This is one of several short-term courses developed to assist in the training of wastewater treatment plant operational personnel in the tests, measurements, and report preparation required for compliance with their NPDES permits. This Staff Guide provides step-by-step guidelines on course planning, development and implementation involving classroom instruction, and laboratory application of critical learning outcomes. Part I is concerned with the administrative aspects of the training program. Part II consists of instructional staff guidelines on technical content, learning objectives, and lesson-by-lesson guides for the self-monitoring procedures contained in this course. Included are a variety of techniques for determining various materials in water including phosphorus, nitrogen, ammonia, cadmium, oil, and grease. (CS)
EFFLUENT MONITORING PROCEDURES: NUTRIENTS

STAFF GUIDE

U.S. ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF WATER PROGRAM OPERATIONS
STAFF GUIDE

for

CONDUCTING THE COURSE

EFFLUENT MONITORING PROCEDURES: NUTRIENTS

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U.S. Environmental Protection Agency
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(ERIC Logo)
Background of Effluent Self-Monitoring Requirement

With passage of the Federal Water Pollution Control Act Amendments of 1972 (Public Law 92-500) a new permit program was created to replace and improve upon the earlier permit system which existed under the 1899 Refuse Act.

Under the 1972 Act, the United States Environmental Protection Agency is required to establish national effluent limitations and national treatment performance standards for all sources of water pollution, including not only municipal discharges, but also factories, animal feedlots, and power plants. These effluent limitations are the maximum amount of a pollutant that any discharger may release into a water body.

In order to insure that the prescribed effluent limits are met, every discharger is required to obtain an NPDES (National Pollutant Discharge Elimination System) Permit. Types of water discharge sources for which a permit is required include municipal wastewater treatment facilities; manufacturing plants; agriculture, forestry, mining and fishing operations, and other service, wholesale, retail, and commercial establishments having operations which result in discharge of water to the Nation's bodies of water.

The NPDES Permit is not a license to pollute. To the contrary, a Permit stipulates what may be discharged, and how much may be discharged over a defined period of time. Each Permit is tailored to the discharger, and sets specific limits on each effluent.

Furthermore, the NPDES Permit also requires dischargers to monitor their effluents, performing specified tests and measurements at designated frequencies specified in the individual Permit, and to report the amount and nature of all waste components discharged.

Training Need Associated with Self-Monitoring Requirements

Compliance with NPDES requires that all specified tests and measurements be performed in accordance with methods specified by EPA and announced in the Federal Register. Only under very limited conditions are deviations from specified methods acceptable.

These requirements are the basis of an immediate, massive, training need to provide the responsible waste water treatment personnel with the knowledge and skills required to comply with the self-monitoring requirement. There is at present a wide range of initial capabilities for performing the tests and measurements. This ranges from the situation in the large, fully-staffed, fully-equipped facility in which little or no training is required, through all shades and levels to the limited staff in small, inadequately-equipped facilities in which at present there is little or no capability for performing the required tests and measurements.

AT.EMP.(164.3).1.10.76
This is one of several special short-term courses recently developed and currently under development by the Environmental Protection Agency and associated educational institutions. This Instructor's Guide is intended to assist other training organizations in conducting training of waste water treatment plant operational personnel in the tests, measurements, and report preparation required for compliance with their NPDES Permits.

TRAINING AVAILABLE OR UNDER DEVELOPMENT TO MEET SELF-MONITORING REQUIREMENTS

<table>
<thead>
<tr>
<th>Title</th>
<th>Workers should take this who:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Basic Laboratory Skills for Self-Monitoring Tests and Measurements</td>
<td>Have little or no laboratory experience or training, and need to develop basic skills including use of balances, preparation of solutions and reagents; names, characteristics, preparation and care of common laboratory equipment supplies. This course prepares students requiring such training for entry into the following named courses in which procedures for designated tests and measurements are learned.</td>
</tr>
<tr>
<td>Self-Monitoring Procedures: Basic Parameters for Municipal Effluents</td>
<td>Have basic laboratory skills, as designated above, but need to learn one or more of the procedures required for most or all municipal effluents including BOD, fecal coliform, pH, suspended solids, flow, and reporting of results.</td>
</tr>
<tr>
<td>Effluent Monitoring Procedures: Metals Analyses</td>
<td>Usually have completed the &quot;Basic Parameters&quot; course, but have a Permit which requires report on one or more metals.</td>
</tr>
<tr>
<td>Effluent Monitoring Procedures: Nutrients</td>
<td>Usually have completed the &quot;Basic Parameters&quot; course, but have a Permit which requires periodic report on one or more of the so-called &quot;nutrients&quot; including nitrogen and phosphorus.</td>
</tr>
<tr>
<td>Effluent Monitoring Procedures: Flow Measurement and Sampling Techniques</td>
<td>Have made arrangements whereby the required laboratory procedures are performed elsewhere by contract or other special arrangement, and who are required only to collect and care for samples, and to make such tests and measurements as must be made at the sampling site.</td>
</tr>
<tr>
<td>Any of the analytical courses of EPA National Training and Operational Technology Center for specific Permits</td>
<td>Are professional chemists, microbiologists, or key laboratory technicians in large treatment facilities where they work full-time doing a designated series of analytical tests and measurements.</td>
</tr>
</tbody>
</table>
A Personal Note to the User of this Guide

The need for providing this training, and the nature of current efforts of the Environmental Protection Agency to meet this need have been discussed in foregoing paragraphs.

It is an oversimplification of a classic quotation to say that all that is needed to conduct training is to "put Mark Hopkins on one end of a log and a student on the other." It is almost as much an oversimplification to say that all that is needed to conduct a course is to bring together the students and the instructional staff in a classroom and laboratory equipped with requisite equipment and supplies for the instruction to be delivered.

A short course such as this, involving both classroom instruction and laboratory application, and with critical learning outcomes to be achieved within a limited period of time, absolutely requires careful, detailed planning, preparation, and implementation. Meticulous attention to detail and effective staff teamwork are essential at every step of course planning, development, and implementation.

The purpose of this Staff Guide is to provide you, the training staff member, with useful, practical assistance in presenting this course in your own facilities. The Guide is a resource. It is not a blueprint to be followed rigidly or unthinkingly. Even with this guide or any other form of assistance, you will have to plan, to think and to prepare in order to perform effectively in conducting this course. On the other hand, this Guide should be helpful in reducing the amount of original development work you will have to do, and it should be helpful in suggesting factors in course planning and presentation which otherwise might be ignored or glossed over.

You are invited, in fact requested, to participate actively in making this Guide a living document which effectively represents the best experience of all in planning, preparing for, conducting and terminating this course. Please provide this office with your constructive suggestions for strengthening and improving upon this Guide, based on your own experiences in conducting the course. Your recommendations will be fully considered in future editions of this and other Staff Guides for other courses of this series. In the event that questions arise in interpretation of any aspect of this Instructors' Guide, please write or call:

Director, National Training and Operational Technology Center
Office of Water Program Operations
U.S. Environmental Protection Agency
Cincinnati, OH 45268

Telephone: (513) 684-7501

Format of this Manual

This manual consists of two major parts. Part I is concerned with administrative aspects of planning, preparing, and conducting the course. Part II consists of guidance to the instructional staff on the technical content, learning objectives to be achieved by the students, and lesson-by-lesson instructional guides for each of the several self-monitoring procedures covered in this course.
Part I, which immediately follows, will have greatest value to those conducting this course for the first time or for the first few times. With practice in conducting the training most organizations will develop their own adaptations and ramifications of this plan to meet their own requirements and the specific situation.

It is strongly urged that each institution conducting this training course develop its own formal version of such a guide as this. Staff does change; new personnel require indoctrination and training on standard procedures of the organization they have joined. Rules and procedures promulgated by regulatory authority do change; such changes will require adjustment in course content. With repetition of specified tasks, many staff members, however dedicated, do slip into unconscious variations in practice or omissions in details which can subtly change the character of the course. A formal plan and guide, consciously studied and followed, can help the training instructor and the administrator to avoid many pitfalls.

Intelligent use of a plan such as this, with variations as proven necessary, can go far in keeping this course in harmony with the needs of the students and with requirements of regulatory authorities. Above all, the course should remain free of the distractions and last-minute corrections of errors or omissions discovered at untimely stages in course preparation and presentation.
PART I - COURSE PLANNING AND MANAGEMENT

A. Course Plan and Working Schedules

This section considers five topics:

1. Responsibilities in self-monitoring training

It is an inherent responsibility of any teacher to provide a learning situation which gives the student the best possible opportunity to develop the target level of knowledge and skills.

In addition, and not to be overlooked, this course involves a responsibility to the regulatory agency—to assure that students acquire specified knowledge and skills and that these skills are acquired to a level such that data reported by the student will be accepted as reliable by the regulatory authority.

Announcing the course

This section gives examples of course announcements, and identifies specific information which should be provided in any course announcement.

Summary plan for the course

This is a one-page summary of the course plan in which the reader can discover the subject matter coverage, days and approximate time allocations and the designation of the instructional specialty involved in presenting the instruction.

Sample course schedule

This covers the same information as the summary plan. The format is different, providing a day-by-day, hour-by-hour class schedule. This schedule format has been found most practical at EPA training centers for more than 20 years. It works. It is recommended for your use.

Milestones in course planning and preparation

Each member of the training staff has individual and cooperative duties in planning and conducting the course. Much of the responsibility of each staff member is outlined later in this Guide in the section on Training Staff (Outline 6). The milestone chart shown here is an example of a plan to accomplish necessary tasks in a timely manner. It is necessary that each training institution develop its own logistics of course preparation to meet the situation at hand. It is urged that a formal milestone chart be developed.

1. Responsibilities in Self-Monitoring Training

   a. Implications of NPDES

Each Permit issued under the National Pollutant Discharge Elimination System (NPDES) includes a program of required self-monitoring analyses of effluents and reporting of results at prescribed intervals.

AT. EMP. (164.3) 2. 10. 76
1) The methodology to be followed in performing the self-monitoring tests and measurements is described in issuances in the Federal Register (FR).

2) In some cases, two or more alternative procedures are available to the analyst for compliance with monitoring requirements.

3) Provisions do exist whereby the regulatory agencies (State and EPA) can recommend and permit use of methods not listed in the FR. Procedures for orderly application of accepted methods are described in the FR issuance at the end of this section. It cannot be too strongly emphasized that the Permit-holder does not have the authority to make a unilateral decision to introduce analytical procedures not found in the FR issuances or not specifically authorized by the appropriate regulatory authorities.

b. Training Response to NPDES/FR Requirements

1) Methods taught in this and associated courses developed by EPA are limited to those most recently prescribed in issuances of the FR.

2) Usually, when alternative methods are available, the course will include only one of the alternatives. In planning the details of a specific course offering, a training representative should in every case consult with responsible representatives of the regulatory agency in the area in which the course is conducted. This consultation should include determination of:

   a) Which, if any, of more than one alternative analytical method is preferred (or required) by the regulatory agency for the Permit-holders under its jurisdiction?

   b) What, if any, changes have occurred since the last course offerings which require adjustment in course content?

   c) What advice does the regulatory agency have to offer about the planned course presentation based on problems encountered in implementation of self-monitoring procedures, and in observations of performance of past graduates from previous offerings of the course?

3) The training institution which fails to apply these elementary practices, with a result of sometimes teaching inappropriate methods, stands in danger of committing a serious disservice to those it purports to serve:

   a) To the students enrolled in the course; and

   b) To the Permit-holding organization, which is being led to believe that through this training the qualifying student will perform self-monitoring tests and measurements in accordance with methods approved by the regulatory authority.
c. Responsibilities in Accreditation of Students

1) Successful completion of this course of training will be a factor used by many regulatory agencies in the accreditation or certification of treatment plant personnel to perform self-monitoring tests and measurements.

2) The instructional staff has a responsibility to provide a learning experience through which the qualifying student will have every expectation of being able to perform the required self-monitoring procedures in a satisfactory manner on return to his normal working environment.

3) Student Evaluation and Testing

For permanent record or qualification and performance, a record of student performance should be made.

a) Tests (both written and applied) should be criterion tests, i.e., designed to demonstrate ability of the student to perform the required analyses, as contrasted with tests designed to develop a comparative rating of the individual students.

b) Written tests should be strictly applied to the procedures being taught and should be appropriate to a written answer. Thus, definitions, matters of specific information, solving of problems, and similar matters are appropriate to written tests.

c) Many students will take alarm at written tests, and may not perform up to expectations due to this alarm. One means of alleviating this fear is to give open-book quizzes but to expect a high standard of performance. It is pointed out here that it is more important for the student to know where to refer for needed facts and to check these facts, than it is to demonstrate rote memory. The memory will come with practice of the test procedures. The student should learn to check the facts when in doubt, rather than to risk a blunder.

d) Much of the student evaluation will be based on performance in the laboratory. It will be appropriate to keep records on such factors in student performance as:

(1) Accuracy in following directions as given;

(2) Demonstration of acceptable manipulative skills;

(3) Reporting of laboratory results falling within acceptable relationship to a class norm; and other factors as determined by the instructor.

e) Each instructor is responsible for reporting the quality of performance of each student for the procedures for which he has primary responsibility. These reports should be written, and they should be made a part of the permanent course record. This course plan provides for having at least two instructors in the laboratory during all laboratory phases of the course. At any
given session, one instructor will be the designated primary instructor, with others in a supporting role. To make a system like this work satisfactorily in evaluation of students and to assure consistence in student instruction each member of the instructional team has definite, if implied, responsibilities. These include:

(1) The primary instructor is the individual who reports on acceptability or non-acceptability of student performance.

(2) The instructor-assistant(s) must instruct students in the same way as designated by the primary instructor. Introduction of individuality in manipulative procedures, order of proceeding with a test, etc., can only confuse the student.

(3) The instructor-assistant(s) should bring the problem-student to the attention of the primary instructor at the earliest possible moment. This is to provide maximum opportunity to correct problems which might result in failure of the student to qualify in a given analytical procedure.

(4) Primary instructor and instructor-assistants have, of course, a continuing responsibility of preserving the highest standards of professional and ethical relationships with each other throughout the course. Differences of opinion will arise among individuals of any instructional staff. These differences must be resolved outside the classroom and laboratory. Furthermore, these differences are not subjects for airing with students at any time.

4) The Nonqualifying Student

a) There is a job/financial implication to any student failing to qualify for any of the analytical tests and measurements included in this course. Therefore instructors should be particularly careful to document the reasons for any nonqualification.

b) Any nonqualifying student should be entitled to be informed on the reasons why he is judged nonqualifying.

c) Ideally, an opportunity should be provided for nonqualifying students to have another chance to correct deficiencies in their performance. This could be accomplished through a number of different approaches such as makeup work (evenings) during the course itself, through repeating the training module in which acceptable results were not achieved (at a mutually agreeable time for the student and for the instructor), or by enrollment in a future offering of the course with participation only in the module which was failed the first time around.
PART II:

ENVIRONMENTAL PROTECTION AGENCY

WATER PROGRAMS

Guidelines Establishing Test Procedures for the Analysis of Pollutants

Amendments
Title 40—Protection of Environment
CHAPTER I—ENVIRONMENTAL PROTECTION AGENCY
SUBCHAPTER D—WATER PROGRAMS
[FRL 530-4]
PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS
Amendment of Regulations
On June 9, 1975, proposed amendments to the Guidelines Establishing Test Procedures for the Analysis of Pollutants (40 CFR 136) were published in the Federal Register. This proposed amendment is an outgrowth of an Executive Order directing Federal agencies to improve the controls for the subvention of pollutants. It is the purpose of this proposal to provide a uniform test method for the analysis of pollutants that shall be consistent with the controls for the discharges of pollutants.

(1) In response to three requests from Federal, State, and local governments, and dischargers, “hardness,” may be measured as the sum of calcium and magnesium, analyzed by atomic absorption and x-ray fluorescence.

(2) The proposal to limit measurement of fecal coliform bacteria in the presence of chlorine to only the “Most Probable Number” (MPN) procedure has been withdrawn in response to a coordinated effort from forty-five commenters including State pollution control agencies, permit holders, analysts, treatment plant operators, and a manufacturer of analytical equipment. The membrane filter (MF) procedure will continue to be an approved technique for the routine measurement of fecal coliform in the presence of chlorine. However, the MF procedure must be used to report on the data.

(3) A total of fifteen objections, representing the entire spectrum of commenters, addressed the drying temperatures used for measurement of residues. The use of different temperatures in drying of total residue, dissolved residue, and residue with water adjustment has been withdrawn in response to requests from forty-five commenters including State pollution control agencies, major dischargers, and analytical professionals.

(4) The clarification of test parameters. Some measurement techniques that had been proposed have been deleted and new techniques have been proposed. The six comments received were carefully evaluated and based upon the review of comments, the following principal changes to the proposed regulations were made:

(A) Definitions: Section 136.2 has been amended to update references. Twenty-five commenters representing the entire spectrum of responding groups pointed out that the definition of “Residual Chlorine” was not consistent with the 1974 EPA Manual of Methods for the Analysis of Water and Waste Water. The definition has been changed to “Residual Chlorine” to mean chlorine, free and combined as free, inorganic, and organic chlorine, including chloramines, hypochlorites, and hypochlorous acid, as determined by a suitable method approved by the Administrator after due consideration of the comments received. The revised definition is given in a footnote of Table I.

(B) Identification of Test Procedures: Both the content and format of Table 136.3, “Table I. List of Approved Test Procedures” have been revised in response to twenty-one comments received from State and local governments, major dischargers, professional and trade associations, and analytical laboratories. Table I has been revised by:

(1) The addition of a fourth column of references for procedures which are equivalent to previously approved methods.

(2) The addition of a fifth column of references for procedures which are equivalent to previously approved methods.

(3) Listing generically related parameters alphabetically within four subcategories: bacteria, metals, radiological and residue, and by listing these subcategory headings in alphabetic sequence relative to the remaining parameters.

(4) Deleting the parameter “Alginic Acid” and by entering the single relevant algicid, “Pentachlorophenol” by its chemical name.

(C) Clarification of Test Parameters. The conditions for analysis of several parameters have been more specifically defined. The result of comments received by the Agency:

(1) In response to five commenters representing State or local governments, major dischargers, or analytical laboratories, the end point for the alkalinity determination is specifically designated as pH 5.5.

(2) Manual digestion and distillation are still required as necessary preliminary steps for the Kjeldahl nitrogen procedure. Analysis after such distillation may be by Nessler color comparison, titration, electrode, or automated phenolate procedures.

(3) In response to eight commenters representing State or local governments, major dischargers, or analytical laboratories, the parameters have been more specifically defined. The result of comments received by the Agency:

(1) In response to comments from forty-five commenters, the method for measurement of chlorides has been withdrawn in response to requests from State pollution control agencies, permit holders, analysts, tax relief plant operators, and a manufacturer of analytical equipment. The membrane filter (MF) procedure has been replaced by a new procedure that will continue to be an approved technique for the routine measurement of chlorides. However, the MF procedure must be used to report on the data.

(2) The proposed infrared spectroscopy technique has been withdrawn. The measurement of alcohols and ketones has been withdrawn in response to comments from forty-five commenters including State pollution control agencies, permit holders, analysts, treatment plant operators, and a manufacturer of analytical equipment. The membrane filter (MF) procedure has been replaced by a new procedure that will continue to be an approved technique for the routine measurement of alcohols and ketones. However, the MF procedure must be used to report on the data.

(3) The proposed infrared spectroscopy technique has been withdrawn. The measurement of alcohols and ketones has been withdrawn in response to comments from State pollution control agencies, permit holders, analysts, treatment plant operators, and a manufacturer of analytical equipment. The membrane filter (MF) procedure has been replaced by a new procedure that will continue to be an approved technique for the routine measurement of alcohols and ketones. However, the MF procedure must be used to report on the data.

(D) New Parameters and Analytical Procedures. Forty-four new parameters have been added to Table I. In addition to the designation of analytical procedures, the following modifications have been made in analytical procedures designated in response to comments:

(1) The ortho-tolidine procedure was not approved for the measurement of residual chloride because of its poor accuracy and precision. Its approval had been requested by seven commenters representing State or local governments, major dischargers, or analytical laboratories. The method has been withdrawn in response to comments from State pollution control agencies, major dischargers, and analytical professionals.

(2) The Environmental Protection Agency concurred with the American Dyestuff Manufacturers Association in approving its procedure for measurement of color and the procedure is now available at the Atmospheric Monitoring and
Support Laboratory, Cincinnati (EMSL-CL).

(F) Sample Preservation and Holding Times. Criteria for sample preservation and sample holding times were also deleted in response to comments. Reference to sample preservation and holding time criteria applicable to the Table I parameters is given in footnote (1) of Table I.

(G) Alternate Test Procedures. Comments pertaining to Table 136.4, Application for Alternate Test Procedures, included objections to various obstacles within the proposed methodology.
these procedures for expedient approval of alternate test procedures. Four analytical instrument manufacturers commented that by limiting of application for review and/or approval of alternate test procedures to NPDES permit holders, the EPA placed an impediment to the commercial development of new or improved measurement devices based on new measurement principles. Applications for such review and/or approval were not be accepted from any person not a NPDES permit holder. The intent of the alternate test procedure is to allow the use of measurement systems which are known to be equivalent to the approved test procedure for wastewater discharges.

Applications for approval of alternate test procedures applicable to specific discharges will continue to be made only by NPDES permit holders, and approval of an alternate test procedure will be made on a case-by-case basis by the Regional Administrator in whose Region the discharge is made.

Applications for approval of alternate test procedures which are intended for nationwide use can now be submitted by any person directly to the Director of the Environmental Monitoring and Support Laboratory in Cincinnati. Such applications should include a complete methods write-up, any literature references, comparability data between the proposed alternate test procedure and those already approved, and all other information necessary for approval. The Regional Administrator should include precision and accuracy data of the proposed alternate test procedure and data confirming the general applicability of the test procedure to wastewater categories for which it is intended. The Director of the Environmental Monitoring and Support Laboratory, after review of the submitted information, will recommend approval or rejection of the application to the Administrator, or he will return the application to the applicant for more information. Approval or rejection of applications for test procedures intended for nationwide use will be made by the Administrator, after considering the recommendations made by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati. Since the Agency considers these procedures for approval of alternate test procedures for nationwide use to be interim procedures, the Agency will welcome suggestions for criteria approval of alternate test procedures. Interested persons should submit their written comments in triplicate on or before June 1, 1977 to: Dr. Robert S. Medlin, Environmental Protection Technology, Monitoring and Research, Office of Environmental Monitoring and Technical Support (RD-660), Environmental Protection Agency, Washington, D.C. 20460.

For Freedom of Information A copy of all public comments: an analysis by parameter of those comments; and documents providing further information on the rationale for the changes made in the final regulation are available for inspection and copying at the Environmental Protection Agency Public Information Reference Unit, Room 2022.

Waterside Mall, 401 M Street, SW., Washington, D.C. 20460, during normal business hours. The EPA information regulation 40 CFR 3 provides that a reasonable fee may be charged for copying such documents.

Effective date: These amendments become effective on April 1, 1977.

JOHN QUARLES
Acting Administrator, Environmental Protection Agency.

Chapter I, Subchapter D, of Title 40, Code of Federal Regulations is amended as follows:

1. In §136.3, paragraphs (f), (g), and (h) are amended to read as follows:


(h) "EPA Methods" means Methods for Chemical Analysis of Water and Waste, 1974. Methods Development and Quality Assurance Research Laboratory, National Environmental Research Center, Cincinnati, Ohio 45268; U.S. Environmental Protection Agency, Office of Technology Transfer, Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268. This publication is available from the Office of Technology Transfer.

2. In §136.3, the second sentence of paragraph (b) is amended, and a new paragraph (c) is added to read as follows:

§ 136.3 Identification of test procedures.

(b) Under certain circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator or the Director upon the recommendation of the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(c) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring and Support Laboratory, Cincinnati, additional alternate test procedures for nationwide use.

3. Table I of §136.3 is revised by listing the parameters alphabetically; by adding 44 new parameters; by adding a fourth column under references listing equivalents; and by adding 24 new footnotes to read as follows:

<table>
<thead>
<tr>
<th>Table I.—List of approved test procedures</th>
</tr>
</thead>
<tbody>
<tr>
<td>Parameter and unit</td>
</tr>
<tr>
<td>---------------------</td>
</tr>
<tr>
<td>pH</td>
</tr>
<tr>
<td>BOD</td>
</tr>
<tr>
<td>Ammonia (as N), milligrams per liter</td>
</tr>
<tr>
<td>Nitrate</td>
</tr>
<tr>
<td>Nitrite</td>
</tr>
<tr>
<td>Fluoride</td>
</tr>
<tr>
<td>Bacteria</td>
</tr>
</tbody>
</table>

* AAS: American Association for the Advancement of Science

FEDERAL REGISTER, VOL. 41, NO. 233—WEDNESDAY, DECEMBER 1, 1976
<table>
<thead>
<tr>
<th>Parameter and units</th>
<th>Method</th>
<th>EPA standard methods</th>
<th>Inter agency methods</th>
<th>Other approved methods</th>
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<tr>
<td>Cobalt—Total, milligrams per liter</td>
<td>Digestion followed by atomic absorption</td>
<td>107 142 346 90 (287)</td>
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<tr>
<td>Cobalt—Dissolved, milligrams per liter</td>
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<td>106 142 345 233 (419) (287)</td>
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<td>Copper—Total, milligrams per liter</td>
<td>Digestion followed by atomic absorption or by colorimetric (Nessler's)</td>
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<td>Mercury—Total, milligrams per liter</td>
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<td>Manganese—Total, milligrams per liter</td>
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<td>Palladium—Total, milligrams per liter</td>
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<td>Platinum—Total, milligrams per liter</td>
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<td>Rhodium—Total, milligrams per liter</td>
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<td>Ruthenium—Total, milligrams per liter</td>
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<td>Selenium—Total, milligrams per liter</td>
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<td>146 148 349 115 (419) (287)</td>
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<td>Silver—Total, milligrams per liter</td>
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See footnotes at end of table
### RULES AND REGULATIONS

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<td>14th ed (page nos.)</td>
<td>Pt. 31, USGS 1973</td>
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<td>Condensation—Evaporation</td>
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<td>Nitrate, as NO₃, milligrams per liter</td>
<td>Condensation—Evaporation</td>
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<tr>
<td>Nitrite, as NO₂, milligrams per liter</td>
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<td>Oil and grease, milligrams per liter</td>
<td>Liquid—Aqueous extraction with trichloroacetic acid</td>
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<td>Organic arsenic, total, arsenic, milligrams per liter</td>
<td>Graphite furnace atomic absorption</td>
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<td>Organic phosphorus, total, phosphorus, milligrams per liter</td>
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<td>Arsenic, as As, milligrams per liter</td>
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<td>Phosphorus, elemental, milligrams per liter</td>
<td>Liquid—Aqueous extraction</td>
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<td>Phosphorus, as P, milligrams per liter</td>
<td>Graphite furnace atomic absorption</td>
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<tr>
<td>Specific conductance, microsiemens per centimeter at 25°C</td>
<td>Winkler end point titrations</td>
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<td>350</td>
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<tr>
<td>Nitrate, as NO₃, milligrams per liter</td>
<td>Gravimetric, 105 to 106°C</td>
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<tr>
<td>Nitrates, dissolved, as NO₃, milligrams per liter</td>
<td>Gravimetric, 105 to 106°C</td>
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<tr>
<td>Total suspended (undissolved), milligrams per liter</td>
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<td>Total soluble, milligrams per liter</td>
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<td>Oxygen, as O₂, milligrams per liter</td>
<td>Winkler end point titrations</td>
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<tr>
<td>Total dissolved, as NO₃, milligrams per liter</td>
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<tr>
<td>Specific conductance, microsiemens per centimeter at 25°C</td>
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</table>

### RECOMMENDATIONS
Recommendations for sampling and preservation of samples according to parameter measured may be found in Method No. 119: Methods for Chemical Analysis of Water and Waste, 1974 (U.S. Environmental Protection Agency, table 3, pp. 83-85).
In §136.5, paragraph (a) is amended by inserting the phrase "proposed by the responsible person or firm making the discharge" immediately after the words "discharge" immediately after the period that ends the paragraph.

In §136.5, paragraph (b) is amended by inserting in the first sentence 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 "Methodology Development and Quality Assurance Research Laboratory" immediately after the phrase "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, Ohio 45268."
§ 136.5 Approval of alternate test procedure.

(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 76-35092 Filed 11-30-76; 8:45 am]
2. Announcing the Course

a. Course Availability

Course availability is most likely to be established through one of two mechanisms:

1) Management or regulatory authority determines that training is required, makes arrangements for course presentation and instructs designated personnel to appear at a specified time and place for training; or,

2) The course may be planned by a training organization which schedules and publicly announces the course either as a special offering or as an element of an overall curriculum of training.

b. Responsibility for Course Announcement

All training organizations should establish and maintain mailing lists of officials, organizations and interested individuals to whom training announcements should be addressed.

Course announcements should be released by the training organization and/or the sponsoring agency (if applicable).

When a special course offering is planned at the request of management or regulatory authority with identified class participants, it usually is best for the requesting authority to make the announcement.

c. Types of Course Announcements

1) Training bulletins, or catalogues are widely used by established training organizations, and should be used for announcement of this course when offered as part of an on-going curriculum of courses.

2) Special fliers or brochures should be developed for public announcement through established mailing lists. These releases may be used for regular offerings of an on-going curriculum of courses; but they are particularly applicable when a special offering of the course is planned.

3) The course may be announced in a journal, newsletter or other periodical widely read by the personnel for whom the training is intended.

4) The course may be announced by personal letter or other direct communication with a student assigned to take the training.

d. Timing of Course Announcements

Training catalogues or bulletins usually are for a period of one year or more. Accordingly, the prospective student should have from three months to one year of advance notice of the training.
When the course is a special offering announced through a flier or other special mechanism, at least 90 days should be provided between the release of the announcement and the start of the course. There are at least two reasons for this:

1) Permits course applicants to secure necessary approvals for attendance, and to make personal scheduling arrangements; and,

2) Provides course presentation staff with lead time for course preparations, acquiring special instructional materials, preparation of laboratory supplies and equipment, and related tasks.

e. Information Provided in Course Announcements

The following list should be helpful as a checklist to those preparing a course announcement. Samples of an announcement for this course as it might appear in a catalogue of courses, and as it might appear in a special flier are shown in the section of this Guide titled SECRETARIAL SUPPORT.

In the event that the course is announced in a periodical, the editor may apply constraints on style and format which make it impossible to provide all the pertinent information on the course. In such cases the announcement must provide the name and address of an office from which further information can be obtained. The information to be provided should be as complete as that given in a course catalogue or flier and, naturally, should include any additional special information specifically requested.

The following will be helpful as a checklist to those preparing an original course announcement:

1) Course title, dates and location

2) Name of organization conducting the course (and name of co-sponsor, if applicable)

3) Description of intended student body, reason why this training is needed and summary of Course content

4) Prerequisites for attendance (special skills or training which the applicant must have for admission)

5) Description of the training environment to be used (classroom, laboratory, field, in-plant, etc.)

6) Identification of knowledge and skills the participant will have on satisfactory completion of training

7) Tuition (if applicable)

8) How and where to apply for admission to this course
3. Summary Plan for the Course

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 authors 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. Then other considerations should be applied. Some examples are:

a. 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.

b. 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.

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

d. If most students need only 80% of the procedures taught, schedule the remaining 20% of the procedures as a group so students can conveniently schedule their attendance for training pertinent to their needs.
## ESTIMATED AGENDA FOR COURSE 164.3

<table>
<thead>
<tr>
<th>MONDAY</th>
<th>Time Hours</th>
<th>TUESDAY</th>
<th>Time Hours</th>
<th>WEDNESDAY</th>
<th>Time Hours</th>
<th>THURSDAY</th>
<th>Time Hours</th>
<th>FRIDAY</th>
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<td>Evaluation</td>
<td>1/2</td>
<td>Evaluation TKN</td>
<td>1/2</td>
<td>Titrations</td>
<td>1/2</td>
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<td>1/4</td>
<td>Total Nitrogen and Organic Nitrogen</td>
<td>3-1/2</td>
<td>Evaluation NH₃</td>
<td>1/2</td>
<td>Chemical Oxygen Demand</td>
<td>3-1/2</td>
<td>Reporting Data</td>
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<td>Nitrate and Nitrite</td>
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<td>Oil and Grease</td>
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<td>Nitrate and Nitrite</td>
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<td>Phosphorus</td>
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<td>Ammonia</td>
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<td>8</td>
<td>TOTAL</td>
<td>9</td>
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### NOTES:
1. Teaching time is available Monday. Thursday is a long day.
2. Phosphorus glassware requires special cleaning and is used for other tests. Schedule this earlier in week.
3. Use of Spec 20 and Calibration Curves must be taught before Total Nitrogen, Nitrate and Nitrite, and Phosphorus.
4. COD can be taught in two segments, oxidation in the afternoon, titrations the next morning.
5. Keep Oil and Grease on Friday. A different test could be taught then if local needs suggest it.
### 4. Sample Course Schedule

**Effluent Monitoring Procedures:**
**Nutrients (164.3)**

(Location)  
(Date)

Course Coordinator:

<table>
<thead>
<tr>
<th>DAY AND TIME</th>
<th>SUBJECT</th>
<th>OUTLINE</th>
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<tr>
<td>9:30 - 8:50</td>
<td>Registration - Introductions</td>
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<td>8:50 - 9:30</td>
<td>NPDES Requirements</td>
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<td>Course Coordinator</td>
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<td>EMP Format</td>
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<td>9:30 - 9:45</td>
<td>Laboratory &amp; Safety Orientation</td>
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<td>Chemist #1</td>
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<tr>
<td>9:45 - 11:15</td>
<td>Use of a Spectrophotometer</td>
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<td>Chemist #2</td>
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<td>(Chemist #1)</td>
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<td>11:15 - 12:15</td>
<td>Preparation of Calibration Graphs</td>
<td>2</td>
<td>Chemist #2</td>
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<td>(Chemist #1)</td>
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<td>12:15 - 1:15</td>
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<tr>
<td>1:15 - 2:00</td>
<td>Determination of Total Phosphorus</td>
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<td>(Chemist #2)</td>
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<td>2:00 - 3:30</td>
<td>Determination of Total Phosphorus: Glassware Preparation</td>
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<td>3:30 - 5:00</td>
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<td>Chemist #3</td>
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<td>1:00 - 1:45</td>
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<td>(Chemist #2)</td>
</tr>
<tr>
<td>1:45 - 4:30</td>
<td>Determination of Chemical Oxygen Demand</td>
<td>4</td>
<td>Chemist #1</td>
</tr>
<tr>
<td></td>
<td>(Chemist #3)</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Wednesday</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8:30 - 10:00</td>
<td>Determination of Chemical Oxygen Demand</td>
<td>4</td>
<td>Chemist #1</td>
</tr>
<tr>
<td></td>
<td>(Chemist #3)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10:00 - 10:15</td>
<td>Break</td>
<td></td>
<td>Chemist #2</td>
</tr>
<tr>
<td>10:15 - 12:00</td>
<td>Determination of Total Kjeldahl Nitrogen</td>
<td>5</td>
<td>(Chemist #1)</td>
</tr>
</tbody>
</table>

AT. EMP. (164.3). 5.10.76
<table>
<thead>
<tr>
<th>DAY AND TIME</th>
<th>SUBJECT</th>
<th>OUTLINE</th>
<th>INSTRUCTOR*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monday</td>
<td></td>
<td></td>
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<tr>
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<tr>
<td>Wednesday (Continued)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12:00 - 1:00</td>
<td>Lunch</td>
<td></td>
<td>Chemist #2</td>
</tr>
<tr>
<td>1:00 - 3:00</td>
<td></td>
<td></td>
<td>(Chemist #1)</td>
</tr>
<tr>
<td>3:00 - 5:00</td>
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<td>Chemist #3</td>
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<td></td>
<td></td>
<td></td>
<td>(Chemist #2)</td>
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<tr>
<td>Thursday</td>
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<tr>
<td>8:00 - 8:15</td>
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<td></td>
<td>Chemist #1</td>
</tr>
<tr>
<td>8:15 - 12:15</td>
<td></td>
<td></td>
<td>(Chemist #3)</td>
</tr>
<tr>
<td>12:15 - 1:15</td>
<td>Lunch</td>
<td></td>
<td>Chemist #1</td>
</tr>
<tr>
<td>1:15 - 5:00</td>
<td></td>
<td></td>
<td>(Chemist #3)</td>
</tr>
<tr>
<td>Friday</td>
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<td></td>
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<tr>
<td>8:30 - 11:30</td>
<td></td>
<td></td>
<td>Chemist #2</td>
</tr>
<tr>
<td>11:30 - 12:15</td>
<td></td>
<td></td>
<td>(Chemist #1)</td>
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<tr>
<td>12:15 - 12:30</td>
<td></td>
<td></td>
<td>(Chemist #2)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Course Coordinator</td>
</tr>
</tbody>
</table>

*NOTE: For actual course presentation, insert the name of the instructor instead of the technical specialty, as shown.
5. 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. The Staff Guide (Chapter) number of the related outline is given for each listing so that the user can quickly find the details about his/her assignments.

The table headings are job titles associated with the listed tasks. A maximum 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.

About three months before the course, decisions must be made regarding specific procedures to be taught, because several of the Effluent Monitoring Procedures (EMPs) contain a choice of procedures.

The ammonia EMP is written for the distillation of a macro sample followed by either Nesslerization or titration. The Kjeldahl Nitrogen EMP contains a choice between macro or micro digestion and distillation followed by either Nesslerization or titration. There is also an EMP for determining ammonia by using a selective ion electrode. The Chemical Oxygen Demand EMP is written for the low level range of 5-50 mg/liter, but includes the modifications for concentrations greater than 50 mg/liter COD. The Phosphorus EMP can be used to determine total phosphorus or only orthophosphate. These different possibilities affect the equipment preparations needed prior to the course.

The requirements of area NPDES permits and/or the type of equipment readily available will help the course planners make choices among these possibilities.
<table>
<thead>
<tr>
<th>Time Frame</th>
<th>Task</th>
<th>Staff</th>
<th>Course Coordinator</th>
<th>Chemist #1</th>
<th>Chemist #2</th>
<th>Chemist #3</th>
<th>Lab Assistant</th>
</tr>
</thead>
<tbody>
<tr>
<td>5 TO 6 MONTHS BEFORE COURSE</td>
<td>Determination of the need and decision to have course.</td>
<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td>Designation of Course Director and Course Secretary.</td>
<td></td>
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<tr>
<td></td>
<td>Review responsibilities.</td>
<td></td>
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<tr>
<td></td>
<td>Review responsibilities:</td>
<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td>Commit classroom and laboratory facilities.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
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</tr>
<tr>
<td></td>
<td>Develop and release Course Announcement including location, date,</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
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</tr>
<tr>
<td></td>
<td>general statement of course content and training objectives.</td>
<td></td>
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</tr>
<tr>
<td></td>
<td>Prepare all forms and information sheets related to student registration procedures.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
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</tr>
<tr>
<td></td>
<td>Decide on staff members.</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>4 TO 5 MONTHS BEFORE COURSE</td>
<td>Receive, review, act upon Course Applications, continuing until course begins.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
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</tr>
<tr>
<td></td>
<td>Maintain records on deposition of each application, continuing</td>
<td></td>
<td></td>
<td>x</td>
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<tr>
<td></td>
<td>through course.</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Inventory Staff Guides, Order needs.</td>
<td></td>
<td></td>
<td>x</td>
<td>x</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

PREFACE, 1 thru 12
PREFACE, 1, 6, 10, 11, 12
7
10, 11, 12
6
10, 11, 12
9
# Staff Guide Outline No. 3

## 3 MONTHS BEFORE COURSE

<table>
<thead>
<tr>
<th>Task</th>
<th>Supervisor</th>
<th>Coordinator</th>
<th>Secretary</th>
<th>Chemist #1</th>
<th>Chemist #2</th>
<th>Lab Assistant</th>
<th>Staff Guide</th>
</tr>
</thead>
<tbody>
<tr>
<td>Commit all staff members who will participate in course.</td>
<td>x x</td>
<td>x x x x x x</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
<td>6</td>
</tr>
<tr>
<td>Develop Milestone Checklist for Course.</td>
<td>x x</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>5</td>
</tr>
<tr>
<td>Distribute copies to staff of Milestones, Staff Guide, Student Text and any other pertinent training resources.</td>
<td>x x x</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>5, 9</td>
</tr>
<tr>
<td>Review responsibilities.</td>
<td>x x x</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>PREFACE, 1, 6, 10, 12, 13</td>
</tr>
<tr>
<td>Assign topics to Primary (P) and Assistant (A) Instructors:</td>
<td>x x</td>
<td>A P</td>
<td>A P A P A</td>
<td></td>
<td></td>
<td></td>
<td>14</td>
</tr>
<tr>
<td>Spectrophotometer</td>
<td></td>
<td></td>
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<td>15</td>
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<tr>
<td>Calibration Graphs</td>
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<td></td>
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<td></td>
<td></td>
<td>16</td>
</tr>
<tr>
<td>Phosphorus</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>17</td>
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<tr>
<td>Chemical Oxygen Demand</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>18</td>
</tr>
<tr>
<td>Kjeldahl Nitrogen</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>19</td>
</tr>
<tr>
<td>Ammonia</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>20</td>
</tr>
<tr>
<td>Ammonia Electrode</td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>21</td>
</tr>
<tr>
<td>Nitrate-Nitrite (Cd)</td>
<td></td>
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</tr>
<tr>
<td>Oil and Grease</td>
<td></td>
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</tr>
<tr>
<td>Decide which procedures to teach if EMP has choices.</td>
<td>x x</td>
<td>x x x x x x</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
<td>8 and 13 thru 22</td>
</tr>
<tr>
<td>Record on related IPW.</td>
<td></td>
<td></td>
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<td></td>
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</tr>
<tr>
<td>Mark which procedures are to be taught on the summary of laboratory equipment and supply needs for course.</td>
<td>x x x</td>
<td>x x x x x x</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
<td>8</td>
</tr>
<tr>
<td>Develop summary plan for course.</td>
<td>x x x x x x</td>
<td>x x x x x x</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>3</td>
</tr>
<tr>
<td>Inventory chemicals and laboratory equipment/supplies.</td>
<td>x x</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>8</td>
</tr>
<tr>
<td>List and commit lending sources.</td>
<td>x x x x x x</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Order rest of needs.</td>
<td></td>
<td></td>
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<td></td>
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</tr>
<tr>
<td>Inventory classroom equipment/supplies. List and commit lending sources. Order rest of needs.</td>
<td>x x</td>
<td></td>
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<td>7</td>
</tr>
<tr>
<td>(Continued)</td>
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</tbody>
</table>

(Continued)
<table>
<thead>
<tr>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Course Secretary</th>
<th>Chemist #1</th>
<th>Chemist #2</th>
<th>Chemist #3</th>
<th>Lab Assistant</th>
<th>Staff Guide Outline No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
</tr>
</tbody>
</table>

### 2 MONTHS BEFORE COURSE

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

### 6 WEEKS BEFORE COURSE

- **Check out operation of all items listed as "A. Capital Equipment" plus COD reflux apparatus.**
- **Primary and Assistant Instructors go through EMP laboratory procedures in student reference texts, using IPWs to standardize instructions for students.**

### 1 MONTH BEFORE COURSE

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

(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. (Special for Phosphorus, COD, Ammonia, distillation apparatus)

Reserve all specially-cleaned glassware.

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 effluent samples.

Determine range of concentration of desired constituent in effluent sample from source of course samples.

Arrange for disposal of special test wastes (COD, alkaline wastes, Cd)
<table>
<thead>
<tr>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Chemist #1</th>
<th>Chemist #2</th>
<th>Chemist #3</th>
<th>Lab Assistant</th>
</tr>
</thead>
<tbody>
<tr>
<td>Give Laboratory Assistant final list of equipment and supplies to be at each laboratory position. Discuss arrangement of shared equipment.</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>13 thru 22</td>
</tr>
</tbody>
</table>

**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 texts, 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>Task</th>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Course Secretary</th>
<th>Chemist #1</th>
<th>Chemist #2</th>
<th>Chemist #3</th>
<th>Lab Assistant</th>
<th>Staff Guide</th>
<th>Outline No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prepare course certificates and give to Day's Instructor.</td>
<td>x</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>10, 12</td>
</tr>
<tr>
<td>EVERY DAY OF COURSE</td>
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<tr>
<td>Maintain general supervision of course.</td>
<td>x</td>
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<td></td>
<td>6</td>
</tr>
<tr>
<td>Prepare unstable reagents and/or samples on day of test.</td>
<td>x x x x x</td>
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<td></td>
<td>8, 16, 18, 19, 20, 22</td>
</tr>
<tr>
<td>Obtain effluent samples for each test on day of test.</td>
<td>/ x x x x</td>
<td></td>
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<td></td>
<td></td>
<td>8, 16, 17, 18, 19, 21</td>
</tr>
<tr>
<td>When assistant instructor, make any student evaluation records requested by the lead instructor.</td>
<td>x x x</td>
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<td></td>
<td>1, 6, 10, 13 thru 22</td>
</tr>
<tr>
<td>When primary instructor, compile evaluation record for each student.</td>
<td>x x x</td>
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<td></td>
<td>1, 6, 10, 13 thru 22</td>
</tr>
<tr>
<td>When primary instructor, sign certificate of each student who satisfactorily performs test.</td>
<td>x x x</td>
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<td></td>
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<td>1, 12, 13 thru 22</td>
</tr>
<tr>
<td>After signing certificates, give them to next primary instructor.</td>
<td>x x x</td>
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<td></td>
<td>1, 13 thru 22</td>
</tr>
<tr>
<td>When primary instructor, make arrangements to work with each non-qualifying student.</td>
<td>x x x</td>
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<td></td>
<td></td>
<td>17, 18, 19, 20</td>
</tr>
<tr>
<td>SECOND-LAST DAY OF COURSE</td>
<td>Training Supervisor</td>
<td>Course Coordinator</td>
<td>Course Secretary</td>
<td>Chemist #1</td>
<td>Chemist #2</td>
<td>Chemist #3</td>
<td>Lab Assistant</td>
<td>Staff Guide Outline No.</td>
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<tr>
<td>Distribute course critique sheet to students.</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>LAST DAY OF COURSE</th>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Course Secretary</th>
<th>Chemist #1</th>
<th>Chemist #2</th>
<th>Chemist #3</th>
<th>Lab Assistant</th>
<th>Staff Guide Outline No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Assemble certificates, check for completeness and sign.</td>
<td>x</td>
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<tr>
<td>Collect students' critique sheets.</td>
<td>x</td>
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</tr>
<tr>
<td>Conduct closing exercises and distribute certificates.</td>
<td>x</td>
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</tr>
<tr>
<td>Participate in course closing.</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>6</td>
</tr>
<tr>
<td>Clean up classroom and laboratory.</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>x</td>
<td>6</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>WITHIN A WEEK OF COURSE PRESENTATION</th>
<th>Training Supervisor</th>
<th>Course Coordinator</th>
<th>Course Secretary</th>
<th>Chemist #1</th>
<th>Chemist #2</th>
<th>Chemist #3</th>
<th>Lab Assistant</th>
<th>Staff Guide Outline No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Return or replace any borrowed classroom equipment/supplies.</td>
<td>x</td>
<td>x</td>
<td></td>
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<td></td>
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</tr>
<tr>
<td>Return or replace any borrowed laboratory equipment/supplies.</td>
<td>x</td>
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<td>x</td>
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<tr>
<td>Return or replace any borrowed training aids.</td>
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<td>x</td>
<td>x</td>
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<td>Order repairs or replacements of own equipment used in course.</td>
<td>x</td>
<td>x</td>
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<td>x</td>
<td>x</td>
<td>x</td>
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<td>7, 8, 9</td>
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<td>File evaluation records on all student in predetermined area.</td>
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<td>Course Coordinator</td>
<td>Course Secretary</td>
<td>Chemist #1</td>
<td>Chemist #2</td>
<td>Chemist #3</td>
<td>Lab Assistant</td>
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<td>10, 11, 12</td>
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</table>

- Participate in staff session on evaluation of course and recommendations for future offerings.
- Prepare course summary/evaluation report.
- Complete and file entire course records in mutually determined area.
PART I - COURSE PLANNING AND MANAGEMENT

B. Training Resources

This section considers four topics: Training Staff, Training Facilities, Laboratory Equipment and Supply Requirements, and Instructional Resources.

Staffing plans, facilities, equipment and supplies described in this Guide are based on a class of 18 students. For at least the first several (3 or 4) course offerings, it is urged that this maximum number of students per class be strictly adhered to.

A staff experienced in presentation of this course may be able to increase class size to, say, 24 students. On no account should this laboratory-oriented course exceed 24 students with an instructional staff of the size described here. Large numbers of students per instructor simply cannot be provided with the individual attention and instruction required for effective training and assurance that they have indeed learned to perform the analyses which are the subject of this course.

If the number of workers requiring this course is quite large, the best course of action is to meet the training need through providing a greater number of course offerings.

The required instructional resources, shown in outline 9, are compiled from the individual instructional package worksheets shown in Part II. Training administrators should coordinate requests for instructional resources to be acquired, through loan or through purchase, from other sources. This outline provides a basis for such coordination.

1. Training Staff

Each member of the training staff for this, as for any short course, is a member of a team. This team can function effectively only through each member's understanding of the training goals to be met and the plans for meeting these goals, through performance of his/her own duties, and through providing mutually supportive activity with other team members for the effective conduct of the course as a whole.

a. Qualifications of Instructional Staff

1) Each should have a thorough knowledge of the subject matter for which he/she has responsibility, including a high order of technical skill in any procedures to be carried out.

2) Each should be able to perform effectively as an instructor, both in the classroom and in the laboratory. This includes ability to make rapid adjustments in the style and technical level of instruction in order to work with students having a varied range of entry-level knowledge, skills, and prior education.

3) Each should be willing to accept a certain rigidity in the choice of analytical procedures to be taught, in accordance with policies...
and formal directives of the applicable regulatory authority (-ies). The basis for, and recommended procedures to be followed, in introducing variations in methods to be taught in tests and measurements of municipal effluents is discussed elsewhere in this Guide.

b. Estimated Time Allocations for Training Staff

Each member of the training staff has specific duties before, during, and after the scheduled course dates. For planning purposes, it is assumed that pre-course activity will begin three months or more, as required, before classroom instruction begins. During this period, the estimated time allocations will permit the phasing-in of work activity for this course with other duties of all personnel. During the course, all instructional staff and laboratory assistant (if used) are fully occupied, and should not be given any other duty assignments. Post-course activities should be completed within one or two weeks after the last day of instruction. In the post-course period, all staff may begin to phase in other duties pending final completion of all details associated with this training effort.

<table>
<thead>
<tr>
<th>Staff Identification</th>
<th>Working Days (estimate)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Before</td>
</tr>
<tr>
<td>Course Administration</td>
<td></td>
</tr>
<tr>
<td>Course Coordinator (ordinarily, this is one of the instructional staff, who is assigned double-duty as coordinator-instructor)</td>
<td>12</td>
</tr>
<tr>
<td>Course Secretary</td>
<td></td>
</tr>
<tr>
<td>Instructional Staff</td>
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</tr>
<tr>
<td>Chemist #1</td>
<td></td>
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<td>Chemist #2</td>
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<tr>
<td>Chemist #3</td>
<td></td>
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<tr>
<td>Laboratory Support</td>
<td></td>
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<tr>
<td>Laboratory Assistant (optional)</td>
<td></td>
</tr>
</tbody>
</table>

C. Duties and Responsibilities of Training Staff

1) Course Coordinator

This individual may be known variously as Course Director, Course Leader, or by some other term suitable to the situation. In most cases the Course Coordinator will function in a dual capacity as one
of the instructional staff for the course. In principle, any one of the instructors could function as Course Coordinator. In the absence of other factors, it may be best to have the individual who bears the lightest teaching load to act as Course Coordinator.

a) Before the course, the Coordinator receives assignment from management to lead the course, after which he/she:

(1) Obtains commitment of the other members of the training team for the course, including Secretary, Instructors, and (if used) Laboratory Assistant;

(2) Determines the composition and geographic origin of the student body to be trained;

(3) Develops a working schedule and specific staff assignments for all significant milestone stages of course preparation and implementation;

(4) Meets with course staff, distributes instructional guides and related training materials, and reviews the preparation plans and day-to-day working schedule; assigns specific topics and time allocation for which each Instructor has prime instructional responsibility and for which each Instructor serves as assistant to prime Instructor;

(5) Identifies which, if any, available options in procedures should be taught for compliance with directives of the governing regulatory authority(ies);

(6) Prepares announcement of the course, and distributes it as appropriate to the potential student body;

(7) Reviews classroom and laboratory facilities, making arrangements for any required repairs or adaptations;

(8) In consultation with Instructors, reviews availability and condition of all equipment, supplies, and other training resources required for the course, and makes timely arrangements for repair and maintenance, reorder, or borrowing of needed items;

(9) Arranges for supplies of student reference texts and associated training materials;

(10) Works with Secretary in registration of students and in all pre-course communications on schedules, local housing and transportation, and other pertinent matters;

(11) If the course is to be conducted in a "field" location, coordinates timely arrangements for staff travel, transportation of equipment and supplies, arrangements for training facilities, local housing and transportation, determination of availability and location of dining facilities, and any other preparations required for course conduct away from normal base location;
(12) Initiates periodic and timely checks with other training team members to assure that their pre-course preparations are on schedule and that preparation of required resources is moving ahead according to plan. Takes action as necessary to identify problems and to expedite solutions as the need arises;

(13) Represents the training team in all formal communications with management, host organizations, students, and with commercial or private sources of equipment and supplies.

b) During the course, the Course Coordinator:

(1) Takes charge of course opening exercises including student registration, welcome and staff introductions. Presents and discusses course objectives;

(2) Maintains general supervision of course, assuring that all activities are kept on schedule; maintains liaison between staff members and other interfacing organizations/individuals as necessary;

(3) Maintains consolidated summary record of student performance based on information provided by other Instructors; with staff determines which students should/should not be recorded as having satisfactorily met training objectives;

(4) Provides Course Secretary with timely information necessary for preparation of course certificates; signs course certificates as representative of the course training staff;

(5) Presides over course closing activities, including award of certificates.

c) After the course, the Course Coordinator:

(1) Reviews and evaluates with the instructional staff all matters considered pertinent to the effective implementation of the course as planned, developing proposals and plans for adjustments as necessary for future offerings of the course;

(2) Orders repair, renovation, and replacement of any equipment or supplies that require such attention;

(3) Coordinates return of any borrowed resources used in the course;

(4) If course was conducted in the field, coordinates repacking and return shipment of all equipment and supplies;

(5) Drafts course summary/evaluation report;

(6) Prepares and forwards any reports required by other supervising, coordinating, or financing authority.
2) Course Secretary

The Course Secretary works under the direct supervision of the Course Coordinator, and prepares or arranges preparation of all formal communications; procurement documents, training materials, and records required for course preparation and implementation. The Course Secretary also provides office support work for the course instructional staff in all matters pertaining to course preparation and implementation.

a) Before the course, the Course Secretary:

(1) Works with the Course Coordinator in identification and preparation of copies of all pertinent course materials for use in planning conferences between Course Coordinator and the instructional staff, including Instructors' copies of working schedules for course preparation, course agenda from preliminary to final draft, staff assignments, Instructors' instructional package worksheets, all student instructional materials and associated data sheets, student evaluation sheets, quizzes, and any other typed or printed material projected for course use;

(2) After-Course Coordinator's conference with instructional staff, and resolution of decision making issues, arranges for printing (or reproduction) and assembly of all materials indicated under (1) above, in a quantity adequate for projected course requirements;

(3) Arranges for printing or reproduction, and distribution of the projected course announcement as directed by Course Coordinator;

(4) Serves as Registrar, maintaining roster and records of students submitting application and accepted for admission to the course, prepares routine response to students announcing acceptance with information as appropriate on course dates and schedule, local "geography" including key addresses, hotel/motel/dining information, local transportation information, and any other information which will simplify personal planning of registered students;

(5) Prepares, or arranges for, all individual student supplies, including registration cards, course manuals, note paper, pencils, name tags (1 for wearing and 1 for use at classroom seating position), course certificates, etc.

(6) Prepares orders or procurement requests for equipment and supplies needed for the course based on specifications provided by the instructional staff;

(7) If the course is a "field" presentation, makes arrangements for shipment of equipment and supplies to course site and return, staff travel schedules and order of tickets, hotel reservations, and associated functions;
(8) Prepares the classroom for use in the course, including
distribution of individual student materials to seating
positions, arrangements for classroom organization of
audiovisual projection or playback equipment, chalkboards
and associated supplies, and other classroom needs.
(Ordinarily, the Course Secretary does not travel to a
"field" presentation; this function will have to be pro-
vided through special arrangements with the host organization
at the course site.)

b) During the course, the Course Secretary functions as an
"unofficial staff hostess," and:

1. Attends course opening exercises, assisting students in
   completion of registration cards and associated records;
2. Prepares course summary registration information, prepares
   class roster on first day of course and distributes copies
   to students and instructors, keeping copies for future
   records;
3. Provides clerical/secretarial support to Course Coordinat-
   tor and instructional staff as required;
4. Receives incoming mail and messages for staff and students,
   expediting communications to extent practical and feasible;
5. Prepares course certificates as specified by Course Coordinator
   on last day of course;
6. Inspects classroom daily, making arrangements as necessary
   for coordination of deficiencies in janitorial services,
   and personally corrects minor deficiencies to extent feasible;
   Attends and participates in course closing exercises.

C) After the course, the Course Secretary:

1. Prepares typed copy of all reports drafted by the Course
   Coordinator, and forwards reports as indicated;
2. Places purchase orders or procurement requests for repair,
   renovation, or replacement of equipment and supplies as
   directed by Course Coordinator;
3. Removes all course supplies from the classroom, returns
   borrowed resources, leaves classroom in condition suitable
   for use by following class (this does not imply janitorial
   services);
4. If course is conducted on a repetitiye basis, inventories
   all consumable classroom supplies including data sheets,
   worksheets, quizzes, course schedules, and the like, and
   reorders or provides for reproduction of any items coming
   into short supply.
3) Instructors-Chemists

b) Before the course, each Instructor receives course duty assignment from management, and:

(1) Meets with Course Coordinator for discussions of course plans, objectives, and for development of day-to-day course preparation schedule;

(2) In conference with Course Coordinator determines which any options in tests and measurements will be taught, determines lesson guides to be followed and student reference materials to be used, and resolves any other problems on instructional materials, their content, and related matters which should be determined beforehand;

(3) Reviews requirements for equipment, supplies, audiovisual training aids, and other training resources to be used in individual instructional assignments. Performs equipment upkeep and maintenance procedures, prepares supplies and reagents required to be available for student use. Provides Course Coordinator with timely, detailed information on specifications for all equipment, supplies and other training resources which must be purchased, rented, or borrowed for the course;

(4) If the course is to be conducted in a "field" location, packs equipment and supplies for shipment so that they will arrive at destination in good condition; identifies to the Course Coordinator the equipment and supplies which should not or cannot be shipped which should be provided otherwise at the course site;

(5) Rehearses all classroom and laboratory instructional presentations to the extent necessary to assure effective performance within the scheduled time allocation;

(6) Reviews and practices all tests for which he/she has responsibility as primary Instructor to assure personal proficiency and adequacy of pre-course plans and preparations. Supervises pre-course practice of those who will serve as assistant instructors for the tests;

(7) Prepares to serve as assistant Instructor for specified tests and measurements, developing personal proficiency through pre-course practice under supervision of the applicable primary Instructor, and prepares to teach the tests and measurements in accordance with techniques specified by the primary Instructor;

(8) Reports periodically as requested to the Course Coordinator on status of course preparations, and cooperates in working out timely procedures for their accomplishment.
b) During the course, each Instructor:

(1) Attends and participates in course opening exercises;

(2) Serves as primary Instructor for the tests and measurements assigned to him/her. In this capacity he/she is responsible for all classroom instruction on the designated subjects, leadership of all laboratory instruction, collection of (or arrangements for) samples for laboratory examination by class, student performance evaluation and associated records for submission to Course Coordinator, and student counseling on pertinent matters related to area of personal responsibility;

(3) Assists primary Instructor in laboratory instruction phases of the tests and measurements as assigned, including preparation and organization;

(4) Attends and participates in all classroom start-of-day evaluation sessions on previous day's student performance;

(5) Attends and participates in course closing exercises.

c) After the course, each Instructor:

(1) Reviews the course implementation experience with the Course Coordinator, mutually developing proposals and plans for future offerings of the course;

(2) Evaluates condition of all equipment and supplies, initiating action to repair, renovate, or replace any items found deficient or in short supply;

(3) Takes necessary action to put laboratory into state of neatness and order for occupancy of the next course (this does not imply janitorial service!);

(4) If course was conducted in the field, repacks all equipment and supplies for return to home institution, after at least superficial cleaning of all dirty or contaminated glassware;

(5) On return of shipment to home institution, unpacks all equipment and supplies, returning it to designated custodial site, including return of borrowed equipment and other returnable resources.

4) Laboratory Assistant

The Laboratory Assistant is designated as "optional" in the staffing plan, but services of a Laboratory Assistant are strongly recommended. This is particularly urged in a fixed, training installation where this and other courses are being conducted on a continuing or repetitive basis. The Laboratory Assistant works particularly in support of the
instructional staff. The Assistant will be given routine tasks which will free the instructional staff for more specialized or complicated tasks associated with the planning, preparation, and implementation of the training.

a) Before the course, the Laboratory Assistant:

(1) Works closely with the instructional staff members, performing standardized tasks as specified in the course preparation plan;

(2) Organizes laboratory supplies and equipment for each procedure in such a way as to permit distribution to the students or to their working sites with maximum efficiency during the course presentation;

(3) Assists Course Secretary wherever feasible in assembly and organization of student instructional materials, classroom preparation, and related tasks.

b) During the course, the Laboratory Assistant:

(1) Performs all possible tasks in support of primary Instructors in setting up student work positions, collecting and returning used glassware, supplies and equipment, etc., to central repository;

(2) Cleans and maintains all glassware and supplies excepting those stipulated for student performance;

(3) Notifies primary Instructor promptly of any noted discrepancies or deficiencies in supplies, equipment, or planning which would lead to problems in implementing the course;

(4) In field courses, packs equipment in shipping cases as rapidly as its use has been completed for the course.

c) After the course, the Laboratory Assistant:

(1) Assists Instructors in all equipment and supply inspection, renovation, and return to proper location;

(2) Puts laboratory in state of neatness and order preparatory to use for next class;

(3) Prepares any stable supplies required for next offering of the course, within limits of technical capability.
2. Training Facilities

This course requires both a classroom and a laboratory for class use. Effective presentation of the course requires staff attention to many details related to these facilities. Problems more often occur in field courses (i.e., away from "home base"). In any location it is unusual that all desired features of a training facility will be met, but with timely attention most problems can be solved or at least partially resolved.

a. General Considerations

1) Spatial Relationships

Classroom and laboratory should be separate, but close together. Much of the instruction requires frequent shifts between classroom and laboratory. Therefore, the classroom and laboratory must not be in separate buildings, and should not be far apart within any structure.

2) Associated Comforts

a) The classroom and the laboratory should have a comfortable temperature, be free of obvious drafts, be well-ventilated, and should be well-lighted. It is, of course, possible to develop specifications for acceptable temperature ranges, light intensity ranges, humidity, etc.; but there is no substitute for exercise of good judgment.

b) Suitable restroom and drinking fountain facilities should be convenient to the classroom and laboratory.

c) Smoking:

(1) NO SMOKING IN THE LABORATORY. There should be no compromise on this.

(2) Some schools permit smoking in the classrooms. If this is the practice, it is advisable to locate ashtrays so that smokers sit in an area where their smoking will not disturb others.

3) Lunchroom Facilities

Most schedules for this course will allow a one-hour lunch break. It is advisable that the course staff identify and make known to the class the names and locations of convenient dining facilities where service, variety, quality, and price are satisfactory.

4) Comments to Class about Facilities

a) On the first day of the course the general orientation should include such information as the class needs on the location and use of facilities and conveniences for class use.
b) It is strongly urged that the entire training staff never at any time indulge in apologies or criticisms of the classroom or laboratory facilities being used. Such remarks serve no useful purpose and can only detract from an effective program, provided that everything possible has been done beforehand to resolve existing problems with facilities. Student comments and complaints should be given an honest response, but such comments from students should not be regarded as an excuse for staff to enlarge on the subject.

b. Classroom

1) General Features

   a) Door at rear of room is preferred; this permits entry of late-comers without excessive distraction of class.

   b) The classroom should be free from excessive extraneous noises, such as from construction projects, heavy traffic, or from aircraft.

   c) The classroom should have adequate electric power outlets (115 V) for use of audiovisual equipment. The receptacles should be inspected for assurance that they are compatible with the plugs on the audiovisual projector equipment being used, and adapters and extension cords secured as required.

   d) Room size should be adequate for seating 18 students, plus providing for instructor equipment, projection equipment, and a modest number (4 to 8) of intermittent visitors to the classroom.

   e) The classroom should be capable of being darkened quickly and effectively for use of projection equipment or television. Room dimmer lights for indirect lighting (not striking the screen directly) are recommended in fixed training installations, but can be dispensed within a field training situation.

2) Student Facilities

   a) Ideally, students should be seated at tables, with all seats facing the instructor's area at the front of the classroom. Each student should be allocated 30" or more of table width. The sidearm chairs so familiar in the classrooms of secondary schools and colleges may be used if absolutely necessary, but are distinctly inferior to tables for student work.

   b) Student seating should be at least two screen widths from the projection screen (assuming a 6' screen, no student closer than 12' from the screen) and not more than 6 screen widths from the screen (again assuming a 6' screen, no student more than 36' from the screen). Furthermore, all students should be seated within a 30° angle to the left and to the right of a line from the middle of the projection screen to the projector.
3) Classroom Instructional Facilities

a) Lectern, either freestanding or table-type; suitable for standing instructor

b) Demonstration table at front of classroom, approximately 3' x 5'

c) Chalkboard, at least 3' x 5' (preferably larger), with chalk, erasers, pointer

d) Audiovisual equipment

(1) Public address system (optional but recommended) with lavalier microphone with adequate cord length to permit instructor to move about at front of classroom with relative freedom

(2) Projection screen (for size consideration see 2) above), matte, beaded, or lenticular surface

(3) Projector, 35-mm slide projector for slides mounted in cardboard or plastic mount; carousel type preferred. Should have projection lens with cord length suitable for use from rear of room

(4) Projector, overhead type, for use with projectuals approximately 7" x 9"

(5) Cassette type playback unit, with cueing feature for automatic operation of cassette type slide projector; compatible with National Training and Operational Technology Center (EPA) tape/slide instructional units

(6) Television tape playback unit (3/4" cassette type, "U-Matic" or equivalent)

(7) Television receiver, commercial type, color, 19" diagonal picture, or larger At least one receiver, preferably two

c) Laboratory

1) General Features

a) Should be well-lighted, adequately ventilated. It is particularly important that the laboratory be free from strong drafts in student working areas.

b) Should provide for students to stand at laboratory benches which are approximately 30" from floor to bench surface.

c) Conventional laboratory services should be available at student work areas, including electricity (115 V), gas, and vacuum.
d) Space between benches should be adequate for students to work without interfering with each other, and to permit free movement of instructors in the student working area.

e) Safety features of the laboratory should be checked, including location and condition of first-aid kits, fire extinguishers, emergency showers, eye-wash facilities, and other emergency equipment.

2) Student Facilities

a) Provide at least 6' of bench width per student pair. While students will work in pairs to the extent that they will share certain limited equipment, each student will perform all tests and measurements.

b) Provide bench space or floor space as necessary for laboratory equipment described in the equipment and supply lists, such as balances, ovens, waterbaths and other items not assigned to individual student work.

3) Laboratory Instructional Facilities

a) A chalkboard and demonstration table are recommended.

b) Provide at least 20 square feet for reserve supplies or equipment of each instructor.

c) For field courses, provide area for packing and unpacking areas for equipment to be shipped. This should be at least 100 square feet of floor space, with at least 20 square feet of table space.

d) Security

Valuable property is used both in the classroom and in the laboratory. Some of the items are particularly susceptible to theft. Accordingly:

1) Provide for locking of both classroom and laboratory when not in use, or assure that adequate security is provided in the facility by other means.

2) Be sure that the necessary keys are available to the instructional staff at their need.

3) With field courses, often it is necessary for the training staff to work in the evening or weekends to prepare for coming classwork. Arrangements must be made well in advance to secure authorized entry to the training facilities being made available by a host organization.

4) Thefts during normal working hours may be a special problem. Maintain surveillance to the extent practical, and keep out-of-service theft-prone items out of exposed locations.
3. Laboratory Equipment and Supply Requirements

The consolidated list in this section is for overall planning purposes. For day-to-day laboratory requirements, see the "IPW Equipment and Supply Requirements" and the "IPW Reagent Requirements" in each Instructional Package Worksheet contained in Part II of this Guide.

Before using the listing in this section decisions must be made about the specific procedure to be taught for each test in the course because several of the Effluent Monitoring Procedures (EMPs) contain a choice of procedures. The Ammonia EMP is written for the distillation of a macro sample followed by either Nesslerization or titration. The Kjeldahl Nitrogen EMP contains a choice between macro or micro digestion and distillation, followed by either Nesslerization or titration. There is an EMP for determining ammonia by using a selective ion electrode. The Chemical Oxygen Demand EMP is written for the low level range of 5-50 mg/liter but includes the modifications for concentrations greater than 50 mg/liter COD. The Phosphorus EMP can be used to determine total phosphorus or only orthophosphate. These different possibilities affect the equipment and supply needs. The requirements of area NPDES permits and/or the type of equipment readily available will help the course planners make the choices among these possibilities.

The quantities of laboratory materials needed for a course depend on the specific laboratory work assigned to each student. For the consolidated list in this section, the basis for quantities required is the assignment stated in each Instructional Package Worksheet (IPW).

The abbreviations used in the listing to refer to the various procedures that might be taught and the assignment given for each in the related IPW are stated in a table preceding the laboratory listing.

The listing itself is divided into three sections: capital equipment of more than $100 unit value, reusable equipment of less than $100 unit value, and consumable equipment of less than $100 unit.value. The first column in each section contains a description of the item in the language and the qualifications required for ordering the item from commercial catalogs. The second column lists the minimum quantity per student required for each procedure presented in the EMPs for the course. The third column lists the minimum quantity for a class of 18 students. The numbers found here are often based on multiple use of the item. The fourth and final column contains remarks that may be useful when deciding on class needs or when ordering equipment.

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.
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 which will provide needed resources, on an item-by-item basis. When the responsibility is assigned/accepted, this can be annotated in the "remarks" column, with a copy of the annotated list in the hands of the Course Coordinator and a copy for 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.
LABORATORY EQUIPMENT AND SUPPLY REQUIREMENTS

Following is the key for abbreviations used for course topics. Quantities per test are based on the IPW assignments as noted.

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Test Description</th>
<th>IPW Assignments</th>
</tr>
</thead>
<tbody>
<tr>
<td>ADma</td>
<td>Ammonia Distillation, macro</td>
<td>1 sample per 2 students</td>
</tr>
<tr>
<td>ADmi</td>
<td>Ammonia Distillation, micro (as part of Kjeldahl N EMP)</td>
<td>1 sample</td>
</tr>
<tr>
<td>AE</td>
<td>Ammonia Electrode</td>
<td>2 standards + 2 samples</td>
</tr>
<tr>
<td>AN</td>
<td>Ammonia Nesslerization (as part of Kjeldahl N EMP)</td>
<td>1 blank + 2 standards + 1 digested sample</td>
</tr>
<tr>
<td>ATma</td>
<td>Ammonia Titration, macro</td>
<td>1 sample</td>
</tr>
<tr>
<td>CG</td>
<td>Calibration Graphs</td>
<td>1 graph</td>
</tr>
<tr>
<td>COD</td>
<td>Chemical Oxygen Demand (low level)</td>
<td>1 sample or blank</td>
</tr>
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<td></td>
<td></td>
<td>1 FAS standardization</td>
</tr>
<tr>
<td>KDma</td>
<td>Kjeldahl Digestion, macro (alternative to micro)</td>
<td>1 sample</td>
</tr>
<tr>
<td>KDmi</td>
<td>Kjeldahl Digestion, micro</td>
<td>1 sample</td>
</tr>
<tr>
<td>NO₃</td>
<td>Nitrate + Nitrite and Nitrate</td>
<td>Prepare one reduction column.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2 blanks + 2 standards</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2 samples</td>
</tr>
<tr>
<td>OG</td>
<td>Oil and Grease</td>
<td>1 sample</td>
</tr>
<tr>
<td>OP</td>
<td>Orthophosphate as P (alternative to Total Phosphorus)</td>
<td>1 blank + 1 standard</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 sample</td>
</tr>
<tr>
<td>SP</td>
<td>Spectrophotometer</td>
<td>1 instrument</td>
</tr>
<tr>
<td>TP</td>
<td>Total Phosphorus as P</td>
<td>1 blank + 1 standard</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1 sample</td>
</tr>
</tbody>
</table>
# A. CAPITAL EQUIPMENT (More than $100 Unit Value per One Item)

<table>
<thead>
<tr>
<th>DESCRIPTION</th>
<th>EACH STUDENT</th>
<th>QUANTITY FOR:</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>BALANCES, analytical; 0.1 mg sensitivity at a load of 200 g; with Instructions and Manuals</td>
<td>OG 1/4</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td></td>
<td>OP 1/3</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>TP 1/3</td>
<td></td>
<td></td>
</tr>
<tr>
<td>BALANCES, Trip, 500 g capacity</td>
<td>NO₃ 1/3</td>
<td>6</td>
<td>To weigh cadmium</td>
</tr>
<tr>
<td>DIGESTION APPARATUS, for Kjeldahl determination. See diagrams of equipment in EMP. Quantity is according to heat sources required.</td>
<td>KĐma 1</td>
<td>18</td>
<td>Apparatus is readily available commercially.</td>
</tr>
<tr>
<td></td>
<td>KĐmi 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DISTILLATION APPARATUS, for ammonia and/or Kjeldahl determinations. See diagrams of equipment in Kjeldahl Nitrogen EMP.</td>
<td>TP 1/4</td>
<td>4</td>
<td>ADmi set-ups shown in EMP require some fabricated parts. (Inquire EPA-NTOTC) ADmi total assemblies are also available commercially.</td>
</tr>
<tr>
<td>ELECTRICAL OUTLETS, 120 volts</td>
<td>TP 1</td>
<td>?</td>
<td>Number depends on number (size) of HOT PLATES used.</td>
</tr>
<tr>
<td>HEATING SURFACES, flat for Erlenmeyer flasks or else heating mantles for round bottom flasks. Choice should produce at least 9 watts per inch and have adjustable heat control.</td>
<td>COD 1</td>
<td>18</td>
<td>A 16 amp line is usually required for a series of 6 reflux set-ups.</td>
</tr>
<tr>
<td>HOT PLATES with continuous settings, not just &quot;low,&quot; &quot;medium&quot; and &quot;high.&quot; If doing AE EMP, magnetic stirring feature is desirable.</td>
<td>OG 1</td>
<td>?</td>
<td>Number depends on size. OG for 1 - 1 liter-beaker for each, TP for 3 - 125 ml Erlenmeyers for each.</td>
</tr>
<tr>
<td></td>
<td>TP 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>HOOD SPACE - equivalent well-ventilated area.</td>
<td>OG 1</td>
<td>1/3</td>
<td>OP/TP: three are needed if students clean glassware.</td>
</tr>
<tr>
<td></td>
<td>OP 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>TP 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY FOR:</td>
<td>CLASS</td>
<td>REMARKS</td>
</tr>
<tr>
<td>-------------</td>
<td>---------------</td>
<td>-------</td>
<td>---------</td>
</tr>
<tr>
<td>pH METERS, ELECTROMETRIC accurate to at least 0.1 pH unit and preferably with a single electrode</td>
<td>EACH STUDENT</td>
<td>OF 18</td>
<td>To conserve time, this quantity is preferable.</td>
</tr>
<tr>
<td>SPECIFIC ION METERS, Orion* Model 404, 407 or 407A, each with AMMONIA ELECTRODE, Orion* Model 95-10</td>
<td>AE 1/3</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>SPECTROPHOTOMETERS, Bausch and Lomb* Spectronic 20 or equivalent, with Instruction Manuals</td>
<td>AN 1/3</td>
<td>SP 1/3</td>
<td>6</td>
</tr>
<tr>
<td>SPECTROPHOTOMETER CELLS for instruments used</td>
<td>AN 1/3</td>
<td>SP 1/3</td>
<td>6</td>
</tr>
<tr>
<td>SPECTROPHOTOMETER PHOTOTUBES, Infared range (B&amp;L* #33-29-72) plus Infared FILTERS (B&amp;L* #33-29-18) or equivalent</td>
<td>OP 1/3</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>SPECTROPHOTOMETER PHOTOTUBES, visible range (B&amp;L* #33-29-71) or equivalent</td>
<td>AN 1/3</td>
<td>SP 1/3</td>
<td>6</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>OP 1/3</td>
<td>6</td>
<td>OP for filtration of turbid samples</td>
</tr>
<tr>
<td>WATER BATHS to maintain 80°C for 125 ml distilling flasks</td>
<td>DG 1</td>
<td></td>
<td>Number depends on size: 1 - 125 ml flask for each student</td>
</tr>
<tr>
<td>WATER STILL with an anion-cation exchange system to produce water free of ammonia</td>
<td>ADma 1</td>
<td>KDma 1</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>ADmi 1</td>
<td>KDmi 1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>AE 1</td>
<td>ATma 1</td>
<td></td>
</tr>
</tbody>
</table>

* Mention of a specific brand name does not constitute endorsement of the U.S. Environmental Protection Agency.
### REUSABLE EQUIPMENT (Less than $100 Unit Value)

<table>
<thead>
<tr>
<th>DESCRIPTION</th>
<th>EACH STUDENT</th>
<th>CLASS OF 18</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>APRONS, laboratory (plastic acceptable)</td>
<td>All 1</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>BEAKERS, glass, 50 ml</td>
<td>ADma 1</td>
<td>ADmi 1</td>
<td></td>
</tr>
<tr>
<td>BEAKERS, glass, 150 ml</td>
<td>AE 4</td>
<td>NO3 1</td>
<td>36-72 only if AE is taught</td>
</tr>
<tr>
<td>BEAKERS, glass, 250 ml</td>
<td>NO3 3</td>
<td>54</td>
<td></td>
</tr>
<tr>
<td>BEAKERS, glass, 600 ml</td>
<td>ADma 1/2</td>
<td>OP 1</td>
<td>18 OP/TP only if cleaning glassware</td>
</tr>
<tr>
<td>BEAKERS, glass, 1000 ml</td>
<td>OG 1</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>BOILING BEADS, glass 5 mm</td>
<td>ADma 6</td>
<td>KDma 6</td>
<td>738</td>
</tr>
<tr>
<td>BOTTLES, glass dispensing, 100 ml</td>
<td>AT 1/2</td>
<td>9</td>
<td></td>
</tr>
<tr>
<td>BOTTLES, glass, for aliquots of samples, 225 ml minimum</td>
<td>COD 1/3</td>
<td>6</td>
<td>Each needs 75 ml</td>
</tr>
<tr>
<td>BOTTLES, plastic, for aliquots of samples, 250 ml minimum</td>
<td>KDmi 1/3</td>
<td>OP 1/3</td>
<td>18 Each test volume is 50 ml per one student.</td>
</tr>
<tr>
<td>BOTTLES, plastic, for aliquots of samples, 1000 ml</td>
<td>KDma 1/2</td>
<td>AE 1/3</td>
<td>24 Macro tests require 400-500 ml. AE requires 2-100 ml samples for each.</td>
</tr>
<tr>
<td>RUSHES, assorted, for cleaning glassware</td>
<td>All 1/2</td>
<td>9</td>
<td></td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>EACH STUDENT</td>
<td>CLASS OF 18</td>
<td>REMARKS</td>
</tr>
<tr>
<td>-------------</td>
<td>-------------</td>
<td>-------------</td>
<td>---------</td>
</tr>
<tr>
<td>BRUSHES, balance</td>
<td>NO\textsubscript{3} 1</td>
<td>OP 1</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>OG 1</td>
<td>TP 1</td>
<td></td>
</tr>
<tr>
<td>BURETS, 25 ml, 0.1 ml graduations, teflon stopcock plug preferred</td>
<td>COD 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Put by balances.</td>
</tr>
<tr>
<td>BURETS, 50 ml, 0.1 ml graduations, teflon stopcock plug preferred</td>
<td>ATma 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>ADma 1/2</td>
<td></td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>ADm1 1/6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>BURNERS, gas, Meker type or equivalent heat source</td>
<td></td>
<td></td>
<td>9</td>
</tr>
<tr>
<td>GLAMPS, buret for titration stand, to hold two burets</td>
<td>ATma 1/2</td>
<td>NO\textsubscript{3} 1/2</td>
<td>9</td>
</tr>
<tr>
<td></td>
<td>COD 1/2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CLAMPS, for titration stand to fit neck of 125 ml distilling flask</td>
<td>OG 1/2</td>
<td></td>
<td>9</td>
</tr>
<tr>
<td>CLAMPS, screw to regulate flow through 4 cm ID rubber tubing</td>
<td>NO\textsubscript{3} 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td>CLAMP, pinchcock for 4 cm ID rubber tubing</td>
<td>NO\textsubscript{3} 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td>CONDENSERS, 12 inch Allihn or equivalent with a ground glass joint to fit into heat-resistant FLASK. (24/40 is a commonly used joint size)</td>
<td>COD 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td>CONDENSERS, 16 inch Graham or Allihn. Extend tip with rubber and glass tubing for delivery into 500 ml graduated Erlenmeyer FLASK.</td>
<td>ADma 1/2</td>
<td></td>
<td>9</td>
</tr>
<tr>
<td>CONTAINERS for acid and mercury wastes, about 2 liter</td>
<td>COD 1/4</td>
<td></td>
<td>5</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Up to 400-500 ml per student</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>EACH STUDENT</td>
<td>CLASS OF 18</td>
<td>REMARKS</td>
</tr>
<tr>
<td>-----------------------------------------------------------------</td>
<td>--------------</td>
<td>-------------</td>
<td>----------------------------------------------</td>
</tr>
<tr>
<td>CYLINDERS, graduated, 10 ml</td>
<td>ADmi 2</td>
<td>OG 1</td>
<td>36</td>
</tr>
<tr>
<td></td>
<td>KDmi 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CYLINDERS, graduated, 25 ml</td>
<td>COD 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td>CYLINDERS, graduated, 50 ml</td>
<td>NO3 1</td>
<td>OG 1</td>
<td>18</td>
</tr>
<tr>
<td>CYLINDERS, graduated, 100 ml</td>
<td>ADma 2</td>
<td>KDmi 1</td>
<td>18-36</td>
</tr>
<tr>
<td></td>
<td>AE 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>COD 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>KDma 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CYLINDERS, graduated, 500 ml</td>
<td>ADma 1</td>
<td>KDma 1</td>
<td>18</td>
</tr>
<tr>
<td>DESICCATORS with effective desiccant and large enough for 1 - 125 ml distilling flask per student</td>
<td>OG 1</td>
<td></td>
<td>? Number depends on size of desiccator</td>
</tr>
<tr>
<td>EVAPORATING DISHES, or similar pieces to place between heating surface and flask for cooling</td>
<td>COD 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td>FILTERING ASSEMBLIES for 60 ml or 10 ml volumes, filtrate to be transferred</td>
<td>OP 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>TP 1</td>
<td></td>
<td></td>
</tr>
<tr>
<td>FLASKS, distilling, 125 ml with a 24/40 ground glass neck (Corning® #4100 is an example)</td>
<td>OG 1</td>
<td></td>
<td>18</td>
</tr>
<tr>
<td>FLASKS; Erlenmeyer, wide mouth, 125 ml</td>
<td>OP 3</td>
<td>TP 3</td>
<td>54</td>
</tr>
<tr>
<td>FLASKS; Erlenmeyer, wide mouth, 250 ml</td>
<td>NO3 3</td>
<td></td>
<td>54</td>
</tr>
</tbody>
</table>

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<table>
<thead>
<tr>
<th>DESCRIPTION</th>
<th>QUANTITY FOR:</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>DESCRIPTION</strong></td>
<td><strong>EACH STUDENT</strong></td>
<td><strong>CLASS</strong> <strong>OF 18</strong></td>
</tr>
<tr>
<td>FLASKS, Erlenmeyer, wide mouth, graduated, 500 ml</td>
<td>ADma 1</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>COD 1</td>
<td></td>
</tr>
<tr>
<td>FLASKS, heat resistant glass, 500 ml graduated Erlenmeyer or 300 ml round bottom with a ground glass neck to fit CONDENSER of choice.</td>
<td>COD 1</td>
<td>18</td>
</tr>
<tr>
<td>FLASKS, Kjeldahl, 100 ml</td>
<td>ADmi 1</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>KDmi 1</td>
<td></td>
</tr>
<tr>
<td>FLASKS, Kjeldahl, 800 ml</td>
<td>ADma 1</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>KDma 1</td>
<td></td>
</tr>
<tr>
<td>FLASKS, volumetric, 50 ml, with glass stoppers</td>
<td>NO3 1</td>
<td>54</td>
</tr>
<tr>
<td></td>
<td>TP 3</td>
<td></td>
</tr>
<tr>
<td>FLASKS, volumetric, 100 ml with glass stoppers</td>
<td>NO3 2</td>
<td>36</td>
</tr>
<tr>
<td>FLASKS, volumetric, 1000 ml with glass stoppers</td>
<td>OP 1</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>TP 1</td>
<td></td>
</tr>
<tr>
<td>FORCEPS, pairs to manipulate filter paper</td>
<td>OP 1</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>TP 1</td>
<td></td>
</tr>
<tr>
<td>FUNNELS, Buchner, to fit CONTAINER for COD wastes (for glass beads)</td>
<td>COD 1/4</td>
<td>5</td>
</tr>
<tr>
<td>FUNNELS, separatory, 2 liter with Teflon stopcock</td>
<td>OG 1</td>
<td>18</td>
</tr>
<tr>
<td>FUNNELS; short stem, diameter about 50 mm to fit neck of 50 ml volumetric flask and 25 or 50 ml buret</td>
<td>COD 1</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>OP 1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>OG 1</td>
<td></td>
</tr>
<tr>
<td></td>
<td>TP 1</td>
<td></td>
</tr>
<tr>
<td>GLASSES, pairs of safety</td>
<td>ALL 1</td>
<td>18</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>EACH STUDENT</td>
<td>CLASS OF 1B</td>
</tr>
<tr>
<td>------------------------------------------------------------</td>
<td>--------------</td>
<td>-------------</td>
</tr>
<tr>
<td>GLOVES, pairs of rubber material</td>
<td>NO$_3$ 1</td>
<td>TP 1</td>
</tr>
<tr>
<td></td>
<td>OP 1</td>
<td></td>
</tr>
<tr>
<td>MAGNETIC STIRRERS with magnetic bars</td>
<td>AE 1/3</td>
<td>6</td>
</tr>
<tr>
<td>NESSLER TUBES, scored at 50 ml, low form desirable</td>
<td>AN 3</td>
<td>72</td>
</tr>
<tr>
<td></td>
<td>KDmi 1</td>
<td></td>
</tr>
<tr>
<td>NESSLER TUBE SUPPORTS</td>
<td>AN 1/2</td>
<td>9</td>
</tr>
<tr>
<td>PANS for ice water to cool 500 ml Erlenmeyer flasks</td>
<td>COD 1/2</td>
<td>9</td>
</tr>
<tr>
<td>PIPETS, dropping (medicine droppers) with bulb about 1 ml</td>
<td>OG 1</td>
<td>18</td>
</tr>
<tr>
<td>PIPETS, measuring, Mohr glass, 1 or 2 ml, graduated at 0.5 ml</td>
<td>NO$_3$ 1/4</td>
<td>5</td>
</tr>
<tr>
<td>PIPETS, measuring, glass, 1 ml graduated at 0.1 ml</td>
<td>OP 1</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>TP 1</td>
<td></td>
</tr>
<tr>
<td>PIPETS, measuring, Mohr glass, 10 ml</td>
<td>AN 2</td>
<td>OP 3</td>
</tr>
<tr>
<td></td>
<td>COD 1</td>
<td>TP 3</td>
</tr>
<tr>
<td></td>
<td>NO$_3$ 1</td>
<td></td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 1 ml</td>
<td>AE 1/4</td>
<td>OP 1/8</td>
</tr>
<tr>
<td></td>
<td>NO$_3$ 1/4</td>
<td>TP 1/8</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 2 ml</td>
<td>NO$_3$ 1/4</td>
<td>5</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 3 ml</td>
<td>OP 1/8</td>
<td>TP 1/8</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY FOR:</td>
<td>EACH STUDENT</td>
</tr>
<tr>
<td>-----------------------------------------------------------------</td>
<td>---------------</td>
<td>--------------</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 5 ml</td>
<td>NO₃</td>
<td>TP 1/8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>OP 1/8</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 10 ml</td>
<td>COD 1</td>
<td>OP 1/8</td>
</tr>
<tr>
<td></td>
<td>NO₃ 1/4</td>
<td>TP 1/8</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 20 ml</td>
<td>OP 1/8</td>
<td>OP 1</td>
</tr>
<tr>
<td></td>
<td>TP 1/8</td>
<td>TP 1</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 25 ml</td>
<td>COD 1</td>
<td>NO₃ 1</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 30 ml</td>
<td>OP 1/8</td>
<td>TP 1/8</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 40 ml</td>
<td>OP 1/8</td>
<td>TP 1/8</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 50 ml</td>
<td>COD 1</td>
<td>TP 1</td>
</tr>
<tr>
<td></td>
<td>NO₃ 3</td>
<td>OP 1/8</td>
</tr>
<tr>
<td></td>
<td>OP 1</td>
<td>TP 1/8</td>
</tr>
<tr>
<td>PIPETS, volumetric transfer, 100 ml with top and tip cut off for reduction column</td>
<td>NO₃ 1</td>
<td></td>
</tr>
<tr>
<td>PIPETS, volumetric transfer of size required if sample dilution is necessary</td>
<td>KD ml 1</td>
<td>TP 1</td>
</tr>
<tr>
<td></td>
<td>OP 1</td>
<td></td>
</tr>
<tr>
<td>PIPET BULBS, large with shaped opening to fit variety of pipet sizes</td>
<td>AlJ 1</td>
<td></td>
</tr>
<tr>
<td>PROPPIPET BULB</td>
<td>NO₃ 1</td>
<td></td>
</tr>
<tr>
<td>PIPETTERS, automatic (OPTIONAL), glass with delivery setting for at least 10 ml for acids</td>
<td>COD 2</td>
<td></td>
</tr>
<tr>
<td></td>
<td>16</td>
<td></td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>EACH STUDENT</td>
<td>GLASS OF 18</td>
</tr>
<tr>
<td>-----------------------------------</td>
<td>--------------</td>
<td>-------------</td>
</tr>
<tr>
<td>REAGENT SPOONS, 0.4 gram</td>
<td>TP 1/3</td>
<td>6</td>
</tr>
<tr>
<td>REAGENT SPOONS, 1 gram</td>
<td>COD 1/3</td>
<td>6</td>
</tr>
<tr>
<td>RINGS, metal for stand to support a 2 liter separatory funnel</td>
<td>OG 1</td>
<td>18</td>
</tr>
<tr>
<td>RULERS, 12 inches long</td>
<td>CG 1</td>
<td>18</td>
</tr>
<tr>
<td>SIEVES, 60 mesh</td>
<td>NO₃ 1</td>
<td>18</td>
</tr>
<tr>
<td>SPATULAS</td>
<td>NO₃ 1</td>
<td>30</td>
</tr>
<tr>
<td></td>
<td>Balances 12</td>
<td></td>
</tr>
<tr>
<td>SPRAY TRAP, Kjeldahl, connecting bulb, 55 mm. See diagram in Kjeldahl Emp.</td>
<td>AODma 1/2</td>
<td>9</td>
</tr>
<tr>
<td>STANDS, titration with porcelain bases</td>
<td>ATma 1/2</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>NO₃ 1/2</td>
<td></td>
</tr>
<tr>
<td></td>
<td>COD 1/2</td>
<td></td>
</tr>
<tr>
<td></td>
<td>OG 1</td>
<td></td>
</tr>
<tr>
<td>STEAM BATHS for 125 ml distilling flask</td>
<td>OG 1</td>
<td>?</td>
</tr>
<tr>
<td>STOPPER/GLASS TUBING APPARATUS for applying suction to a 125 ml distilling FLASK. See OG EMP, Figure 2.</td>
<td>OG 1/6</td>
<td>3</td>
</tr>
<tr>
<td>STOPPERS, rubber #3 or #6 to fit NESSLER TUBES</td>
<td>AN 1</td>
<td>18</td>
</tr>
<tr>
<td>STOPPERS, rubber #3 to fit CONDENSER (Allihn) or spray trap connection</td>
<td>AODma 1/2</td>
<td>9</td>
</tr>
<tr>
<td>STOPPERS, rubber #7 to fit neck of 800 ml Kjeldahl FLASK for spray trap connection</td>
<td>AODma 1/2</td>
<td>9</td>
</tr>
<tr>
<td>TONGS, crucible, pairs</td>
<td>TP 1</td>
<td>18</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY FOR:</td>
<td>EACH STUDENT</td>
</tr>
<tr>
<td>-------------</td>
<td>---------------</td>
<td>--------------</td>
</tr>
<tr>
<td>TUBING, soft rubber, 4 cm ID, 7.5 cm length</td>
<td>NO₃</td>
<td>1</td>
</tr>
<tr>
<td>TUBING, tygon for CONDENSER as connection to cooling water source. Lengths depend on apparatus</td>
<td>COD</td>
<td>1</td>
</tr>
<tr>
<td>WASH BOTTLES, plastic squeeze type, 500 ml</td>
<td>All</td>
<td>1</td>
</tr>
</tbody>
</table>

C. CONSUMABLE SUPPLIES (Less than $100 Unit Value)

<table>
<thead>
<tr>
<th>DESCRIPTION</th>
<th>QUANTITY FOR:</th>
<th>EACH STUDENT</th>
<th>CLASS OF 18</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>FILTER DISCS, Whatman No. 40 to fit FUNNEL with diameter of about 50 mm</td>
<td>OG</td>
<td>1</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>FILTER DISCS, phosphorus-free, 0.45 micron pore size, Gelman GA-6 or equivalent, to fit FILTERING ASSEMBLIES</td>
<td>OP</td>
<td>1</td>
<td>TP 3</td>
<td>18-54</td>
</tr>
<tr>
<td>GLASS WOOL, small wad for end of 100 ml cut-off pipet</td>
<td>NO₃</td>
<td>1</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>GRAPH PAPER, sheets, arithmetic</td>
<td>CG</td>
<td>1</td>
<td>18</td>
<td>Can substitute printed graph sheets in EMP</td>
</tr>
<tr>
<td>ICE SUPPLY, trays of cubes</td>
<td>COD</td>
<td>1/3</td>
<td>6</td>
<td></td>
</tr>
<tr>
<td>PENCILS, WAX MARKING</td>
<td>All</td>
<td>1</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>pH PAPER, strips, short range to test at pH 9.5</td>
<td>Adga</td>
<td>5</td>
<td>90</td>
<td></td>
</tr>
</tbody>
</table>

REAGENTS are listed according to test at the end of this section

SAMPLES are listed according to test at the end of this section
<table>
<thead>
<tr>
<th>DESCRIPTION</th>
<th>QUANTITY FOR: EACH STUDENT</th>
<th>CLASS OF 18</th>
<th>REMARKS</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAMPLE TAGS, preferably pre-printed with blanks for information required by NPDES reports</td>
<td>All</td>
<td>~45</td>
<td></td>
</tr>
<tr>
<td>TISSUES, soft in boxes</td>
<td>SP 6 BALANCES 6</td>
<td>12</td>
<td></td>
</tr>
<tr>
<td>TOWELS, paper, in boxes or packets</td>
<td>SINKS 1</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>WATER, distilled, ammonia-free</td>
<td>ADma 0.1 l ADmii 0.1 l AE 0.5 l AN 0.15 l</td>
<td>13.5 l</td>
<td></td>
</tr>
<tr>
<td>WATER, distilled, ammonia-free and nitrate-nitrite free</td>
<td>NO 3 4 l</td>
<td>72 l</td>
<td>The large quantity is required to prepare Cu-Cd.</td>
</tr>
<tr>
<td>WATER, distilled, phosphorus-free</td>
<td>OP 2.5 l TP 2.5 l</td>
<td>45 l</td>
<td>The large quantity is required for cleaning glassware.</td>
</tr>
<tr>
<td>WATER, distilled, negligible organic concentrations</td>
<td>COD 0.2 l</td>
<td>2 l</td>
<td></td>
</tr>
<tr>
<td>WATER, distilled</td>
<td>SP 0.1 l</td>
<td>2 l</td>
<td></td>
</tr>
<tr>
<td>WEIGHING BOATS; disposable</td>
<td>NO 3 1</td>
<td>18</td>
<td>To weigh cadmium</td>
</tr>
<tr>
<td>REAGENTS:</td>
<td></td>
<td></td>
<td>See p. 160 or p. 176 EPA Methods Manual. Also see Ammonia EMP or the Kjeldahl N EMP, as applicable.</td>
</tr>
<tr>
<td>AMMONIA DISTILLATION, macro or micro</td>
<td>Amounts are minimum.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(AD is used to isolate ammonia from samples. It is also used to isolate ammonia from digested samples as part of the Kjeldahl N test.)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Boric Acid solution, 2%: 10 g boric acid/500 ml</td>
<td>ADma 150 ml ADmii 5 ml</td>
<td>450 ml 90 ml</td>
<td>For ADmii, make 250 ml.</td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>EACH STUDENT</td>
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<td>REMARKS</td>
</tr>
<tr>
<td>-----------------------------------------------------------------------------</td>
<td>--------------</td>
<td>-------------</td>
<td>------------------------------</td>
</tr>
<tr>
<td>1N sodium hydroxide solution: 10 g sodium hydroxide/250 ml</td>
<td>ADma ^10 ml</td>
<td>^180 ml</td>
<td>For pH adjustment</td>
</tr>
<tr>
<td>Borate buffer solution - 500 ml: ^0.5 g sodium hydroxide</td>
<td>ADma 25 ml</td>
<td>226 ml</td>
<td>For ADma, it is convenient to make the 500 ml volume.</td>
</tr>
<tr>
<td>^2.5 g sodium tetraborate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sodium hydroxide-sodium thiosulfate solution - 250 ml: 125 g sodium hydroxide</td>
<td>ADma 100 ml</td>
<td>1800 ml</td>
<td>For AD as part of Kjeldahl-N test. See that EMP.</td>
</tr>
<tr>
<td>^7 g sodium thiosulfate pentahydrate</td>
<td>ADmi 10 ml</td>
<td>180 ml</td>
<td></td>
</tr>
<tr>
<td>Phenolphthalein solution: 1 g phenolphthalein/100 ml</td>
<td>ADma 10 drops</td>
<td>180 drops</td>
<td>For AD as part of Kjeldahl-N test. See that EMP.</td>
</tr>
<tr>
<td>ADmi 2 drops</td>
<td>36 drops</td>
<td></td>
<td></td>
</tr>
<tr>
<td>AMMONIA ELECTRODE</td>
<td></td>
<td></td>
<td>Preparations are according to electrode manufacturer. See EMP.</td>
</tr>
<tr>
<td>Stock ammonium chloride solution: 0.3819 g ammonium chloride/100 ml</td>
<td>10 ml</td>
<td>10 ml</td>
<td>Dilute this twice for 200 ml of 1.0 and 0.1 mg/liter for each student for standards plus sample preparations.</td>
</tr>
<tr>
<td>10 M sodium hydroxide solution: 80 g sodium hydroxide/200 ml</td>
<td>4 ml</td>
<td>72 ml</td>
<td></td>
</tr>
<tr>
<td>AMMONIA NESSLERIZATION</td>
<td></td>
<td></td>
<td>See p. 177 in 1974 EPA Methods Manual. Also see Kjeldahl-N EMP.</td>
</tr>
<tr>
<td>Nessler reagent - 100 ml: 10 g mercuric iodide 7 g potassium iodide 16 g sodium hydroxide</td>
<td>4 ml</td>
<td>72 ml</td>
<td></td>
</tr>
<tr>
<td>Stock ammonium chloride: 0.381 g ammonium chloride/100 ml</td>
<td>0.18 ml</td>
<td>3.25 ml</td>
<td>Dilute this for up to 18 ml of 0.01 mg/liter per student.</td>
</tr>
<tr>
<td>DESCRIPTION</td>
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</tr>
<tr>
<td>-------------------------------------------------</td>
<td>--------------</td>
<td>-------------</td>
<td>-------------------------------------------------------------------------</td>
</tr>
<tr>
<td>AMMONIA TITRATION, macro</td>
<td>Amounts are minimum</td>
<td></td>
<td>See p. 160 in 1974 EPA Methods Manual. Also see Ammonia EMP.</td>
</tr>
<tr>
<td>Mixed indicator solution - 150 ml:</td>
<td>3 drops</td>
<td>27 drops</td>
<td></td>
</tr>
<tr>
<td>200 mg methyl red</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100 mg methylene blue</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.1 N sulfuric acid solution:</td>
<td>To make 1 ml of 0.02 N</td>
<td>810 ml</td>
<td>Dilute this for up to 90 ml of 0.02 N acid per student.</td>
</tr>
<tr>
<td>1.5 ml concentrated sulfuric acid/500 ml</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CHEMICAL OXYGEN DEMAND, low level</td>
<td>Amounts are minimum</td>
<td></td>
<td>See p. 22 in 1974 EPA Methods Manual. Also see EMP.</td>
</tr>
<tr>
<td>Mercuric sulfate</td>
<td>1 g</td>
<td>18 g</td>
<td></td>
</tr>
<tr>
<td>Concentrated sulfuric acid</td>
<td>25 ml</td>
<td>450 ml</td>
<td>OPTIONAL: Place in automatic pipetters and check for 5 ml delivery.</td>
</tr>
<tr>
<td>Sulfuric acid - silver sulfate solution -</td>
<td>70 ml</td>
<td>1260 ml</td>
<td>OPTIONAL: Place in 2 automatic pipetters and check for 10 ml delivery.</td>
</tr>
<tr>
<td>2 liters:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2 liters concentrated sulfuric acid</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>21.6 g silver sulfate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.250 N potassium dichromate solution -</td>
<td>100 ml</td>
<td>200 ml</td>
<td>Dilute this for 50 ml of 0.025 N per student.</td>
</tr>
<tr>
<td>2.452 g potassium dichromate/200 ml</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferroin indicator solution - 100 ml:</td>
<td>11 drops</td>
<td>198 drops</td>
<td>Ferroin solution can be purchased.</td>
</tr>
<tr>
<td>1.48 g 1-10 orthophenanthroline monohydrate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.70 g ferrous sulfate heptahydrate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.250 N ferrous ammonium sulfate solution -</td>
<td>100 ml</td>
<td>200 ml</td>
<td>Dilute this for 75 ml of 0.025 N per student</td>
</tr>
<tr>
<td>200 ml:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>19.6 g ferrous ammonium sulfate hexahydrate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 ml concentrated sulfuric acid</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
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</tr>
<tr>
<td>------------------------------------------</td>
<td>--------------</td>
<td>-------------</td>
<td>-------------------------------------------------------------------------</td>
</tr>
<tr>
<td><strong>KJELDAHL DIGESTION</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Digestion reagent - 2000 ml:</td>
<td>KDma 100 ml</td>
<td>1800 ml</td>
<td>See p. 176 in 1974 EPA Methods Manual. Also see EMP. For KDmi, use 0.1 amounts to make 200 ml.</td>
</tr>
<tr>
<td>267 g potassium sulfate</td>
<td>KDmi 10 ml</td>
<td>180 ml</td>
<td></td>
</tr>
<tr>
<td>400 ml concentrated sulfuric acid</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4 g mercuric oxide</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 ml concentrated sulfuric acid for</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>20% solution</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>NITRATE + NITRITE and NITRATE</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Concentrated ammonium chloride - EDTA</td>
<td>480 ml</td>
<td>8640</td>
<td>Dilute this for 800 ml dilute solution per student.</td>
</tr>
<tr>
<td>solution - 14 liters</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>130 g ammonium chloride</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>17 g disodium ethylenediamine tetraacetate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>~10 ml concentrated ammonium hydroxide</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Color reagent - 500-ml:</strong></td>
<td>12 ml</td>
<td>216 ml</td>
<td>For pH adjustment</td>
</tr>
<tr>
<td>50 ml concentrated phosphoric acid</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 g sulfanilamide</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.5 g (1-naphthyl)-ethylenediamine</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>dihydrochloride</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Concentrated ammonium hydroxide</td>
<td>~10 ml</td>
<td>~180 ml</td>
<td>For pH adjustment</td>
</tr>
<tr>
<td>Concentrated hydrochloric acid</td>
<td>~10 ml</td>
<td>~180 ml</td>
<td>For pH adjustment</td>
</tr>
<tr>
<td>6 N hydrochloric acid</td>
<td>60 ml</td>
<td>1080 ml</td>
<td></td>
</tr>
<tr>
<td>600 ml concentrated hydrochloric acid/</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1200 ml</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2% copper sulfate solution:</td>
<td>200 ml</td>
<td>3600 ml</td>
<td></td>
</tr>
<tr>
<td>80 g copper sulfate/4 l</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stock potassium nitrate solution:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3/609 g potassium nitrate/500 ml</td>
<td>11 ml</td>
<td>11 ml</td>
<td>Dilute this for 25 ml of 1 mg/l and for up to 15 ml of 0.01 mg/l and for 50 ml sample per student.</td>
</tr>
<tr>
<td>(1 ml chloroform as preservative)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DESCRIPTION</td>
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</tr>
<tr>
<td>-------------</td>
<td>-------------</td>
<td>-------------</td>
<td>---------</td>
</tr>
<tr>
<td>Stock potassium nitrite solution: 3.036 g potassium nitrite/500 ml (1 ml chloroform as preservative)</td>
<td>10 ml</td>
<td>10 ml</td>
<td>Dilute this for up to 15 ml of 0.01 mg/l and for 50 ml sample per student</td>
</tr>
<tr>
<td>Cadmium coarse powder, 40-60 mesh can be filed off stick. Should pass a 10 mesh sieve and be retained on a 40, then a 60 mesh sieve.</td>
<td>20 g</td>
<td>360 g</td>
<td>Powder is available from M Laboratories, 500 Executive Blvd., Elmsford, NY 10523.</td>
</tr>
<tr>
<td>pH buffers 4, 7 and 10</td>
<td>~100 ml of each</td>
<td>~1500 ml of each</td>
<td>For students to calibrate pH meters.</td>
</tr>
<tr>
<td>OIL AND GREASE</td>
<td>Amounts are minimum</td>
<td></td>
<td>See p. 230 in 1974 EPA Methods Manual. Also see EMP.</td>
</tr>
<tr>
<td>1,1,2-trichloro-1,2,2-trifluoroethane Du Pont*, Freon 113 TF or Allied Chemical*, Genosolv D</td>
<td>125 ml</td>
<td>2250 ml</td>
<td></td>
</tr>
<tr>
<td>Anhydrous sodium sulfate</td>
<td>3 g</td>
<td>54 g</td>
<td></td>
</tr>
<tr>
<td>SPECTROPHOTOMETER</td>
<td>Amounts are minimum</td>
<td></td>
<td>See IPW for preparation.</td>
</tr>
<tr>
<td>See of synthetic phosphorus standards made from a methylene blue indicator which is diluted to give a series of standards for each instrument.</td>
<td>0.1 g</td>
<td>This is enough for several sets of &quot;standards.&quot;</td>
<td></td>
</tr>
<tr>
<td>TOTAL PHOSPHORUS or ORTHOPHOSPHATE</td>
<td>Amounts are minimum</td>
<td></td>
<td>See p. 252 in 1974 EPA Methods Manual. Also see EMP.</td>
</tr>
<tr>
<td>HCl hydrochloric acid (to clean glassware): 1500 ml concentrated hydrochloric acid/3000 ml</td>
<td>150 ml</td>
<td>2700 ml</td>
<td></td>
</tr>
<tr>
<td>40 N sodium hydroxide solution: 40 g sodium hydroxide/100 ml</td>
<td>5 ml</td>
<td>100 ml</td>
<td>Use 10 ml for 5 ml of 0.1 N per student.</td>
</tr>
</tbody>
</table>

*Mention of a specific brand name does not constitute endorsement of the U.S. Environmental Protection Agency.*
<table>
<thead>
<tr>
<th>DESCRIPTION</th>
<th>EACH STUDENT</th>
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</tr>
</thead>
<tbody>
<tr>
<td>11 N sulfuric acid:</td>
<td>4 ml</td>
<td>82 ml</td>
<td>Use 10 ml for 5 ml of 1.1 N per student.</td>
</tr>
<tr>
<td>31 ml concentrated sulfuric acid/100 ml</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Combined reagent - 3 liters:</td>
<td>150 ml</td>
<td>2700 ml</td>
<td>Includes 125 ml per student to check glassware.</td>
</tr>
<tr>
<td>210 ml concentrated sulfuric acid</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.6857 g antimony potassium tartrate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>20 g ammonium molybdate</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.76 g ascorbic acid</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>21.6 g ammonium persulfate</td>
<td>1.2 g</td>
<td>21.6 g</td>
<td></td>
</tr>
<tr>
<td>Stock phosphorus solution:</td>
<td>20 ml</td>
<td>40 ml</td>
<td>Dilute this for up to 50 ml of 1.0 µg /l per student. Make 30 ml per student if they dilute the stock.</td>
</tr>
<tr>
<td>0.2197 g potassium dihydrogen phosphate</td>
<td>30 ml</td>
<td>540 ml</td>
<td></td>
</tr>
<tr>
<td>Potassium dihydrogen phosphate</td>
<td>0.2197 g</td>
<td>4 g</td>
<td>For analytical balance exercise. Students can make own stock solution with this.</td>
</tr>
<tr>
<td>SAMPLES</td>
<td>Amounts are minimum</td>
<td></td>
<td>Distribute in plastic BOTTLES except the COD sample.</td>
</tr>
<tr>
<td>Sample, Ammonia Distillation, macro, followed by Ammonia Titration:</td>
<td>400 ml</td>
<td>9 l</td>
<td>Test range is 1 to 25 mg/liter ammonia nitrogen</td>
</tr>
<tr>
<td>WWT plant effluent with at least 1 mg/l NH₃-N</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample, Ammonia Electrode:</td>
<td>100 ml</td>
<td>2.5 l</td>
<td>Dilute the standard ammonium chloride solutions.</td>
</tr>
<tr>
<td>Synthetic, between 0.03 to 0.1 mg/l NH₃-N</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Synthetic, between 0.1 to 1.0 mg/l NH₃-N</td>
<td>100 ml</td>
<td>2.5 l</td>
<td></td>
</tr>
<tr>
<td>Sample, Chemical Oxygen Demand:</td>
<td>75 ml</td>
<td>2 l</td>
<td>Distribute in glass BOTTLES.</td>
</tr>
<tr>
<td>WWT plant effluent of 5-50 mg/l COD.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DESCRIPTION</td>
<td>QUANTITY FOR:</td>
<td>CLASS OF 18</td>
<td>REMARKS</td>
</tr>
<tr>
<td>----------------------------------------------------------------------------</td>
<td>----------------</td>
<td>-------------</td>
<td>-------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Sample, Kjeldahl Digestion, micro, followed by Ammonia Distillation, micro, and Ammonia Nesslerization: WWT plant effluent of 0.03-1.0 mg/l NH₃-N</td>
<td>50 ml</td>
<td>1.5</td>
<td>Dilute standard potassium nitrate and potassium nitrite solutions.</td>
</tr>
<tr>
<td>Sample, Nitrate + Nitrite and Nitrate: Synthetic sample up to 1 mg/liter NO₂ + NO₃</td>
<td>50 ml</td>
<td>1.5</td>
<td>Test range is 0.01 to 1.00 mg/liter P</td>
</tr>
<tr>
<td>Example: 40 ml NO₃ standard plus 40 ml NO₂ standard into 1 liter gives 0.8 mg/l total.</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample, Oil and Grease: WWT plant effluent, acidified to pH 2</td>
<td>14</td>
<td>2</td>
<td>Test range is 5 to 1000 mg/liter extractable material</td>
</tr>
<tr>
<td>Sample, Total Phosphorus: WWT plant effluent of known mg/liter P so dilutions can be assigned</td>
<td>50 ml</td>
<td>1.5</td>
<td>Test range is 0.01 to 1.00 mg/liter P</td>
</tr>
</tbody>
</table>
4. Instructional Resources

a. Introduction

Most training institutions will make the fullest possible use of pre-existing 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) The Instructional Package Worksheets (IPWs) in Part II of this Manual and the student reference text refer to a number of different instructional resources. These include:
   a) The student reference text itself;
   b) Audiovisual training aids; and
   c) Supportive references

2) Instructional resources are discussed in this section from the viewpoint of the sources of the materials:
   a) Resources developed by United States Environmental Protection Agency (U.S. EPA)
   b) Resources developed by other sources; and,
   c) Resources already in possession of the training institution conducting this course.

b. Instructional Resources Developed by U.S. EPA

1) Student Reference Text and Staff Guide for the course "Effluent Monitoring Procedures Nutrients":
   a) While present supplies last, a sample copy (can be duplicated) is available on specific request to:

   National Training and Operational Technology Center
   ATTN: Training Information Clerk
   U.S. Environmental Protection Agency
   Cincinnati, Ohio 45268

   b) NTOTC has negatives of the Text and Guide which are available for temporary loan on request of a sponsoring Agency wishing to duplicate the materials.
c) NTIS can supply copies of the Text and Guide. A paper copy of the Text (PB-261-290/AS) costs $9.75. Contact NTIS for the identification number and cost of a paper copy of this Guide.

A microfiche copy of either the Text or Guide is also available from NTIS at $3.00 each.

U.S. Department of Commerce
National Technical Information Service
5285 Port Royal Road
Springfield, Virginia 22151

d) Your state agency may be able to arrange a direct supply of the texts and/or guides. Before ordering reproducing materials, you might consult with your state agency about this possibility.

2) Audiovisual Training Aids Developed for the Course:

a) What is available, according to topic:

(1) Use of a Spectrophotometer
   (a) X-8, Slides, unassembled-16

   (b) XT-51, Tape/Slide Unit, "Use of the Spectronic 20 Spectrophotometer", 10 minutes, 35 slides

   (c) TC-57, Videocassette Tape Unit "Use of a Spectrophotometer", 21 minutes

(2) Preparation of Calibration Graphs:
   (a) X-9, Slides, unassembled-6

(3) Determination of Total Phosphorus (asP) or Orthophosphate (asP); Single Reagent Method
   (a) OT-7, Overhead Projectuals-7

   (b) XT-44, Tape/Slide Unit, "Determination of Phosphorus", 15 minutes, 65 slides

(4) Determination of Chemical Oxygen Demand
   (a) OT-8, Overhead Projectuals-4

   (b) X-7, Slides, Assembled-4
(5) Determination of Total Kjeldahl Nitrogen
   (a) OT-9, Overhead Projecturals-9
   (b) Two Videocassette Tape Units are proposed for development by Charles County Community College

(6) Nitrogen, Ammonia Determination (wet)
   (a) XT-67, Tape/Slide Unit, "Determination of Ammonia Nitrogen", 8 minutes, 45 slides

(7) Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method
   (a) OT-10, Overhead Projecturals-11
   (b) TC-58, Videocassette Tape Unit, "Preparation of the Cadmium Reduction Column", 21 minutes

(8) Determination of Oil and Grease
   (a) XT-56, Tape/Slide Unit, "Determination of Grease and Oil", 15 minutes, 54 slides
   (b) TC-55, Videocassette Tape Unit, Part I, "Basic Skills", 17 minutes and Part II, "Use of a Separatory Funnel", 13 minutes
   (c) TC-56, Videocassette Tape Unit, "Distillation Set-Up", 15 minutes

(9) Determination of Ammonia by an Ammonia Selective Electrode
   (a) X-10, Slides, unassembled-6

b) How to request loan of audiovisual training aids:

(1) All items described in b.2)a) 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 (next page). Send requests to NTOTC at the address given in b.1)a) above.
REQUEST FOR LOAN
AUDIOVISUAL INSTRUCTIONAL UNIT

Title and Catalog No ________________________________

Intended Use ______________________________________

Preferred Date of Use ______________________________

Alternate Date _____________________________________

BORROWER'S NAME __________________________________

Title ______________________________________________

Organization ________________________________________

Address ____________________________________________________________________________________________

Phone Number (include Area Code) ______________________ (Zip)

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 (Cin), (8-74)

(2) It is urged that materials desired from NIESC 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.
(3) Requests should be timely. To assure effective delivery in time for use in the course, requests should be received at NTOTC at least 41 days prior to the course date. The Center 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.

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

(5) With returned borrowed training resources, it is requested that the user provide the Center 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 use of such resources should include the number of students with whom the material was used.

3) Supportive References:

a) Manual: EPA-EMS L, "Methods for Chemical Analysis of Water and Wastes". This is the reference source of all the methods presented in this course in the Effluent Monitoring Procedure (EMP) format. Address requests for a copy to:

U.S. EPA
Office of Technology Transfer
Industrial Environmental Research Laboratory
Cincinnati, Ohio 45268

b) AV Catalog: NTOTC, "Audiovisual Instructional Units". This is a catalog of slide-tape instructional units developed by the Center. 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 in b. 1) a) above.

c) IRIS: A Water Quality Instructional Resources Information System has been developed through an EPA training grant. The "Master Reports" contain availability information and descriptions for 2300 entries of instructional and resource materials. These are printed matter, slides, films, slide-tape units and video tapes developed by varied sources for training personnel involved in all aspects of water quality assessment and control. Thus, IRIS serves as an information source of available, water quality training resources covering a wide range of subjects.
The user does not need data processing equipment to use the system. Four of the commonly used elements of IRIS (Users Manual, Tables, Master Report, Subject Index) are currently available.

1) While present supplies last, a set is available qualifying educational institutions and training agencies from NTOTC at the address given in b.1) above.

2) Paper copies can be ordered as a set of four volumes from NTIS (PB-262-223/AS, Set, 6120 pp. "Water Quality Instructional Resources Information System, Volumes I through IV) for $31.00. The NTIS address is given in b.1)c) above. Microfiche copies cost $12.00 per set.

3) Paper or microfiche copies of individual volumes can also be ordered from NTIS at the address given in b.1)c) above using this information:

   (a) PB-262-224/AS, 99 pp., "Water Quality Instructional Resources Information System, Volume I - Users Manual" @ $5.00. (microfiche $3.00)

   (b) PB-262-225/AS, 96pp., "Water Quality Instructional Resources Information System, Volume II - IRIS Tables" @ $5.00 (microfiche $3.00)

   (c) PB-262-226/AS, 494 pp., "Water Quality Instructional Resource Information System, Volume III - Identification Number Master Report" @ $12.50 (microfiche $3.00)

   (d) PB-262-227/AS 434 pp., "Water Quality Instructional Resources Information System, Volume IV - Subject Index" @ $11.75 (microfiche $3.00)

Instructional Resources Developed by Other Sources

1) Minimum technical references which should be in possession of the institution include:


2) Audiovisual and Other Training Aids

a) A wide variety of training resources is listed in the EPA sponsored publication, "Water Quality Instructional Resources Information System" (IRIS), cited in b. 3) c) above.

b) Information on sources of the listed items is provided.

c) Training institutions having information about training resources applicable to this course, which are not currently listed in IRIS, are invited to relay this information to the Director, National Training and Operational Technology Center. Such resources, as applicable, will be made known to other organizations which could benefit from their use.

d) Instructional Resources Already in Possession of the Training Institution

1) Many training organizations prefer to develop their own texts and audiovisual training resources.

2) To the extent that these resources can be released for free reproduction and use by others, institutions are invited to make such resources available to other training organizations.

3) The National Training and Operational Technology Center is prepared to serve as a focal point for making information about such resources widely available, provided that copyright or other restrictions on reproduction do not limit availability of such materials.

a) Before encouraging other training institutions to use such resources in relation to this Course, elements of EPA will:

   (1) Review the training resources to determine whether the instruction is consistent with existing laws, regulations, and Agency policy;

   (2) Review the resource for technical validity and educational quality.

b) Materials found suitable by EPA would be recommended to other institutions known to be presenting this Course.

4) All training resources referred to NTOTC as available for use by others will be added to the overall inventory listing cited in IRIS, b. 3) c), above. It is hoped that a mutually supportive activity in this area will, in time, result in:

   a) making IRIS a diversified, total resource system for training materials which will be of the highest technical quality;

   b) offering training institutions a wide variety of types of training resources;

   c) reducing the amount of duplication of effort that so often results from lack of information on what is available, from what sources, and how obtained.
PART I – COURSE PLANNING AND MANAGEMENT

C. Secretarial Support

The key role of the office worker(s) designated "Course Secretary" cannot be overemphasized.

This function has many elements including being the right arm of the Course Coordinator, being Course Registrar, being Course Secretary and being a "Course Watchdog" to give the alarm when essential milestone stages of course planning and preparation are being overlooked.

In this section, these elements are considered under the headings: Course Records and Record-keeping, Suggested Student Registration Procedures and Printed and Reproduced Materials - Summary.

1. Course Records and Record-keeping
   a. General Considerations

   1) Complete, detailed, and accurate records should be established for each course presentation. Each course record will be a separate file. In addition to the individual course files, it may be necessary to establish a finder-system for locating the records of individual students.

   2) Response to Inquiries about Former Students

      a) Students enroll in this course in order to acquire necessary knowledge and skills to perform the self-monitoring procedures required for municipal effluents.

      b) In many, if not all cases, satisfactory completion of this course will be a factor in the accreditation of individuals to perform the analyses and measurements required for compliance with NPDES Permits.

   c) It is anticipated that numerous inquiries from former students and from regulatory agencies will be addressed to the training institution. Typical requests for information may include any or all of the following:

      (1) Verification of attendance and satisfactory completion of training;

      (2) Identification of the specific analyses and measurements covered in the course, as well as designation of the method which was taught;

      (3) Quality of student performance in the course;
(4) Documentation of any specific analyses, tests, or measurements in which the student did not meet the required standard of performance, and the nature of such failure; and

(5) Documentation of any other events which made the student unusual. This could be a record of exceptionally high performance, or it could be a record of any specific difficulty which arose in connection with the student, within or outside the scheduled training activities.

3) Reports

Most training institutions require submission of periodic reports on progress and achievements. It is safe to predict that management or cognizant regulatory agencies from time to time will call for information not provided in routine reports. If such demands are to be met, complete course records will be the most reliable source for such information.

4) Retention of Records

The length of time course record files should be retained is uncertain, and must be determined by each institution.

Institutions having a system of archives for inactive files may find it convenient to retain course records in active office files for approximately two years, then retire them to archives storage. Institutions not having archives storage probably should retain the complete file on each course presentation for at least five years.

b. Contents of Course Files

1) In the planning and development stage, and until completion of each course presentation, course records are kept most effectively in two sections.

These are:

a) A file folder, kept in the filing cabinet or in the desk of the Course Secretary; and,

b) A student record notebook, usually a 3-ring binder, kept on the Course Secretary's desk, or in a convenient bookcase.

Both sections of the Course files should be maintained by the Course Secretary, and should be made available to other staff members under rigid controls providing for direct examination and immediate return. After completion of the course, the two sections can be combined in a single large file packet for future retention.
2) The file folder is best suited for such records as:

a) Copies of all correspondence, memoranda, and records of telephonic conferences related to course planning and development;

b) Copies of course schedules;

c) Records of equipment and supply acquisition for the course, through purchase or through loan (with information and records on return to owner);

d) Records of staff assignments, classroom and laboratory reservations;

e) Copy of course announcement and/or description (See pages 12-7 and 8);

f) Sample record copies of all routine informational material sent to students accepted for training; (See pages 12-15 through 18);

g) Records of arrangements for travel personnel and transportation of equipment and supplies, arrangements for field facilities, and other records pertaining to a field course; and

h) Course evaluation commentaries by Course Coordinator and other staff members as appropriate.

3) The student record notebook is best suited as a vehicle for all records and copies of communications related to individual students. This may be organized effectively in a 3-ring notebook, containing separator sheets with alphabetical tabs. The personal records of each student will be retained under the alphabetical tab corresponding with his last name. In the student record notebook may be found:

a) At the front (before the "A" of the series of tabbed dividers):

(1) A registration summary sheet showing record of standard communications with each accepted student, fees paid, etc... (See page 12-19);

(2) A waiting list summary sheet showing record of standard communications with each student placed on a waiting list prior to the course due to early maximum enrollment. (See page 12-20)

(3) A non-attendance summary sheet showing record of students who applied for admission but could not be admitted for lack of qualification, or due to an already-filled class. This summary also is used to identify applicants who applied for admission, who were admitted, and who failed to appear without due explanation ("no shows"). (See page 12-21);
(4) A summary sheet recording student performance (acceptable or not acceptable) for each of the units of instruction in the course. (Such a summary worksheet has not been developed at the writing of this Guide).

b) In the alphabetical section of the student record notebook, each student's personal record will contain such items as:

1. The application for admission to training (See pages 12-9 and 10);

2. Copies of all correspondence with the student (See pages 12-11 through 14), except for the routine local information sheets (See pages 12-15 through 18), one set of which is kept in the loose file folder;

3. Record copies of student quizzes, data sheets, and other individual records of class performance provided by Instructor. (See pages 12-25 through 39).

4. Copy of the certificate awarded at end of course which is a record of the measurements completed by the student in a satisfactory manner. (See page 12-24);

5. Documentation of any information about the student judged to be of possible future concern or inquiry. (An example of such a sheet had not been developed at the time of writing this Guide).
2. Suggested Student Registration Procedures

a. Introduction

1) Purpose

Formal registration and enrollment procedures are intended to assure that:

a) The class consists of students for whom the training is intended and designed;

b) The accepted students meet minimum knowledge and skills required for reasonable assurance of satisfactory completion of the course;

c) Accepted students are provided with adequate pre-training information so that they will make their personal arrangements and travel schedules to assure arrival at the appointed time and place, with full participation throughout the program of training;

d) The size of the class is in accordance with the course plan; and

e) Those not accepted for training are provided with suitable advice which can lead to future admission.

2) Alternative Approaches to Registration

Three different approaches to registration are considered here, though only the first is described in detail. Most details of the second and third identified alternatives can be inferred through study of the first alternative. The three approaches considered are:

a) Registration by priority of receipt of application;

b) Registration by comparative evaluation of all applicants; and

c) Registration for special course offering at request of another organization. Students are nominated by the requesting organization and are accepted without further evaluation of entry-level qualifications by the training institution.

b. Registration by Priority of Receipt of Application (Recommended)

1) Applicant

a) Receives course announcement;

b) Completes application and secures internal approvals as required in his own organization;

c) Sends application to registration office of the institution conducting the training.
2) Course Secretary
   a) Receives application;
      b) As received, checks status of registration for availability of
         class space, and makes appropriate notation on the application
         or on attached transmittal slip;
      c) Depending on b), records application either in the registration
         summary, or the waiting list summary, in front of student notebook;
      d) Delivers application to Course Coordinator.

3) Course Coordinator
   a) Evaluates the student application and the space availability
      situation;
   b) Directs the Course Secretary by notation on the application to:
      (1) Accept the applicant, or
      (2) Notify the applicant of closed class enrollment, or
      (3) Refer the applicant to the "Basic Laboratory Skills" course
           before entry into this course;
   c) Prepares a special letter for non-admissible applicants
      (or memorandum record of other form of communication with
      applicant) which sets forth the reasons why the applicant
      is being rejected. Because of the compulsory nature of
      the self-monitoring tests on municipal effluents for NPDES
      Permit compliance, rejection of a bona fide applicant may
      be a very serious matter, and should be handled with care.

4) The Course Secretary
   a) Prepares a standard letter or special letter as directed, and
      obtains signature of Course Coordinator;
   b) Places a file copy of the letter and the application in the
      appropriate place under the alphabetical tab section of the
      student record notebook;
   c) Mails the original letter to the applicant, and records the
      the date of mailing in the appropriate place in the summary
      records at the front of the student notebook.
   d) Approximately 30 days before the course, mails to each accepted
      applicant a standard communication, consisting of:
      (1) A form letter of welcome to the course, including information
          on starting and closing dates and hours of the course, direc-
          tions and how to proceed to the classroom area, and related
          information (See page 12-14); and
(2) Local information helpful to outside visitors, such as hotel/motel information, local transportation and schedules; a schematic map of the area, and related information. (See pages 12-15 through 18);

e) Records the mailing of the standard packet on the registration summary sheet.

f) When applications are still being accepted within 30 days before the start of the course, includes the general informational material with the letter of acceptance for admission.

g) On the first day of the course:

(1) Obtains a detailed registration card from each student. Some institutions may dispense with this record, though it can be of value in report preparation (See page 12-22);

(2) Prepares any registration tallies required by requesting organization(s) and/or administrative regulations. (See page 12-23);

(3) Prepares a class roster of those in attendance, distributes to class, staff, and keeps a permanent record copy in course files;

(4) Records any "no shows" (applicants accepted for training but who did not appear) on the student non-attendance summary record sheet. (See page 12-21)

c. Registration by Comparative Evaluation of all Applicants

1) Applicant

a) Receives course announcement;

b) Completes application and secures internal approvals as required in his own organization;

c) Mails application to registration office of the institution conducting the training.

2) The Course Secretary

a) Receives the applications;

b) Records receipt of application in a summary record in student record notebook;

c) Files application in student notebook;

d) Sends standardized letter acknowledging the application, and briefly explaining the registration procedure, with assurance that decision on admission will be announced not less than 30 days prior to start of the course;
e) Delivers all applications to the Course Coordinator, approximately 35 days before start of the course.

3) The Course Coordinator
a) Reviews and evaluates all applications;
b) Selects students to be admitted for training;
c) Directs Course Secretary to send appropriate standardized letters and information packets as described in b. above;
d) Special note should be taken of the particular attention which should be given to rejected applicants. See b., 3), c). above.

4) The Course Secretary
a) Sends communications;
b) Prepares records and student files as described in b.4) above.

d. Registration for Special Course Offering

Here a requesting organization has designated a student body which it wishes to have trained. It is the duty of the training institution to provide the requesting organization with admission standards for the course. It becomes the duty of the requesting organization to screen its candidates for conformance to these standards, and to provide the training institution with the names of the students to be trained. The requesting organization usually notifies the students.

1) For record purposes, it is best that students complete a course application form, though it will not be evaluated as in b. and c. above.

2) On receipt in the training institution, the Course Secretary makes the necessary entries showing record of receipt and class composition. Files are kept in the usual way. Approximately 30 days before the course, the individual standardized welcome and information packets are sent to students in the usual way. The training organization follows its usual practices in preparation of records, rosters, and any other data required for reports.
3. Printed and Reproduced Materials - Summary

a. General Information

In addition to the student reference text, standardized letters and administrative forms/materials, presentation of this course also requires calculation forms, laboratory data sheets, and graph forms which must be prepared in quantity by the Course Secretary.

1) In the following summarizing table, all of the standardized materials noted above are identified and supported with additional information on due date, the number to be prepared (for a class of 18 students), and the ultimate fate of the materials in permanent course records. Institutions offering this course may find it necessary to add to or to modify these standardized materials. It is suggested that plans to do so be noted on the summarizing table, with samples or examples provided on separate pages.

2) A sample or example of each item listed (except the student reference text) is shown following the summarizing table.

a) Samples can be copied directly, if meeting requirements of the training institution.

b) The examples are shown in recognition that a corresponding item probably will be needed by the training institution, but probably will have to be modified to fit the situation.

b. Responsibilities for Printed and Reproduced Material

1) Course Coordinator

a) Reviews the administrative materials for conformance to the requirements of the regulatory authority;

b) Makes modifications as necessary to the samples and examples provided in this Guide;

c) Decides upon and designs any additional administrative documents or records needed; and

d) Provides the Course Secretary with complete identification of material to be copied directly or to be modified, and also provides samples of any new material required for course administration.

2) Instructors

a) Review all materials identified for the procedures for which they have instructional responsibility;

b) Design new supportive instructional material as required; and
c) Provide the Course Secretary with complete information on material to be copied directly or to be modified, and also provide samples of any new material required for student instruction.

3) Course Secretary

a) Receives from Course Coordinator and Instructors identification of existing materials, samples of modified and new material;

b) Adds to the summarizing table, in the appropriate locations, the identifying information, together with the supporting information on due date, quantity, confidentiality, and ultimate fate of any new or revised material designed by Course Coordinator or by Instructors;

c) Reproduces, or arranges reproduction of, the needed course materials so that they will be available for use at the time and place required.

c. Special Warnings

1) All staff members should be particularly alert to adjustments in "Due Date" which must be made when the course is conducted in the field, if training equipment and supplies must be shipped to the course site.

2) Preparation of these training materials is a potential source for great difficulty in course development and presentation. Few activities in course planning and development require a greater amount of effective teamwork among all staff members. The greatest problem here is one of timing.

a) All staff members must provide necessary information and samples of new or modified materials with adequate lead time to meet "Due Dates." The amount of lead time is not specified here; this will vary from one institution to another.

b) The Course Secretary must be diligent in advising Course Coordinator and Instructors of impending logistic problems if delays occur in submission of materials, and must give prompt attention to printing or reproduction of needed materials when delivered by staff members.

c) The author of this guide ruefully confesses that the worst and most frequent breakdowns in this area usually are the result of belated delivery of needed material from Instructional Staff to the Course Secretary.
<table>
<thead>
<tr>
<th>Description</th>
<th>Lesson</th>
<th>When Needed</th>
<th>Number to be Prepared</th>
<th>Confidential?</th>
<th>Permanent Record?</th>
<th>Remarks</th>
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<tr>
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<td>Course Announcement</td>
<td>-</td>
<td>6 months before course</td>
<td>Indeterminate</td>
<td>No</td>
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<td>Distribute to target group 6 months before course.</td>
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<td>1 copy</td>
<td>Same as announcement. Can be used in conjunction with chronological</td>
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<td>Application for Admission</td>
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<td>No</td>
<td>No</td>
<td>Usually part of course announcement. May be separate sheet.</td>
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<tr>
<td>Prerequisite Verification</td>
<td>-</td>
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<td>No</td>
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<td>Standard Letter: Acceptance</td>
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<td>5 months before course</td>
<td>100</td>
<td>No</td>
<td>No</td>
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<tr>
<td>Standard Letter: Standby: Full Class</td>
<td>-</td>
<td>5 months before course</td>
<td>100</td>
<td>No</td>
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<tr>
<td></td>
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<td>Standard Letter: Referral to Basic</td>
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<td>100</td>
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<tr>
<td>Lab Skills Course</td>
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<td>Standard Letter: Welcome and Local</td>
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<td>1 copy</td>
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<tr>
<td>Information: Hotels/Motels,</td>
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<td>Transportation Schedule,</td>
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<tr>
<td>Schematic Area Map</td>
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<td>Classroom Location</td>
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<td>Registration Summary Record</td>
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<td>1</td>
<td>No</td>
<td>Yes</td>
<td>In Registrar's three-ring notebook.</td>
</tr>
<tr>
<td>Description</td>
<td>Lesson</td>
<td>When Needed</td>
<td>Number to be Prepared</td>
<td>Confidential</td>
<td>Permanent Record?</td>
<td>Remarks</td>
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<td>Waiting List Summary</td>
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<td>1</td>
<td>No</td>
<td>Yes</td>
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<tr>
<td>Non-attendance Summary</td>
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<td>First day of course</td>
<td>1</td>
<td>No</td>
<td>Yes</td>
<td>In Registrar's three-ring notebook</td>
</tr>
<tr>
<td>Trainee Registration Card</td>
<td></td>
<td>First day of course</td>
<td>20</td>
<td>No</td>
<td>Yes</td>
<td>Institution's Records</td>
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<td>Registration Tally and Class Roster</td>
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<td>First day of course</td>
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<td>No</td>
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<tr>
<td>Course Certificates</td>
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<td>18</td>
<td>No</td>
<td>Yes</td>
<td>In student file</td>
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<tr>
<td>Student Performance Summary Record</td>
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<td>18</td>
<td>Yes</td>
<td>Yes</td>
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<tr>
<td>Classroom/Laboratory Use of a Spectrophotometer</td>
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<td>Day 1</td>
<td>20</td>
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<tr>
<td>Summary of Student Performance</td>
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<td>18</td>
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<td>Keep in students' files.</td>
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<td>Preparation of Calibration Graphs</td>
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<td>18</td>
<td>No</td>
<td>Yes</td>
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<td>Graph Paper</td>
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<td>Day 1</td>
<td>20</td>
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<td>18</td>
<td>No</td>
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<td>Lesson</td>
<td>When Needed</td>
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<td>Confidential?</td>
<td>Permanent Record?</td>
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<tr>
<td>Total Phosphorus or Orthophosphate</td>
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<tr>
<td>Example Data Sheet</td>
<td>2 of 5</td>
<td>Day 2</td>
<td>20</td>
<td>No</td>
<td>Yes</td>
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<td>EMP Calibration Graph</td>
<td>4 of 5</td>
<td>Day 2</td>
<td>20</td>
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<td>Yes</td>
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<td>Standardization of FAS Calculation Sheet</td>
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<td>Day 3</td>
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<td>Total Kjeldahl Nitrogen</td>
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<td>Keep in students' files.</td>
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<tr>
<td>Description</td>
<td>Lesson</td>
<td>When Needed</td>
<td>Number to be Prepared</td>
<td>Confidential?</td>
<td>Permanent Record?</td>
<td>Remarks</td>
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<td><strong>Nitrogen Ammonia</strong></td>
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<td>Day 3</td>
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<td><strong>Nitrate + Nitrite Nitrogen</strong></td>
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<td>Example Data Sheet</td>
<td>7 of 11</td>
<td>Day 4</td>
<td>20</td>
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<td>EMP Calibration Graph for NO₂ + NO₃⁻N</td>
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<td>EMP Calibration Graph for NO₂⁻N</td>
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<td>Day 4</td>
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<tr>
<td><strong>Oil and Grease</strong></td>
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<tr>
<td><strong>Ammonia-Electrode</strong></td>
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<tr>
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<td></td>
<td>Final day of course</td>
<td>18</td>
<td>No</td>
<td>Yes</td>
<td>Keep in students' files.</td>
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</tbody>
</table>
ANNOUNCING

USEPA SPONSORED INSTRUCTOR TRAINING FOR PERMIT COMPLIANCE

The National Training and Operational Technology Center has developed a series of "package" courses for training municipal treatment plant personnel how to sample, measure, and analyze their wastewater discharges so as to comply with NPDES Permit requirements. Instructional materials for each course have been prepared in modular form, with each module containing detailed step-by-step procedures for the performance of a specific monitoring task. A description of each course so far developed appears below.

The National Training and Operational Technology Center provides tuition-free courses for instructors engaged in this type of training in Federal, State, and Local water pollution control programs, and in educational institutions. In these courses, the student-instructors perform each self-monitoring task. In addition, they become thoroughly familiar with the organization and recommended use of the instructional materials, with training aids and other technical assistance available from USEPA for this type of training, with the planning and implementation of intensive training programs, with recommended institutional techniques, and with USEPA regulatory requirements for NPDES self-monitoring. These latter subjects are dealt with in depth only in course 164.1 which is an 8 day offering for student-instructors. It is recommended that interested instructors attend this course prior to any of the others.

164.1 Effluent Monitoring Procedures: Basic Parameters for Municipal Wastewaters 8 days (8 days for student-technicians)

This course is intended for municipal wastewater treatment plant technicians and others engaged in analysis of wastewater treatment plant effluents for compliance with effluent discharge permits issued under the National Pollutant Discharge Elimination System. Upon completion of training, the student technician will be able to collect samples, perform the tests, and report results for 

| BOD | COD | pH | Suspended Solids | Total Solids | Ammonia | Nitrite | Nitrate
|-----|-----|----|----------------|-------------|---------|--------|--------
|     |     |    |                |             |         |        |        |

...and other required parameters, using both manual and automatic laboratory test procedures, conform to Agency-approved methods as promulgated in the Code of Federal Regulations.

164.2 Effluent Monitoring Procedures: Metals Analysis 8 days

This course is designed for municipal wastewater treatment plant technicians who are responsible for performing selected metals analyses on treatment plant effluents. Participants will perform selected metal analyses including iron, copper, lead, mercury, sodium, and zinc. Classroom instruction is limited to information about performing these analyses and reporting the results. Most of the time is given to laboratory experiences for the trainee who gains detailed, stepwise procedures to analyze typical samples. Procedures will be in conformance with Agency-approved methods as promulgated in the Code of Federal Regulations.

164.3 Effluent Monitoring Procedures: Nutrients 8 days

This course is designed for municipal wastewater treatment plant technicians who are responsible for self-monitoring of nutrient concentrations in treated effluents to comply with requirements of discharge permits issued under the National Pollutant Discharge Elimination System. Upon completion of training, the participants will be able to perform selected analyses for Chemical Oxygen Demand, the Nitrogen Species (Total, Ammonia, Nitrate, Nitrite), Total Phosphorus. The test procedures conform to Agency-approved methods as promulgated in the Code of Federal Regulations.

164.4 Effluent Monitoring Procedures: Flow Measurement & Sampling Technique 8 days

This course is designed for the treatment plant operator or technician who is required to monitor effluent discharges under a National Pollutant Discharge Elimination System (NPDES) Permit, and who has had little or no previous experience in measuring wastewater flows. Following classroom discussions and demonstrations, participants measure flows and collect samples at a wastewater treatment plant using both manual methods and automatic devices, and also measure effluent parameters on-site, using portable field instrumentation.

For additional information about these courses, call the NTOTC Registry - 512-444-2801.
EFFLUENT MONITORING PROCEDURES:
NUTRIENTS (Course 1643).

5 Days

November 3-7, 1975
June 28-July 2, 1976

This course is designed for the treatment plant operator or technician who is required to monitor effluent discharges under a National Pollutant Discharge Elimination System (NPDES) Permit, and who has had little or no previous experience in wastewater analysis.

The course includes procedures for measuring Total Phosphorus (as P), Chemical Oxygen Demand, Kjeldahl (Total) Nitrogen, Ammonia (as N), Organic Nitrogen by difference of Kjeldahl N and Ammonia N, Nitrate-Nitrite (as N), Nitrite (as N) by difference of Nitrate-Nitrite N and Nitrite N, Nitrate (as N) by difference of Nitrate-Nitrite N and Nitrite N, and Oil and Grease. The course also includes procedures for related skills--using a spectrophotometer and preparing a calibration graph.

During the course, the student will perform an approved analytical procedure for each of the measurements. At the conclusion, he will be given a certificate verifying which measurements he performed in a satisfactory manner.

PRE-REQUISITES

Self-Monitoring Procedures: Course I - Basic Laboratory Skills or equivalent experience is pre-requisite for the course. A "Student Skills Checklist," signed by the applicant's employer, must be submitted before an application for the course can be processed.

Tuition: $175.00
COURSE APPLICATION FORM

1. Name of Applicant:  
   - Mr.  
   - Miss.  
   - Mrs.  
   (last)  
   (first)  
   (middle initial)

2. Course desired:  
   Course Title  
   Place  
   Dates

3. Previous Courses Attended:  
   Course Title  
   Dates  
   Course Title  
   Dates

4. Sponsor or Employer:  
   (name of organization or firm)  
   (street address)  
   (city)  
   (state)  
   (zip code)  
   (telephone)

5. Mailing address of applicant:  
   (if different from above)  
   (street address)  
   (city)  
   (state)  
   (zip code)  
   (telephone)

6. Job Duties: (Briefly describe your present position)

7. Education:  
   Last year of school completed

8. Experience:  
   Total years in wastewater treatment plant work

9. Student Skills Checklist: This checklist must be submitted before Application can be processed.

   signature of supervisor (where applicable)  
   (where applicable)  
   signature of applicant  
   date
(SAMPLE, STUDENT SKILLS CHECKLIST)

<table>
<thead>
<tr>
<th>Name</th>
<th>Employer</th>
</tr>
</thead>
</table>

To assist us in processing applications, please check YES or NO for each of the following items:

<table>
<thead>
<tr>
<th>Item</th>
<th>YES</th>
<th>NO</th>
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</thead>
<tbody>
<tr>
<td>I have operated a laboratory gas burner</td>
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<td></td>
</tr>
<tr>
<td>I have operated a laboratory hotplate/stirrer</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have operated an autoclave</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have operated a laboratory drying oven</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have used a vacuum source to filter liquids</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have used a desiccator</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have weighed items on an analytical balance</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have weighed items on a single pan balance</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have used a graduate to measure liquids</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have used a volumetric pipet to measure liquids</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have used a graduated (Mohr) pipet to measure liquids</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have used a pipet bulb to fill a pipet</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have used a volumetric flask to prepare solutions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have used chromic acid to clean glassware</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have operated a laboratory safety shower</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have operated a laboratory eye washer</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have operated a fume hood</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have prepared 0.0375N potassium biiodate solution</td>
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</tr>
<tr>
<td>I have made out labels for bottles of reagents</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have recorded a reading at a meniscus</td>
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<td></td>
</tr>
<tr>
<td>I have titrated one solution against another to a color change end point</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have calculated the normality (N) of a solution</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have recorded laboratory data in a laboratory notebook</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have entered laboratory data on a pre-printed form</td>
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<td></td>
</tr>
<tr>
<td>I have recorded information about samples on record sheets</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have located required purchase information in a catalog of laboratory equipment</td>
<td></td>
<td></td>
</tr>
<tr>
<td>I have written a purchase order for chemicals</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*Volume means space occupied by a solid, liquid, or gas*

- mg/l means milligrams per liter
- 1 kilogram equals 0.001 gram
- 1 inch equals 2.54 cm
- 1000 ml equals 1 liter
- 85 times 4.1 equals 42.5
- 7 minus 2 divided by 0.02 equals 250
- 3.26 rounded to the nearest tenth is 3.2
- 84.55147 rounded to the nearest thousandth is 84.551
Dear (Name):

A reservation has been confirmed for your participation in the course "Effluent Monitoring Procedures: Nutrients" to be conducted at (address, including building and room identification if pertinent).

Formal class activities will begin promptly at 8:30 AM on Monday, (date) and the course will be completed by 12:30 PM on Friday, (date). Please arrange your travel schedule so that you will be in the classroom at the start of course activities on Monday and that you will not have to hurry your departure on Friday.

Information about local travel, transportation, and local hotels is enclosed for your assistance. We believe that you will wish to make your own hotel or motel reservations.

We look forward to seeing you at the course, and we will do everything in our power to make this course a pleasant and rewarding experience for you.

Sincerely yours,

(signature)
Course Coordinator

Note: If something develops which makes it impossible for you to attend the course, please telephone (number) or write this office immediately, in order that another applicant may be admitted to the course in your place. Please do not arrange for a substitute without first getting the approval of this office.
To

Dear (Name):

We have received your application for admission to the course "Effluent Monitoring Procedures: Nutrients" to be conducted at (name of institution) during the period (date to date).

We would be most pleased to enroll you in this course, but by the time we received your application, all available positions in the class had been reserved. As you may know, we limit the class size to a fixed number in order to provide for the greatest possible amount of personal instruction during the course, and to provide each participant with the greatest possible opportunity for actual practice in the laboratory.

We have made a tentative reservation for you in the next offering of the course, which is scheduled to be given (dates). If this will be satisfactory to you, please write or call us within (number) days, so that we can confirm your reservation.

In the meantime, we have placed your name on the waiting list for the course dates which you requested. If a vacancy does become available, we will let you know immediately.

Sincerely yours,

(signature)
Course Coordinator

119
(SAMPLE LETTER OF REFERRAL)

(Institutional Letterhead)

(DATE)

To

Dear (Name):

We have received your application for admission to the course "Effluent Monitoring Procedures: Nutrients," to be conducted at (name of institution) during the period (date to date).

Based on our review of your previous laboratory experience, we consider that it would be very doubtful whether this course would provide you with all the knowledge and skills you will require to perform the self-monitoring tests and measurements on your municipal wastewater effluents.

Accordingly, we are confirming your reservation in this course, subject to your first satisfactorily completing the course (title of "Basic Skills" course). This course will be conducted at (location) (dates). We have made a tentative reservation for you to attend this course. Please let us know if you can attend this offering of the course.

If you must delay taking the course (name of "Basic Skills" course) at this time, then it will be necessary to delay your acceptance in the course which you have requested.

In the course (name of "Basic Skills" course) you will learn many things not covered in the later course, including use of the analytical balance, preparation and standardization of laboratory reagents, care and maintenance of laboratory supplies and equipment, and related tasks.

We are most anxious to help you learn to perform all the tasks required for self-monitoring of your municipal wastewater effluents. Please let us know if you can come to both courses.

Sincerely yours,

(signature)
Course Coordinator
(SAMPLE LETTER OF WELCOME)

(Institutional Letterhead)

(Date)

TO: COURSE PARTICIPANTS

We are looking forward to your participation in the course "Effluent Monitoring Procedures: Nutrients", scheduled for presentation at this Center during the period (date to date). If you find you cannot attend the course, please call us (telephone number).

To assist your planning and preparation for this course, the following items are enclosed:

1. List of hotels and motels
2. Information on local bus transportation and city map (Note: If bus service is used to the Center, you must have exact fare of (amount) on boarding bus).

On your arrival in the classroom you will be provided a course manual and related materials. Production schedules make it impossible to mail manuals to you in advance of course date.

The course will start at 8:30 AM on Monday, (date) in (room identification) and will close no later than 12:30 PM, Friday, (date). At the conclusion of the course, a certificate will be awarded verifying which analytical measurements you have performed in a satisfactory manner. Please arrange your travel schedule for after closing exercises. (Approximately (number) hours should be allowed for travel from (name of institution) to the airport.)

This course involves a considerable amount of work in the laboratory, using concentrated acids and bases. You will want to bring appropriate clothing.

(Name), of our staff, is serving as Course coordinator and will be available to assist you in solving any special problems you encounter while attending the course.

Should you have questions or desire assistance in any way, please do not hesitate to contact us.

Sincerely yours,

(signature)
Director
Institution Name
<table>
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<tr>
<th>Hotel Name</th>
<th>Address</th>
<th>Phone</th>
<th>Single</th>
<th>Double</th>
<th>Twin</th>
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<td></td>
<td></td>
<td></td>
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<td>462 rooms, meeting rooms 11, cap. 50-550</td>
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<td>Family plan, 800 rooms, TV, meeting rooms 15 - Cap. 70-1500, special Gov't. rates - $16.00 single, $23.00 double, $24.00 - twin</td>
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<td>245 rooms, meeting rooms 4 - cap. 25-135</td>
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<td>Family plan, 350 rooms, meeting rooms 4 - cap. 75-400, color TV, special Gov't. rates - $18.00 single, $25.00 double, $26.00 - twin</td>
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**NOTE:** We recommend you checking the rate at the time you make your reservation in the event there has been a price increase.

These hotels and motels are listed for your information to assist you in planning for your accommodations during your stay in Cincinnati while attending our training course, and does not imply endorsement by the Office of Water Program Operations, U.S. Environmental Protection Agency.

ERI(__) REQUIRES TRANSFER TO SECOND BUS.
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<td>Sixth &amp; Vine</td>
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**NOTE:** The above two routes are the most direct routes to the Environmental Research Laboratory, 26 W. St. Clair Street, Cincinnati, Ohio. Information has been provided by the Queen City Metro. Times selected are those most apt to be used by students attending NTC Training Courses. Arrival times are approximate, and will vary because of road and traffic conditions.
FROM GREATER CINCINNATI AIRPORT TO U.S. EPA ST. CLAIR FACILITY:

Interstate 75 (North or South)
Exit HOPPLE ST. to CENTRAL PKY.,
Right to MARSHALL AVE., Left to RIDDLE RD., Right to ST. CLAIR and EPA FACILITY.

Interstate 71 (North)
Exit READING RD. to BURNET AVE.,
Left to H. TAFT RD., Left to JEFFERSON AVE.,
Right to EPA FACILITY.

FROM GREATER CINCINNATI AIRPORT TO U.S. EPA RIDGE RD. FACILITY

Interstate 71 (North)
Interstate 75 to Interstate 71
To RIDGE RD., (North) Exit to EPA FACILITY.

FROM ST. CLAIR FACILITY TO RIDGE RD. FACILITY

JEFFERSON to MCMILLAN, Left to
Interstate 75 North, Exit RIDGE RD., North to Facility.

FROM U.S. EPA ST. CLAIR FACILITY TO GREATER CINCINNATI AIRPORT

Right on ST. CLAIR to RIDDLE RD.,
Left on MARSHALL AVE., Right on CENTRAL PKY.,
Left on HOPPLE ST.,
Right to Interstate 75 South.

FROM U.S. EPA RIDGE RD. FACILITY TO GREATER CINCINNATI AIRPORT

Right on RIDGE RD., Right to
Interstate 75 South to Interstate 75 South.
(SAMPLE REGISTRATION SUMMARY)

(Course Title)

REGISTRATION

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126

12-19
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(SAMPLE WAITING LIST SUMMARY)
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## Example Registration Card

**Employer Category**
- EPA
- DHEW
- DEPT. OF DEFENSE
- OTHER FEDERAL
- STATE GOVERNMENT
- LOCAL GOVERNMENT
- FOREIGN GOVT
- UNIV. FACULTY
- UNIV. STUDENT
- INDUSTRY
- CONSULTANT
- REGIONAL AGENCY
- OTHER: Specify

**Years of Experience**
- 0-1
- 2-4
- 5-7
- 8-10
- 11-15
- 16-20
- 21 OR OVER

**Profession or Occupation**
- ADMINISTRATOR
- BIOLOGIST
- CHEMIST
- CIVIC ORGANIZATION
- CONSERVATIONIST
- EDUCATOR
- ENGINEER
- GEOLOGIST
- MICROBIOLOGIST
- OCEANOGRAPHER
- PHARMACIST
- SANITARIAN
- STATISTICIAN
- TECHNICIAN
- TREATMENT PLANT OPERATOR
- OTHER: Specify

**Education**
- HIGH SCHOOL NON-GRADUATE
- HIGH SCHOOL GRADUATE
- COLLEGE NON-GRADUATE 1-3 YEARS
- COLLEGE NON-GRADUATE 4 YEARS
- BACHELOR DEGREE
- MASTER DEGREE
- DOCTOR DEGREE
- OTHER (describe)

---

**Notes:**
- One item in each category only.
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| 0-1 YEARS                     |       |       |
| 2-4                           |       |       |
| 5-7                           |       |       |
| 8-10                          |       |       |
| 11-15                         |       |       |
| 16-20                         |       |       |
| 21 OR OVER                    |       |       |
| ADMINISTRATOR                 |       |       |
| BIOLOGIST                     |       |       |
| CHEMIST                       |       |       |
| CIVIC ORG.                    |       |       |
| CONSERVATIONIST               |       |       |
| EDUCATOR                      |       |       |
| ENGINEER                      |       |       |
| GEOLOGIST                     |       |       |
| MICROBIOLOGIST                |       |       |
| OCEANOGRAPHER                 |       |       |
| PHARMACIST                    |       |       |
| SANITARIAN                    |       |       |
| STATISTICIAN                  |       |       |
| TECHNICIAN                    |       |       |
| TREAT PLANT OPER              |       |       |
| HS NON-GRAD                   |       |       |
| HS GRAD                       |       |       |
| COL 1-3 YEARS                 |       |       |
| COL OVER 3 YEARS              |       |       |
| BACHELOR DEGREE               |       |       |
| MASTER DEGREE                 |       |       |
| DOCTOR DEGREE                 |       |       |
| EPA PAID                      |       |       |

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</table>
This certifies that

THOMAS JONES

has completed the course

Effluent Monitoring Procedures: Nutrients (164.3)

and has performed the following measurements in a satisfactory manner:

- Measurement of Total Phosphorus
- Measurement of Ammonia Nitrogen (Macro Method)
- Measurement of Chemical Oxygen Demand
- Measurement of Nitrate & Nitrite Nitrogen
- Measurement of Total Kjeldahl Nitrogen
- Measurement of Oil & Grease

INSTRUCTOR

19 to 19

(SIGNATURE)

Director, (Institution Name)

(SIGNATURE)

Course Coordinator
**EFFLUENT MONITORING PROCEDURE:** Use of a Spectrophotometer

**EXAMPLE DATA SHEET**

**SPEC 20**

<table>
<thead>
<tr>
<th>A (mg/l)</th>
<th>C (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td></td>
</tr>
<tr>
<td>0.0</td>
<td></td>
</tr>
</tbody>
</table>

A of sample = 0.0
### RECORDS AND REPORTS Section IX

**EXAMPLE DATA SHEET FOR TOTAL PHOSPHORUS OR FOR ORTHOPHOSPHATE, mg/liter P**

<table>
<thead>
<tr>
<th>E.1c</th>
<th>Sampling Location</th>
<th>Final Effluent</th>
</tr>
</thead>
<tbody>
<tr>
<td>E.1c</td>
<td>Sample Identification</td>
<td>E.S. Is</td>
</tr>
<tr>
<td>E.1c</td>
<td>Type of Sample</td>
<td>Grab or Composite</td>
</tr>
<tr>
<td>E.1c</td>
<td>Date and Time Collected</td>
<td>1/17/75 9:00 a.m.</td>
</tr>
<tr>
<td>E.1c</td>
<td>Sample Collector</td>
<td>Tom Sampler</td>
</tr>
<tr>
<td>E.1c</td>
<td>Date and Time Analysis Began</td>
<td>1/17/75 9:30 a.m.</td>
</tr>
<tr>
<td>N.5a</td>
<td>Analyst</td>
<td>Dick Analyst</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>J.2.1d</th>
<th>Calibration Standards</th>
<th>Absorbance</th>
<th>Check Standards</th>
<th>Absorbance</th>
<th>Curve</th>
</tr>
</thead>
<tbody>
<tr>
<td>L.3a</td>
<td>mg/liter P</td>
<td></td>
<td>mg/liter P</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L.8a</td>
<td>0.02</td>
<td></td>
<td>0.10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L.8a</td>
<td>0.06</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>L.8a</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>L.8a</td>
<td>0.20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>L.8a</td>
<td>0.40</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>L.8a</td>
<td>0.60</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>L.8a</td>
<td>0.80</td>
<td></td>
<td>0.80</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L.8a</td>
<td>1.00</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**EXAMPLE CALCULATIONS**

<table>
<thead>
<tr>
<th>M.1c</th>
<th>Amount of Sample Dilution</th>
<th>Absorbance</th>
<th>Curve mg/liter P</th>
<th>Dilution Factor</th>
<th>Final mg/liter P</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Straight Sample</td>
<td>off scale</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>25 ml sample, 50 ml total</td>
<td>off scale</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>10 ml sample, 50 ml total</td>
<td>0.3525</td>
<td>0.520</td>
<td>2.60</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>5 ml sample, 50 ml total</td>
<td>0.1775</td>
<td>0.260</td>
<td>10</td>
<td>2.60</td>
</tr>
<tr>
<td>5</td>
<td>2.5 ml sample, 50 ml total</td>
<td>0.0875</td>
<td>0.130</td>
<td>20</td>
<td>2.60</td>
</tr>
</tbody>
</table>

**Note:** The document appears to be a page from a technical manual or report, detailing procedures for monitoring effluent, specifically focusing on the determination of Total Phosphorus or Orthophosphate in mg/liter P.
**Effluent Monitoring Procedure:** Determination of Total Phosphorus (as P) or of Orthophosphate (as P), Single Reagent Method

**Records and Reports**

**Determination of Total Phosphorus (as P)**

- **Determination of Orthophosphate (as P)**
- or
- **Determination of Digested Phosphorus Standards**
- **Determination of Non-Digested Phosphorus Standards**

**Calibration Graph**

**Signature of Preparer:**

**Date Graph Was Prepared:**

**Absorbance**

**Concentration of Phosphorus, mg/liter**

**Graph Data:**

- Absorbance values range from 0.00 to 1.00.
- Concentration of Phosphorus ranges from 0.00 to 1.00 mg/liter.

**Graph Axes:**

- Abscissa: Concentration of Phosphorus, mg/liter.
- Ordinate: Absorbance.
<table>
<thead>
<tr>
<th>Name of Plant</th>
<th>Typical Laboratory, Data Sheet for Chemical Oxygen Demand, mg/liter</th>
</tr>
</thead>
<tbody>
<tr>
<td>A.5 Identification</td>
<td>Blank</td>
</tr>
<tr>
<td>A.5 Type (grab, composite)</td>
<td>Composite</td>
</tr>
<tr>
<td>A.5 Date and Time Collected</td>
<td>3/17/75 0600-1200</td>
</tr>
<tr>
<td>A.5 Sample Collector</td>
<td>Tom Sampler</td>
</tr>
<tr>
<td>F.35 Date and Time Boiling Began</td>
<td>3/17/75 1300</td>
</tr>
<tr>
<td>F.9 RECORD: S, ml Sample Used</td>
<td>50.0</td>
</tr>
<tr>
<td>H.15 ml FAS * at END of titration</td>
<td>38.55</td>
</tr>
<tr>
<td>H.22 ml FAS * at START of titration</td>
<td>15.00</td>
</tr>
<tr>
<td>H.23 A, ml FAS * used to titrate the Blank</td>
<td>23.55</td>
</tr>
<tr>
<td>H.16 B, ml FAS * used to titrate the Sample</td>
<td>15.00</td>
</tr>
<tr>
<td>J.3 SUBTRACT B (line 10) from A (line 9)</td>
<td>8.55</td>
</tr>
<tr>
<td>J.4 RECORD: N, normality of FAS * (Calculated on Standardization Sheet, C.29)</td>
<td>0.024</td>
</tr>
<tr>
<td>J.6 MULTIPLY ml Difference of FAS * (line 11) by Normality of FAS (line 12)</td>
<td>0.2052</td>
</tr>
<tr>
<td>J.8 DIVIDE 8000 by S, ml Sample Used (See line 6)</td>
<td>160</td>
</tr>
<tr>
<td>J.10 MULTIPLY line 13 by line 14</td>
<td>328320</td>
</tr>
<tr>
<td>J.12 ROUND OFF line 15 to the nearest whole number of mg/liter</td>
<td>33</td>
</tr>
<tr>
<td>J.13 Signature</td>
<td>Jim Analyst</td>
</tr>
</tbody>
</table>

FAS means Ferrous-Ammonium Sulfate Solution. CALCULATION FORMULA: COD, mg/liter = (A-B)N x 8000
<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
<th>Flask 1</th>
<th>Duplicate</th>
</tr>
</thead>
<tbody>
<tr>
<td>C.31</td>
<td>ml FAS at END of titration</td>
<td>24.60</td>
<td>35.15</td>
</tr>
<tr>
<td>C.23</td>
<td>ml FAS at START of titration</td>
<td>14.00</td>
<td>24.60</td>
</tr>
<tr>
<td>C.33</td>
<td>ml FAS used for Standardization</td>
<td>10.60</td>
<td>10.55</td>
</tr>
<tr>
<td></td>
<td>(SUBTRACT ml FAS at START on line 2 from ml FAS at END on line 1)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C.36</td>
<td>DIVIDE 0.250* by the ml difference on line 3 to a 4 decimal place answer.</td>
<td>0.0236</td>
<td></td>
</tr>
<tr>
<td>C.38</td>
<td>Normality of the FAS solution</td>
<td>0.024</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(ROUND OFF line 4 to 3 decimal places)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>C.39</td>
<td>Date</td>
<td>3/17/75</td>
<td></td>
</tr>
<tr>
<td>C.40</td>
<td>Signature</td>
<td>Jim Analyst</td>
<td></td>
</tr>
</tbody>
</table>

* From the formula:

\[
\text{Normality FAS} = \frac{\left(10.0 \text{ ml potassium} \right) \left(0.025 \text{ N potassium} \right)}{\frac{\text{dichromate}}{\text{dichromate}}} \frac{\text{ml FAS}}{\text{ml FAS}}
\]
**EFFLUENT MONITORING PROCEDURE: Determination of Total Kjeldahl Nitrogen**

### RECORDS AND REPORTS

<table>
<thead>
<tr>
<th>TRAINING GUIDE NOTE</th>
<th>REFERENCES/RESOURCES</th>
</tr>
</thead>
<tbody>
<tr>
<td>Typical Laboratory Data Sheet for Total Kjeldahl Nitrogen, mg/l Name of Plant</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>E.1.c.</th>
<th>Sampling Location</th>
</tr>
</thead>
<tbody>
<tr>
<td>E.1.c.</td>
<td>Type of Sample</td>
</tr>
<tr>
<td>E.1.c.</td>
<td>Date and Time Collected</td>
</tr>
<tr>
<td>E.1.c.</td>
<td>Sample Collector</td>
</tr>
<tr>
<td>E.1.c.</td>
<td>Date and Time Analysis Began</td>
</tr>
<tr>
<td>E.1.c.</td>
<td>Analyst</td>
</tr>
<tr>
<td>E.1.c.</td>
<td>Method Used (Macro or Micro)</td>
</tr>
<tr>
<td>E.1.c.</td>
<td>ml sample used</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>D.4.12a</th>
<th>B. ml total distillate including boric acid (H₃BO₃) and dilution water</th>
</tr>
</thead>
<tbody>
<tr>
<td>E.4.12a</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>F.1.5c.</th>
<th>C. ml distillate taken for Nesslerization</th>
</tr>
</thead>
<tbody>
<tr>
<td>F.3.5a.</td>
<td>A. mg NH₃-N/50.0 ml, from curve</td>
</tr>
</tbody>
</table>

Use this formula in calculating the results for the colorimetric method:

\[
\text{TKN mg/l} = A \times 1000 \times \frac{B}{\text{ml sample}} \quad \text{(See pp. 5-31 and 5-32)}
\]

If Organic Nitrogen (mg/l) is needed and a separate ammonia analysis has been performed, use the following equation to determine this.

Since: \( \text{TKN} = \text{Organic/N} + \text{Ammonia/N} \)

Then: \( \text{Organic/N} = \text{TKN - Ammonia/N} \)

**Final Results**

<table>
<thead>
<tr>
<th>TKN mg/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{NH}_3-N, \text{ mg/l} )</td>
</tr>
<tr>
<td>( \text{Org-N, \text{ mg/l}} )</td>
</tr>
</tbody>
</table>
**RECORDS AND REPORTS**

<table>
<thead>
<tr>
<th>Training Guide Note</th>
<th>References/Resources</th>
</tr>
</thead>
<tbody>
<tr>
<td>F.2.6a Values from Nesslerization Procedure</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Tube #</th>
<th>Concentration mg NH₃-N/50.0 ml</th>
<th>Absorbance</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.005</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.010</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.020</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.040</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>0.050</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>0.080</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>0.10</td>
<td></td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>Sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Sample</td>
<td></td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>Sample</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
EFFLUENT MONITORING PROCEDURE: Determination of Total Kjeldahl Nitrogen

DETERMINATION OF AMMONIA NITROGEN

CALIBRATION GRAPH

SIGNATURE OF PREPARER: __________________________

DATE GRAPH WAS PREPARED: _______________________

ABSORBANCE

0.50

0.40

0.30

0.20

0.10

0.00

0.01 0.02 0.03 0.04 0.05 0.06 0.07 0.08 0.09 0.10

CONCENTRATION OF AMMONIA NITROGEN mg/50.0 ml
<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Date/Time Sampled</th>
<th>Sample Point</th>
</tr>
</thead>
</table>

(Sulfuric acid 0.02N ml | (0.28)(1000) |

Sample ml |  |

mg/liter NH₃-N | 147
LABORATORY DATA SHEET

Nitrogen, Ammonia Determination
(Sample Contains 0.05 to 1.0 mg/liter NH₃-N)

Sample No.  Date/Time Sampled  Sample Point

\[
\frac{(\text{mg of NH}_3\text{-N})(1000)}{\text{Sample ml}} \times \frac{(\text{Total Distillate* Collected ml})}{(\text{Distillate Taken for Nesslerization ml})} = \text{mg/l NH}_3\text{-N}
\]

*Include boric acid plus dilution water

Analyst  Date
**EXAMPLE DATA SHEET**

**SAMPLE NUMBER** | mg/liter TOTAL NO$_2$+NO$_3$-N (A) | mg/liter NO$_2$-N (B) | mg/liter NO$_3$-N (C) | mg/liter NO$_2$-N (D) | mg/liter NO$_3$-N (E) | mg/liter NO$_2$-N (F) | mg/liter NO$_3$-N (G) |
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Reduced Nitrate Working Standards:</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.05</td>
<td>0.05</td>
<td>0.22</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>0.10</td>
<td>0.10</td>
<td>0.44</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>0.20</td>
<td>0.20</td>
<td>0.89</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>0.50</td>
<td>0.50</td>
<td>2.22</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>1.00</td>
<td>1.00</td>
<td>4.43</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Reduced Sample(s):</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Non-reduced Nitrate Working Standards</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>0.05</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.16</td>
</tr>
<tr>
<td>3</td>
<td>0.10</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.33</td>
</tr>
<tr>
<td>4</td>
<td>0.20</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.66</td>
</tr>
<tr>
<td>5</td>
<td>0.50</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1.69</td>
</tr>
<tr>
<td>6</td>
<td>1.00</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>3.29</td>
</tr>
<tr>
<td>Non-reduced Sample(s):</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
**EFFLUENT MONITORING PROCEDURE:** Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method

<table>
<thead>
<tr>
<th>RECORDS AND REPORTS</th>
<th>SECTION IX</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>DETERMINATION OF TOTAL NO₂+NO₃⁻ N</strong></td>
<td></td>
</tr>
<tr>
<td>(Reduced Nitrate Standards)</td>
<td></td>
</tr>
<tr>
<td><strong>CALIBRATION GRAPH</strong></td>
<td></td>
</tr>
</tbody>
</table>

**SIGNATURE OF PREPARER:**

**DATE GRAPH WAS PREPARED:**

---

**ABSORBANCE**

---

**CONCENTRATION OF NITRATE NITROGEN, mg/liter**

RECORDS AND REPORTS

DETERMINATION OF NO₂⁻ N
(Non-reduced Nitrite Standards)

CALIBRATION GRAPH

SIGNATURE OF PREPARED:

DATE GRAPH WAS PREPARED:

CONCENTRATION OF NITRITE NITROGEN, mg/liter
EFFLUENT MONITORING PROCEDURE: Determination of Oil and Grease

Blank Determination

Weight of distilling flask after evaporation of the 100 ml of TF/D = E

Weight of the empty distilling flask used to determine the blank = F

Value of blank, D = E - F

Sample Determination

Weight of distilling flask + the oil/grease residue = A

Weight of empty distilling flask used for the sample = B

Volume of sample = C

Milligrams of oil/grease residue per liter sample = \( \frac{A - B - D}{C} \times 1000 \times 1000 \)
For each Effluent Monitoring Procedure (commonly termed "EMP" by Instructors) in the Student Reference Manual, there is an Instructional Package Worksheet (IPW) in this Guide. The Worksheet is for guidance to the Instructor for development of 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 Packages do provide a perspective on the background of each analytical procedure, lesson-by-lesson learning achievement levels the students should attain, an indication of available audiovisual and other instructional resources, and a recommended course of action in pre-course preparation and classroom/laboratory instruction.

Application of these Instructional Packages will help the Instructor to reduce the time required for planning and organizing a strategy of preparation and instruction. But 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 a Course Guide as this; ultimately the Instructor is the key person in assuring that the student acquires the needed knowledge and skills.
A prototype for development of routine operational procedures
for the
use of a spectrophotometer

as applied in
wastewater treatment facilities
and in the
monitoring of effluent wastewaters

Instructional package worksheet

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U.S. Environmental Protection Agency

CH.IN.sp.ipw.2d.7.77
PART II INSTRUCTIONAL PACKAGE WORKSHEETS

A. Use of a Spectrophotometer

1. This skill is required in order to determine Phosphorus, Total Kjeldahl Nitrogen and Nitrate-Nitrite Nitrogen using the Effluent Monitoring Procedures (EMPs) for this course.

2. It was most practical to write the EMP for one instrument, the Bausch and Lomb* Spectronic 20-Spectrophotometer, and to use the manufacturer's instructions as the source of the procedure.

3. The steps in the EMP can be readily adapted for application to other instruments.

4. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for processing a provided set of synthetic standards in a spectrophotometer, then a provided synthetic sample.

5. Each student is to process one set of standards and a sample made according to the directions in the IPW. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust materials and quantities accordingly.

ERRATA: Please add this note in the EMP, Operating Procedure C at Step 7 in the third column:

7a. Always use a soft tissue to handle phototubes or to wipe off fingerprints.

* Mention of a specific brand name does not constitute endorsement by the Office of Water Program Operations, U.S. Environmental Protection Agency.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Use of a Spectrophotometer

UNIT OF INSTRUCTION:

LESSON NUMBER: 1 of 1

ESTIMATED TIME: 75 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Several determinations required under the permit system involve the measurement of color intensity. The Bausch and Lomb Spectronic 20 spectrophotometer is one instrument which may be used to make the color intensity measurements.

ENTRY LEVEL BEHAVIOR: The learner must be able to:
1. Perform basic mathematical computations (addition, subtraction, multiplication and division).
2. Handle solutions of acids safely.
3. Clean laboratory glassware.

A. INSTRUCTIONAL OBJECTIVE
1. Terminal Behavior - The learner will exhibit proper technique while using the Bausch and Lomb Spectronic 20 spectrophotometer.

2. Conditions - The learner will have the use of the attached EMP, and all materials on the equipment list.

3. Accepted Performance - The use of proper technique will be judged by the instructor.

B. INSTRUCTIONAL RESOURCES
1. Available Media
   a. XT-51, Use of the Spectronic 20 Spectrophotometer, 10 minutes, tape and 35 slides.
   b. TC-57, Videocassette Tape Unit, "Use of a Spectrophotometer", 21 minutes, showing the steps listed in EMP Procedure B and a discussion of EMP Procedure C.
   c. NOTE: Either the Tape/Slide Unit or the Videocassette Tape Unit may be used.
   d. X-8, sixteen unassembled slides.

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH (Sequencing)
1. Class (Using 16 assembled slides)
   a. Discuss the concept of light being absorbed by a chemical solution.
   b. Define wavelength.
   c. Define absorbance.
   d. Define the Lambert-Beer law and the four terms in it. Absorbance and concentration values are needed to prepare a calibration graph. The calibration graph is the basis for quantitation in colorimetric measurements.
   e. Discuss how a series of colorimetric standards (e.g., as for the EPA phosphorus determination) would be prepared. Concentrations of the standards are known.
f. Mention that a Bausch & Lomb Spectronic 20 Spectrophotometer is one type of instrument which could be used to obtain absorbance values.

g. Show XT-51, Use of the Spectronic 20 Spectrophotometer, or the TV tape.

h. Discuss that it is now known how to obtain concentration and absorbance values.

i. Discuss the concept of a dilution factor and how it is used.

j. Have the learners read the Brief Description of Analysis.

2. Laboratory

a. Have the learners begin the EMP at section A.3 by removing the "wrong" visible phototube from the Spectronic-20 (which you had previously inserted) following the EMP procedure. Have them next insert the "correct" infrared phototube and filter, and proceed with section B. The terms "wrong" and "correct" are used in reference to the methylene blue synthetic color standard. A break may be given during the warm-up period.

b. Have the learners obtain absorbance values at 660 nm for a series of synthetic color standards prepared using methylene blue. (Before class, check that 660 is the proper wavelength for each Spectronic 20; i.e., the value may be a few nm more or less than 660.) In order to prepare the stock methylene blue solution, add a few drops of the indicator to about 300 ml of distilled water. The absorbance of the solution should be about 0.6. If it is not, add more distilled water or indicator as needed. Arbitrarily, mark this solution as having a concentration of 8.0 mg/l. Successive 50% dilutions will then give concentrations of 4.0, 2.0, 1.0, and 0.5 mg/l. Supply a set of methylene blue standards (including plain distilled water for 0.0 mg/l) with each Spectronic 20. Prepare a sample from the methylene blue such that the learners will have to dilute it 10 ml to 100 ml to obtain an "on-scale" absorbance reading; this will introduce the idea of using a dilution factor when the calibration graph is prepared. If desired, actual color standards could also be prepared using the phosphorus effluent monitoring procedure. An example data sheet for the methylene blue color standards is attached. Copies should be prepared and distributed to the learners. Note that the actual "concentration" of the diluted methylene blue sample will be determined after completion of the effluent monitoring procedure on the preparation of calibration graphs.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS

1. For each student:
   - Laboratory apron
   - Safety glasses
   - One pen or pencil
   - Notebook or data sheet

2. Shared:
   - One Bausch and Lomb Spectronic 20 Spectrophotometer per three students
   - One manufacturer's manual for each spectrophotometer
   - Still, or other source of distilled water
One visible phototube (Bausch & Lomb catalog number 33-29-71) for each Spec 20
One infrared phototube (Bausch & Lomb catalog number 33-29-72) for each Spec 20
One infrared filter (Bausch & Lomb catalog number 33-29-18) for each Spec 20
Ten soft tissues (for wiping the cells)
One plastic squeeze distilled water bottle
Sink or 1 liter container for rinsing solutions
One 1 cm cell for each Spectronic 20
One 100 ml volumetric flask
One 10 ml volumetric pipet
One pipet bulb

E. IPW REAGENT REQUIREMENTS (Minimum amounts per group sharing a Spectronic 20)

One set of six methylene blue color standards. See C.2.b. above.
One bottle of methylene blue "sample" (to be diluted 10 to 100)
## EXAMPLE DATA SHEET
for
Methylene Blue Color Standards

<table>
<thead>
<tr>
<th>Concentration in mg/l</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>0.00</td>
</tr>
<tr>
<td>0.5</td>
<td>0.00</td>
</tr>
<tr>
<td>1.0</td>
<td>0.00</td>
</tr>
<tr>
<td>2.0</td>
<td>0.00</td>
</tr>
<tr>
<td>4.0</td>
<td>0.00</td>
</tr>
<tr>
<td>8.0</td>
<td>0.00</td>
</tr>
</tbody>
</table>

Absorbance of diluted sample = 0.00

Dilution factor = 10
A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES for the PREPARATION OF CALIBRATION GRAPHS

as applied in WASTEWATER TREATMENT FACILITIES and in the MONITORING OF EFFLUENT WASTEWATERS

Instructional Package Worksheet

National Training and Operational Technology Center Municipal Operations and Training Division Office of Water Program Operations U. S. Environmental Protection Agency
PART II - INSTRUCTIONAL PACKAGE WORKSHEETS

B. Preparation of Calibration Graphs

1. This step is required in order to determine Phosphorus, Total Kjeldahl and Nitrates-Nitrites Nitrogen using the Effluent Monitoring Procedures (EMPs) for this course.

2. The procedure was written in the EMP format using a traditional method of plotting interdependent variables on an arithmetical graph form.

3. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for each student to prepare one graph. If you choose a different assignment, adjust quantities of materials accordingly.
SUBJECT MATTER: Preparation of calibration graphs

UNIT OF INSTRUCTION:

LESSON NUMBER: 1 of 1

ESTIMATED TIME: 60 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: If absorbance or transmittance determinations are made as part of an analysis being done for the permit system, the learner must know how to prepare a calibration graph in order to arrive at a number value for the chemical constituent being determined.

ENTRY LEVEL BEHAVIOR: The learner must:
1. Understand the terms milligram (mg), microgram (µg) and liter (l).
2. Have successfully completed the EMP, "Use of a Spectrophotometer."

A. INSTRUCTIONAL OBJECTIVE:
1. Terminal Behavior: The learner will prepare a calibration graph and use it to determine the amount of chemical constituent in a wastewater effluent sample.
2. Conditions: The learner will have the use of the attached EMP, a ruler, a pencil, and a sheet containing analysis data.
3. Accepted Performance: The graph will be prepared to the satisfaction of the instructor.

B. INSTRUCTIONAL RESOURCES:
1. Available Media:
   a. Six unassembled slides.
2. Suggested Media: None.

C. INSTRUCTIONAL APPROACH (sequencing):
All steps below are done in the classroom.
1. Have the learners read to themselves the Analysis Objectives and the Brief Description of Analysis. Ask for questions.
2. Distribute pencils and rulers to the learners.
3. Outline the procedure for preparing the calibration graph, using the six unassembled slides.
4. Have the learners do the entire EMP. Use the example data on Page 2-18 of the EMP, or the data obtained from the methylene blue color standards (see the EMP on the Use of a Spectrophotometer). In the latter case, note that the numbering of the two axes would be different than if the data on Page 2-18 were used.
5. Check the calibration graphs for use of the entire mg/l on A axes, and for reasonable neatness.
6. Final questions/discussion.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS
1. For each student:
   - One ruler, 12 inches long
   - Pencil
   - Eraser
   - Graph paper (one piece for each calibration graph). There are many kinds of graph paper. In ordinary water pollution analyses, a simple type of graph paper is used. Figure 1 is an example of one type of simple graph paper. The main feature of simple graph paper is that it is divided into a certain number of large squares of equal size. (For example, one inch might be the length of one side of the large squares). These large squares are subdivided into a certain number of smaller squares of equal size. (For example, a one inch square might be subdivided into one hundred small squares).
2. Shared:
   - None

E. IPW REAGENT REQUIREMENTS (minimum amounts per student)
   - None
### Example Data Sheet

<table>
<thead>
<tr>
<th>Absorbance Values of Standards</th>
<th>Concentration of Standards in mg/l</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.000</td>
<td>0.0</td>
</tr>
<tr>
<td>0.130</td>
<td>5.0</td>
</tr>
<tr>
<td>0.290</td>
<td>10.0</td>
</tr>
<tr>
<td>0.420</td>
<td>15.0</td>
</tr>
<tr>
<td>0.570</td>
<td>20.0</td>
</tr>
<tr>
<td>0.690</td>
<td>25.0</td>
</tr>
</tbody>
</table>

Twenty-five ml of the sample were diluted to 500 ml in a volumetric flask. The absorbance of the diluted sample was 0.310. What is the concentration of the undiluted sample in mg/l?
A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES
for the DETERMINATION OF TOTAL PHOSPHORUS (as P) OR OF ORTHOPHOSPHATE (as P), SINGLE REAGENT METHOD
as applied in WASTEWATER TREATMENT FACILITIES and in the MONITORING OF EFFlUENT WASTEWATERS

Instructional Package Worksheet

National Training and Operational Technology Center Municipal Operations and Training Division Office of Water Program Operations U. S. Environmental Protection Agency

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PART II INSTRUCTIONAL PACKAGE WORKSHEETS

C. Determination of Total Phosphorus (as P) or Orthophosphate (as P), Single Reagent Method

1. The Federal Register (FR) issuance in Outline No. 1. of this Guide lists one manual method to determine Total Phosphorus (as P) or Orthophosphate (as P): ascorbic acid reduction. To determine Total Phosphorus, the sample is pretreated by persulfate digestion to convert non-orthophosphate phosphorus forms to orthophosphate.

2. The persulfate digestion treatment and the ascorbic acid reduction method have been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Total Phosphorus (as P) or Orthophosphate (as P), Single Reagent Method."

The reference source for the EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 249. Other FR sources for the procedures to determine Total Phosphorus (as P) or Orthophosphate (as P) are:


4. The procedures in this EMP can be used for two determinations. Total Phosphorus (as P) can be determined using both the digestion and reduction procedures. If only Orthophosphate (as P) is the required data, only the reduction procedure is used.

5. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for the determination of Total Phosphorus (as P) in a wastewater treatment plant effluent sample.

6. Each student is to process one blank, one standard and one sample. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.

7. OPTIONAL assignments from this EMP have been used to check student proficiency at prerequisite basic skills for this course, i.e., using an analytical balance and making dilutions of stock solutions. These exercises can be used between lesson 1 and lesson 2 and require 14 hours laboratory time. Details are in the IPW. C. INSTRUCTIONAL APPROACH. The related equipment and reagent requirements are noted on the IPW listings.

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GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus, mg/liter P.

UNIT OF INSTRUCTION: Summary of 5 Elements

ESTIMATED TIME: Three hundred ninety minutes exclusive of any break time. If instructor chooses to check weighing and diluting skills (See C note), estimated time is 480 minutes.

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The determination of total phosphorus is required by many NPDES permits.

ENTRY LEVEL BEHAVIOR: Learner must have mastered the skills in Course I Basic Laboratory Skills, or have equivalent experience. In addition, learner must know how to operate a pH meter, assemble and use a membrane filtration assembly, operate a Bausch and Lomb Spectronic 20 Spectrophotometer, and construct and use a calibration graph.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will prepare glassware for the test, prepare a blank, a calibration standard and a sample dilution, digest them, filter them, adjust their pH, add color reagent, determine their absorbance, construct a calibration graph and use it to convert the absorbance reading for the sample into mg/liter P.

2. Conditions: The learner will be provided with classroom instruction, the required equipment, a suitable sample, a copy of the EMP, a copy of the data sheet, a copy of the EMP calibration graph form, and a total working time of 240 minutes.

3. Accepted Performance: His techniques must be satisfactory according to an instructor's rating and his answers must be the same as an instructor's answers calculated from the same data.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: 1974 EPA "Methods for Chemical Analysis of Water and Wastes" EMP "Determination of Total Phosphorus (as P) or Orthophosphate (as P), Single Reagent Method," eight overhead projecturals: (1) Federal Register listing of acceptable procedures; (2) definitions of reportable phosphorus fractions; 1974 EPA Methods Manual, p. 251; (3) EMP procedural flow sheet, p. 6; (4) word equations for "Basis of Test" and "Digestion"; (5) overview of the four lessons divided according to procedures in each lesson; (6) the Example Laboratory Data Sheet - EMP, p. 49; (7) overview of "Transfers" to be made during Lesson three (Procedures G, I, and J); (8) the Calibration Graph, EMP, p. 50, and a 15 minute slide-tape, XT-44, "Determination of Phosphorus."

2. Suggested Media: None.
C. INSTRUCTIONAL APPROACH:

The method presented in this Effluent Monitor Procedure is according to the 1974 EPA Methods Manual. Of the several reportable phosphorus fractions given in the manual, this EMP covers only total phosphorus and orthophosphate. Of these two, only total phosphorus is given in its entirety, with the additional information for orthophosphate being given as alternate steps in the procedure. Since many orthophosphate samples will need to be filtered before determination, and hence reported as dissolved orthophosphate, this procedure is discussed in the Training Guide, Section VII, E.3d.

The 1974 EPA Methods Manual specifies either a filter photometer or a spectrophotometer for use at either 650 or 880 nm. The steps in the procedure involving use of a spectrophotometer have been written specifically for use in conjunction with a B & L Spectronic 20 (or equivalent). If a filter photometer, uv-visible spectrophotometer or other machine is used, these steps will need to be modified. Even if a B & L Spectronic 20 (or equivalent) is being used, the standard instrument is not sufficient. Both the 650 and 880 nm wavelengths require that the standard phototube be replaced with an accessory infrared sensitive phototube and an accompanying filter.

The strength of the standard phosphorus solution, as given in the EMP, is predicated on the use of standard 1/2 inch colorimeter tubes in the spectrophotometer. As the 1974 EPA Methods Manual only specifies that the light path of the spectrophotometer be "1 cm or longer," Section VI.C.1b of the Training Guide gives a discussion of how to modify the standard phosphorus solution for cells with path lengths other than 1/2 inch, and presents an alternative Table 1 for use with 1 inch cells.

NOTE: Some instructors have utilized parts of this EMP to check student proficiency at the basic skills required to make reagents, i.e., using an analytical balance and making dilutions of stock solutions. EMP step B.12.6 can be used to check weighing technique. The instructor checks readouts on the balance as the student obtains them. If volumetric glassware is available, the student can complete Procedure B.12 to make his own stock phosphorus solution. Alternatively, the instructor can prepare the stock solution for the entire class. Then, to check dilution technique, the students each do Procedure C. The evaluation standard is the calibration curve each obtains by using the standard Phosphorus Solution each prepared. For this to be valid, each student should prepare a calibration curve using only his own results (as opposed to using several absorbances obtained by other students). Assigning at least two calibration standards instead of one makes this feasible. These student exercises have been used between lesson 1 and lesson 2 and require 1 1/2 hours laboratory time.

1. Preparation for Instruction:

   a. Duplicate copies of the EMP data sheet and calibration graph form for each student.

   b. Have on hand a treatment plant effluent of known mg/liter P concentration so that a suitable dilution will be assigned. (A maximum of 50 ml per student will be needed.) Plan the assignment.
c. Make out tags for the sample bottles (1 per 3-4 students) including the information required on the EMP "Example Laboratory Data Sheet."

d. Use the EMP Training Guide VII directions to prepare phosphorus-free filter papers unless such filters were purchased.

e. See that equipment and reagents required for each student to do the planned "Student Performances" are assembled in the laboratory. (Hot 1:1 HCl must be available at the beginning of the first lab session.)

2. Sequencing:

a. Lesson one - 135 minutes
b. Optional - 90 minutes, checking basic skills: use of an analytical balance (B.12.6) and diluting solutions (C)
c. Lesson two - 60 minutes (This lesson does not have to immediately follow lesson one or the optional lesson.)
d. A break can be scheduled now before beginning lesson three, provided someone is available to watch the boiling digestions and remove them from the hot plates to cool at the end of the boiling period. However, no break is necessary as regards teaching the procedure.
e. Lesson three - 120 minutes (If necessary a break can be scheduled after G, filtration and pH adjustment, before beginning colorimetry.)
f. Lesson four - 30 minutes (This lesson on constructing and using the calibration curve to find mg/liter P in the sample does not have to immediately follow lesson three.)
g. Lesson five - 45 minutes (This wrap-up session may be scheduled anytime after the completion of the first four lessons.)

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS

(1 reagent blank, 1 calibration standard and 1 sample dilution)

1. For each student:

- 1 apron
- 2 boiling beads, glass, 4 per assignment
- 1 beaker, 400-600 ml for cleaning glassware
- 1 bottle, squeeze type for distilled water
- 1 bulb, pipet
- 1 filter assembly, smallest volume available to filter 10 ml digestate
  (filtrate gets transferred) Fritted glass funnels not recommended
- 3 pieces filter paper, phosphorus-free, 0.45 micron pore size, diameter to fit filter assembly (Gelman GA6 or equivalent)
- 3 flasks, 125 ml Erlenmeyer with wide mouth
- 3 flasks, 50 ml volumetric with stoppers (Need 4 if diluting sample)
- 1 pair forceps
- 1 funnel, to fit neck of 50 ml volumetric flask
- 1 pair safety glasses
- 1 pair rubber gloves for cleaning glassware
- 1 pH meter, preferably with combination electrode (If students share pH meters, teaching time becomes prohibitively long.)
- 1 pipet, Pasteur or Mohr for 10 N sodium hydroxide during pH adjustment
- 1 pipet, Mohr, 10 ml for combined reagent
- 1 pipet, 1 ml graduated in 0.1 ml for pH adjustments
- 1 pipet, volumetric, X ml on a graduate if diluting sample
- 1 pair tongs

See NOTE, next page, bottom
1 copy data sheet in EMP
1 copy calibration graph form in EMP

If checking use of an analytical balance:
1 flask, 1 liter volumetric with stopper (if available)
1 spatula
1 weighing boat

If checking dilution technique:
1 flask, 1 liter volumetric with stopper
1 pipet, 20 ml volumetric

2. Shared:

1-2 hoods (for cleaning glassware)
XXX hot plates to accommodate 3-125 ml Erlenmeyer flasks for each student
XXX pipets, volumetric, 1, 3, 5, 10, 20, 30, 40 and 50 ml (Numbers of each size needed depend on planned assignment of standards.)
XXX pipets, volumetric, 50 ml or volumes corresponding to planned assignment of sample dilutions
1 reagent spoon, 0.4 g, per 3 students for measuring ammonium persulfate
1 spectrophotometer for every 3 students
1 sample of known mg/liter P concentration split up into smaller containers (tagged), one for every 3 or 4 students

If checking use of an analytical balance:
1 analytical balance per 3 students

E. IPW REAGENT REQUIREMENTS:

(minimum amounts per student)

2.5 liters phosphorus-free distilled water
150 ml 1 N hydrochloric acid for use in cleaning glassware
150 ml combined reagent for test and for cleaning glassware
1.2 g ammonium persulfate
50 ml (maximum) standard phosphorus solution if students do not prepare their own
5 ml 10 N sodium hydroxide
5 ml 0.1 N sodium hydroxide
4 ml 11 N sulfuric acid (strong acid solution)
1 ml 1.1 N sulfuric acid

If checking use of an analytical balance:
0.2197 g potassium dihydrogen phosphate which has been dried for an hour at 105°C, then cooled and stored in a desiccator

If checking dilution technique:
about 30 ml stock phosphorus solution if students do not prepare their own stock

*NOTE: All glassware used for phosphorus tests and for preparation of phosphorus reagents must be acid-washed with hot 1:1 HCl, rinsed with distilled water and checked with combined reagent to assure complete removal of phosphorus contamination. If glassware can be reserved for phosphorus tests, this treatment is only required occasionally. See EMP Procedure A for details.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus, mg/liter P

UNIT OF INSTRUCTION: Overview of the Determination and Cleaning Glassware (EMP Procedure A)

LESSON NUMBER: 1 of 5

ESTIMATED TIME: 135 minutes

JUSTIFICATION FOR THE INSTRUCTIONAL OBJECTIVE: To determine total phosphorus accurately in a sample, the learner must clean all glassware with hot acid.

ENTRY LEVEL BEHAVIOR: Learner must have mastered the skills in Course I, Basic Laboratory Skills, or have equivalent experience

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will clean his glassware according to EMP Procedure A.

2. Conditions: Learner will be given the equipment, a copy of the EMP, no assistance and 90 minutes.

3. Accepted Performance: Learner must accomplish terminal behavior to the satisfaction of an instructor rating. (Particularly note safety technique in handling the HCl acid.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; five overhead projecturals: Federal Register listing of acceptable procedures; definitions of reportable phosphorus fractions, 1974 EPA Methods Manual, p. 251; procedural flow sheet, EMP p. 6; word equations for "Basis of Test" and "Digestion;" overview of the four lessons; and a 15 minute slide-tape XT-44, "Determination of Phosphorus."

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: (45 minutes)
   a. Lecture - Introduction
      - 15 minute slide-tape presentation, XT-44, "Determination of Phosphorus"
      - Overheads - Federal Register, definition of reportable phosphorus fractions - discuss types of phosphorus (ortho vs. condensed and organic, etc.)
   b. Lecture - overheads of EMP flow sheet, "Basis of Test" and "Digestion" word equations, overview of four lessons. Then specifics of Procedure A.
c. Demonstration - cleaning of glassware, warning about dangers of hot HCl acid, and warning against use of phosphate detergents.
d. Assignment - Have students read EMP Procedure A.
e. Question and answer period on material presented
f. Assignment - Instructor should tell students items of glassware to be cleaned according to Procedure A. (Possibly a 1 liter flask, a volumetric pipet, and an Erlenmeyer flask).

2. Student Performance and Evaluation: (90 minutes)

As stated in Instructional Objective above, by instructor rating.
GUIDELINES FOR
INSTRUCTIONAL-PACKAGE WORKSHEET

SUBJECT: Total Phosphorus, mg/liter P

UNIT OF INSTRUCTION: Preparation of Phosphorus Calibration Standards and of Sample; Digestion of Standards, Blank, Sample (EMP Procedures D, E, and F)

LESSON NUMBER: 2 of 5

ESTIMATED TIME: 60 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: These procedures are part of the determination of total phosphorus in a sample.

ENTRY LEVEL BEHAVIOR: Learner must have mastered the skills in Course I, Basic Laboratory Skills, or have equivalent experience.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will prepare a blank, a calibration standard and a sample dilution according to EMP Procedures D and E, then digest these solutions according to EMP Procedure F, all as stated or described in the information column.

2. Conditions: Learner will be given the equipment, a copy of the EMP, a data sheet, no assistance and 45 minutes.

3. Accepted Performance: Learner must accomplish terminal behavior to the satisfaction of an instructor rating. (Particularly note correct pipetting technique and dilution to the exact 50 ml mark.)

B. INSTRUCTIONAL RESOURCES:


2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation (15 minutes)

   b. Assignment - D. 1 Note division of establishing or checking curve.
      Assign blank plus one standard.
   c. Distribution - Give each student a copy of the data sheet.
   d. Overhead of Data Sheet - note keys to divisions
   e. Assignment - Instructor gives students sample information for E, I and assigns to each a sample dilution.
NOTE: Once student solutions are all digesting, a logical continuity could be maintained by giving the students a break, then reassembling in the classroom for the next lesson. A staff member can watch the digestion volumes and remove flasks from the hot plates when digestion is complete.

2. Student Performance and Evaluation: (45 minutes)

As stated in Instructional Objective above, by instructor rating.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus, mg/liter P

UNIT OF INSTRUCTION: Filtration and pH Adjustment of Digestions followed by Colorimetry and Spectrophotometric Measurements (EMP Procedures G, H, I and J)

LESSON NUMBER: 3 of 5

ESTIMATED TIME: 120 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: This is part of the laboratory procedure to determine total phosphorus, mg/liter P.

ENTRY LEVEL BEHAVIOR: Learner must have mastered the skills in Course I, Basic Laboratory Skills, or have equivalent experience. In addition, learner must know how to operate a pH meter, assemble and use a membrane filtration assembly, and operate a spectrophotometer.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will filter his sample, standard and blank, adjust their pH, dilute them back to 50.0 ml, then add combined color reagent and determine their absorbances, according to the steps in EMP Procedures G, H, I and J as stated or described in the Information column.

2. Conditions: Learner will be given the equipment, EMP, no assistance and 90 minutes.

3. Accepted Performance: Learner will perform Procedures G, H, I and J to the satisfaction of an instructor. (Particularly note pH meter technique, accuracy of dilution, and correct technique in the use of the spectrophotometer.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of the EMP, overheads of overview of the four lessons and overview of "Transfers" to be made in these procedures. Variety of filtration assemblies.

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Présentation: Briefing (30 minutes)
   a. Use overhead of overview of four lessons
   b. Assignment - Have students read Procedure G
   c. Discussion
      2. G.7. Go through notes on pH adjustment.
      3. G.8. Note reason for this acid addition.
d. Briefly note contents of Procedures H, I, and J.
e. Use overhead of "Transfers" to be made in these procedures to give overview and to stress the importance of careful technique for quantitative recovery.
f. Assignment - If more than one spectrophotometer is being used, assign students to a particular instrument, emphasizing non-interchangeability of data from one instrument to another.

2. Student Performance and Evaluation: (90 minutes)

As stated in Instructional Objective above, by instructor rating.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus mg/liter P

UNIT OF INSTRUCTION: Construction of Calibration Curve and Reading,
Calculating and Reporting Data from the Calibration Curve (EMP Procedures K, M, N and O)

LESSON NUMBER: 4 of 5

ESTIMATED TIME: 30 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner will be responsible for interpreting the data of the test.

ENTRY LEVEL BEHAVIOR: Learner must be able to construct and use a calibration curve.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will construct a calibration plot and use it to convert the absorbance reading for his sample into a mg/liter P concentration reading according to EMP Procedures K, M, N and O.

2. Conditions: Learner will be given the EMP, a sheet of graph paper, the absorbance data for a set of calibration standards, his data sheet and 15 minutes.

3. Accepted Performance: Learner's calibration curve and obtained result must be the same as the instructor's calibration curve and sample result using the same data.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; overheads of the Example Data Sheet and the calibration curve form in EMP, pp. 49 and 50

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Lecture (15 minutes).
   a. Lecture - Discussion of EMP Procedures K, M, N and O: Use EMP Procedures K and M, and the overheads of the Data Sheet and the calibration curve form from the EMP to illustrate correct plotting procedure and use of a calibration curve.
   b. Distribution - Give each student a piece of graph paper.

2. Student Performance: (15 minutes) As stated in Instructional Objective above, by instructor rating.

   NOTE: The group, or groups, will need to pool data to construct a curve. Instructors may wish to have each group post their calibration standard data on the board.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Total Phosphorus, mg liter P

UNIT OF INSTRUCTION: Follow-up and Summary of Subject Matter

LESSON NUMBER: 5 of 5

ESTIMATED TIME: 45 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Learner should have an opportunity to ask any questions about the procedure and also should receive some final comments on sample dilutions, using other than 1/2 inch colorimeter tubes, the procedure modifications required to run an orthophosphate determination and the method of checking the continued accuracy of the calibration curve.

ENTRY LEVEL BEHAVIOR: Learner will have completed the entire Total Phosphorus EMP

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will have any unresolved questions about the procedure answered and will particularly consider the criteria for choosing sample volumes and rejecting the calibration curve.

2. Conditions: Copies of EMP, data sheet and calibration graph will be available.

3. Accepted Performance: Active participation in the 30 minutes wrap-up session as judged by the instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: EMP, calibration graph and data sheet

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Lecture-Discussion (45 minutes)

   a. Discuss results of test as appropriate. Answer questions.
   b. Note EMP Training Guide Sections VII, II and VI on dilutions, use of dilutions and using other-than-standard 1/2 inch colorimeter tubes. Answer questions.
   c. Note EMP Training Guide Section VII, E.3d on procedure modifications required to run orthophosphate determinations. Answer questions.
   d. Assignment - Have students read EMP Procedure D, steps 7 through 15, and EMP Procedure L. "Checking the Calibration Curve." Discuss and answer questions.
   e. Note EMP Procedure P, Clean-up.
   f. Answer any remaining questions.

2. Student Evaluation: Satisfactory participation in the session as judged by instructor.
A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES for the
DETERMINATION OF CHEMICAL OXYGEN DEMAND

as applied in
WASTEWATER TREATMENT FACILITIES and in the
MONITORING OF EFFLUENT WASTEWATERS

INSTRUCTIONAL PACKAGE WORKSHEET

National Training and Operational Technology Center,
Municipal Operations and Training Division
Office of Water Program Operations
U. S. Environmental Protection Agency
PART II INSTRUCTIONAL PACKAGE WORKSHEETS.

Determination of Chemical Oxygen Demand

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists one method to determine Chemical Oxygen Demand (COD): dichromate reflux.

2. The dichromate reflux method has been written in the Effluent Monitoring Procedure (EMP) format entitled "Determination of Chemical Oxygen Demand."

3. The reference source for the EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes." p. 20. Other FR sources for the procedure are:

4. The procedures in this EMP are for low level COD of 5-50 mg/l but there are references to the Training Guide for the modifications required for greater concentrations.

5. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for the determination of low level COD in a wastewater treatment plant effluent sample.

6. Each student is to process either one sample or one blank, and to standardize his ferrous ammonium sulfate titrant. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Summary of 6 Elements

ESTIMATED TIME: 240 minutes excluding up to 70 additional minutes for a 2 hour oxidation period.

JUSTIFICATION: The reporting of Chemical Oxygen Demand is required by many National Pollutant Discharge Elimination System permits.

ENTRY-LEVEL BEHAVIOR: The learner must know how to use volumetric glassware, pipet bulbs, and burets; how to perform titrations; and how to subtract, multiply, and divide numbers containing decimals.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will record sample identification information, prepare one sample or one blank for oxidation, standardize ferrous ammonium sulfate solution and, after rinsing and removing his flasks from the condensers, use the FAS for titration of the sample or blank. He will clean up his equipment, then use his laboratory data to calculate the chemical oxygen demand of his sample.

2. Conditions - He will be given classroom instruction, the required equipment conditioned and ready to use, the required reagents, a sample, a copy of the related EMP, copies of each of the data sheets in the EMP and total working time of 160 minutes.

3. Accepted Performance - His techniques must conform to those presented in the EMP according to an Instructor's rating. His volume for the ferrous ammonium sulfate standardization should be within ±0.05 ml of a majority of the class results and his COD value should be within ±4mg/liter of the class average unless some explanation of disagreement is accepted by the Instructor.

INSTRUCTIONAL RESOURCES:


C. INSTRUCTIONAL APPROACH:

1. Preparation for Instruction:
   a. Duplicate copies for each student of the two EMP data sheets. Two completed copies of each will be required. You can distribute one and later duplicate the completed sheet or you can distribute two and have the student make the copy.
   b. Duplicate for each student a copy of the two papers about mercury disposal.
   c. Obtain a sample (75 ml per student) of treatment plant effluent with COD between 5-50 mg/liter.
   d. Make out tags for the sample bottles (1 per 5 students) including the information required on the EMP "Typical Laboratory Data Sheet."
   e. See that the equipment and reagents required for each student to do the planned "Student Performances" are assembled in the laboratory.

2. Sequencing:
   a. Lesson one - 100 minutes.
   b. A break can be scheduled next, of any time duration provided someone is available to turn off the reflux apparatus two hours after lesson one ends. However, no break is necessary as regards teaching the procedure.
   c. Lesson two - 60 minutes.
      This standardization procedure should be scheduled on the same day as lesson three if possible.
   d. Lesson three - 45 minutes.
   e. Lesson four - 30 minutes.
      This lesson on calculations does not have to immediately follow lesson three.
   f. Lesson five - 20 minutes.
      This discussion of variable aspects of the test does not have to immediately follow lesson four.
   g. Lesson six - 10 minutes.
      This discussion of the corrected data sheets will have to be scheduled after the Instructor collects (lesson 4) and corrects (outside class time) the data sheets.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS
   (1 sample or blank, 1 FAS standardization, titration)

1. For each student:
   1. Apron
   1. Beaker, glass, 150 ml - 250 ml (to transfer FAS)
   1. Buret, 25 ml; 0.1 ml graduations, teflon stopcock
   1. Clamp, buret, for titration stand
   1. Cylinder, graduated, 25 ml
   1. Cylinder, graduated, 100 ml
   1. Evaporating-dish (to place between flask and heating surface for cooling)

See NOTE, next page, bottom
1. **For each student (continued)**
   - Flask - condenser - heating surface assembly with tubing connections to a source of cooling water.
   - 5 Beads, glass boiling, in the reflux flask
   - 1 Flask, Erlenmeyer, wide mouth 600 ml, with volumes marked on the outside.
   - 1 Funnel, short stem (to fill 25 ml buret).
   - 1 Pair safety glasses
   - 1 Pan for ice water to cool mixture in reflux flask at various stages of the test.
   - 1 Pencil, wax
   - 1 Pipet bulb, preferably propipet to avoid accidents when pipeting \( \text{H}_2\text{SO}_4 \)
   - 1 Pipet, graduated, 10 ml
   - 1 Pipet, volumetric, 10 ml
   - 1 Pipet, volumetric, 25 ml
   - 1 Pipet, volumetric, 50 ml.
   - 1 Stand, titration, support for buret.
   - Towels, paper
   - 1 Wash bottle, squeeze type 500 ml.

   **NOTE:** All glassware should be conditioned for use in the test according to EMP Procedures D and E.

2. **Shared:**
   - Two liter container per 6 students for test wastes containing mercury complexes and significant amounts of sulfuric acid.
   - 1 Buchner funnel for each test wastes container (to catch glass beads from flask).
   - 1 Reagent spoon per 3 students to roughly measure 1 gram of mercuric sulfate.
   - 1 Sample bottle, tagged, per 3 students. Should preferably be glass but plastic may be used if it is known that no organic contaminants are present.

**E. IPW REAGENT REQUIREMENTS:** (minimum amounts per student)
- 17 ml distilled water, high quality, low COD
- 11 drops ferroin
- 75 ml 0.025 N ferrous ammonium sulfate solution (dilute 100.0 ml of 0.250 N per 1 liter)
- 1 g mercuric sulfate
- 50 ml 0.025 N potassium dichromate solution (dilute 100.0 ml of 0.250 N per 1 liter)
- 40 ml concentrated sulfuric acid
- 85 ml sulfuric acid-silver sulfate solution

**NOTE:** Student will need a tray of ice cubes for cooling bath during test.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Procedure A Preparing to Test the Sample and Procedure F Oxidation of the Sample and Blank.

LESSON: 1 of 6

ESTIMATED TIME: 100 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner must perform these procedures to determine COD.

ENTRY LEVEL BEHAVIOR: The learner must know how to use volumetric glassware and pipet bulbs.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will record sample identification information, prepare one sample or one blank in a reflux flask, then attach the flask to a condenser and begin heating it, all according to EMP Procedures A and F.

2. Conditions - He will be given classroom instruction, a copy of the EMP and a related data sheet, the required reagents (Procedure B), the required equipment conditioned and ready to use (Procedures D and E), a sample, minimal supervision and total working time of 40 minutes.

3. Accepted Performance - His techniques must conform to those described in the EMP according to an Instructor's rating. (Particularly note caution in pipeting sulfuric acid and transferring it to the flask).

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP; 19 slides pertaining to overview of test (2), Procedure A. Step 2 Reagents (2), A. Step 3 Conditioning (3), A. Step 4 Conditioning (11) and Procedure F (11); 1 overhead and copies of the EMP "Typical Laboratory Data Sheet."

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH:

1. Presentation (60 minutes)
   b. Lecture using board - EMP p. 4 - Reaction, then Quantitation
      1) Develop equation with essentials first: reagents - oxidation time - temperature.
      2) Equipment: function of condenser
      3) Catalyst: define "catalyst" = Ag$_2$SO$_4$ - why.
      4) Chloride interference - effect on catalyst - effect on potassium dichromate - adding HgSO$_4$.
      5) Want excess K$_2$Cr$_2$O$_7$ to titrate and then calculate COD.
      6) Blank - why necessary - used in calculations.
c. Use 19 slides to teach overview of test (2); Procedure A and its references to Procedures B(2), D(3) and E(1); Procedure F(11)

d. Go through A steps 1-4 to note references and locations of EMP Procedures B, D, E.

e. A Step 5 - Distribute copies of EMP "Typical Laboratory Data Sheet."
   (Option: Two completed sheets will be required for each student. You can distribute 1 and later duplicate the completed sheet or you can duplicate 2 and have the student make the copy).

f. Note left column on data sheet contains step designation, the right column contains example numbers. (Overhead can be used)

g. Have students make a note at the end of A Step 5 to turn to Procedure F for the next part of laboratory assignments.

h. Procedure F is written to do 1 sample and 1 blank.
   1) Assign the blank to 2 students, warning them to read carefully and just do F steps pertaining to a blank.
   2) Rest of class are to do steps pertaining to a sample and omit steps referring to the blank.
   3) F Step 12 - Stress hazards of H₂SO₄. Demonstrate using a pipet bulb or an automatic dispenser.

2. Student Assignment - Do Procedure A step 5 and all of Procedure F in the laboratory.

3. Student Performance and Evaluation (40 minutes)
   As stated in the Instructional Objective above.

4. NOTE: It is not essential that the mixtures boil exactly 2 hours for this exercise. This IPW provides 50 minutes of other instruction before the heat sources must be cut off.
SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Procedure C. Standardizing Ferrous Ammonium Sulfate Solution

LESSON: 2 of 6

ESTIMATED TIME: 50 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: On the day of use, the learner must standardize the ferrous ammonium sulfate solution.

ENTRY LEVEL BEHAVIOR: The learner must know how to use volumetric pipets, pipet bulbs, burets and how to titrate a solution to a color change end point.

A. INSTRUCTIONAL OBJECTIVE:

1. **Terminal Behavior** - The learner will titrate one aliquot of the ferrous ammonium sulfate solution, record his result, use the posted results of the class as "duplicate" results to check his work, and then calculate the normality of the solution, all according to EMP Procedure C.

2. **Conditions** - He will be given classroom instruction, a copy of the EMP and a related data sheet, the required reagents (Procedure B), the required equipment conditioned and ready to use (Procedures D and E), minimal supervision and total working time of 40 minutes.

3. **Accepted Performance** - His techniques must conform to those described in the EMP according to an Instructor's rating. (Particularly note the drop-by-drop addition technique near the end point of the reaction). His total ml added should be equal to or within ±0.05 ml of a majority of the class results as determined by the Instructor who corrects the completed data sheets.

B. INSTRUCTIONAL RESOURCES:

1. **Available Media** - EMP; 1 overhead of EMP-Flow Sheet; copies of the EMP data sheet for "Standardization of Ferrous Ammonium Sulfate Solution" and 1 overhead of same; 6 slides pertaining to overview of Procedure C and the color change at the end of the titration (2); 1 overhead reviewing rules for rounding of numbers.

2. **Suggested Media** - None

C. INSTRUCTIONAL APPROACH

1. **Presentation** (10 minutes)
   a. Use overhead of Flow Sheet so students see where they are in doing the test.
   b. Use 4 slides to give overview of Procedure C.
   c. Distribute copies of EMP data sheet for "Standardization of Ferrous Ammonium Sulfate Solution." (Option: Two completed data sheets will be required for each student. You can distribute 1 and later duplicate the completed sheet or you can distribute 2 and have the student make the copy).
d. Use overhead to show left column on data sheet contains step designation, the right column contains examples for duplicate titrations.
e. The EMP has directions to do duplicate titrations. For this class exercise each student should do one titration and use class results as a check according to Procedure C. Thus all students should omit steps beginning with the word "Repeat" (Steps 3, 10, 15, 32).
f. Present 2 slides which show the color change for the end point of the titration.

2. Student Assignments - Do Procedure C through step 33 then turn off the heat under your test flask and insert an evaporating dish upside down between the flask and heating surface. Go to the classroom and finish Procedure C.

3. Discussion of Duplicates - When all the students are back in the classroom, put their ml results from C. step 33 on the board and go through C. step 34 on allowable differences for duplicates.

4. Review of Rounding Off Numbers - Use overhead to review for C. step 37 rounding off operation which is recorded as C. 38 on the data sheet.

5. Student Performance and Evaluation (40 minutes)
   As stated in the Instructional Objective above.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand


LESSON: 3 of 6

ESTIMATED TIME: 45 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner must perform these procedures to determine Chemical Oxygen Demand.

ENTRY LEVEL BEHAVIOR: The learner must know how to use a buret and how to titrate a solution to a color change end point.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will rinse and remove his reaction flask from the condenser, titrate the excess potassium dichromate in it with the ferrous ammonium sulfate solution he standardized in lesson 2, record his results, and clean up his equipment, all according to EMP Procedures G, H and I.

2. Conditions - He will be given classroom instruction, a copy of the EMP and a related data sheet, the required reagents (Procedure B), the required equipment conditioned and ready to use (Procedures D and E), minimal supervision and total working time of 30 minutes.

3. Accepted Performance - His techniques must conform to those described in the EMP according to an Instructor's rating. (Particularly note the drop-by-drop technique near the end point of the reaction).

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP; copies of the EMP Data Sheet; 1 overhead of EMP Flow Sheet; slides pertaining to Procedure H titration color change (use the 2 from lesson 2) and 3 slides of rinsing and removing the flasks from the condensers and bringing the solution in volume.

2. Suggested Media - None.

C. INSTRUCTIONAL APPROACH:

1. Presentation (15 minutes)
   a. Use overhead of Flow Sheet so students see where they are in doing the test.
   b. Re-project the 2 slides of the color change at the end point of titration in Procedure C to stress that Procedure H is the same process.
   c. Contrast and compare Procedure H to Procedure C.
      1) Only add ferroin to the diluted test mixture, then titrate.
      2) Add 10 drops of ferroin this time since the diluted volume is greater here.
d. Use 3 slides to preview Procedure G, removing flasks.
e. Procedures G and H are written for testing a sample and a blank.
   Use the steps that apply to your assignment. (See next section).

2. Student Assignment - Do Procedures G, H, and I in the laboratory. For
   G, all do steps 1 through 10; for H, all do steps 1 through 16; for I,
   all do all steps.

3. Student Performance and Evaluation (30 minutes)
   As stated in Instructional Objective above.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Procedure J. Calculations

LESSON: 4 of 6

ESTIMATED TIME: 30 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner may be responsible for using the test data to calculate the mg/liter Chemical Oxygen Demand.

ENTRY LEVEL BEHAVIOR: The learner must know how to subtract, multiply, and divide numbers containing decimals and how to round off numbers.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will use his laboratory result to calculate the Chemical Oxygen Demand of his sample according to EMP Procedure J and report that result to the Instructor. He will also turn in his completed data sheets for the sample and for standardizing the ferrous-ammonium sulfate solution.

2. Conditions - He will have the EMP and the related data sheets he used during earlier lessons, the results for titrating the blank, supervision and a total working time of 20 minutes.

3. Accepted Performance - The data sheets must be complete and with the same final answer as the Instructor calculates from the same data. Also, his value for COD must agree within ±4 mg/liter of the class average unless some explanation of disagreement is accepted by the Instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP, copies of EMP Data Sheets, overheads (3) of the EMP Flow Sheet, the EMP Data Sheet and examples of rounding off numbers.

2. Suggested Media - None

C. INSTRUCTIONAL APPROACH:

1. Introduction (1 minute)
   a. Use overhead of Flow Sheet to show students where they are in the EMP.
   b. Procedure J has step-by-step directions on using the data sheet to calculate COD. The steps follow from J.1a, the calculation formula for COD which is also printed at the bottom of the data sheet. Use overhead to show area of data sheet used for calculations.

2. Presentation (4 minutes)
   a. Note A = ml. FAS to titrate the blank. Post the ml values from the 2 students who did blanks.
   b. Discuss the criteria for acceptable blanks in Training Guide VII.
   c. If both blank volumes are acceptable, average the numbers.
   d. If only one blank volume is acceptable, use just that one.
   e. Have the class record the blank value on their data sheets in the column they used for their sample, on line 9.
3. **Student Assignment** - Use the steps in Procedure J and your own results on your data sheet to calculate the COD of your sample. The two students who did the blank should also do the calculation, using 50.0 ml on line 6 and 10.0 ml on line 10. During this time some may need to see the overhead on rounding off numbers.

4. **Discussion (5 minutes)**
   As students get results, see that they are posted on the board. When all are available, discuss them and give the students a chance to deduce causes for any far-ranging numbers. Some points to be covered are novice technique, differing FAS normality values, the effect of the 8000 factor.

5. Collect completed data sheets from the students.

6. **Student Performance (20 minutes)**
   As stated in the Instructional Objective above.

7. **Student Evaluation**
   The instructor will correct the data sheets outside of class time. On the standardization sheet, accepted performance is a result agreeing ±0.05 ml with a majority of the class results. On the laboratory data sheet, accepted performance is a result agreeing ±4mg/liter with the class average unless some explanation of disagreement is accepted by the instructor. A corrected copy of each sheet is to be returned to the student and a second corrected copy of each sheet is to be retained by the instructor for the course records.

   Students often "slip up" on the following items on the data sheet:
   a. They fail to record Name of Plant.
   b. Lines 9 and 10 each require two pieces of information. They often record only one.

   Correcting the sheets gives an opportunity to check:
   a. If student reads and records buret volumes to two decimal places (lines 7 through 11).
   b. If student correctly rounds off numbers (line 12 is result from FAS Sheet; line 16).

   A detail that can also be noted is using a zero with decimal points as in line 6 (50.0) and line 12 (0.02X).
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Final Comments on Training Guide Topics - Smaller Volumes of Sample (VII), Automatic Dispensers (V), Two Hour Oxidation Period (VII), and Disposal of Mercury - Containing Wastes (VI).

LESSON: 5 of 6

ESTIMATED TIME: 20 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: In the future the learner may have to make decisions to modify these aspects of the procedure and he should learn the basis for making these choices.

ENTRY LEVEL BEHAVIOR: Learner should have completed lessons 1 through 4.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will particularly consider the topics for this unit of instruction and have any unresolved questions about this information answered.

2. Conditions - He will have his EMP, copies of two papers on mercury disposal, and an opportunity for asking questions during the 20 minute session.

3. Accepted Performance - Active participation in the session as judged by the instructor.

B. INSTRUCTIONAL RESOURCES:


2. Suggested Media - None

C. INSTRUCTIONAL APPROACH:

1. Introduction (2 minutes)
   Go through the use of keys in the fourth column of the EMP to locate related information in the Training Guide.

2. Discuss the Unit Topics from the Training Guide (18 minutes)
   a. Smaller Volumes of Sample for the Test (VII) - 1 slide
      1) Especially note point 6 regarding volumes.
   b. Automatic Dispensers (V) - 1 slide
      1) Especially for acids
      2) Last two paragraphs have amounts
      3) You must check delivered volumes.
c. Two Hour Oxidation (Boiling) Period (VII) - 1 slide
   1) Must do 7 duplicates over a period of several days so variability of the sample source is more likely to show up.
   2) Results must agree within the precision limits of the test, ±4 mg/liter.
   3) Should re-check periodically by running duplicates.

d. Disposal of Mercury-Containing Wastes (VI) - 3 slides
   1) Pass out copies of the two papers cited in the Training Guide.
   2) Dean, et al: Raise the pH to 10 or higher and use a 40-50% w/v solution of sodium thiosulfate to precipitate the mercury as a sulfide. Note list of reprocessors.
   3) Maag and Hecker: Use aluminum (foil) to displace mercury which can then be recovered in metallic form.

3. Student Performance and Evaluation - As stated in Instructional Objective above.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Chemical Oxygen Demand

UNIT OF INSTRUCTION: Discussion of Corrected Data Sheets

LESSON: 6 of 6

ESTIMATED TIME: 10 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner should have an opportunity to question any corrections made on his data sheet.

ENTRY LEVEL BEHAVIOR: Learner should have completed lessons 2 and 4.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior - The learner will ask any questions or give any explanations regarding his corrected data sheets.

2. Conditions - He will have EMP and corrected data sheets, and 10 minutes.

3. Accepted Performance - Active participation in the session as judged by the instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media - EMP, corrected data sheets.

C. INSTRUCTIONAL APPROACH:

1. Discussion of Corrected Data Sheets (10 minutes)
   a. Distribute corrected data sheets for the sample and for standardizing the ferrous ammonium sulfate solution.
   b. Ask if there are any questions or explanations about notes made on any of the sheets.
   c. Make any observations that are of general interest to the class or which stress critical aspects of the test.

2. Student Performance and Evaluation - As stated in the Instructional Objective above.
A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES for the DETERMINATION OF TOTAL KJELDAHL NITROGEN

as applied in WASTEWATER TREATMENT FACILITIES and in the MONITORING OF EFFLUENT WASTEWATERS

INSTRUCTIONAL PACKAGE WORKSHEET

National Training and Operational Technology Center Municipal Operations and Training Division Office of Water Program Operations U.S. Environmental Protection Agency

CH.N.IPW.1.6.77
PART II INSTRUCTIONAL PACKAGE WORKSHEETS

E. Determination of Total Kjeldahl Nitrogen

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists one method for the first phases of the Total Kjeldahl Nitrogen Test: digestion of the sample to convert certain nitrogen forms to ammonia and distillation to isolate the ammonia for quantification. It lists three manual methods for the subsequent measurement of the ammonia: nesslerization (for concentrations of 0.05-1 mg/l), titration (for 1-25 mg/l) and electrode (for concentrations from 0.05-1400 mg/l).

2. The digestion, distillation, nesslerization and titration methods for macro or micro sample volumes have been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Total Kjeldahl Nitrogen." There is a separate EMP for the electrode method ("Determination of Ammonia by an Ammonia Selective Electrode") which can be used for the ammonia quantification part of the Total Kjeldahl Nitrogen Determination.

To determine only Organic Nitrogen, a sample is processed for Total Kjeldahl Nitrogen, and also for original Ammonia. Organic Nitrogen is the difference between these two. Organic Nitrogen can be determined directly by removal of ammonia from the sample (by boiling or distillation) prior to the digestion procedure of the Total Kjeldahl Nitrogen Test. In addition to the Selective Electrode EMP, there is an EMP for the distillation, nesslerization and titration methods for macro sample volumes, "Nitrogen, Ammonia Determination."

3. The reference source for the Total Kjeldahl Nitrogen EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 175. Other FR sources for the procedure are:

4. The procedures in this EMP provide a choice of macro or micro sample volumes, and a choice of nesslerization or titration for the Ammonia measurement.

5. In the following Instructional Package Worksheet the lesson plan is for digestion and distillation of a micro volume of a WWTP effluent sample followed by nesslerization for the ammonia quantification.

6. Each student is to process one sample through the digestion, distillation and nesslerization procedures and also one blank and two standards through the nesslerization procedure. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Total Kjeldahl Nitrogen

UNIT OF INSTRUCTION: Summary of 3 elements

ESTIMATED TIME: 5 hours

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The reporting of Total Kjeldahl Nitrogen is required by many National Pollutant Discharge Elimination System permits.

ENTRY LEVEL BEHAVIOR: The learner must have completed Course I, Basic Laboratory Skills or have shown competence in the areas covered by this course. Course II, Basic Parameters for Municipal Effluents, is not a prerequisite, but is desirable. The learner must also be able to:

1. Adjust pH of a sample
2. Use a spectrophotometer
3. Plot a calibration curve

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will perform all laboratory work to determine Total Kjeldahl Nitrogen by the micro method and Nesslerization, then will calculate results.

2. Conditions: The learner will have available all necessary glassware, reagents and equipment, and a copy of the Total Kjeldahl Nitrogen Effluent Monitoring Procedure.

3. Accepted Performance: The learner will choose and properly deliver the correct reagents, correctly prepare a calibration curve and correctly perform the final calculations. He must obtain at least 80% on an overall evaluation of these tasks (checklist in Lesson 3), or else repeat the analysis until he obtains the 80% rating.

B. INSTRUCTIONAL RESOURCES

1. Available Media
   a. Total Kjeldahl Nitrogen Effluent Monitoring Procedure (EMP)
   b. 1974 EPA Methods of Chemical Analysis, page 175.
   c. Transparencies available from National Training and Operational Technology Center (NTOTC), OT-9(N) Series of 9: the figures from Total Kjeldahl Nitrogen EMP.
   d. A Performance Checklist is part of Lesson 3.

2. Suggested Media - Video Tape presentation showing both micro and macro methods of the Total Kjeldahl Nitrogen Determination.

The Total Kjeldahl Nitrogen Determination may be performed by using either the micro method or the macro method. The procedure has been written primarily for the micro method, although suggested procedures and equipment are listed in the Effluent Monitoring Procedure for the macro method. It should be noted that the micro method requires a micro Kjeldahl Digestion unit and a steam distillation apparatus. If this equipment is used then the determination may be performed in the 5-hour time frame. The macro method would require a macro digestion and distillation apparatus, and the time required would be 7-hours.

In order to perform the analysis in the 5-hour time frame, all reagents except for the colorimetric standards must be prepared by the Instructor. These are itemized later in this Instructional Package Worksheet.

1. Preparation for Instruction:

a. Duplicate copies of the EMP Data Sheet and Calibration Graph form for each student.
b. Have on hand a treatment plant effluent sample of known TKN concentration, so that a suitable dilution will be assigned. (A maximum of 50 milliliters per student will be needed.)
c. Make out tags for the sample bottles. Include information required on the EMP example laboratory data sheet.
d. Make sure that all equipment and reagents required for each student to do planned student performances are assembled in the laboratory. For the micro method with a maximum class size of 12, two digestion racks are needed and at least three separate steam distillation units should be available. This allows each student to have his/her own sample. Since the steam distillations only require 10 minutes per sample, three are sufficient.

If the macro method is chosen for instruction, then a digestion rack with 12 burners is recommended as well as a distillation rack capable of holding the same number. For the other macro equipment and reagents, see Chapter 8 in this Staff Guide.

The equipment needed for the Total Kjeldahl Nitrogen Determination is somewhat complex and consequently should be checked out thoroughly by the Instructional Staff prior to course time.

2. Sequencing:

a. Lesson one-60 minutes. Theoretical Concepts of the Nitrogen Determination to be discussed in the classroom.
b. Lesson two-45 minutes: It is advantageous to explain only the digestion and to have the student set up and begin digestion of his sample. Then the Instructor can give the complete procedure description, including the utility of the micro and macro methods, sample handling and preservation and a demonstration of both micro and macro pieces of equipment.
c. A break can be scheduled, such as a lunch break, during the digestion of the sample.

d. Lesson three - 3 hours. Analysis of the sample. This includes the completion of the digestion, steam distillation, colorimetric determination, calculations and discussion of results.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS: (1 sample for digestion, plus 1 blank and 2 standards for Nesslerization)

1. For each student:
   a. 1 apron
   b. 6 boiling beads
   c. 1 50 ml beaker, graduated
   d. 1 100 ml beaker, graduated
   e. 2 150 ml beakers, graduated
   f. 2 10 ml cylinders, graduated
   g. 1 100 ml cylinder, graduated
   h. 1 50 ml Erlenmeyer flask, graduated
   i. 1 125 ml Erlenmeyer flask, graduated
   j. 1 100 ml Kjeldahl flask
   k. 4 Nessler tubes scored at 50 ml
   l. 2 10 ml pipets (Mohr) graduated
   m. 1 xx ml pipet, volumetric, of size required if sample dilution is necessary
   n. 1 50 ml pipet, volumetric
   o. 4 No. 3 or No. 6 rubber stoppers or caps to fit the Nessler tubes

2. Shared:
   a. Digestion apparatus, micro, 1 for every 6 students
   b. Distillation apparatus, steam, micro, 1 for every 4 students
   c. Spectrophotometer, 1 for every 3 students
   d. Nessler tube support, 1 for every 2 students

E. IPW REAGENT REQUIREMENTS: (minimum amounts per student)
   a. 5 ml boric acid solution, 2%
   b. 10 ml sodium hydroxide-sodium thiosulfate solution
   c. 2 drops phenolphthalein solution
   d. 4 ml Nessler reagent
   e. 0.18 ml stock ammonium chloride solution
   f. 10 ml digestion reagent
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Total Kjeldahl Nitrogen

UNIT OF INSTRUCTION: Theoretical concepts

LESSON NUMBER: 1 of 3

ESTIMATED TIME: 1 hour

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The student should know what forms of nitrogen this test measures and the significance of the test in waste treatment plant operations.

ENTRY LEVEL BEHAVIOR: The learner must have completed Course I, Basic Laboratory Skills, or have shown competence in the areas covered by this course. Course II Basic Parameters, is not a prerequisite, but is desirable.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will participate in classroom discussion.

2. Conditions: The learner will have a copy of the Total Kjeldahl Nitrogen Effluent-Monitoring Procedure.

3. Accepted Performance: The learner will answer all questions orally given by the instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media:
   a. Effluent Monitoring Procedure Training Guide Notes, Section I

2. Suggested Media:

C. INSTRUCTIONAL APPROACH: (Sequencing)

1. Presentation (45 minutes)

   Instructor should follow the outline in Training Guide, Section I of the Effluent-Monitoring Procedure to introduce the student to the utility of the Total Kjeldahl Nitrogen Determination. The transparencies mentioned above should be used in conjunction with the lecture material.

2. Student Performance & Evaluation (15 minutes)

   As stated in the Instructional Objective above by Instructor rating.
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Total Kjeldahl Nitrogen

UNIT OF INSTRUCTION: Procedure Description

ESTIMATED TIME: 45 minutes  LESSON NUMBER: 2 of 3

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The student must be instructed in the procedure steps so he can perform the determination in the laboratory.

ENTRY LEVEL BEHAVIOR: The learner must have completed Course I, Basic Laboratory Skills or have shown competence in the areas covered by this course. Course II Basic Parameters is not a prerequisite, but is desirable. He must have completed Lesson 1 of this IPW.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will begin digestion of his sample, then participate in classroom discussion of the complete determination.

2. Conditions: The learner will have a copy of the Effluent Monitoring Procedure, Total Kjeldahl Nitrogen Determination.

3. Accepted Performance: The learner will answer all questions orally given by the Instructor.

B. INSTRUCTIONAL RESOURCES:

   a. #1 Macro Digestion Apparatus
   b. #2 Macro Distillation Apparatus
   c. #3 Micro Digestion Apparatus
   d. #4 Micro Steam Distillation Apparatus
   e. #5 Micro Steam Distillation Apparatus

2. Suggested Media: Video Tape showing both micro and macro methods for TKN

C. INSTRUCTIONAL APPROACH: (Sequencing)

1. Presentation (30 minutes)
   a. Instructor should follow the outline of the procedures covering topics listed. He should explain the digestion step and have the students go to the laboratory to start digestion of a sample. Then the students return to the classroom and the Instructor discusses the complete procedure.
b. It should be emphasized again at this point that the Total Kjeldahl Nitrogen can be performed by either the macro or the micro method, and the differences should be noted. In addition, the difference between the colorimetric and titrimetric determinations should be explained. The flow chart on page 5-5 of the SMP should be used for this purpose.

2. Student Performance & Evaluation: (15 minutes)

As stated in Instructional Objective above by Instructor rating.
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Total Kjeldahl Nitrogen.

UNIT OF INSTRUCTION: Analysis

LESSON NUMBER: 3 of 3

ESTIMATED TIME: 3½ hours

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The student should perform the determination with instructor observation and supervision so he can later do the analysis correctly by himself.

ENTRY LEVEL BEHAVIOR: The learner must have completed the Basic Laboratory Skills course or have shown competence in the areas covered by this course. Course II, Basic Parameters, is not a prerequisite but is desirable. He must have completed lessons 1 and 2 of this series. The learner must also be able to perform the following:

1. Adjust pH of a sample
2. Use a spectrophotometer
3. Plot a calibration curve

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will perform the Total Kjeldahl Nitrogen Determination by the micro method and Nesslerization, construct a calibration curve and do the final calculations according to the Effluent Monitoring Procedure.

2. Conditions: The learner will have at his disposal all the necessary equipment and reagents and the EMP.

3. Accepted Performance: The student must obtain an 80% on an Instructor checklist, or repeat until he does. (See C.3. Student Evaluation.) Note: The checklist includes a section for the completion of the student report form.

B. INSTRUCTIONAL RESOURCES:


   a. #6 Adding solution to form alkaline layer, and
   b. #7 %Transmittance and Absorbance Scale

2. Suggested Media:
C. INSTRUCTIONAL APPROACH: (Sequencing)

1. **Presentation (15 minutes)**

   All students steam-distilling the sample should be supervised very, very closely. The Instructor should demonstrate use of steam distillation apparatus prior to the student operation.

2. **Student Performance: (3 hours)**

   a. Student finishes digesting sample.
   b. Student steam distills sample (micro method).
   c. Student prepares standards for calibration curve, (Nesslerization).
   d. Student, using standards and sample, plots calibration curve, and obtains ammonia concentration.
   e. Student calculates final Total Kjeldahl Nitrogen concentration.

   f. **NOTE**: With a time frame of 3½ hours, there is at least 30 minutes available at the end of the analysis for discussion of results.

3. **Student Evaluation**: Accepted performance as mentioned in A.3 above will be evaluated by the Instructor using checklist like the following:

   **EXAMPLE PERFORMANCE CHECKLIST**
   
   **for the**
   
   **TOTAL KJELDAHL NITROGEN DETERMINATION**

   A. Correct reagents used in digestion and distillation. YES __ NO __
   B. Reagents delivered properly. YES __ NO __
   C. Titration procedure performed correctly, (if Titration used.) YES __ NO __
   D. Calibration curve prepared correctly, (if Nesslerization used.) YES __ NO __
   E. Calculations performed correctly. YES __ NO __
   F. Calculations evaluated by written quiz were correctly done. YES __ NO __
   G. Final evaluation for record: Satisfactory _______ Unsatisfactory _______

   STUDENT

   INSTRUCTOR
A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES
for the
NITROGEN, AMMONIA DETERMINATION

as applied in
WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

INSTRUCTIONAL PACKAGE WORKSHEET

National Training and Operational Technology Center
Municipal Operations and Training Division.
Office of Water Program Operations
U. S. Environmental Protection Agency
PART II INSTRUCTIONAL PACKAGE WORKSHEETS

F. Nitrogen, Ammonia Determination

1. The Federal Register (FR) issuance in Outline 1 of this Guide lists one method for the isolation of Ammonia from a water sample, i.e., manual distillation at pH 9.5.

In a footnote, #4, it states: "Manual distillation is not required if comparability data on representative effluent samples are on company file to show this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies."

The same FR issuance lists three manual methods for the measurement of the isolated Ammonia: nesslerization (for concentrations of 0.05-1mg/l), titration (for 1-25mg/l) and electrode (for concentrations from 0.05-1400 mg/l).

2. The distillation, nesslerization and titration methods for macro sample volumes have been written in the Effluent Monitoring Procedure (EMP) format entitled, "Nitrogen, Ammonia Determination." There is a separate EMP for the electrode method, "Determination of Ammonia by an Ammonia Selective Electrode." The distillation, nesslerization and titration methods for both macro and micro sample volumes are contained in another EMP, "Determination of Total Kjeldahl Nitrogen."

3. The reference source for the Nitrogen, Ammonia Determination EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 159. Other FR sources for the procedure are:
   a. 14th ed., APHA, "Standard Methods for the Examination of Water and Wastewater," p. 410 (Distillation) and p. 412 (nesslerization);

4. The procedures in this EMP are for macro sample volumes with a choice of nesslerization or titration for the ammonia measurement.

5. In the following Instructional Package Worksheet the lesson plan is for distillation of a macro volume of a Wastewater Treatment Plant effluent sample followed by titration.

6. Students are to work in pairs to distill and titrate one sample. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrogen, Ammonia Determination

UNIT OF INSTRUCTION:

LESSON NUMBER: 1 of 1

ESTIMATED TIME: 1 1/2 hours

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner should know how to perform the distillation procedure for the determination of ammonia nitrogen since it is required by many National Pollutant Discharge Elimination System permits.

ENTRY LEVEL BEHAVIOR: The learner must be able to:

1. Perform basic mathematical computations.
2. Handle reagent chemicals safely.
3. Be familiar with laboratory apparatus.
4. Understand terms such as liter, milliliter, gram, and milligram.
5. Understand the term normality, to the extent that it is a chemical way of expressing concentration.
6. Perform weighings on analytical and trip balances.
7. Clean laboratory glassware.
8. Prepare chemical solutions.
9. Perform a titration.
10. Use a colorimeter.

A. INSTRUCTIONAL OBJECTIVE

1. Terminal Behavior: The learner will exhibit proper technique while determining the ammonia-nitrogen content of a wastewater treatment plant effluent.

2. Conditions: The learner will have the use of the attached EMP, and all chemicals and equipment listed in it.

3. Accepted Performance: The use of proper technique in performing the test will be judged by the instructor.

B. INSTRUCTIONAL RESOURCES

1. Available Media: 8 minute slide-tape, XT-67, Determination of Ammonia Nitrogen*

C. INSTRUCTIONAL APPROACH (Sequencing)

1. Classroom - discussion of the importance of the \( \text{NH}_3-N \) determination by the instructor and the use of an audiovisual unit, XT-67.

*Available from the U.S. Environmental Protection Agency, Office of Water Program Operations, National Training & Operational Technology Center, Cincinnati, Ohio 45268
2. Laboratory - determination of the NH$_3$-N concentration of a wastewater treatment plant effluent.

3. Classroom - discussion of calculations.


D. EQUIPMENT AND SUPPLY REQUIREMENTS:

(Macro distillation plus titration of 1 sample per 2 students)

1. For each student:

   All equipment is shared; see below

2. Shared:

   - beaker, 50 ml (1 per 2 students)
   - beaker, 600 ml (1 per 2 students)
   - boiling beads, glass, 5 mm (6 per 2 students)
   - bottle, glass, 100 ml dispensing (1 per 2 students)
   - bottle, glass, 1000 ml with glass stopper (1 per 18 students)
   - bottle, glass, 1000 ml with rubber stopper (1 per 18 students)
   - buret, 50 ml with 0.1 ml graduations (1 per 2 students)
   - clamp (1 per 2 students)
   - cylinder, graduated, 100 ml (2 per 2 students)
   - cylinder, graduated, 600 ml (1 per 2 students)
   - distillation assembly (1 per 2 students). Diagram is in Kjeldahl Nitrogen EMP in equipment list.
   - Meker burner
   - 800 ml Kjeldahl flask
   - Kjeldahl spray trap, connecting bulb, 55 mm
   - Graham or Allihn condenser, 16 inch
   - rubber stopper #3
   - rubber stopper #7
   - flask, Erlenmeyer, 500 ml (1 per 2 students)
   - flask, volumetric, 1000 ml with stopper (1 per 18 students)
   - stand, titration with porcelain base for buret.

E. IPW REAGENT REQUIREMENTS:

(minimum amounts per student)

- 10 g suitable detergent
- pH paper, strips, short range to test at pH 9.6
- 10 ml sodium hydroxide (1N)
- 25 ml borate buffer
- 50 ml boric acid solution (2%)
- 90 ml (maximum) standard sulfuric acid solution (0.02N)
- 3 drops mixed indicator
- 100 ml ammonia-free distilled water
A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES FOR THE DETERMINATION OF NITRATE-NITRITE NITROGEN AND OF NITRATE-NITROGEN, CADMIUM REDUCTION METHOD as applied in WASTEWATER TREATMENT FACILITIES and in the MONITORING OF EFFLUENT WASTEWATERS.

Instructional Package Worksheet

National Training and Operational Technology Center Municipal Operations and Training Division Office of Water Program Operations U.S. Environmental Protection Agency
PART II INSTRUCTIONAL PACKAGE WORKSHEETS

G. Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists two manual methods to determine Nitrate (as N): cadmium reduction and brucine sulfate (formation of a complex).

2. The cadmium reduction method has been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Nitrate-Nitrite Nitrogen and of Nitrate Nitrogen, Cadmium Reduction Method". The simpler equipment and techniques required are ideal if equipment and experienced personnel are limited, and the test is required intermittently. Furthermore, the method yields data for concentrations of nitrate plus nitrite, of nitrate and of nitrite.

If only nitrate data is required, the time involved in using this method becomes a significant consideration when testing is done on a daily basis. The brucine sulfate method might be preferred. An EMP for the brucine sulfate method is in preparation.

3. The reference source for the Nitrate-Nitrite, Nitrate Nitrogen, Cadmium Reduction Method EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 201. The only other FR source for the procedure is:


4. The procedures in this EMP are for determining Nitrate plus Nitrite (asN) and Nitrite (as N). Nitrate (as N) concentrations are obtained by calculating the difference of the above. The EMP also includes the calculation of Nitrate (as NO₃) and of Nitrite (as NO₂) in case permits require these expressions of the data.

5. In the following Instructional Package Worksheet (IPW) the lesson plan is for determining nitrate plus nitrite, then nitrite in a synthetic sample, then doing the calculations listed above in 4.

6. Each student is to prepare one reduction column and then process one blank, one standard, and one sample to determine nitrate plus nitrite (as N) concentration. He is also to process one blank, one standard, and the same sample to determine nitrite (asN) concentration. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.
SUBJECT MATTER: Nitrate + Nitrates Nitrogen, Nitrite Nitrogen, and Nitrate Nitrogen

UNIT OF INSTRUCTION: Summary of I1 Elements

ESTIMATED TIME: 400 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The testing of Nitrates and Nitrites \(\text{NO}_3^-\text{N}; \text{NO}_2^-\text{N}; \text{NO}_3; \text{NO}_2\) is required by many National Pollution Discharge Elimination System Permits.

ENTRY LEVEL BEHAVIOR: Learner must know how to weigh to 4 decimal places on an analytical balance, how to weigh to two decimal places using a triple beam balance, how to measure the volume of liquids with a pipet and a graduated cylinder, how to measure pH using a pH meter, how to make spectrophotometric measurements, using a spectrophotometer, how to plot and interpret graphs, how to solve subtraction problems involving decimals and how to multiply and divide factors in a fraction.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will assemble the required equipment (including a reduction column), copperize cadmium and fill a reduction column with it, prepare a blank, one nitrate standard and a sample, pass each through the reduction column, add color reagent, measure the absorbances, construct a calibration curve using absorbances obtained by the class and obtain from it a \(\text{mg NO}_2^-\text{N} + \text{NO}_3^-\text{N}/\text{liter}\) result for his reduced sample. He will also prepare a blank, one nitrite standard and a sample, add color reagent, measure the absorbances, construct a calibration curve using class absorbance values and obtain from it a \(\text{mg NO}_2^-\text{N}/\text{liter}\) result for his non-reduced sample. He will calculate and report the efficiency of his column using the absorbance values for the two standards he tested. Then he will calculate for his sample \(\text{mg NO}_3^-\text{N}/\text{liter}\), \(\text{mg NO}_3^-\text{N}/\text{liter}\), and \(\text{mg NO}_2^-\text{N}/\text{liter}\). He will give the instructor a completed data sheet and will have an opportunity to discuss the test, sample dilutions, applying column efficiency data and interferences to the test. All work must be done according to the EMP.

2. Conditions: being provided with classroom instruction, the required equipment and supplies, one sample, a copy of the EMP, a copy of the data sheet, two pieces of graph paper, and a total working time of 290 minutes.

3. Accepted Performance: The learner's techniques must be satisfactory according to an instructor's rating and the learner's answers must be the same as an instructor's answers calculated from the same data.
B. INSTRUCTIONAL RESOURCES:

1. Available Media: EPA "Methods for Chemical Analysis of Water and Wastes," EMP on Determination of NO\textsubscript{3} + NO\textsubscript{2}-N and NO\textsubscript{3}-N, EMP (p. 48), Example Data Sheet, EMP (pp. 49 & 50), Calibration Graph forms for the test, Laboratory Equipment and Supplies, one 21 minute CCTV cassette on "Preparation of the Cadmium Reduction Column" and the following numbered overheads (11):
   
   a. #1 "Data obtained from the Cadmium Reduction Method"
   b. #2 EMP (p. 9) "Figure 1 Reduction Column"
   c. #3 Overview of steps for "Preparation of Reduction Column"
   d. #4 Overview of EMP Procedures E, F, G, H and L, "Determination of Nitrite Plus Nitrate Nitrogen"
   e. #5 Overview of EMP Procedures I, J, K and L, "Determination of Nitrite Nitrogen"
   f. #6 EMP (p. 48) "Example Data Sheet"
   g. #7 EMP (p. 49) Calibration Graph for Total NO\textsubscript{2} + NO\textsubscript{3}-N with curve drawn from example absorbances on the EMP Data Sheet
   h. #8 EMP (p. 50) Calibration Graph for NO\textsubscript{2}-N with curve drawn from example absorbances on the EMP Data Sheet
   i. #9 "Example of Calculating Column Efficiency"
   j. #10 EMP (p. 47) "Key to Data Sheet" which summarizes how to obtain five nitrogen values from the test data, using format of the EMP Example Data Sheet
   k. #11 A summary of "Removal of Interferences" from EMP Procedures D, F and J

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH: The method presented in the Effluent Monitoring Procedure (EMP) is according to the EPA Methods Manual. Procedures to determine total nitrite plus nitrate nitrogen by the cadmium reduction method and also nitrite nitrogen by colorimetry are included in entirety. The nitrate nitrogen in a sample can be calculated from the data obtained by these procedures. Since some permits require the data as nitrate and nitrite, the EMP includes calculating these concentrations from the data obtained using the EMP procedures.

1. Preparation for Instruction:
   
   a. Use list of equipment in D below to supply laboratory stations for each student.
   b. Make one glass reduction column for each student.
   c. Prepare all necessary reagents as listed in E below.
   d. Clean all glassware.
   e. Duplicate copies of the EMP data sheet, two for each student.
   f. Duplicate copies of the two EMP calibration graph forms for each.
   g. Check pH meters for proper operation.
   h. Check spectrophotometers for proper operation.
2. Sequencing:

a. Lesson one - 70 minutes
b. Break - 10 minutes

(Alternative: A break of any length could come here since lesson one includes storage directions for the newly-prepared column.)

c. Combination Presentation:

Lesson two - 60 minutes
Lesson three - 20 minutes
Lesson four - 40 minutes

There can be no break between lessons two and three since color development of the reduced blank and standard must begin within 15 minutes of the reduction process. To compensate for student differences in accomplishing lesson two, classroom presentations of lessons two and three can be combined into one session so students can do the laboratory work for each with no classroom break between.

Since lesson four involves the same laboratory techniques as lessons two and three, all three lessons can be included in the same classroom session.

(Alternative:
1) Lesson two - 60 minutes
2) No break
3) Lesson three - 20 minutes
4) Break - 15 minutes

If lessons are presented in numerical order, this is the longest break you can allow from the end of lesson three to the end of lesson seven.

If lesson seven [as applicable] immediately follows lesson four, a break up to 60 minutes may be allowed.

(5) Lesson four - 40 minutes)

d. Break - 15 minutes

(Alternative: If the lessons are presented in numerical order and a 15 minute break was given after lesson three, there should be no break given now.)

(Alternative: If lesson seven [as applicable] is to follow lesson four, this break can last up to 60 minutes. Subtract any break given after lesson three.)

e. Successive Presentations:

Lesson seven (as applicable) - 25 minutes
Lesson eight (as applicable) - 25 minutes
Lunch break - 60 minutes
This sequence was chosen so students can do the spectrophotometric measurements and calibration curve for the reduced nitrate standards and sample. This completes this half of the total workload and provides for a logically-placed long break.

(Alternative: If a long break is not desirable at this point, the lessons can be presented in numerical order. For this choice, only 15 minutes is available for a break from the end of lesson three to the end of lesson seven.)

f. Combination Presentation:

Lesson five - 35 minutes
Lesson six - 15 minutes

Lessons five and six involve the same laboratory techniques so the classroom presentations are easily combined.

(Alternative:

(1) Lesson five - 35 minutes
(2) Break - 15 minutes

If lessons are presented in numerical order, this break is allowable only if no break has occurred since the end of lesson three.

If lesson seven (as applicable) followed lesson four, a break up to 85 minutes may be allowed.

(3) Lesson six - 15 minutes)

g. Break - 10 minutes

If lessons are presented in numerical order, 15 minutes is the longest allowable break here provided no other break was given since the end of lesson three.

If lesson seven (as applicable) followed lesson four, a break up to 85 minutes may be allowed. Subtract any break time given after lesson five.

h. Lesson seven (as applicable) - 15 minutes

The classroom presentation and work time applicable to the reduced nitrate standards and sample was done earlier. Students only need a reminder in the classroom before doing the spectrophotometric measurements.

(Alternative: Lesson seven - 40 minutes. If lessons are presented in numerical order, all spectrophotometric measurements are done now.)
i. No break

(Alternative: If lessons have been presented in numerical order, only one 15 minute break can be given from the end of lesson three to the end of lesson seven. In this case, a break of any length could come now since the remaining lessons involve calculations and a summary session.)

j. Lesson eight (as applicable) - 15 minutes

See explanation under lesson seven.

(Alternative: Lesson eight - 40 minutes. If lessons were presented in numerical order, both calibration curves are constructed now.)

k. No break

(Alternative: A break of any length could be scheduled.)

l. Lesson nine - 25 minutes

m. Break - 10 minutes

(Alternative: A break of any length can be scheduled.)

n. Lesson ten - 25 minutes

o. No break

(Alternative: A break of any length can be scheduled.)

p. Lesson 11* - 30 minutes

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS:

a.m. - 1 blank, 1 nitrate standard, 1 sample
p.m. - 1 blank, 1 nitrite standard; 1 sample

1. For Each Student:

1 apron
1 beaker, 150 ml
3 beakers, 250 ml
1 beaker, 400 ml
1 weighing boat (for cadmium)
1 wash bottle, squeeze type
1 glass column for reduction
1 cylinder, graduated, 50 ml
1 cylinder, graduated, 100 ml
3 flasks, 250 ml Erlenmeyer (If lessons are done in numerical order, you need 6.)
1 flask, 50 ml volumetric with stopper (if diluting sample)
2 flasks, 100 ml volumetric with stoppers (If lessons are done in numerical order, you need 4.)
1 pair rubber gloves
1 pair safety glasses
1. three inch length rubber hose (4 cm ID)
1 pH meter (avoid sharing; uses time)
1 pipet, Mohr, 10 ml
1 pipet; volumetric, 25 ml
1-3 pipets, volumetric, 50 ml
1 propipet bulb
2 clamps, one screw-type and one pinch cock
1 sieve, 60 mesh
1 spatula or stirring rod to transfer Cd to column
1 wad of glass wool for column
1 copy EMP data sheet
1 copy of the two EMP calibration graph forms

2. Shared:
1 balance (to weigh approximately 20 g Cd) per 3 students (You can pre-weigh.)
1 very large funnel containing fluted filter paper (for Cd rinsings) set in old bottle per 2 students
1 set volumetric pipets (1.0, 2.0, 5.0, 10.0 ml) per 4 students
1 one or two milliliter pipet graduated at 0.5 ml per 4 students
1 two milliliter volumetric pipet for each bottle of color reagent
1 spectrophotometer per 3 students
1 spectrophotometer cell per instrument
1 spatula by each balance (if students are to weigh out the Cd)
1 support stand per 2 students
1 snap-clamp buret holder per 2 students

E. IPW REAGENT REQUIREMENTS: (minimum amounts per student)

4 liters distilled water, nitrate-nitrite free
480 ml concentrated ammonium chloride - EDTA solution (300 ml for every 500 ml of dilute solution required)
800 ml dilute ammonium chloride - EDTA solution
12 ml color reagent (sulfanilamide, phosphoric acid, Marshalls reagent)
Store in dark bottle in refrigerator.
1 ml concentrated hydrochloric acid in dropper bottle for pH adjustment
1 ml concentrated ammonium hydroxide in dropper bottle for pH adjustment
(may not be required if sample is synthetic)
60 ml hydrochloric acid, 6 N
200 ml copper sulfate solution, 2%
11 ml potassium nitrate stock solution. Refrigerate. One liter is a convenient amount to prepare accurately.
10.0 ml potassium nitrite stock solution. Refrigerate. One liter is a convenient amount to prepare accurately.
20 g cadmium, 40-60 mesh. If not available from a commercial source, file cadmium sticks with a coarse file and collect the granules which will pass a 10 mesh sieve and are retained on a 40, then a 60 mesh sieve. Use a hood. Wear rubber gloves and a mask.
about 100 ml each of pH buffers 4, 7 and 10 if students are to check the calibration of their pH meters
THE FOLLOWING SOLUTIONS ARE UNSTABLE. Prepare them just before use.

25 ml 1.00 mg NO$_3$-N/liter solution to activate the cadmium reduction column. Dilute 1 ml stock potassium nitrate solution to one liter.

15 ml potassium nitrate standard solution. One liter is a convenient amount to prepare accurately. (You might use up to 90 ml more to prepare 1 liter of sample. See below.)

15 ml potassium nitrite standard solution. One liter is a convenient amount to prepare accurately. (You might use up to 90 ml more to prepare 1 liter of sample. See below.)

50 ml sample of up to 1 mg NO$_2$ + NO$_3$-N/liter. You can use the standard solutions of each form to prepare this. Forty ml of each in one liter gives 0.4 mg/liter of each form for a sample of 0.8 mg NO$_2$ + NO$_3$-N/liter.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Preparation of Copper-Cadmium Reduction Column (EMP Procedure C)

LESSON NUMBER: 1 of 11

ESTIMATED TIME: 70 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: To determine NO$_3$-N in a sample, the learner must prepare a reduction column to reduce nitrate to nitrite.

ENTRY LEVEL BEHAVIOR: Learner must be able to measure the volume of liquids with a graduated cylinder.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will set up a functioning copper-cadmium reduction column according to the steps in EMP Procedure C as stated or described in the information column.

2. Conditions: Learner will be given the EMP, the necessary equipment, two copies of data sheet, 40 minutes and no assistance.

3. Accepted Performance: Learner must accomplish terminal behavior to the satisfaction of an instructor rating. (Particularly note the proper flow rate of 7-10 ml/minute.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; three overheads: (1) "Data Obtained from the Cadmium Reduction Method," (2) EMP "Figure 1 Reduction Column," p. 9, (3) overview of steps for "Preparation of Reduction Column;" 21 minute CCTV cassette on "Preparation of the Reduction Column"

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: 30 minutes

   a. Lecture - Introduction

      - Use Training Guide I to introduce the test and the reasons why nitrogen forms are important to water quality.
      - Use overhead (1) "Data Obtained..." to establish the two determinations students will do in the laboratory.
      - Show overhead (2) of EMP Figure of the Reduction Column.
      - Use overhead (3) to give overview of steps to prepare the column. Stress importance of flow rate.
      - Caution re using cadmium (use gloves, do not inhale).
b. Assignment: Read from above
   A. Instructional Objective, 1. Terminal Behavior
   c. Show CCTV cassette on preparing the column.
   d. Question period on material presented

2. Student Performance and Evaluation: (40 minutes)
   As stated in Instructional Objective above, by instructor rating.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite-Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Preparation of Nitrate Working Standards and Reduction of Nitrate to Nitrite (EMP Procedures E and F)

LESSON NUMBER: 2 of 11

ESTIMATED TIME: 60 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: This determination requires a calibration curve derived from nitrate working standard solutions which have been reduced to the nitrite form.

ENTRY LEVEL BEHAVIOR: Learner must know how to use a pipet, a volumetric flask, and a single-electrode pH meter.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will prepare a blank and one assigned nitrate working standard solution and pass each solution through the reduction column to reduce nitrate-nitrogen to nitrite-nitrogen according to the steps in EMP Procedures E and F as stated or described in the information column.

2. Conditions: Learner will be given the equipment, the EMP, 50 minutes, and no assistance.

3. Accepted Performance: Learner will perform procedures E and F to the satisfaction of an instructor rating. (Particularly note pipetting techniques and dilution to volume.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; two overheads: (1) "Data Obtained from the Cadmium Reduction Method" and (4) Overview of steps for "Determination of Nitrite Plus Nitrate Nitrogen" (E, F, G, H, L)

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Briefing (10 minutes)
   a. Use overhead (1) to stress that this is the beginning of the Determination of Nitrite Plus Nitrate Nitrogen.
   b. Use overhead (4) to give overview of EMP Procedures E, F, G, H and L used to do this determination.
   c. Assignment: Read from above
      A. Instructional Objective, 1. Terminal Behavior
      d. Assignment: Have students read Procedures E and F.
e. Assignment: Tell students to prepare a blank and assign one of the concentrations for a working standard. Explain that for instructional purposes, results will be pooled among the class for a calibration curve.

2. Student Performance and Evaluation: (50 minutes)

As stated in Instructional Objective above, by instructor rating.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Color Development of Blank and Reduced Nitrate Working Standard (EMP Procedure G)

LESSON NUMBER: 3 of 11

ESTIMATED TIME: 20 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The analytical basis for this determination is colorimetry.

ENTRY LEVEL BEHAVIOR: Learner must know how to measure volumes of liquids using a pipet.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will add the color developer to the blank and the reduced working standard solutions according to the steps in EMP Procedure G as stated or described in the information column.

2. Conditions: Learner will be given the equipment, the EMP, 15 minutes and no assistance.

3. Accepted Performance: Learner will perform Procedure G to the satisfaction of an instructor rating. (Particularly note pipetting techniques.)

B. INSTRUCTIONAL RESOURCES:


2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing)

1. Presentation: Briefing (5 minutes)
   a. Assignment: Read from above
      A. Instructional Objective, 1. Terminal Behavior
   b. Use overhead (4) so students see where they are in determining NO₂ + NO₃
   c. Assignment: Have students read Procedure G

2. Student Performance and Evaluation: (15 minutes)
   As stated in Instructional Objective above, by instructor rating.

3. NOTE: The solutions can stand up to 2 hours before Procedure L (Lesson 7) Spectrophotometric Measurements.
GUIDELINES FOR 
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Preparation, pH Adjustment, Reduction and Color Development of the Sample (EMP Procedure H)

LESSON NUMBER: 4 of 11

ESTIMATED TIME: 40 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The sample must undergo the same treatment as the nitrate working standard solutions so its total nitrate + nitrite concentration can be determined.

ENTRY LEVEL BEHAVIOR: Learner must be able to use a pipet, a volumetric flask and a single-electrode pH meter.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will prepare a sample dilution if assigned, check and if necessary adjust the pH of the sample, pass it through the reduction column, and add the color developer to it according to the steps in EMP Procedure H as stated or described in the information column.

2. Conditions: Learner will be given the equipment, one sample, the EMP, 30 minutes and no assistance.

3. Accepted Performance: Learner will perform Procedure H to the satisfaction of an instructor rating.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP and one overhead (4), overview of steps for "Determination of Nitrite + Nitrate Nitrogen" (E. F. G. H)

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing)

1. Presentation: Briefing (10 minutes)
   a. Assignment: Read from above
      A. Instructional Objective, 1. Terminal Behavior
      b. Use overhead (4) so students see where they are in determining NO₂ + NO₃
      c. Assignment: Have students read Procedure H.

2. Student Performance and Evaluation: (30 minutes)
   As stated in Instructional Objective above, by instructor rating.
3. NOTE: The solution can stand up to 2 hours before Procedure L, Spectrophotometric Measurements.

4. NOTE: The students can now do Procedure L (lesson 7) for the reduced nitrate standards and sample and also Procedures M, O, and P (lesson 8) or they can do the nitrite analysis (lessons 5 and 6) before lessons 7 and 8.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Preparation and Color Development of Nitrite Working Standards (EMP Procedures I and J)

LESSON NUMBER: 5 of 11

ESTIMATED TIME: 35 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: This determination requires a calibration curve derived from nitrite working standard solutions.

ENTRY LEVEL BEHAVIOR: Learner must be able to use a pipet, a volumetric flask, and a single-electrode pH meter.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will prepare a blank and one assigned nitrite working standard solution and then add color developer to them according to the steps in EMP Procedures I and J as stated or described in the information column.

2. Conditions: Learner will be given the EMP, the necessary equipment, 25 minutes, and no assistance.

3. Accepted Performance: Learner will perform Procedures I and J to the satisfaction of the instructor rating. (Particularly note correct pipetting techniques and dilution to the exact marks.)

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; two overheads: (1) "Data Obtained from the Cadmium Reduction Method" and (5) overview of the steps for "Determination of Nitrite Nitrogen" (I, J, K, L)

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Briefing (10 minutes):
   a. Use overhead (1) to stress that this is the beginning of the Determination of Nitrite Nitrogen.
   b. Use overhead (5) to give overview of EMP Procedures I, J, K and L used to do this determination.
   c. Assignment: Read from above

A. Instructional Objective, 1. Terminal Behavior
d. Assignment: Have students read Procedures I and J.

e. Assignment: Have each do the same concentration nitrite standard as they did for a nitrate standard. They will need absorbances for the same concentrations to check their column efficiency in lesson 9.

2. Student Performance and Evaluation: (25 minutes)

As stated in Instructional Objective above, by instructor rating.

3. NOTE: The solutions can stand up to 2 hours before Procedure L (lesson 7), Spectrophotometric Measurements.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Preparation, pH Adjustment and Color Development of the Sample (EMP Procedure K)

LESSON NUMBER: 6 of 11

ESTIMATED TIME: 15 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The sample must undergo the same treatment as the nitrite working standard solutions so its nitrite concentration can be determined.

ENTRY LEVEL BEHAVIOR: Learner must be able to use a pipet, a volumetric flask and a single-electrode pH meter.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will prepare a sample dilution if assigned, check and if necessary adjust the pH of the sample, then add color developer to it according to the steps in EMP Procedure K as stated or described in the information column.

2. Conditions: Learner will be given the equipment, one sample, the EMP, 10 minutes and no assistance.

3. Accepted Performance: Learner will perform Procedure K to the satisfaction of an instructor rating.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of the EMP and one overhead, (5) overview of steps for "Determination of Nitrite Nitrogen" (I, J, K and L)

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Briefing (5 minutes)
   a. Assignment: Read from above
      A. Instructional Objective, 1. Terminal Behavior
      b. Use overhead (5) so students see where they are in determining NO₂
      c. Assignment: Have students read Procedure K

2. Student Performance and Evaluation: (10 minutes)
   As stated in Instructional Objective above, by instructor rating.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Spectrophotometric Measurements (EMP Procedure L)

LESSON NUMBER: 7 of 11

ESTIMATED TIME: 40 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The absorbance values are necessary for preparation of standard curves and for determination of the concentration of unknown samples.

ENTRY LEVEL BEHAVIOR: Learner must know how to operate a B & L Spectronic 20 (or equivalent).

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will determine and record the absorbance for his reduced nitrate standard solution, the nitrite standard solution, and two sample solutions using the corresponding processed blanks to zero the instrument and according to the steps in EMP Procedure L as stated or described in the information column.

2. Conditions: Learner will be given the equipment, the EMP, 2 copies of the EMP "Example Data Sheet," 30 minutes and no assistance.

3. Accepted Performance: Learner must accomplish terminal behavior to the satisfaction of an instructor rating. (Particularly note correct procedure in using the Spectronic 20: checking infinite absorbance, adjusting to zero using his treated blanks, handling cells.)

B. INSTRUCTIONAL RESOURCES:


C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Briefing (10 minutes)

   a. Use overhead (1) to remind students they have two sets of blanks and standards and 2 aliquots of sample processed differently.

   b. Use overhead (4) to remind students of how they processed solutions for NO₂ + NO₃ data.

   c. Use overhead (5) to remind students of how they processed solutions for NO₂ data.
d. Assignment: Read from above

A. Instructional Objective, 1. Terminal Behavior.

e. Assignment: Have students read Procedure L.

f. Distribute 2 copies of the EMP "Example Data Sheet" to each student.

g. Announce that one data sheet must be completed for the permanent record of the course.

h. Use overhead (6) to show students where to record the respective absorbances on the EMP data sheet.

2. Student Performance and Evaluation: (30 minutes)

As stated in Instructional Objective above, by instructor rating.

3. NOTE: There are two sets of solutions to be measured. This lesson can be split with 25 minutes given to the reduced nitrate standards and sample and 15 minutes given to the non-reduced nitrite standards and sample.
SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Making a Calibration Curve and Reading Results from the Calibration Curve (EMP Procedures M, O and P)

LESSON NUMBER: 8 of 11

ESTIMATED TIME: 40 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The determination requires making calibration curves for reduced nitrate standards and for non-reduced nitrite standards and then determining concentrations for reduced and non-reduced samples from the curves.

ENTRY LEVEL BEHAVIOR: Learner must be able to construct and use a graph.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will construct 2 calibration plots and use them to convert the absorbance readings for his sample into mg \( \text{NO}_2^- + \text{NO}_3^- \text{N/liter} \) and also into mg \( \text{NO}_2^- \text{N/liter} \) values according to EMP Procedures M, O and P, as stated or described in the Information column.

2. Conditions: Learner will be given the EMP, a copy of the two calibration graph forms in the EMP, absorbance data obtained from other students, his data sheet, ruler, 30 minutes and no assistance.

   Accepted Performance: Learner's graphs and obtained results must be the same as the instructor's graphs and sample results obtained from the same data.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; three overheads: (6) EMP "Example Data Sheet," (7) Example Calibration Graph for Total Nitrite Plus Nitrate Nitrogen, (8) Example Calibration Graph for Nitrite Nitrogen

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing)

1. Presentation: Lecture (10 minutes)
   a. Assignment: Read from above

   A. Instructional Objective, 1. Terminal Behavior

   b. Assignment: Have students read EMP Procedures M, O and P.
c. Lecture: Interpretation of Graphs

- Use EMP Procedures M, O and P
- Use overhead (6) of EMP "Example Data Sheet" to show location of absorbances used for each graph.
- Use overheads (7) and (8) to show calibration graphs drawn from example absorbances on an EMP Example Data Sheet and how to use an absorbance value for a sample to obtain a concentration value for it.
- Compile absorbance data for all standards from students.
- Make sure students have absorbance data for the calibration standards recorded on their data sheets.

2. Student Performance and Evaluation: (30 minutes)

As stated in Instructional Objective above, by instructor rating.

3. NOTE: There are two curves to construct and use. This lesson can be split with 25 minutes given to the reduced nitrate standard (mg NO₂ + NO₃-N/liter) curve and 25 minutes for the non-reduced nitrite standard curve.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Checking Column Efficiency (EMP Procedure N)

LESSON NUMBER: 9 of 11

ESTIMATED TIME: 25 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The column efficiency must be near 100% in order to obtain reliable results from the copperized cadmium reduction column.

ENTRY LEVEL BEHAVIOR: Learner must be able to work division and multiplication problems involving decimals.

A: INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will calculate the column efficiency of his column and report the result to the instructor.

2. Conditions: Learner will be given the EMP, his data sheet, 15 minutes and no assistance.

3. Accepted Performance: Learner must obtain the same answer as the instructor calculates from the same data and the column efficiency must be within the range of 96% to 104%.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP and one overhead (9) "Example of Calculating Column Efficiency"

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Briefing (10 minutes)

   a. Assignment: Read from above
      A. Instructional Objective, 1. Terminal Behavior
      b. Assignment: Have students read EMP Procedure N.
      c. Lecture: Use EMP Procedure N and overhead (9) to go through an example of calculating column efficiency.

2. Student Performance and Evaluation: (15 minutes)

   As stated in Instructional Objective above, by instructor rating.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Calculations and Reporting Data (EMP Procedures Q, R and S)

LESSON NUMBER: 10 of 11

ESTIMATED TIME: 25 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner will be responsible for calculating results of the test and reporting this data.

ENTRY LEVEL BEHAVIOR: Learner must be able to work subtraction, multiplication and division problems.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will subtract the mg/liter of NO₂-N from the mg/liter of total (NO₂-N + NO₃-N) and report the results as NO₃-N; multiply NO₂-N by correct factor and report results as NO₂ mg/liter; multiply NO₃-N by correct factor and report the results as NO₃ mg/liter according to the steps in the EMP Procedures Q, R and S as stated or described in the information column.

2. Conditions: On the provided example data sheet, given the EMP Training Guide with directions for doing the calculations, with 15 minutes and no assistance.

3. Accepted Performance: Learner must turn in completed data sheet with the same answer (to the nearest .01 mg/liter) as the instructor calculates from the same data.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: Copies of EMP; two overheads: (6), EMP "Example Data Sheet" and (10), EMP p. 47 "Key to Data Sheet"

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing):

1. Presentation: Briefing (10 minutes)
   a. Use overhead (6), the headings on columns, to show students the values to be obtained.
      - Read from above:
        A. Instructional Objective, 1. Terminal Behavior
   b. Assignment: Have students read EMP Procedures Q, R and S.
   c. While students have data sheet before them, use overhead (10) which lists the ways to calculate the desired values using the data sheet format.
2. **Student Performance and Evaluation**: (15 minutes)

   As stated in Instructional Objective above, by instructor rating.

3. **Collect One Completed Data Sheet**: from each student at the end of this lesson.
GUIDELINES FOR INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Nitrite + Nitrate Nitrogen, Nitrite Nitrogen and Nitrate Nitrogen

UNIT OF INSTRUCTION: Follow-up and Summary of Subject Matter

LESSON NUMBER: 11 of 11

ESTIMATED TIME: 30 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: Learner should have an opportunity to ask any questions about the procedure and also should receive final comments about the reduction column, sample dilutions and removal of interferences.

ENTRY LEVEL BEHAVIOR: Learner will have performed the entire EMP procedure to determine Total Nitrate + Nitrite, Nitrate and Nitrite. (mg NO₃-N/liter + mg NO₂-N/liter, mg NO₃-N/liter and mg NO₂-N/liter.)

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: Learner will have any unresolved questions about the procedure, choosing sample volumes and rejecting calibration curves answered. He should also be aware that the EMP contains Procedure D, Removal of Interferences.

2. Conditions: Copies of the EMP, data sheet, calibration curve and 30 minutes.

3. Accepted Performance: by active participation in the 30 minute wrap-up session as judged by the instructor.

B. INSTRUCTIONAL RESOURCES:

1. Available Media: EMP with Training Guide and one overhead: (11) summarizing "Removal of Interferences." Overheads 1 through 10 should be available for question period.

2. Suggested Media: None

C. INSTRUCTIONAL APPROACH (Sequencing)

1. Presentation: Lecture-Discussion (30 minutes)
   a. Discuss results of test if appropriate. Answer Questions.
   b. NOTE: EMP Training Guide Section VII on checking column efficiency and how to reactivate the column.
   c. NOTE: EMP Training Guide Section VII on use of dilutions.
   d. Use overhead (11) summarizing "Removal of Interferences."
   e. Answer any remaining questions.

2. Student Evaluation: Satisfactory participation in the sessions as judged by the instructor.
A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

for the

DETERMINATION OF OIL AND GREASE

as applied in

WASTEWATER TREATMENT FACILITIES

and in the

MONITORING OF EFFLUENT WASTEWATERS

INSTRUCTIONAL PACKAGE WORKSHEET

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U. S. Environmental Protection Agency
H. Determination of Oil and Grease

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists one method to determine Oil and Grease: liquid-liquid extraction with trichloro-trifluoroethane, gravimetric.

2. The extraction and gravimetric method has been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Oil and Grease."

3. The reference source for the EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 229. The only other FR source for the procedure is:

4. In the following Instructional Package Worksheet (IPW) for the EMP, the lesson plan is for extraction and gravimetric determination of oil and grease in a WWTP effluent sample acidified to pH2.

5. Each student is to process one sample. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Oil and Grease

UNIT OF INSTRUCTION:

LESSON NUMBER: 1 of 1

ESTIMATED TIME: 2 1/2 hours

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The learner should know how to perform an oil and grease determination since it may be required by the permit system.

ENTRY LEVEL BEHAVIOR: The learner must be able to:
1. Perform basic mathematical computations (addition, subtraction, multiplication and division).
2. Handle solutions of acids safely.
3. Understand the term liter, milliliter, gram, and milligram.
4. Perform weighings on an analytical balance.
5. Clean laboratory glassware.
6. Prepare a desiccator for use.
7. Use ordinary glassware such as beakers, flasks, graduated cylinders.
8. Use a laboratory oven for drying glassware.
9. Drill holes through a rubber stopper.
10. Bend and fire polish glass tubing using a burner as a source of heat.
11. Use a laboratory burner.

A. INSTRUCTIONAL OBJECTIVE
1. Terminal Behavior - The learner will exhibit proper technique while performing an oil/grease determination.

2. Conditions - The learner will have the use of the attached EMP, and all chemicals and equipment listed in it.

3. Accepted Performance - The use of proper technique in performing the test will be judged by the instructor.

B. INSTRUCTIONAL RESOURCES
1. Available Media
   a. XT-56, The Determination of Oil and Grease
   b. Color TV tape showing the use of a separatory funnel*
   c. Color TV tape showing the following items/techniques:*
      i. Use of pH sensitive paper
      ii. Folding a piece of filter paper
      iii. Placing the filter paper in a 60° funnel
      iv. Use of thermometers
   d. Color TV tape showing a distillation setup (To be used if the solvent will be distilled off at the end of the procedure)

* b and c above, are on one cassette

2. Suggested Media - None
C. INSTRUCTIONAL APPROACH (Sequencing)

1. Classroom
   a. Have the learners read the Brief Description of Analysis.
   b. Discuss oil and grease using XT-56, Determination of Oil and Grease, and, or the TV tape(s).
   c. Explain the symbol TF/D (see A.1.4 and A.1.4.4a.).
   d. Have the learners read sections C. and D.1.1. through D.1.9.
   e. Read A.4.1 and A.4.1.1b. (Have the flasks in a desiccator in the laboratory).

2. Laboratory
   a. Have the learners do D.1.10 through D.2.14. (A break may be given during the 30 minute cooling period).
   b. If desired, assign someone to run a blank (see section F).

3. Laboratory
   a. Have the learners do section E.

4. Classroom
   a. Have the learners do section G.
   b. Final questions and discussion.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS

1. For each student:
   - One stopper/glass tubing apparatus for applying suction to the distilling flask. See figure 2 in the EMP
   - Source of vacuum (water aspirator or vacuum pump)
   - Hot water bath (80°C temperature needed)
   - Hot plate (must have continuous setting between its lower and upper limit; cannot have only low, medium and high settings)
   - Steam bath (large enough to accommodate at least 1 distilling flask, 125 ml size)
   - Laboratory apron
   - Safety glasses
   - Pen or pencil
   - Notebook (for recording data)
   - Distilling flask, 125 ml, with a 24/40 ground glass neck (Corning number 4100 is an example) - One flask is used for each determination.
   - Desiccator (large enough to hold at least one 125 ml distilling flask)
D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS (Cont'd.)

Graduated cylinders, 10 ml and 50 ml
Ring stand
Funnel, 60°, about 50 mm size
Separatory funnel with Teflon stopcock, 2 liter
Ring (to support the separatory funnel)
Clamp (to fit neck of distilling flask)
Beakers, 1000 ml (1), 100-150 ml (2)

The 2 liter separatory funnel should be mounted on the ring and ring stand, and should contain 1 liter of sample already acidified to pH2.

2. Shared:
Analytical balance (200 g capacity), 1 per 4 students
Still, or other source of distilled water

E. IPW REAGENT REQUIREMENTS (MINIMUM AMOUNTS PER STUDENT)

1,1,2-trichloro-1,2,2-trifluoroethane* (125 ml)
Lintless tissues (may be used in place of crucible tongs) (5)
Whatman number 40 filter paper (to fit the funnel in D above), 1 piece
Anhydrous sodium sulfate, Na₂SO₄ (3 g)

*Freon 113 is a general name used by E. I. DuPont de Nemours, Inc., for the above solvent. TF and PCA are two specific grades of Freon 113. TF is the better of the two. Genosolv D is the name used by Allied Chemical Company for the above solvent. Either Freon TF or Genosolv D may be used in the determination.
A PROTOTYPE FOR DEVELOPMENT OF ROUTINE OPERATIONAL PROCEDURES

FOR THE

DETERMINATION OF AMMONIA BY AN AMMONIA SELECTIVE ION ELECTRODE

...applied in...

WASTEWATER TREATMENT FACILITIES
and in the
MONITORING OF EFFLUENT WASTEWATERS

Instructional Package Worksheet

National Training and Operational Technology Center
Municipal Operations and Training Division
Office of Water Program Operations
U.S. Environmental Protection Agency

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PART II INSTRUCTIONAL PACKAGE WORKSHEETS

I. Determination of Ammonia by an Ammonia Selective Ion Electrode

1. The Federal Register (FR) issuance in Outline No. 1 of this Guide lists one method for the isolation of Ammonia from a water sample, i.e., manual distillation at pH9.5. In a footnote, #4, it states: "Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary step is not necessary; however, manual distillation will be required to resolve any controversies."

The same FR issuance lists three manual methods for the measurement of the isolated ammonia: nesslerization (for 0.05-1 mg/l), titration (for 1-25 mg/l) and electrode (for 0.05-1400 mg/l).

2. The electrode method has been written in the Effluent Monitoring Procedure (EMP) format entitled, "Determination of Ammonia by an Ammonia Selective Ion Electrode." The distillation, nesslerization and titration methods for macro sample volumes are contained in the EMP, "Nitrogen, Ammonia Determination." The distillation, nesslerization and titration methods for both macro and micro sample volumes are contained in another EMP, "Determination of Total Kjeldahl Nitrogen."

3. The reference source for the Ammonia Selective Ion Electrode EMP is 1974 EPA "Methods for Chemical Analysis of Water and Wastes," p. 165. This manual refers the analyst to the directions of the manufacturer if he is using a specific ion meter. Thus, the second reference for this EMP is the Orion,* "Instruction Manual for Probe and Meter." No other sources for the procedure are listed in the FR.

4. The procedures in this EMP are only for the measurement of ammonia. The distillation procedure for both macro and micro sample volumes are contained in the EMP, "Determination of Total Kjeldahl Nitrogen." The distillation procedure for macro sample volumes is also in the EMP, "Nitrogen, Ammonia Determination."

5. In the following Instructional Package Worksheet, the lesson plan is for determination of ammonia in a synthetic sample which may or may not have been distilled.

6. Each student is to process two standards and two samples. The IPW equipment and reagent requirements are based on this assignment. If you choose a different assignment, adjust quantities accordingly.

*Mention of a specific brand name does not constitute endorsement by the U. S. Environmental Protection Agency.
GUIDELINES FOR
INSTRUCTIONAL PACKAGE WORKSHEET

SUBJECT MATTER: Determination of Ammonia by Selective Ion Electrode Method

UNIT OF INSTRUCTION:

LESSON: 1 of 1

ESTIMATED TIME: 90 minutes

JUSTIFICATION FOR THIS INSTRUCTIONAL OBJECTIVE: The determination of ammonia is required by many NPDES permits. It is carried out in most wastewater treatment plants. The Selective Ion Electrode Method is one of three approved methods for this determination.

ENTRY LEVEL BEHAVIOR: The learner should have completed the Basic Skills course or have the skills taught there.

A. INSTRUCTIONAL OBJECTIVE:

1. Terminal Behavior: The learner will exhibit proper technique while determining the ammonia-nitrogen content of a distilled ammonia sample.

2. Conditions: The learner will have the use of the related EMP and all chemicals and equipment listed in it. In addition the learner should be given a copy of the EMP "Nitrogen, Ammonia Determination" for use in the distillation procedure should this be required.

3. Accepted Performance: The use of proper technique in performing the test shall be judged by the instructor.

B. INSTRUCTIONAL RESOURCES:


C. INSTRUCTIONAL APPROACH (Sequencing)

1. Classroom (30 minutes) The instructor should discuss the advantages and disadvantages of using the electrode. Cover the procedures necessary to place the electrode in operation including the calibration of the meter.

2. Laboratory Briefing-classroom (15 minutes) Cover what the learner should do in the laboratory, including the equipment and use and preparation of standards.

3. Laboratory (40 minutes) Determine the ammonia content of a sample which has been previously distilled.
4. Classroom (5 minutes) Final discussion of the procedure.

D. IPW EQUIPMENT AND SUPPLY REQUIREMENTS: (2 standards, 2 samples)

1. For each student:
   1 apron
   4 beakers, glass, 150 ml
   1 cylinder, graduated, 100 ml
   1 pair safety glasses
   1 pencil, wax
   1 pipet bulb
   1 pipet, 1 ml volumetric
   1 stir plate and bar
   1 wash bottle

2. Shared:
   1 Orion* Specific Ion Meter; Model 401, 407 or 407A per 3 students
   if can alternate use with other lab work.
   1 Orion* Ammonia Electrode, Model 95-10 per 3 students if alternating
   lab work.

E. IPW Reagent Requirements:

(minimum amounts per student)

100 ml 0.1 mg NH₃-N/liter
100 ml 1.0 mg NH₃-N/liter
100 ml sample one; concentration between 0.01 to 1.0 mg NH₃-N/liter
100 ml sample two; concentration between 0.01 to 1.0 mg NH₃-N/liter
  4 ml sodium hydroxide 10M
  500 ml distilled water, ammonia-free

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