle)
5. Four hundred ml sample with total chlorine residual around 3mg/L
   (A chlorine solution may be diluted. Another source is a hypochlorite bleach or a chlorinated wastewater treatment plant effluent may be used. Check any solution to be used as a sample to be sure chlorine is present in the applicable range of the test).

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of chlorine-free distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Thirty-six slides, X-ZI: Chlorine - See next pages:
Cartoon-type of drawing showing plant operator killing disease organisms with chlorine.

SLIDE 1

- Chlorine gas
- Chlorine gas dissolved in water
- Calcium hypochlorite

SLIDE 2

- Chlorine gas
- Chlorine gas dissolved in water
- pH Decrease

SLIDE 3

PRODUCTS of Cl₂

- H⁺ - hydrogen ion
- Cl⁻ - chloride ion
- HOCI - hypochlorous acid
- H⁺ - hydrogen ion
- OCl⁻ - hypochlorite ion

SLIDE 4

PRODUCTS of Ca(OCl)₂

- Ca²⁺ - calcium ion
- OH⁻ - hydroxide ion
- HOCI - hypochlorous acid
- H⁺ - hydrogen ion
- OCl⁻ - hypochlorite ion

SLIDE 5

SLIDE 6

SLIDE 7

Ammonia

SLIDE 8

Monochloramines

Tetrachloramines

SLIDE 9

Demand from side reactions

SLIDE 10
Disinfection

Free Chlorine residuals

Combined Chlorine

Hypochlorine ion

Hypochlorous acid

Hypochlorite ion

Total residual chlorine = free chlorine residual + combined chlorine residual

Disinfecting power

Concentration

Contact time

Free Chlorine - mg/l

Combined Chlorine - mg/l

Total residual chlorine

Reducing agent

Live of titration assembly
TUO STAGE TITRATION
ph 7 FREE CHLORINE RESIDUAL
ph 4 COMBINED CHLORINE RESIDUAL

SLIDE 19

CURRENT PRODUCED
PROPORTIONAL TO FREE CHLORINE PRESENT

SLIDE 20

LIVE OF ADDING TITRANT, CURRENT REDUCED

SLIDE 21

LIVE OF ADJUSTING POINTER TO RIGHT ON SCALE

SLIDE 22

LIVE OF ADJUSTING MORE TITRANT

SLIDE 23

LIVE OF ADDING TITRANT

SLIDE 24

LIVE OF POINTER AT REST

SLIDE 25

LIVE OF POINTER STAYING AT REST

SLIDE 26

1 ml PHENTLARSEN OXIDE, EQUALS 1 mg/l OF CHLORINE

SLIDE 27

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Phenylarsine oxide reacts with free chlorine only.

Free chlorine reacts with combined chlorine.

Starch-iodide method: chlorine oxidizes iodide to produce free iodine.

Combined chlorine determination: pH 4 and addition of potassium iodide.

Combined chlorine compounds oxidize iodide to free iodine.

Live of adding KI solution.

Live of adding pH4 buffer.

Live of repeating titration process.

Free chlorine - mg/l.

Combined chlorine - mg/l.

Total residual chlorine.

SLIDE 28

SLIDE 29

SLIDE 30

SLIDE 31

SLIDE 32

SLIDE 33

SLIDE 34

SLIDE 35

SLIDE 36 (END OF SLIDES)
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Use of a Spectrophotometer and Calibration Graphs

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 45 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Participants need to use a spectrophotometer and prepare calibration graphs to determine total phosphorus, nitrate and nitrite nitrogen, and fluoride.

V. ENTRY LEVEL BEHAVIOR:

A. Ability to perform calculations involving decimals
B. Knowledge of weight and volume terms like "mg", "μg", "L", "ml", "standard solution"
C. Ability to use arithmetic graph paper

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know how to zero a spectrophotometer at a selected wavelength, make absorbance measurements for standards and samples, use the results to prepare a calibration graph and then use the graph to determine the concentration of the desired constituent in a sample, all according to the outline in the training manual (see VII. A. 1.).

B. Conditions: He/she will be given the training manual and 45 minutes of instruction. Later in the course, he/she will be given the time and the necessary equipment to perform spectrophotometric measurements and to prepare calibration graphs.

C. Accepted Performance: Subsequent satisfactory use of a spectrophotometer and ability to prepare calibration graphs for the total phosphorus and the nitrate and nitrite nitrogen determinations, all according to an Instructor's rating.

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. One outline in training manual: "Use of a Spectrophotometer". Terms and operational sequence are for B & L Spectronic 20 models, but can be easily adapted for any spectrophotometer.


4. Thirty-five slides, 8 minute tape and script, XT-51: Use of the
Spectronic 20 Spectrophotometer (See XI Description of Visual Materials)

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Review the course laboratory procedures which utilize a spectrophotometer: phosphorus, fluoride, nitrate and nitric acid, nitrogen.
2. Review visual materials and outline in course manual.
3. Prepare the lesson using the Sequencing below or your own organization of the material.

B. Sequencing:


   a. General principles of absorbance and colorimetry; X-8, 1.
   b. Definition of wavelength; X-8, 2.
   c. Characteristics of wavelength; X-8, 3.
   d. Relation of wavelength values to inches; X-8, 4.
   e. Lambert-Beer Law; X-8, 5.
   f. Shapes of absorption cells; X-8, 6.
   g. Use of the EPA phosphorus procedure to exemplify the preparation of standards; X-8, 7.
   h. Preparation of the stock phosphorus solution (known composition and concentration); X-8, 8, 9, and 10.
   i. Preparation of the standard phosphorus solution (known composition and concentration); X-8, 11, 12 and 13.
   j. Preparation of a series of phosphorus solutions of known composition and concentration; X-8, 14, 15 and 16.

2. Classroom - Discuss preparation of a calibration graph using X-9, Calibration Graphs.
   a. Information needed to prepare a calibration graph; X-9, 1.
   b. Drawing and labeling the axes of a calibration graph; X-9, 2.
      \[ (A = \text{absorbance}, \ C = \text{concentration}) \]
c. Locating one point using absorbance concentration values. (mg P/liter is an example label for the abscissa). X-9, 3.

d. Locating five points; X-9, 4.

e. Connecting the five points by the line of best fit. X-9, 5.

f. Determining the concentration of the sample using the calibration graph. X-9, 6.

3. Classroom - If the background of the participants warrants it, go through an example of calculating amounts of standard solution to be diluted to achieve calibration standards within a range given in a Method write-up.


5. Classroom - Discuss the use of a spectrophotometer using XT-51. Use of the Spectronic 20 Spectrophotometer. The slide-tape is a presentation of:

a. Warm-up

b. Adjustments prior to use

c. Making measurements

d. Changing photo tube

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

A. Visual Materials

X. IPW REAGENT REQUIREMENTS:

A. None

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Sixteen slides, X-8; Use of a Spectrophotometer - See next pages.

B. Six slides, X-9: Calibration Graphs (Follows X-8)

C. Thirty-five slides (also 3-minute tape), XT-51: Use of the Spectronic 20 Spectrophotometer (Follows X-9)
SERIES X-8: USE OF A SPECTROPHOTOMETER

SLIDE 1

Color Intensity Measurement

SLIDE 2

WAVELENGTH
A VERY SMALL DISTANCE
NANOMETER (nm)
MILLIMICRON (mu)
COLOR INTENSITIES ARE MEASURED AT A SPECIFIC WAVELENGTH

SLIDE 3

SLIDE 4

1 nm = 0.000000001 METER
1 METER = 39.37 INCHES

SLIDE 5

LAMBERT-BEER LAW
A = Ibc
A = ABSORBANCE
b = A CONSTANT
c = CELL LIGHT PATH LENGTH
b = CONCENTRATION (USUALLY mg/l)

SLIDE 6

Top view of two types of cells
5 cm is a common value of b

SLIDE 7

PHOSPHORUS DETERMINATION
EPA METHOD
BLUE COLOR INDICATES PRESENCE OF PHOSPHORUS

SLIDE 8

Distilled water to 1000 ml mark

SLIDE 9

1000 ml mark
1000 ml volumetric flask

0.3197 g of potassium dihydrogen phosphate

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SERIES 18 USE OF A SPECTROPHOTOMETER

SLIDE 10

SLIDE 11

SLIDE 12

SLIDE 13

SLIDE 14

SLIDE 15

SLIDE 16
Absorbance and concentration values for each standard. Absorbance value of sample.
Spectrophotometers

% transmittance

Absorbance (optical density)

% transmittance

concentration

Photo of Spectronic 20 with Numbers on the Controls

Photo Featuring #1 - On/Off Knob

Photo Featuring #2 Cell Compartment (closed)
I. SUBJECT MATTER: Phosphorus

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 195 minutes (45+45+95+10)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:
   A. Fundamental knowledge of inorganic chemistry
   B. Ability to correctly use volumetric glassware
   C. Ability to perform calculations involving decimals
   D. Completion of the lessons (or, equivalent):
      1. pH
      2. Use of a Spectrophotometer and Calibration Graphs
      3. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will know pertinent facts about phosphorus forms (see VIII B.) and will determine total phosphorus in a municipal wastewater treatment plant effluent using the procedures in the training manual (see VII A. 1. b & c). He/she will draw up a calibration curve using absorbances posted by class members and will use his/her absorbance for the sample to obtain a result. He/she will also participate in a class discussion of the topics and laboratory procedures.
   B. Conditions: He/she will be given the training manual, 45 minutes of instruction, 140 minutes laboratory time, all necessary equipment and reagents, a sample, and ten minutes for class discussion.
   C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedure to the satisfaction of an instructor, and obtain a result for the sample within ± 10% of the class average (unless the reason for lack of agreement is acceptable to the instructor).

VII. INSTRUCTIONAL RESOURCES:
   A. Available Media:
      1. Three outlines in the training manual:
a. "Phosphorus in the Aqueous Environment"
b. "Laboratory Procedure for Total Phosphorus" according to 1979 EPA Methods for Chemical Analysis, p. 365.2-1
c. "Use of a Spectrophotometer"

2. Fifty-three slides, 14 minute tape and script, XT-44: The Determination of Phosphorus (See XI. Description of Visual Materials).

B. Suggested Media:
1. None.

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:
1. Check equipment and supplies. See IX below.
   Caution:
   a. All glassware, including that used to prepare reagents, and filtering apparatus must be cleaned using special procedures. See the training outline, "Laboratory Procedure for Total Phosphorus" section III for details.
   b. Filter discs must be phosphorus-free. See section IV in the training outline for details.

2. Prepare all necessary reagents and sample as in X below.

3. Review the laboratory procedure by performing the student assignment using the outline in the training manual.

4. Review the topic outline in the training manual and the Visual materials. See XI below.

5. Prepare the lesson using the Sequencing below or your own organization of the material.

6. Check pH meters and spectrophotometers (880 or 650 nm) for accurate operation. The spectrophotometers may need a special filter and phototube for operation at 880 or 650 nm.

7. Distribute supplies and reagents to laboratory stations.

B. Sequencing:

2. Present XT-44:
   a. Introduction - slides 1-5
b. Reasons why phosphorus determinations are done - slides 6-8

c. Phosphorus terminology, ortho- and polyphosphates - slides 9-15

d. Overview and chemistry of the analytical method - slides 16-20

e. Sample collection, preservation, holding time - slides 21-24

f. Total vs. dissolved phosphorus - slide 25

g. Digestion procedures for various forms - slides 26-33

h. pH adjustment of digested standards and sample - slides 34-35

i. Preparation of combined reagent and color development - slides 36-41

j. Absorbance readings and graph - slides 42-43

k. Care of glassware - slides 44-45

l. Applicability of procedure - slide 46.

m. Interferences - slides 47-49

n. Summary - credits - slides 50-53

3. Answer any questions about XT-44 topics.

4. Briefing for laboratory - class will prepare standards and sample, then begin digestion. Using the laboratory procedure in the training manual, they should do section VI. Procedures A, B, and C. Each station prepares the zero standard, one assigned standard and the sample. Stop after placing flasks on hot plates (VI. C 6). A staff member will monitor the boiling, turn off the hot plates and cover the flask mouths.

5. Laboratory-Student performance of assignment (45 minutes).

6. There can be a break of 45 minutes or up to several hours at this point. The "Use of a Spectrophotometer" could be presented during this time (See IPW on the topic).

7. Briefing for laboratory - Do section VI, D, E, F and G; i.e., pH adjustment, filtration, volume adjustment and colorimetry. Refer students to "Use of a Spectrophotometer" outline for stepwise procedure to use the instrument.

8. Laboratory-Student performance of assignment (95 minutes).

9. Classroom (10 minutes)

   a. Participants post absorbance for standards.

   b. Exclude outlier absorbance values.

   c. Sections VII and VIII of Phosphorus procedure - Each makes a "calibration curve" using posted absorbances and obtains a result using own absorbance for sample. (Stress that this method is for convenience and cannot be used for real work).
d. Discuss results and any items from the laboratory session.

e. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

**Assignment:** 2 standards (zero plus one other) and 1 sample

Filter the sample

We have the entire class do the procedure on assigned standards and the sample from the beginning through starting the digestion (manual outline VI-A through C). Reagents and equipment required this far have an asterisk. Then, to minimize the number of pH meters required, we present the classroom sessions on "Use of a Spectrophotometer" and "Fluoride". The remaining laboratory work on phosphorus is done as "split" sessions, with half the class doing fluoride while half finish phosphorus.

A. For each station (no more than 2 persons per station):

1. One 100 ml beaker (for buffers)

2. Twelve boiling beads, glass

3. One filtration apparatus, small enough to filter between 10 and 50 ml of digested solution.

4. One piece phosphorus-free filter paper (may require pre-treatment), to fit the filtration apparatus, 0.45 μm pore size (Gelman GA6 or equivalent)

5. Three flasks, 50 ml volumetric with stoppers

6. Three flasks, 125 ml Erlenmeyer

7. One pair forceps

8. One funnel, to fit neck of 50 ml volumetric flask

9. One pH meter with electrode(s) in a beaker with enough 6.8 buffer or distilled water to cover the tip(s). A single, combination electrode is the most convenient. Also, provide a thermometer.

10. One 10 ml graduated pipet

11. One 50 ml volumetric pipet (for the sample). If sample has suspended solids that may plug the pipet, use a 50 ml graduated cylinder.

12. One pipet bulb

13. One scoop, 0.4g capacity

14. One squeeze bottle of phosphorus-free distilled water

15. One marking pencil
*16. One apron for each student
*17. One pair safety glasses for each student

B. Shared:

*1. One set of volumetric pipets (1, 3, 5, 10, 20, 50 ml) per 6 stations if 1 standard is assigned to each.

*2. Enough hot plates in a hood to accommodate 3-125 ml Erlenmeyer flasks per station.

3. One spectrophotometer (for use at 680 nm or 650 nm and having a 1 cm cell) per 3 stations.

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 1979 EPA "Methods for Chemical Analysis", p. 365. 2-1, EXCEPT for A.5.*

A. Minimum Amounts per Station (no more than 2 persons per station):

1. One hundred ml pH 6.5 buffer
2. One hundred ml pH 4.5 buffer
3. One point two g ammonium persulfate
4. Twenty-four ml combined reagent
5. Seventy ml standard phosphorus solution (1.0 ml = 1.0 mg P) Most will use less than 70 ml. *(20 ml stock per liter for 1 cm cells)
6. Four ml 1N sulfuric acid in dropper bottle
7. Five ml 0.1N sulfuric acid in dropper bottle
8. Ten ml 10N sodium hydroxide in dropper bottle
9. Five ml 0.1N sodium hydroxide in dropper bottle
10. Fifty ml municipal wastewater treatment plant effluent for sample. If not available, use 10 ml of the stock solution per liter to make a sample containing 0.5 mg/liter phosphorus.

B. Prepare excess of each solution to allow for rinsing out glassware, waste, and re-runs.

C. A source of phosphorus-free distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Fifty-three slides-(with 14 minute tape), XT-44: The Determination of Phosphorus - See next pages.
Phosphates $\xrightarrow{\text{heat}}$ Polyphosphates $\xrightarrow{\text{water}}$

Cleaning Agents
- Detergents

Polyphosphates
- Acid
- Heat
- Water
- Orthophosphate ion

Orthophosphate Derivatives
- Trisodium Phosphate
- Disodium Phosphate
- Monosodium Phosphate
- Diammonium Phosphate

Polyphosphate Derivatives
- Sodium hexametaphosphate
- Sodium tripolyphosphate
- Tetrasodium pyrophosphate

Orthophosphates come out.

Cartoon of mad scientist dumping organic phosphorus compounds into grinder. Orthophosphates come out.

Sample collection and preservation
1. Sample collection and preservation
2. Possible filtration
3. Possible digestion
4. pH adjustment
5. Color development
6. Spectrophotometric determination

Ammonium molybdate plus potassium antimonyl tartrate plus phosphorus plus acid
- Antimony phosphomolybdate complex

Antimony phosphomolybdate complex plus ascorbic acid
- Blue complex
<table>
<thead>
<tr>
<th>SLIDE</th>
<th>Image Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>19</td>
<td>Cartoon of 'mad' scientist staring at test tube of blue liquid.</td>
</tr>
<tr>
<td>20</td>
<td>0.01 - 0.5 mg P/l</td>
</tr>
<tr>
<td>21</td>
<td>Drawing of two hands holding sample bottles under water. One bottle is collecting bottom deposits. Yes</td>
</tr>
<tr>
<td>22</td>
<td>Photo of Plastic and Glass Containers</td>
</tr>
<tr>
<td>23</td>
<td>2 ml H₂SO₄ 40 mg HgCl₂/l Storage at 4°C</td>
</tr>
<tr>
<td>24</td>
<td>Drawing of man in laboratory looking at clock</td>
</tr>
<tr>
<td>25</td>
<td>Photo of Membrane Filter Apparatus</td>
</tr>
<tr>
<td>26</td>
<td>ammonium persulfate &amp; sulfuric acid digestion yes sulfuric acid digestion no digestion</td>
</tr>
<tr>
<td>27</td>
<td>59.0 ml</td>
</tr>
</tbody>
</table>
Oxidizable organics and polyphosphates

ammonium persulfate + sulfuric acid digestion
Orthophosphate ion

Polyphosphates and some organics
sulfuric acid digestion
Orthophosphate ion

Orthophosphate
No digestion
Orthophosphate

Drawing of man scratching head. In cloud above head are "polyphosphates, organic phosphates, orthophosphates"

Photo of Hot Plate & Clock

Photo of Autoclave

Adjustment of pH

pH meter
sodium hydroxide

50.0 ml

Photo of Adding Combined Reagent
Sulfuric acid
Potassium antimony tartrate
Alumino-molybdate
Ascorbic acid

50 ml H₂SO₄
5 ml K(SbO)C₆H₄O₆ · 1/2H₂O
Mix

15 ml (NH₄)₃PO₄ · 4H₂O
Mix

10 ml ascorbic acid
Mix

Photo of flask & clock

Hot 1:1 HCl
Distilled water
Color reagent
Distilled water

Drawing of closed safe with glassware
Domestic wastes
Industrial wastes
Saline waters
Surface waters
Drinking waters

Arsenate
Copper
Iron
Silicate

Phosphorus Adsorption
Iron
Aluminum
Manganese

Salt concentration 5-30%
Analytical error less than 1%

Reasons for the determination
Phosphorus terminology

Credit Slide

Clean Water

Photo of EPA Chemical Analysis Manual
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Determinate Error-Accuracy

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 40 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: An analyst must know how to control or quantify determinate errors in laboratory results, and how to check for acceptable analytical recovery of a constituent to substantiate the validity of his/her data.

V. ENTRY LEVEL BEHAVIOR:
A. Understanding of how to use formulas involving simple calculations
B. Though not essential, it is orderly to have completed the lessons (or equivalent):
   1. Sample Handling
   2. Compliance Methodology

VI. INSTRUCTIONAL OBJECTIVE:
A. Terminal Behavior: The participant will know the difference between accuracy and precision, the causes, synonyms, effects and means to detect sources of determinate error, where to find formulas and examples, of each for % recovery, % bias and % relative error, some laboratory techniques regarding accuracy, how to establish one's accuracy in performing an analysis and how to continue to monitor accuracy on a daily basis.
B. Conditions: He/she will be given the training manual and 40 minutes instruction.
C. Accepted Performance: He/she must correctly identify at least 70% of the items pertaining to the objective in the post-course evaluation.

VII. INSTRUCTIONAL RESOURCES:
A. Available Media:
   1. Two outlines in the training manual:
      a. "Accuracy-Precision-Error"
      b. "Elements of a Quality Assurance Program"
   2. Fourteen slides, X-25: Accuracy (See XI Description of Visual Materials).
VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:
1. Review visual materials and available handouts.
2. Prepare the lesson using the Sequencing below or your own organization of the material.
3. Duplicate copies of any handout materials.

B. Sequencing:
Slides are series X-25: Accuracy (optional: slides from X-26: Quality Assurance)

1. Introduction

   a. Before considering accuracy of results, one must have a valid sample and a skilled analyst using recognized analytical methodology.

   b. Overview: consideration of accuracy, i.e. ability to recover known quantities in chemical analyses, causes of error, how to prevent "preventable" errors and how to quantitate the effect of errors we cannot control. Statistics presented will be those most commonly used in recognized method references: percent recovery and percent bias (percent relative error).

   c. Outline, "Accuracy-Precision-Error" (A.P.E.) has details and illustrations, unless noted otherwise.

   d. Slides are from series X-25 unless otherwise noted.

2. Accuracy and Precision

   a. Analytical results are imperfect. Slide 1

   b. Define/exemplify accuracy. Results are close to the true value-standard. Accuracy can also be checked by recovery of a constituent-spike. Slide 2. Stress that both accuracy and precision are requirements for reliable data. Slide 3.

3. Error

   a. Overview: Classification + Causes of error. Determinate error has causes which can be corrected and/or quantitated. Slide 4

   b. Synonyms for determinate error. Use A.P.E.
c. Effects of determinate error: Use A.P.E., figures 2 and 3. Slide 5

d. Detection of determinate error with unknowns - Slide 6

e. Correction of determinate error with laboratory methods: Slide 7

(1) Handout 2 pp. on "Method of Standard Additions" and discuss at point 3 on Slide 7.

4. Percent Recovery Statistic for Accuracy

a. Formula for using a standard to check percent recovery. The "observed" can be the average of several results. Slide 8

b. Formula for using a spiked sample to check percent recovery. Slide 9

c. Explanation of term, "background" in Slide 9 formula. Slide 10

d. Go through handout, "Example of Calculating Percent Recovery for a Spike" to demonstrate the correct use of the information discussed on Slides 9 and 10.

e. Checking own accuracy using a spiked sample. Slide 11. Note that one can use a standard instead. The latter calculations are easier.

f. OPTIONAL: X-26, Slide 4 shows a percent recovery control chart for monitoring accuracy on a daily basis.

5. Percent Bias (Percent Relative Error) Statistic for accuracy

a. Two major reference manuals use different names for this statistic. Slide 12

b. Formula for using a standard to check percent bias. Slide 13

c. OPTIONAL: X-26, Slide 5 shows a percent bias control chart for monitoring accuracy on a daily basis.


7. Daily Application

a. Suggested (EPA Handbook for AQC). - 10% of tests be spikes to check accuracy.

8. Summary

a. Again stress valid sample and skilled analyst using recognized analytical methodology are prior to checking accuracy.

b. Summarize presentation.
IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:
   A. Visual materials

X. IPW REAGENT REQUIREMENTS:
   A. None

XI. DESCRIPTION OF VISUAL MATERIALS
   A. Fourteen slides, X-25: Accuracy - See next pages.
ANALYTICAL RESULT

An Imperfect Estimate of Some Desired Real Value

SLIDE 1

DETERMINATE ERROR
METHOD
OPERATOR
INSTRUMENT

INDETERMINATE ERROR
RANDOM
UNCONTROLLABLE

SLIDE 4

ACURACY
EFFECTS OF DETERMINATE ERROR

ADITIVE
PROPORTIONAL

SLIDE 5

ACURACY
CORRECTION OF DETERMINATE ERROR

1. BLANKS
2. CORRECTION FACTORS
3. STANDARD ADDITION
4. STANDARD COMPENSATION
5. IMPROVED TECHNIQUE

SLIDE 7

% RECOVERY FOR STANDARDS - OBSERVED X 100
KNOWN

SLIDE 9

% RECOVERY
FOR SPIKED SAMPLES
OBSERVED-BACKGROUND SPIKE X 100

SLIDE 9
"BACKGROUND"
The analytical result for the unspiked sample with regard to its volume as a constituent of the spike which yields the "observed" analytical result.

**SLIDE 10**

**ACCURACY**
- Run sample unspiked
- Run spiked sample (double lowest, I to 75%)
- 7 replicates of each
- 2 hour lab time (min.)
- Calculate % recovery

**SLIDE 11**

**DIFFERENT NAMES, SAME STATISTIC:**
- EPA - % bias
- STD methods - % relative error

**SLIDE 12**

**ACCURACY STATISTICS**

\[
\text{% bias (or)} = \frac{\text{mean} - \text{true value}}{\text{true value}} \times 100
\]

% bias or % relative error equals % recovery - 100 (gives sign)

**SLIDE 13**

**SLIDE 14 (LAST SLIDE)**

**SLIDE**
8.5 Method of Standard Additions: In this method, equal volumes of sample are added to a deionized distilled water blank and to three standards containing different known amounts of the test element. The final volumes of all the solutions must be the same. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations from the weight of analyte added to each of the original volumes of sample plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 1.

![Graph showing standard addition plot]

**FIGURE 1: STANDARD ADDITION PLOT**

The method of standard additions can be very useful, however, for the results to be valid the following limitations must be taken into consideration:

a) the absorbance plot of sample and standards must be linear over the concentration range of concern. For best results the slope of the plot should be nearly the same as the slope of the aqueous standard curve. If the slope is significantly different (more than 20%) caution should be exercised.

*from: 1979 EPA "Methods for Chemical Analysis of Water and Wastes"
b) the effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes and the standard addition should respond in a similar manner as the analyte.

c) the determination must be free of spectral interference and corrected for non-specific background interference.
EXAMPLE OF CALCULATING PERCENT RECOVERY FOR A SPIKE

A sample was collected and a 25 ml aliquot was analyzed for nitrate nitrogen. The result of the analysis was 500 µg/liter. A 25 ml spiked aliquot of the same sample was prepared using 1 ml of a spike solution which contained 10 mg/liter nitrate nitrogen. The result of analyzing the spiked sample was 898 µg/liter. Calculate the percent recovery.

\[ p = \frac{\text{observed} - \text{background}}{\text{spike amount}} \times 100 \]

observed is \( \left( \frac{898 \text{ µg}}{0.025 \text{ liter}} \right) = \frac{22.45 \text{ µg}}{} \)

background is \( \left( \frac{500 \text{ µg}}{0.024 \text{ liter}} \right) = \frac{12.00 \text{ µg}}{} \)

spike amount is \( \left( \frac{10,000 \text{ µg}}{0.001 \text{ liter}} \right) = \frac{10.60 \text{ µg}}{} \)

\[ p = \frac{22.45 \text{ µg} - 12.00 \text{ µg}}{10.60 \text{ µg}} \times 100 \]

\[ p = 104.5\% \]
GUIDELINES FOR  
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Fluoride

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 160 minutes (60+95+5)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality program requirements.

V. ENTRY LEVEL BEHAVIOR:
   A. Fundamental knowledge of inorganic chemistry
   B. Ability to use volumetric glassware
   C. Completion of the lessons (or equivalent):
      1. Use of Spectrophotometer and Calibration Graphs
      2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will know pertinent facts about fluoride, the manual distillation procedure and four analytical procedures for determining fluoride (see VIII B.). He/she will test four samples by the SPADNS (by calculation) and the electrode methods (8 tests) using the procedures in the training manual (see VII. A. 1. b and c) and will report the results. He/she will also participate in a class discussion of the laboratory procedures and the topic in general.

   B. Conditions: He/she will be given the training manual, 60 minutes of instruction, 95 minutes laboratory time, all necessary equipment and reagents, four samples and five minutes for final questions.

   C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an instructor and obtain results within ±0.1 mg/liter of the results obtained by the Instructor (unless the reason for the lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:
   A. Available Media:
      1. Three outlines in the training manual:
         a. "Control of Interfering Ions in Fluoride Determinations"

c. "Fluoride Analytical Procedures - Electrode" according to Orion Research, Inc. instrument directions. For other instruments, use the manufacturer's operating instructions. Also see 14th ed. Standard Methods, p. 391.

2. Forty-six slides and 16 minute tape, XT-80: Fluoride Analytical Procedures (see XI., Description of Visual Materials).

B. Suggested Media:
1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.

2. Prepare all necessary reagents and samples as in X below.

3. Review the topic outline in the manual, then the laboratory procedures by performing the student assignment using the outlines in the training manual. Notice that two of the samples contain interferences for the SPADNS method to exemplify interfering ions.

4. If necessary, review other aspects of the topic by consulting Standard Methods, 14th ed, Section 414, Fluoride. Also review the visual materials (see XI below).

5. Prepare the lesson using the Sequencing below or your own organization of the material.

6. Check specific ion meters (and/or pH meters), the electrodes and the spectrophotometers (570 nm) for accurate operation.

7. Distribute supplies and reagents to laboratory stations.

B. Sequencing:

1. Classroom - Introduction
   a. Existence of fluoride in waters
   b. How fluoride ions are introduced

2. Classroom - Introduce and present XT-80: Fluoride Analytical Procedures. Tell the participants about the information to expect during the presentation:
   a. (Title and Credits, slides 1-4)
   b. Fluoride and dental effects (slides 5-6)
c. Titles and overview of four methods approved for analysis of drinking water samples (slides 7-14)

d. Stepwise procedure and calculations for the SPADNS method (slides 15-21)

e. Stepwise procedure using the electrode method (slides 22-30)

f. Interferences in fluoride analyses (slides 31-32)

g. Steps and apparatus for the distillation procedure to remove interferences before analysis (slides 33-39)

h. Summary of information presented (slides 40-45)

i. (Logo, slide 46)

3. Classroom - Discuss approved methods. Differentiate between water supply regulations and NPDES regulations. Section IV in the training manual outline, "Control of Interfering Ions, etc," is a summary of Compliance Methodology.

a. Electrode (Distillation may be required for NPDES).

b. Distillation and SPADNS

c. Automated complexone (Method includes distillation).

d. Distillation and Eriochrome cyanine R (only approved for drinking water samples)

4. Classroom - Summary

5. Laboratory - 95 minutes

a. Briefing and assign four samples to be tested by the SPADNS and also by the electrode method (8 tests in all). If every other station begins with the SPADNS test and every other begins with the electrode method, the required spectrophotometers and electrode-meter apparatus can be shared, one per two stations.

b. Student performance of assignment.

c. Students report results.

6. Classroom Final Discussion - 5 minutes

a. Any items from laboratory session

b. Resolve any questions

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

**Assignment:** Four samples to analyze by two methods - SPADNS and an electrode with selective ion meter. (If a pH meter is to be used, a blank and three standards for calibration should be assigned). To minimize the number of spectrophotometers and electrodes
with meters required, the laboratory session could be conducted twice with half the class doing another procedure requiring about the same time, e.g., Phosphorus pH adjustment and colorimetry.

A. For each station (no more than two persons per station):
1. Six 50 ml plastic beakers (eight for pH meter)
2. Six 125 ml Erlenmeyer flasks
3. Three 10 ml volumetric pipets
4. One 50 ml volumetric pipet
5. One pipet bulb
6. One squeeze bottle fluoride-free distilled water
7. One marking pencil
8. One apron for each student
9. One pair safety glasses for each student
10. Optional: Magnetic stirring apparatus

B. Shared:
1. One spectrophotometer for use at 570 nm and having a 1 cm cell per two stations (see VIII. B. 5. a.).
2. One fluoride specific ion electrode and meter per two stations (see VIII. B. 5. a.).
3. One stop watch or timing device.

X. pH REAGENT REQUIREMENTS: For details of reagent preparations, see 14th edition "Standard Methods", pages 392 and page 393.

A. Minimum amounts per station (no more than two persons per station):
1. Fifty ml distilled water
2. Three hundred ml TISAB (400 ml for pH meter)
3. One hundred ml fluoride standard, 1 mg/liter F⁻ (selective ion meter and SPADNS)
4. One hundred ml fluoride standard, 2 mg/liter F⁻ (only if a pH meter is used)
5. Sixty ml fluoride standard, 10 mg/liter F⁻ (selective ion meter)
6. Seventy-five ml SPADNS reagent
7. One hundred ml sample #1 containing 0.55 mg/liter F⁻ (5.5 ml fluoride stock solution per liter). Both of the methods (SPADNS and electrode) give correct results for this sample.

8. One hundred ml sample #2 containing 1.05 mg/liter F⁻ (10.5 ml fluoride stock solution per liter). Both of the methods give correct results for this sample.

9. One hundred ml sample #3 containing 0.75 mg/liter F⁻ plus 300 mg/L sulfate (7.5 ml fluoride stock solution plus 0.4437g anhydrous sodium sulfate per liter). Only the electrode gives correct results for this sample.

10. One hundred ml sample #4 containing 0.95 mg/liter F⁻ plus 3 mg/L aluminum (9.5 ml fluoride stock solution plus 52.752 mg aluminum potassium sulfate with .12H₂O). Only the electrode gives correct results for this sample.

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of fluoride-free distilled water should be available during laboratory sessions.

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Forty-six slides (with 17 minute tape), XT-80: Fluoride Analytical Procedures. See next pages.
EPA Presents

SLIDE 1

Technical Direction By:
John Pfaff
Ervin Bellack
Thomas Hushower

SLIDE 4

List of EPA Approved Methods and the Related References

SLIDE 7

SLIDE 2

Fluoride Analytical Procedures

SLIDE 5

Photo Child Drinking Water From Fountain

SLIDE 8

Automated Complexone Method Notable Features

SLIDE 9

Zirconium-Eriochrome Cyanine R Method Notable Features

SLIDE 3

PREPARES BY

EPA
ONP
MOTD
NTOTC

SLIDE 6

Photo Operator in Lab Conducting Analysis
Preparing Standards & Sample
SPADNS Procedure
50 ml 50 ml 50 ml
Dist. 1 mg/l Sample
Water F sol
0-Std. 1 mg/l Sample

The SPADNS Zirconium Fluoride Reaction

Analyst Using Ion Meter

Analyst Adding Reagent

Close-up of Spec 2D Showing Wavelength Adjustment
PHOTO
ANALYST USING SPECTRONIC 20

SLIDE 19

ABSORPTION EQUATION

\[ \frac{A_0 - A_1}{A_0} = \text{mg/l} \]

TERMS DEFINED

SLIDE 20

Graph of Absorbance vs. Conc.

SLIDE 21

PHOTO

ANALYST USING AN EXPANDED SCALE pH METER

SLIDE 23

PHOTO

ANALYST USING A pH METER

SLIDE 24

PHOTO

ANALYST USING AN ION METER

SLIDE 25

Preparing Standards & Samples
Electrode Procedure
10 ml 10 ml 10 ml
of of of
1.0 mg/l 10.0 mg/l sample

3-100 ml plastic beakers

SLIDE 26

PHOTO

ANALYST ADDING TISAB

SLIDE 27

PHOTO

ANALYST CALIBRATING METER
PHOTO
ANALYST ADJUSTING TEMPERATURE
COMPENSATOR KNOB

PHOTO
ANALYST READING SAMPLE

PHOTO
SET-UP WITH MAGNETIC STIRRER

LIST: INTERFERING SUBSTANCES IN
FLUORIDE ANALYSIS

SPAOUS METHOD INTERFERENCES
LIST: INTERFERING SUBSTANCES

TITLE
REMOVAL OF INTERFERENCES BY
DISTILLATION

DISTILLATION REACTION EQUATION

PHOTO
ANALYST WITH
DISTILLATION SET-UP

PHOTO
DISTILLATION IN PROGRESS
PHOTO
ANALYST ADDING SAMPLE TO FLASK

PHOTO
CLOSE-UP OF THE DISTILLATE RECEIVER

MOST PREVALENT INTERFERING SUBSTANCES:
- ALUMINUM
- HEXAMETAPHOSPHATE

PHOTO
SAME AS SLIDE #5:
CHILD DRINKING WATER FROM A FOUNTAIN

PHOTO
SAME AS SLIDE #7:
LIST OF EPA-APPROVED METHODS AND THE RELATED REFERENCES

PHOTO
SAME AS SLIDE #12:
ANALYST USING A SPEC 20

PHOTO
DISTILLATION IN PROGRESS
SAME AS SLIDE #36

PHOTO
ANALYST USING ION METER
SAME AS SLIDE #24

PHOTO
OPERATOR IN LAB DOING ANALYSIS
SAFE DRINKING WATER EMBLEM

SLIDE 46

SERIES XT-80 FLUORIDE ANALYTICAL PROCEDURES
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Nitrate and Nitrite Nitrogen

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 310 minutes (30+10+90+30+105+45)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:
   A. Fundamental knowledge of inorganic chemistry
   B. Ability to correctly use volumetric glassware
   C. Ability to perform calculations involving decimals
   D. Completion of the lessons (or equivalent):
      1. pH
      2. Use of a Spectrophotometer and Calibration Graphs
      3. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will know pertinent facts about nitrate and nitrite (see VIII B). Using the procedures in the course manual (see VII A 1 b and c), he/she will prepare a cadmium reduction column, process a blank and assigned standard or sample through it and obtain absorbance readings. He/she will also process a non-reduced blank and assigned standard or sample and obtain absorbance readings. Using his/her own absorbance values, the participant will calculate the efficiency of his/her column. He/she will use results pooled from the class and construct calibration curves (2) for reduced and non-reduced standards, then use them to obtain results for the samples. He/she will calculate NO$_3$-N from these results and will also learn how to express results as NO$_3$ or as NO$_2$. He/she will give the completed graphs to the Instructor. He/she will also participate in a class discussion of the topics and laboratory procedures.
   B. Conditions: He/she will be given the training manual, 70 minutes of instruction, 195 minutes laboratory time, all necessary equipment and reagents, a sample, and 45 minutes for drawing up graphs, calculations, and class discussion.
   C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an Instructor, and obtain results.

...
for the sample within ± 15% of the theoretical answers provided by the Instructor (unless the reason for a lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the training manual:
   a. "Ammonia, Nitrites and Nitrates"
   c. "Use of a Spectrophotometer"

2. Twenty slides, X-23: Nitrate/Nitrite Nitrogen (See XI Description of Visual Materials)

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.
2. Prepare all necessary reagents and samples as in X below.
3. Review the laboratory procedure by performing the student assignment using the outline in the training manual. It is suggested that the Instructor prepare a few columns for back-up if participants let theirs go dry, etc., when it is too late to start over.
4. Review the topic outline in the training manual and the visual materials. See XI below.
5. Prepare the lesson using the Sequencing below or your own organization of the material.
6. Check pH meters and spectrophotometers (540nm) for accurate operation.
7. Distribute supplies and reagents to laboratory stations.

B. Sequencing:

Slides are series X-23: Nitrate/Nitrite Nitrogen

1. Classroom - Analysis of Nitrate and Nitrite Nitrogen (30 minutes)
   Use outline on "Ammonia, Nitrites and Nitrates"
   a. Introduction
      1) Nitrogen Cycle, note conversions, sources of nitrate and nitrite - Slide 1.
2) Types of nitrogen analyses, desired forms - Slide 2.

b. Nitrite

1) Sources (from nitrogen cycle), bacterial oxidation of ammonia or reduction of nitrate

2) Analytical Method - Slide 3.

3) Scope, Range, Preservation - Slide 4.

4) Procedure Steps - Slide 5.

5) Interferences - Slide 6.

6) Compliance Methodology in training manual. Diazotization is the only method cited as of 8/80.

c. Nitrate

1) Source (from nitrogen cycle), bacterial oxidation of nitrite, two mechanisms from atmospheric nitrogen

2) Analytical Methods - Slide 7.

3) Sample Preservation in training manual, Section II

4) Brucine Sulfate Method, procedure and interferences - Slide 8 and 9.


6) Note: Class will do the third method, cadmium reduction, in the laboratory. It is presented next as a main topic. Give an overview of it now since it utilizes the same approach as hydrazine reduction - Slide 11

7) Compliance Methodology in training manual, Section V. D., as of 8/80.

2. Classroom - Cadmium Reduction Columns (10 minutes)
Use outline on "Determination of Nitrate/Nitrite Nitrogen".

a. Students are to prepare the cadmium and pack a column - Slide 12.


c. Briefing on laboratory procedure in training manual, Sections II, III, IV:

1) Note Section II on apparatus has details on making a column.

2) Section III has reagent preparations. The reagents are made up.

3) Note Section IV, division titles for overview. Note on copper sulfate treatment that one addition is usually enough to get the brown copper precipitate. Make any other comments on the laboratory work.
4) Remarks on toxicity of cadmium

3. Laboratory - Student performance of assignment, all of Section IV (90 minutes).

4. Note: There can be a break of unlimited time at this point.

5. Classroom (5 minutes)
   a. Follow up on preparation-of-column laboratory session, discussing any occurrences in the laboratory of general instructional interest.

6. Classroom - Determination of NO$_2$ + NO$_3$-N and of NO$_2$-N using the column. (25 minutes)
   a. Role of ammonium chloride to provide the H$^+$ - Slide 15.
   b. Procedure steps for NO$_2$ + NO$_3$-N, - Slide 16.
   c. Procedure steps for NO$_2$-N are the same as for NO$_2$ + NO$_3$-N, except for the reduction step - Slide 17.
   d. Briefing on procedures in manual, Sections VI and VII. Have participants circle the roman-numeral and capital letter division titles to impress on them the divisions of the procedures.

1) Assign blank, and either a nitrate standard or the sample for each station.

2) Assign blank, and either a nitrite standard or the sample for each station. For standards, assign the same concentration as assigned for nitrate.

3) Refer students to "Use of a Spectrophotometer" outline for stepwise procedures to use this instrument for VI.D. and VII.D.

4) They cannot do calibration curves (VI.E. and VII.E) until results are posted.

7. Laboratory - Student performance of assignment (105 minutes)

8. Class Calculations, Graphing, Discussion (45 minutes)
   a. Participants post absorbance value for nitrate and for nitrite standards.
   b. Discuss and have participants calculate their column efficiency according to formula in manual procedure, Section VIII - Slide 18. (Those who analyzed the sample should use the results obtained by someone who did a pair of standards).
c. Discuss posted results. Discard questionable values.

d. Copies of arithmetic graph paper are in the training manual.

e. Assignment:

1) Participants construct a graph for the reduced nitrate standards and a graph for nitrite standards - Slide 19 and 20. (Some may need assistance).

2) They use their absorbance values for their reduced sample on one graph and for their non-reduced sample on the other to obtain concentrations for each.

3) They calculate NO₃⁻N in the sample by subtraction.

f. Participants report results. Announce theoretical "true" results (if known).

g. Use manual, Section IX in procedure to show how to calculate NO₃ or NO₂ from the data. Have participants do this, using theoretical results.

h. Summary Remarks on the Procedure

1) column rate, checking efficiency, re-activation, advantage of duplicates

2) cautions on cadmium disposal

3) preservation and holding times for samples (Ammonia, etc.

i. Collect graphs. These can be returned later after Instructor checks them.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Prepare and activate a reduction column.

1 blank and either one nitrate standard or the sample
1 blank and either one nitrite standard of the same concentration as assigned for nitrate, or the sample

A. For each station (no more than two persons per station):

1. Two beakers, 150 ml
2. Two beakers, 250 ml
3. One beaker, 400 ml
4. One cut-off 100 ml pipet (for reduction) mounted on a stand
5. One cylinder, graduated, 50 ml
6. One cylinder, graduated, 100 ml
7. Four flasks, 250 ml Erlenmeyer
8. Three flasks, 100 ml volumetric with stoppers
9. One very large funnel containing fluted filter paper (for Cd rinsings) set in an old, 2-3 liter bottle
10. Small wad glass wool for column
11. One 3 inch length rubber hose (4 cm ID)
12. One paper clip
13. One 25 ml volumetric pipet
14. One to three 50 ml volumetric pipets
15. One prop pipet bulb
16. One screw-type clamp to regulate column flow
17. One snap-type clamp to start and stop flow
18. One 60 mesh sieve
19. One spatula or stirring rod to transfer moist cadmium
20. One squeeze bottle of nitrate-nitrite-free distilled water
21. One marking pencil
22. One apron for each student
23. One pair of safety glasses for each student

B. Shared:
1. One pH meter per 2 stations. Each should have the electrode(s) in a beaker with enough 6.0 buffer or distilled water to cover the tip(s). Also, one thermometer for each meter.
2. Two sets of volumetric pipets (1, 2, 5, 10 ml) per 4 stations
3. Two 1 or 2 ml pipets graduated at 0.5 ml per 4 stations
4. One 10 ml graduated pipet for each "common" beaker of color reagent
5. One spectrophotometer (for use at 540 nm and having a 1 cm cell) per 3 stations


A. Minimum amounts per station (no more than two persons per station):
1. 610 ml concentrated ammonium chloride - EDTA solution. Use 210 ml of this to prepare 350 ml dilute ammonium chloride - EDTA solution to prepare the column. The remaining 400 ml is used for the test procedure.
2. Eight ml color reagent
3. ~ 1 ml concentrated hydrochloric acid in dropper bottle
4. ~ 1 ml concentrated ammonium hydroxide in a dropper bottle
5. Sixty ml 6 N hydrochloric acid
6. Two hundred ml 2% copper sulfate solution
7. ~ 20 g cadmium, 40-60 mesh. Pre-weigh into container for stations.
8. ~ 100 ml pH 4.5 buffer
9. ~ 100 ml pH 6.5 buffer
10. Twenty-five ml 1.00 mg NO₃⁻ N/liter (to activate column).
    UNSTABLE - Prepare just before use by diluting 1 ml stock potassium nitrate solution to one liter.
11. Fifteen ml potassium nitrate standard solution
    UNSTABLE - Prepare just before use by diluting 10 ml stock potassium nitrate solution to one liter.
12. Fifteen ml potassium nitrite standard solution
    UNSTABLE - Prepare just before use by diluting 10 ml stock potassium nitrite solution to one liter.
13. Fifty ml sample containing up to 1 mg NO₂⁻ + NO₃⁻ N/liter
    Example: 40 ml of nitrate standard plus 40 ml nitrite standard solutions per liter gives 0.4 mg N/liter of each form for a total of 0.8 mg NO₂⁻ + NO₃⁻ N/liter. A municipal wastewater treatment plant effluent known to contain preferably both forms can be used for a sample. You may need to assign dilutions so test is applicable.

B. Prepare excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of nitrate-nitrite-free distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Twenty slides, X-23 Nitrate/Nitrite Nitrogen - See next pages.
Types of nitrogen analysis (manual)
- Ammonia nitrogen
- Total Kjeldahl nitrogen
- Nitrate nitrogen
- Nitrite nitrogen

DETERMINATION OF NITRITE NITROGEN
BY DIAZOTIZATION
(Manual or automated)

Nitrite Nitrogen Interferences
- Strong oxidizing or reducing agents
- High alkalinity
- No other ions unless their concentration is 1000 times that of the nitrite concentration

Nitrate Nitrogen Determination
Manual Method
1. Adjust pH to 10.5
2. Filter if necessary
3. Place samples and standards into tubes
4. Add NaCl for saline samples
5. Chill to 0-10°C
6. Add H2SO4 solution
7. Add color reagent
8. Place tubes into boiling water for 25 min.
9. 410 nanometers

Nitrate Nitrogen Interferences
- Interferences:
  1. Strong oxidizing or reducing agents
  2. Iron Sidney
  3. Salinity
  4. Dissolved organic material
  5. Chlorine

Delination of Nitrate Nitrogen
1. Brucine Sulfide
   (Manual)
2. Hydrazine reduction
   (Automated)
3. Cadmium reduction
   (Manual or automated)
Nitrate Nitrogen - Technicon
Hydrazine Reduction,
1. Reduce nitrate to nitrite with hydrazine sulfate
2. Determine nitrite

DATA OBTAINED FROM THE CADMIUM REDUCTION METHOD
1. Determine nitrite plus nitrate
   Use nitrate standards and the reduction column
2. Determine nitrite
   Use nitrite standards and no reduction is involved
3. Calculate nitrate by subtracting
   Nitrite + Nitrate = Nitrite + Nitrate

SLIDE 10

Nitrite Plus Nitrate Cadmium Reduction
Removal of Interferences to Column Function
1. Prior to analysis
   Filtration removes suspended solids
2. During analysis
   Extraction removes oil and grease
   Complex metals with EDTA

SLIDE 11

Determination of NitratePlus Nitrite Nitrogen
1. Prepare nitrate working standards
2. Adjust pH of standard and sample to 5-9
3. Measure molar extinction standard
4. Add NH₄Cl - EDTA
5. Pass through activated reduction column
6. Immediately add nitrite color reagent
   into aliquot
7. Repeat steps 3, 4, 5, 6 for each standard and sample
8. Read absorbance at 540 nanometers

SLIDE 12

SLIDE 13

SLIDE 14

SLIDE 15

SLIDE 16

SLIDE 17

SLIDE 18

Series X-23: NITRATE/NITRITE NITROGEN
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Solids

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 190 minutes (15+10+60+45+30+10+10+10)

Note: This is time per one station. See VIII. B.2. for estimating weighing times according to number of stations and balances.

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Understanding of equations for inorganic reactions
B. Ability to use analytical balance
C. Ability to use graduates and filtration apparatus
D. Ability to perform calculations involving decimals
E. Completion of the lessons (or equivalent):
   1. Compliance Methodology
   2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know pertinent information about the procedures to do five gravimetric determinations of residues. He/she will use procedures in the training manual (see VII. A. 1. b. and c.) to determine total residue in a municipal wastewater treatment plant influent and nonfilterable (suspended) residue for a plant influent (membrane filter apparatus) and for a plant effluent (Gooch crucible), checking for constant weight after a second drying and reporting results.

B. Conditions: He/she will be given the training manual, 105 minutes of instruction, 75 minutes laboratory time, all necessary equipment, reagents and samples, and 10 minutes for class discussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of an Instructor and obtain results for each sample within + 10% of the class average if constant weight after drying was achieved during the allotted time.
VII. INSTRUCTIONAL RESOURCES:

A. Available Media:

1. Three outlines in the training manual:
   
a. "Testing for Solids in Water Samples"
   
b. "Laboratory Procedure for Total Solids" according to 1979 EPA Methods for Chemical Analysis, p. 160.3-1
   
c. "Laboratory Procedure for Suspended (Non-Filterable) Solids" according to 1979 EPA Methods for Chemical Analysis, p. 160.2-1


B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.

2. Clean and permanently mark identification on evaporating dishes and on Gooch crucibles to be used by the class. Weigh and record rough weights for each on a sheet(s) to post by balances.

3. Fire the evaporating dishes at 550 ± 50°C for 1 hour in a muffle furnace, partially cool and put in a desiccator, ready for weighing by class.

4. Put a glass fiber filter disc into each Gooch crucible and wash and dry each according to the procedure in the training manual. Discard any nonusable discs. Prepare one for each station plus a few extra. Place in a desiccator ready for weighing by class.

5. Mark identification numbers on planchets (or watch glasses). Use a membrane filter apparatus and wash glass fiber filter discs. Place each on a marked planchet (or watch glass) and store in a desiccator, ready for weighing by class. Prepare a few extras.

6. Check ovens for use, at 98°C to evaporate total residue samples and at 103-105°C to dry total and suspended residue samples.

7. Arrange to get influent and effluent samples from a municipal wastewater treatment plant, as listed in X below.

8. Review the laboratory procedures by performing the student assignments using the outlines in the training manual.

9. Review the topic outline in the training manual and the visual materials. See XI below.
10. Prepare the lesson using the sequencing below or your own organization of the material.

11. Assemble items for classroom briefing on initial weighings: Gooch crucible containing disc, membrane filter funnel and support and corresponding disc, watch glass for disc, forceps, 100-150 ml evaporating dish.

12. Distribute supplies and reagents to laboratory stations.

13. OPTIONAL: You might want to plan to demonstrate the Imhoff cone method to determine settleable solids if your participants need this information. You will need 1 liter of sample containing settleable solids, an Imhoff cone in a support, and a timer. The demonstration can be done during a laboratory session.

B. Sequencing: Slides are series X-28: Solids:

1. Classroom - Briefing (15 minutes) on weighings to be done during combined laboratory-lunch break. This plan works well when the participant to balance ratio is large.

   a. Explain why this scheduling. Lectures will be after lunch.

   b. Use outline on Total Solids. Show evaporating dish; give overview of test; III. A. B. and C. are done; assign D.; record weight on outline sheet in manual. Give location of desiccators and balances.

   c. Use outline on Suspended Non-Filterable Solids. Show membrane filter funnel and support and corresponding disc on a watch glass; note its use for influents; disc must be carefully transferred to balance pan with forceps and weighed. Show Gooch crucible containing disc; it stays in crucible for weighings; note its use for effluents; use tissue when handling. III. A. B. C. D. E. are done. Assign F. Desiccator and balance locations are the same as above. Stress that the constant weight check should be done for "real" tests, but in the interest of time they will omit this part of III F.

   d. A data sheet for Suspended and Non-Filterable solids is in the manual. They mark sample at top of column as "INF-MF" and "EFF-G" to ensure correct apparatus is used for the two samples. Note blank (1) for identification record on filters, and blank (14) for initial weight and the referenced* footnote. (Save any other discussion of the data sheet for lecture session). They should use same balance for all subsequent weighings. Rough weights of crucibles are posted by balances.

   e. Summarize - Three weighings are to be accomplished and recorded: an evaporating dish, a 4.7 cm disc and a 2.2, cm disc in a Gooch crucible.

2. Laboratory - 10 minutes per 3 weighings. Time allotment on agenda depends on number of stations and number of available balances. (Ex. 12 stations and 4 balances required 30 minutes). One way
to avoid back-ups is to schedule half of class first while others have a break or go to lunch, then first has a break or is at lunch while others use balances.

3. Classroom - Introduction - 8 minutes.
Participants should use the training outline, "Testing for Solids in Water Samples" as a reference for the following discussions.

a. Section II Solids and Water Quality - Overview of why tests are done.

b. Section IV lists NPDES Solids Tests.

c. Section V - accuracy of tests and term, "residue"

d. Section VI - All the tests are gravimetric determinations of residues remaining after test treatments. Settleable solids may also be reported in terms of volume. As required by the class, highlight information in this section. Do include B, Drying Temperature, to emphasize the differences and why they were chosen.

e. Section III, Sample Handling - Slide 1

4. Classroom - Total Residue - 10 minutes
Participants should use outline Section VII, "Total Residue"

a. Definition from view of components - Slide 2
Operational definition is in manual, Briefly note manual sections on why test is done.

b. Stepwise procedure - Slides 3, 4
1) Composition of dish
2) Sample volume limits - in manual
3) Steps 1 and 2 done. They did 3 and will do 4 through 7 in laboratory
4) Sources of error - see manual
5) Constant weight limits - in manual

5. Classroom - Total Dissolved Residue - 10 minutes
Participants should use outline Section VIII, "Total Dissolved (Filterable) Residue."

a. Terms filterable and non-filterable illustrated - Slide 5

b. Definition filterable (dissolved) - Slide 6

c. Effects - Slide 7
WWTP significance is in manual

d. Stepwise Procedure - Slides 8, 9, 10, 11
1) Sample volume limits - in manual
2) Constant weight limits - in manual

e. Interferences - Sources of Error - Slide 12

6. Classroom - Total Suspended Residue (Nonfilterable Solids) - 11 minutes
Participants should use outline Section IX, "Total Suspended (Non-Filterable Residue)".

   a. Definition - Slide 13
   b. Effects - Slide 14
      WWTP significance is in manual
   c. NPDES regulations - Slide 15
      1) This is in the manual
      2) May be different for lagoons - this is in the manual
   d. Stepwise Procedure - Slides 16, 17
      1) Can use crucible (effluents) or membrane filter support with
coarse fritted disc (influents) for the disc.
      2) Steps 1 and 2 done, they did step 3 and will do 4 through 6
in the laboratory for an effluent and an influent sample
   3) Sample volumes - in manual
   4) Sources of error - in manual
   5) Constant weight limits - in manual

7. Classroom - Settleable Residue - 10 minutes
Participants should use outline Section X, "Settleable Residue".

   a. Definition, relationship to suspended - Slide 18
   b. Effects - Slide 19
      1) Blanket effect - affects biological activity which affects
CO₂ content and, thus, pH. pH affects adsorption/release of
organics and heavy metals.
      2) WWTP significance is in manual.
   c. Procedure by weight - Slide 20
   d. Procedure by volume - Slide 21
   e. Interferences and sources of error for both procedures are in
manual.
8. Classroom - Conclusion - 3 minutes
   a. Repeat temperatures for drying
   b. Remind about the constant weight condition - may need several weighings
   c. Note accuracy variables, especially representative sample. It is very important to have consistent technique.
   d. Suspended solids quality control samples are available through EPA Regional Quality Assurance Coordinators.

9. Classroom - Lab Briefing - 8 minutes
   a. Total - Influent sample - In the training manual procedure for "Total Solids", do III E. F. G. H. For I, put the dish in a 98°C oven. The Staff will do J. After cooling, the participants will do K and return the dish to a 103 - 105°C oven for a second drying cycle and weighing.
   b. Suspended (Non-Filterable) - In the training manual, for "Suspended (Non-Filterable) Solids", do III G through R for an influent sample using a large disc in a membrane filter funnel. Do G through Q, then S for an effluent sample using a disc in a Gooch crucible. The staff will remove items from the oven. After cooling, participants will weigh the items and return them to a 103-105°C oven for a second drying cycle and weighing. The data sheet has blanks for all the data.

10. Break - 10 minutes

11. Laboratory - 45 minutes
    Student performance of the assignment up to putting items in the ovens for the first time.

12. Classroom - Volatile Solids - 30 minutes
    Participants should use outline Section XI, "Total Volatile Residue".
    a. Introduction, title - Slide 22
    b. Definition and relationship to other solids (residues) - Slide 23
    c. Why test is done - Slides 24, 25
       Effects and WTP significance is in manual
    d. Steps of test - Slides 26, 27
    e. Define fixed residue - Slide 28
    f. Preparation of dish or crucible - Slide 29
    g. Negative errors in the determination, examples - Slides 30, 31
    h. General sources of error - Slide 32 (last slide)
       Also are in manual

2:0
i. Conclusion

13. Staff - For total residue, increase temperature in oven from 98°C to 103-105°C after samples evaporate. For all residues, transfer dishes, discs and supports to desiccators after first drying so they are at room temperature for first weighing.

14. Laboratory - First weighing of two Non-filterable (suspended) residues - 7 minutes (See 9 above). Put discs back in oven.
   First weighing of one Total residue - 3 minutes (See 9 above). Put dish back in oven.

15. Staff - Later, remove discs and supports from oven and put into desiccators so they are at room temperature for second weighing.

16. Laboratory - Second weighing of all three residues - 10 minutes

17. Class Discussion - 10 minutes
   a. Review the constant weight check limit for each test. Have participants compare the two weights they obtained for each of the three tests and calculate the differences.
   b. Participants should post the differences on the board.
   c. Discuss the results posted for the constant weight check.
   d. Participants should use the calculation formulas (IV in laboratory procedures or, for suspended residue, a stepwise calculation is on the data sheet). They should post results on the board. Discuss the results. Check on very low results, especially for the two suspended (non-filterable) tests. EPA recommends having at least 0.47 mg residue on a 2.2 cm disc and 1.0 mg on a 4.7 cm disc. Participants should check their residue weights accordingly.
   e. Give concluding cautions on techniques involved, e.g. handling the discs, finger prints on dish, etc.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: One total residue test, and one non-filterable (suspended) residue test on an influent sample
One non-filterable (suspended) residue test on an effluent sample

A. For each station: (no more than two persons per station):
   1. Enough desiccator space for one 150 ml evaporating dish, one Gooch crucible, and one small planchet (or watch glass). Desiccator should be usable.
   2. One 150 ml evaporating dish with a permanent identification mark on it, stored in a desiccator, ready for weighing.
   3. One glass fiber filter disc in a Gooch crucible (permanent identification mark), washed, dried and in a desiccator, ready for weighing.
4. One glass fiber filter disc to fit a membrane filter apparatus, washed, dried and stored on a marked planchet (or watch glass) in a desiccator, ready for weighing.

5. One 25 or 50 ml graduated cylinder

6. One 100 ml graduated cylinder

7. One suction flask with hose and pinch clamp

8. One membrane filter holder assembly with rubber stopper to fit neck of suction flask

9. One pair blunt-tipped forceps

10. One rubber adapter to hold crucible in neck of suction flask

11. One pair tongs

12. One vacuum source or pump drawing 15 inches of mercury

13. One squeeze bottle of distilled water

14. One marking pencil

15. One apron for each student

16. One pair safety glasses for each student

B. Shared:

1. Desiccators with usable desiccant. See A.1. above for space requirement per station.

2. Box small tissues by each desiccator

3. Balances, analytical - One per 3 stations

4. Box small tissues by each balance

5. Drying oven or steam bath for use at 98°C and to accommodate one 150 ml evaporating dish per station

6. Oven for use at 103-105°C and to accommodate three items per station: one 150 ml evaporating dish, one Gooch crucible, and one small planchet (or watch glass).

X. IPW REAGENT REQUIREMENTS:

A. Minimum amounts per station (no more than two persons per station):

1. Enough plant influent sample to yield 25 to 200 mg total or suspended residue

2. Enough plant effluent sample to yield 25 to 200 mg suspended residue
3. EPA-EMSL, Cincinnati, Ohio 45268 supplies free analytical quality control samples containing known amounts of suspended solids. This might be an alternative if plant samples are not available.

B. Obtain excess of each sample to allow for rinsing out glassware, waste and re-runs.

C. A source of distilled water should be available during laboratory sessions.

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Thirty-two slides, X-28: Solids' - See next pages.
SAMPLE HANDLING

- Resistant glass bottles or plastic-containers
- No preservative
- Analyze as soon as possible
- Remove large particulates (leaves, twigs, etc.)

TOTAL SOLIDS

The sum of the homogeneous suspended and dissolved materials in a sample

FILTERABLE SOLIDS

Those solids: passing through a standard glass fiber filter and dried to constant weight at 180 degrees C
SERIES X-28 SOLIDS

Slide 10

Slide 11

Slide 12

Slide 13

Slide 14

Slide 15

Slide 16

Slide 17

Slide 18

NON-FILTRABLE, SUSPENDED SOLIDS

1) Water Supplies
   - Possible contamination
   - Possible aggregation
2) Irrigation
   - Plant growth and quality
   - Soil quality
   - Equipment
3) Recreational
4) Aesthetics

NON-FILTRABLE SOLIDS INTERFENCES

1) High salt concentration
2) A lack of solids
3) Too much residue

SETTLEABLE SOLIDS

THAT MATTER WHICH
WILL NOT STAY IN SUSPENSION
DURING A 1 HOUR SETTLING PERIOD

1245
NON FILLER, SETTLEABLE SOLIDS

- 10 CLAYETE VALUES
- 27 RECIRCULATION VALUES
- 23 MECHANICAL VALUES
- BOTTOM FILL
- BOTTOM FILL / BLOTS
- SPURTING GROUND
- T. DEG. BATHS

ORGANIC COMPOUNDS
- COMPLETE BOTTOM CARBON
- PRODUCE INNOUS GASES

SETTLEABLE SOLIDS - WEIGHT

1. DETERMINE MG/L NON-FILTERABLES
2. DETERMINE M/L NON-SETTLEABLES
3. DETERMINE MG/L NON-SETTLEABLES
4. SG/SODA 250 ML FROM CENTER OF SETTLEABLES
5. DETERMINE MG/L NON-SETTLEABLES
6. REPORT RESULTS IN MG/LITER
7. HINUS (2) SETTLEABLE SOLIDS

TOTAL VOLATILE RESIDUE
mg/L

ROUGH MEASURE OF ORGANIC MATTER

IGNITION AT 550°C ± 50°C
15 - 20 min.

REASONS FOR DETERMINATION
- WATER QUALITY REQUIREMENT
- PLANT CONTROL
1. Ignite residue at 550°C to constant weight
2. Cool in desiccator
3. Weigh dish plus residue
FIXED RESIDUE

550°C ± 50°C PRE-IGNITION OF DISH OR CRUCIBLE

MgCO₃ → MgO + CO₂

SOURCES OF ERROR
- Uncontrolled temperature
- CaCO₃ → CaO + CO₂
  (major)
- Volatiles loss before ignition
- Incomplete organics oxidation
- Loss of water of crystallization
- Handling of dish

NH₄₂CO₃ → 2NH₃ + H₂O + CO₂
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Elements of a Quality Assurance Program

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 75 minutes

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: An analyst must know and practice requirements for quality assurance when conducting analytical procedures and reporting results.

V. ENTRY LEVEL BEHAVIOR:
   A. Fundamental knowledge of quantitative analyses
   B. Familiarity with equipment and supplies normally found in a water quality chemistry laboratory
   C. At least six months experience in chemical laboratory work
   D. Completion of the lessons (or equivalent):
      1. Sample Handling
      2. Compliance Methodology
      3. Analytical Techniques
      4. Volumetric Analysis
      5. Precision
      6. Accuracy
      7. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will be able to utilize the given outline (see VII. 1. a.) of activities required to effectively fulfill the demands of a quality assurance program for a chemistry laboratory. (VIII. B, Sequencing, in this IPW lists the divisions of these activities)
   B. Conditions: He/she will be given the training manual, handouts and 75 minutes of discussion.
   C. Accepted Performance: He/she must recall from memory at least 70% of the items pertaining to the objective in the post-course evaluation.
VII. INSTRUCTIONAL RESOURCES:
A. Available Media:
1. Three outlines in the training manual:
   a. "Elements of a Quality Assurance Program" which is primarily an outline of the information in resource 3. below.
   b. "Methodology for Chemical Analysis of Water and Wastewater" which defines the principles utilized in recognized methodology.
   c. "Use of a Spectrophotometer" has a section (IV) on calibration standards.
2. Five slides, X-26: Quality Assurance (See XI Description of Visual Materials)
4. Two Handouts - To ensure that current information is distributed, request this information from U.S. EPA, EMSL - Quality Assurance Branch, Cincinnati, Ohio 45268:
   a. The current list of quality control samples available from EPA through the EPA Regional Quality Assurance Coordinators.
   b. (Optional): Roster of EPA Regional Quality Assurance Coordinators if students are from several regions
5. One Handout - Sheet with examples of significant figures and basics on "rounding off" and calculating rules. A copy is at the end of this IPW.
B. Suggested Media:
   1. None
VIII. INSTRUCTIONAL APPROACH:
A. Preparation for Instruction:
1. Review visual materials and available handouts.
2. Prepare the lesson using the Sequencing below or your own organization of the material.
3. Duplicate copies of handout materials (See VII. A. 4.) for each student. Assemble in packets.
4. Assemble demonstration items: volumetric flask, volumetric pipet and small tissues for demonstration of glassware markings and swiping technique for pipets.
B. Sequencing:

Use indicated sections of topic outline in the training manual to discuss these topics unless otherwise noted.

Slides are series X-26: Quality Assurance

1. Introduction - I. A. and B. Note that there are divisions in the outline with details on the 5 elements in I. C. This is primarily an outline of highlights of reference 1. Then briefly cover I.D. and I.E.

2. II. Sample - This was considered in detail in prior lesson, "Sample Handling"

3. III. Recognized Methodology - This was considered in detail in prior lesson, "Compliance Methodology."
   a. D. - definitions of commonly-used types of methodology are in course manual outline, "Methodology for Chemical Analysis" of Water and Wastewater.
   b. F. - Stress strict adherence to reference source when using recognized procedures.

4. IV. Services, Instruments, Supplies - Give highlights. Some notes:
   a. B. 2. g. - blank plus one, preferably two, standard(s) may be used to check a curve for some methods, rather than running a complete set of standards each run.
   b. C. 2. the four functions: a, b, c, d
   c. C. 2. d. Show volumetric flask, volumetric pipet and measuring pipet to demonstrate location of glassware information and also TD and TC terms - Also use pipet and tissue to demonstrate swiping excess off outside of pipet before final reading.
   d. D. 2. General guides are here (a, b, c, d) for their future reference.
   e. D. 5. Standard solutions are in lesson, "Volumetric Analysis."
   f. D. 5. a. 6) Outline "Use of a Spectrophotometer" has Section on Preparation of Calibration Standards, IV. B. Discuss stability information there.
   g. D. 5. c. This is especially important for "Compliance Methodology."

5. V. Quality Analytical Performance
   a. A. Summarize
   b. B. and C. Recall prior lessons, "Precision" and "Accuracy"
c. D. Note 1, 2, 3, 4

d. E. Present items 1, 2, 3, 4, 5

e. Slides of normal curve and control charts using some of the statistics presented in lessons on precision and accuracy:

1) Normal curve - Relate midline, 2 s and 3 s limits to control chart central line, warning and control limits. Slide 1.

2) Precision Chart - R as estimate of s using duplicates. Plot succeeding R of duplicates. Slide 2 (This approach is recommended in the 1979 EPA Handbook for Analytical Quality Control).

3) Precision Chart - X and s, using one check standard. Plot succeeding results. Slide 3.

4) Accuracy Chart - X and s of % recoveries using spiked samples. Plot succeeding % recoveries of spikes. Slide 4 (This approach is recommended in the 1979 EPA "Handbook for Analytical Quality Control").

5) Accuracy Chart - X and s of % bias using spiked samples. Plot succeeding % bias (% relative error). Slide 5

f. F. Interlaboratory Checks on Performance

1) F. 1. Hand out the list of EPA quality control samples currently available through EPA Regional Quality Assurance Coordinators. Give the Participants the telephone and address information for their Regional Coordinator.

2) F, 2, 3, 4, 5 - Note these checks

6. VI. Data Handling and Reporting

a. A/1, 2, 3

b. A. 4 Hand out and discuss the sheet on significant figures. Relate it to course methodology. Note reference for more detail.

c. D. 1. Note current Federal Register regulations regarding storage of data.

7. VII. Safety Considerations - Note highlights. This was considered in detail in a previous lesson. Encourage appointment of Safety Officer.

8. VIII. EPA Regional Quality Assurance Coordinators.

a. Encourage contact with EPA coordinator.

9. References - Note the book.
IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:
   A. For each student:
      1. Copies of handout materials

X. IPW REAGENT REQUIREMENTS:
   A. None

XI. DESCRIPTION OF VISUAL MATERIALS:
SIGNIFICANT FIGURES - EXAMPLES

32.2 has 3 significant figures
32.20 has 4 significant figures
0.0014 has 2 significant figures (zeros used to fix decimal location)
0.1400 has 4 significant figures
0.14 has 2 significant figures (the zero indicates no whole number)

1. Retain as many significant figures in a result or in laboratory data as will give only one uncertain figure. (Possibly, in very accurate work involving lengthy computations, two uncertain figures may sometimes be retained.)

2. When rounding off, if the rejected figure is 0, 1, 2, 3, or 4, just let the remaining significant figure stand as is. If the rejected figure is 5, round the remaining figure to the nearest even number. 2.25 becomes 2.2 and 2.35 becomes 2.4. If the rejected figure is 6, 7, 8, or 9, increase the remaining significant figure by one unit.

3. As a practical operating rule for multiplication or division, keep as many significant figures in an answer as were contained in the factor having the least number of significant figures. The answer should not "suffer" if the other factors are limited to a minimum of one more significant figure than that least number of significant figures during the calculation.

4. For addition or subtraction, the number with the fewest decimal places determines the number of decimal places retained. However, no one number involved in the calculation operation limits the number of significant figures in the result.

GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Turbidity

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 95 minutes (45+40+10)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:

A. Ability to correctly use volumetric glassware

B. Completion of the lesson (or equivalent):
   1. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:

A. Terminal Behavior: The participant will know pertinent facts about turbidity and procedures to determine turbidity (see VIII. B.). He/she will dilute a stock solution to make standards (see VIII. B. 2.), standardize a turbidimeter ensuring that the cell will be consistently oriented in the meter, measure the turbidity of a sample using the procedure in the training manual (see VII. A. 1. b.), report results and participate in a class discussion of the topic and laboratory procedures.

B. Conditions: He/she will be given the training manual, 45 minutes of instruction, 40 minutes laboratory time, all necessary equipment, a stock solution and sample, and 10 minutes for class discussion.

C. Accepted Performance: He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedure to the satisfaction of an Instructor and obtain results for the sample within ± 10% of the class average (unless the reason for the lack of agreement is acceptable to the Instructor).

VII. INSTRUCTIONAL RESOURCES:

A. Available Media:
   1. Two outlines in the training manual:
      a. "Turbidity"
      b. "Calibration and Use of a Turbidimeter (Nephelometer)" according to 1979 EPA Methods for Chemical Analysis, 180.1-1
   2. Ten slides, X-30: Turbidity (See XI Description of Visual Materials)
VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.

2. Prepare all necessary reagents and sample as in X below.

3. Review the laboratory procedure by performing the student assignment using the outline in the training manual.

4. Review the topic outline in the training manual and the visual materials. See XI below.

5. Prepare the lesson using the Sequencing below or your own organization of the material.

6. Check turbidimeters for accurate operation.

7. Distribute supplies and reagents to laboratory stations.

B. Sequencing: Slides are series X-30: Turbidity

1. Classroom instruction on turbidity - 45 minutes
   a. Introduction
      1. Cause - Slide 1
      2. Why measure turbidity - Slide 2
      3. Sample handling - preservation and holding time - Slide 3
         (For compliance monitoring, maximum holding times proposed during 1979 were 24 hours for drinking water samples and 48 hours for NPDES samples. Consult current federal registers for current status).
      4. Original Jackson Candle Turbidimeter - Slide 4
   b. Regulations - NPDES and Water Supply both require nephelometers.
   c. Acceptable instrumentation
      1) Nephelometry - Slides 5, 6,
      2) Formazin Stock Solution - Slides 7, 8, 9
         Note: A final decision on use of styrene divinylbenzene polymer standards for drinking water calibration standards is to be made after 10/27/80. Check on the status of approval before presenting this.
      3) Interferences - Slide 10
   d. Summary
2. Laboratory - 40 minutes
   a. Briefing - Participants use training manual "Calibration and Use ..." outline, III. A., 1 through 17 to use the provided 400 NTU standard for preparing three (40, 4, and 0.4 NTU) standards for the DRT instrument or four (40, 4, 0.4 and 0.04 NTU) standards for the Hach 2100 A instrument, standardizing the instrument, then determining turbidity on the provided sample and on tap water.
   b. Student performance of the assignment

3. Class Discussion - 10 minutes
   a. Participants report results.
   b. Cover any items from laboratory sessions of general nature.
   c. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Dilute the stock solution to make one calibration standard for each range of the instrument to be used, e.g., 40, 4, 0.4, 0.04.

Test two samples (tap water plus 1 synthetic).

To minimize the number of turbidimeters required, the laboratory could be conducted twice with half the class doing another procedure requiring about the same time, e.g., Specific Conductance.

A. For each station (no more than two persons per station):
   1. One pipet bulb
   2. Three or four 100 ml volumetric flasks with stoppers
   3. Three or four 10 ml volumetric pipets if it contains measurable turbidity
   4. One turbidimeter (nephelometer) with cell.
      CAUTION: To ensure that the cell is oriented consistently during the procedure, the participants should mark the cell and instrument during the calibration procedure.
   5. One squeeze bottle turbidity - free distilled water
   6. One marking pencil
   7. One apron for each student
   8. One pair safety glasses for each student

B. Shared:
   1. None
X. IPW REAGENT REQUIREMENTS. For details of reagent preparations, see 1979
EPA "Methods for Chemical Analysis", 180.1-1

A. Minimum amounts per station (no more than two persons per station):

1. Ten ml 400 NTU stock Formazin suspension

2. About 50 ml sample (It is convenient to make 1 liter of sample by
diluting 5 ml of a 40 NTU standard to 1000 ml for a resulting
turbidity of 0.2 NTU)

3. Note: Tap water is the second sample. Also, a treatment plant
effluent sample could be used if it contains measurable turbidity.

B. Prepare excess of each solution to allow for rinsing out glassware, waste
and re-runs.

C. A source of turbidity-free distilled water should be available during
laboratory session(s).
WHY MEASURE TURBIDITY?

1. CAUSE OF TURBIDITY
   a. can impede chlorine disinfection
   b. can be injurious to industrial processes and equipment

2. SERVES AS INDEX TO:
   a. aesthetic quality of water
   b. efficient plant operation
   c. filter breakthrough

SAMPLE HANDLING

STORAGE: STORE IN THE DARK

HOLDING TIME:

ANALYZE AS SOON AS POSSIBLE

SLIDE 1

SLIDE 2

SLIDE 3

SLIDE 4

SLIDE 5

SLIDE 6

SLIDE 7

SLIDE 8

SLIDE 9
INTERFERENCES
RAPID SETTLING COARSE SEDIMENTS
FLOATING DEBRIS
AIR BUBBLES
COLOR
DIRTY OR SCRATCHED SAMPLE TUBES

SLIDE 10
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

I. SUBJECT MATTER: Specific Conductance

II. UNIT OF INSTRUCTION: Summary of Topic Presentation

III. ESTIMATED TIME: 105 minutes (45+50+10)

IV. JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE. The participant will be responsible for this determination to meet water quality control program requirements.

V. ENTRY LEVEL BEHAVIOR:
   A. Understanding of terms "ions", "ionization", "dissociation"
   B. Ability to perform calculations involving decimals
   C. Completion of the lessons (or equivalent):
      1. Volumetric Analysis (terms "acid", "base", "salt", "normality")
      2. Laboratory Safety Practices

VI. INSTRUCTIONAL OBJECTIVE:
   A. Terminal Behavior: The participant will know pertinent facts about specific conductance (see VIII B). He/she will use the procedures in the training manual (see VII A 1 b) to check out a conductivity meter, use it to determine cell constants for three different probes, and determine specific conductance on three samples. He/she will report results and participate in a class discussion of the topic and laboratory procedures.
   B. Conditions: He/she will be given the training manual, 45 minutes of instruction, 50 minutes of laboratory time, all necessary equipment, standards, samples and 10 minutes for class discussion.
   C. Accepted Performance. He/she must correctly answer 70% of the items on this topic in the post-course evaluation, perform the laboratory procedures to the satisfaction of the instructor and obtain results for each sample within ± 10% of the class average (unless the reason for a lack of agreement is acceptable to the instructor).

VII. INSTRUCTIONAL RESOURCES:
   A. Available Media:
      1. Two outlines in the training manual:
         a. "Specific Conductance"
         b. "Calibration and Use of a Conductivity Meter" according to 14th edition Standard Methods, p. 71 and manufacturer's instructions for Beckman's Solubridge instrument. The outline might serve as
a guide if you need to develop laboratory directions for instruments other than this one.

2. Fifteen slides, X-29: Specific Conductance (see XI Description of Visual Materials).

B. Suggested Media:

1. None

VIII. INSTRUCTIONAL APPROACH:

A. Preparation for Instruction:

1. Check equipment and supplies. See IX below.

2. Prepare all necessary reagents and sample as in X below.

3. Review the laboratory procedure by performing the student assignment using the Outline in the training manual. If you have different meter-probe equipment, you will have to revise the training outline or even prepare a new one to fit your needs.

4. Review the topic outline in the training manual and the visual materials. See XI below.

5. Prepare the lesson using the Sequencing below or your own organization of the material.

6. As appropriate, check batteries, conductivity meters and probes for accurate operation.

7. Distribute supplies and reagents to laboratory stations.

B. Sequencing:

Slides are series X-29: Specific Conductance

1. Classroom Instruction on Specific Conductance - 45 minutes
   a. NPDES methodology unit, temperature condition, and measurement requirement (slide 1).
   
   b. Conductivity is an expression of the ability of a water sample to carry an electric current. Discuss the processes resulting in ions (slide 2), the classes of compounds that produce ions and the role of the number of ions produced and their charges (slide 3).

   c. Conductivity is the reciprocal of resistivity. Many have probably seen this demonstration of the property of conductivity (slide 4). Relate it to resistivity.

   d. Representation of a Wheatstone bridge which measures resistance, and a list of types of sensors (slide 5).

   e. Units used for resistance and conductance (slide 6).
Discuss electrode characteristics: one type of construction (slide 7), and the critical factors (slide 8). A "standard" cell is one in which electrodes are spaced exactly 1 cm apart and each has a surface area of 1 cm². Laboratory cells are usually not standard.

How to determine a cell constant for cells which are not standard (slide 9).

If a cell constant is calculated from standard cell conductance values, this formula converts meter results to the value that would have been obtained using a standard cell. The result is called "specific" conductance (slide 10).

Application of a cell constant has limits (slide 11).

Temperature is a critical factor affecting conductivity. (See figure 5 in outline). Therefore, results are to be reported as values that would be obtained at 25°C. This chart from 13th ed. Standard Methods (slide 12) demonstrates the significant effect of temperature by showing the factor required to convert results to a 25°C equivalent. Most meters give results which are temperature-compensated, so it is not necessary to convert the results.

Discuss the problems encountered in this measurement (slide 13).

Briefly note various applications of conductivity measurements. Details about these applications, including graphs as appropriate, are in the training manual outline (slides 14 and 15).

2. Laboratory - 50 minutes. Use the outline, "Calibration and Use of Conductivity Meter" or its equivalent.
   a. Assignment of training manual procedures
      1) Check instrument using II B and II C
      2) Obtain readings to calculate cell constants for the three probes using section III
         a) Note most accurate part of scale
         b) Note Cell Information and Table 1.
      3) Obtain readings for 3 samples. Note section IV and Table 2
      4) As time permits, calculate cell constants (note IV B.1) and apply to readings for samples to get results to report.
   b. Student performance of assignment

3. Classroom Discussion - 10 minutes
   a. Students finish calculations and post results.
   b. Discuss results.
   c. Discuss any items from laboratory session.
d. Answer any questions.

IX. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

Assignment: Cell constant for three probes
Three specific conductance samples

To minimize the number of conductivity meters and probes required, the laboratory session could be conducted twice with half the class doing other procedures requiring about the same time, e.g., Turbidity and Second Weighing of Solids.

A. For each station (no more than two persons per station):

1. Seven 400 ml beakers
2. One 600 ml beaker (probe rinse)
3. One box tissues
4. One thermometer
5. One conductivity meter with Wheatstone Bridge (Ex. Beckman Solu Bridge)
6. One 10,000 ohm resistor
7. Three conductivity probes - VS2, VS02, VS20, or equivalent
8. One squeeze bottle distilled water
9. One marking pencil
10. One apron for each student
11. One pair safety glasses for each student

B. Shared:

1. None

X. IPW REAGENT REQUIREMENTS: For details of reagent preparations, see 14th edition "Standard Methods", page 74.

A. Minimum amounts per station (no more than two persons per station):

1. Four hundred ml each of four potassium chloride standards - 0.1N, 0.01N, 0.001N, 0.0001N
2. Four hundred ml municipal wastewater treatment plant effluent sample. A synthetic sample could be used instead. See next.
3. Four hundred ml sample “A”, the same concentration as the 0.01N KCl standard (or can mix standards).
4. Four hundred ml sample "B", the same concentration as the 0.001N KCl standard (or can mix standards).

B. Prepare/obtain excess of each solution to allow for rinsing out glassware, waste and re-runs.

C. A source of distilled water should be available during laboratory session(s).

XI. DESCRIPTION OF VISUAL MATERIALS:

A. Fifteen slides, X-29: Specific Conductance – See next pages.
Specific Conductance
µmhos/cm
25°C
Wheatstone Bridge

SLIDE 1

SLIDE 2

SLIDE 3

SLIDE 4

SLIDE 5

SLIDE 6

SLIDE 7

SLIDE 8

SLIDE 9
DETERMINATION OF SAMPLE Kon

SPECIFIC CONDUCTANCE = CELL CONSTANT X MEASURED CONDUCTIVITY OF SAMPLE

THE CELL CONSTANT IS APPLICABLE FOR A PARTICULAR

1) PROBE
2) TEMPERATURE
3) CONCENTRATION RANGE

PROBLEMS

1) DIRTY ELECTRODES
2) LARGE TEMPERATURE DIFFERENCES BETWEEN SAMPLE AND STANDARD SOLUTIONS
3) STATIC ELECTRICITY
4) GASEOUS CONTAMINANTS

APPLICATIONS OF CONDUCTIVITY

1) RELATIONSHIP TO DISSOLVED SOLIDS
2) 10 - 1000 uS/cm/CH FOR FRESH NATURAL WATERS
3) < 1.0 FOR DISTILLED AND BAKED WATER
4) 30,000 FOR SEA WATER
5) FINDING UNDERGROUND CHANNELS
6) LOCATION OF SPRING IN ROUTES OF WATER
7) SEA WATER LEAKAGE INTO FRESH WATER WELLS

APPLICATIONS OF CONDUCTIVITY (CONTINUED)

6) MAPPING UNDERGROUND TERRIANES
7) LOCATING OIL
8) ENCROACHMENT OF SEA WATER INTO RIVERS
9) LOCATING POLLUTION SOURCES