



PROPOSED REGULATIONS GOVERNING
LABORATORY CERTIFICATION
AND
STANDARDS OF PERFORMANCE

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REGULATIONS GOVERNING LABORATORY CERTIFICATION
AND STANDARDS OF PERFORMANCE

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REGULATIONS GOVERNING LABORATORY CERTIFICATION AND STANDARDS
OF PERFORMANCE

SUBCHAPTER 1. GENERAL PROVISIONS

7:18-1.1 Scope and authority

This chapter, adopted pursuant to the Safe Drinking Water Act, N.J.S.A. 58:12A-1 et seq. and the Water Pollution Control Act, N.J.S.A. 58:10A-1 et seq., constitutes the Department's regulations governing certification of laboratories performing water analyses required to be performed by regulations or orders issued pursuant to those acts. This chapter establishes the procedures for obtaining and maintaining certifications, and the criteria and procedures laboratories shall follow in analyzing water samples.

7:18-1.2 Construction

These rules shall be liberally construed to permit the department to discharge its statutory functions, and to effectuate the purposes of the laboratory certification program.

7:18-1.3 Purpose of the regulations

(a) This chapter is promulgated for the following purposes:

1. To establish the administrative procedures to be followed by certified laboratories and laboratories seeking certification.
2. To establish the categories and parameters in which laboratories may be certified.
3. To establish the standards, criteria and procedures as to laboratory equipment and supplies, practices, methodology, quality control, personnel, facilities, data reporting and data maintenance which a certified laboratory or laboratory seeking certification shall continually meet.
4. To establish the enforcement procedures the Department shall follow to ensure that all certified laboratories or laboratories seeking certification are in compliance with this chapter.

7:18-1.4 Certification program requirements

(a) Any laboratory wishing to analyze water samples for compliance with regulations adopted or orders issued pursuant to the Water Pollution Control Act, N.J.S.A. 58:10A-1 et seq. or the Safe Drinking Water Act, N.J.S.A. 58:12A-1 et seq., shall follow the procedure set forth herein in order to obtain and maintain certification.

- (b) Certified laboratories and laboratories seeking certification shall analyze all water samples in accordance with the procedures and methods required by this chapter.

7:18-1.5 Incorporation by reference

- (a) The Department hereby adopts and incorporates into these regulations the "National Interim Primary Drinking Water Regulations," as amended, 40 CFR 141, and the "Guidelines Establishing Test Procedures for the Analysis of Pollutants," as amended, 40 CFR 136, and future supplements and amendments to those regulations, both of which have been duly promulgated as regulations by the Administrator of the United States Environmental Protection Agency.
- (b) The above Federal regulations are available from either the U.S. Environmental Protection Agency Regional Library, 26 Federal Plaza, New York, New York 10007, or the New Jersey Department of Environmental Protection, Bureau of Collections and Licensing, P.O. Box CN 402, Trenton, New Jersey 08625, (609) 292-4071.

7:18-1.6 Program information

Unless otherwise specified, any questions concerning the requirements of this chapter should be directed to the Office of Quality Assurance, Division of Water Resources, New Jersey Department of Environmental Protection, P.O. Box CN-029, Trenton, New Jersey 08625.

7:18-1.7 Definitions

The following words and terms, when used in this chapter, shall have the following meanings, unless the context clearly indicates otherwise.

"Accredited" means having the approval conferred upon schools, institutions, or programs where appropriate by a nationally recognized accrediting agency or association as determined by either the U.S. Commissioner of Education, N.J. Commissioner of Education, or Chancellor of Higher Education, or any combination of the three.

"Analytical reagent (AR) grade, ACS reagent grade, and reagent grade" are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

"Category" means a group of parameters for which certification is offered.

"Certified thermometer" is a thermometer that has documentation from the manufacturer showing that it has been calibrated with a National Bureau of Standards thermometer and the correction factors from that calibration.

"Clean Water Act of 1977" means P.L. 95-217, 33 U.S.C. 1251 et seq. and amendments made thereto.

"Commissioner" means the Commissioner of the New Jersey Department of Environmental Protection or an authorized representative.

"Compliance analysis" means the analysis of a sample that is required to be analyzed by a Departmental regulation or order.

"Confluent growth" means a bacterial growth (coliform and non-coliform) covers the entire filtration area of the filter with no discrete colonies.

"Department" means the New Jersey Department of Environmental Protection.

"40 CFR 136" means the "Guidelines Establishing Test Procedures for the Analysis of Pollutants", which were duly promulgated as regulations by the Administrator of the United States Environmental Protection Agency.

"Laboratory pure water" means distilled or deionized water which is free of contaminants that may interfere with the analytical test in question.

"Laboratory seeking certification" means an uncertified laboratory which has submitted an acceptable application and the appropriate fee for the category in which it desires certification, and a laboratory holding a valid interim approval.

"Maximum contaminant level (MCL)" means the maximum permissible level of a contaminant allowed in drinking water under the National Interim Primary Drinking Water Regulations.

"Membrane filtration (MF) method" means a method for determining the coliform count in a water sample. In this method, a known volume of water is filtered through a membrane filter of optimum pore size for full bacterial retention. The filter is incubated in contact with culture medium to provide nutrients for bacterial growth. After incubation at a prescribed time and temperature, the cultures are examined for coliform colonies that are counted and recorded per 100 mL. of water sample.

"Microbiological Methods - EPA" means "Microbiological Methods for Monitoring the Environment, Water and Wastes", U.S.EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, EPA-600/8-78-017, December 1978. Available from ORD Publications, CERL, U.S.EPA, Cincinnati, Ohio 45268.

"Most probable number (MPN)" means a quantitative designation of microbial population which is determined by a statistical method. In this method, a multiple dilution tube technique is used with a standard culture medium. The tubes are incubated and observed for gas production. Results of these tubes are translated by mathematical probability tables into population numbers.

4. "Mysid Shrimp" means the bay mysid shrimp, *Mysidopsis bahia*.

"40 CFR 141" means the "National Interim Primary Drinking Water Regulations", which were duly promulgated as regulations by the Administrator of the United States Environmental Protection Agency.

"New Jersey Pollutant Discharge Elimination System Regulations" or "NJPDES" means the regulations adopted by the Department at N.J.A.C. 7:14A-1.1 et seq., governing the New Jersey system for issuing, modifying, suspending, revoking and reissuing, terminating, monitoring, and enforcing discharge permits pursuant to the New Jersey Water Pollution Control Act.

"New Jersey Safe Drinking Water Act Regulations" means the current Safe Drinking Water Act Regulations promulgated at N.J.A.C. 7:10-1.1 et seq. by the Department pursuant to the New Jersey Safe Drinking Water Act.

"Personal and direct supervision" means that a qualified supervisor is available at all times when laboratory procedures are being performed.

"Primary standard" is a highly pure reagent used as a reference for standardizing other reagent solutions.

"Proficiency sample" is a sample containing a known amount of a specific or combination of parameters used to evaluate the analytical performance of a laboratory.

"Proficiency test average value" means the average of all singularly determined values on a given radiological proficiency sample for one parameter.

"Replicate sample" is a sample prepared by dividing a homogeneous sample into separate parts so that each part is also homogeneous and representative of the original sample.

"Safe Drinking Water Act" or "NJSDWA" means N.J.S.A. 58:12A-1 et seq.

"Standard curve" is a curve plotting 5 or more concentrations of a known parameter standard minus a blank, versus the standard's absorbance or percent transmission.

"Standard methods, 14th Edition" means Standard Methods for the Examination of Water and Wastewater, American Public Health Association, 14th Edition.

"State Primary Drinking Water Regulations" means those regulations promulgated at N.J.A.C. 7:10-5.1 et seq.

"State Secondary Drinking Water Regulations" means those regulations promulgated at N.J.A.C. 7:10-7.1 et seq.

"Subsequent to graduation" means laboratory training and experience acquired after receipt of the degree specified.

"Too numerous to count (TNTC)" means a bacterial count that is usually greater than 200 colonies per membrane. The count is designated in this way when a membrane filter culture has grown in such a way that the total number of bacterial colonies (coliform and non-coliform) are too numerous or not sufficiently distinct to obtain an accurate count, or both.

"USEPA" means United States Environmental Protection Agency.

"Water Pollution Control Act" or "NJWPCA" means N.J.S.A. 58:10A-1 et seq.

7:18-1.8 Severability

If any section, subsection, provision, clause, or portion of this chapter is adjudged unconstitutional or invalid by a court of competent jurisdiction, the remainder of this chapter shall not be affected thereby.

SUBCHAPTER 2. PROGRAM PROCEDURES AND REQUIREMENTS

7:18-2.1 Requirement of certification

- (a) All water sample analyses performed for the purpose of determining compliance with microbiological, chemical and radiological requirements of the State Primary and Secondary Drinking Water Regulations, N.J.A.C. 7:10-5.1 and 7:10-7.1, and microbiological, chemical and bioassay requirements of the New Jersey Pollutant Discharge Elimination System Regulations, N.J.A.C. 7:14A-1, or when required by order issued by the Department pursuant to the authority of the Safe Drinking Water Act, N.J.S.A. 58:12A-1 et seq., the Water Pollution Control Act, N.J.S.A. 58:10A-1 et seq., or any other regulations adopted pursuant to those acts, shall be performed in laboratories certified for this purpose pursuant to this subchapter. Analyses performed in laboratories not so certified shall not be accepted by the Department as being in compliance with the requirements, regulations or orders of the Water Pollution Control Act or the Safe Drinking Water Act.
- (b) Laboratories in other States, certified under conditions no less stringent than those required by this chapter by the agency having primary enforcement responsibility under the Federal Safe Drinking Water Act, 42 U.S.C. 300 et seq., or the agency delegated administrative responsibility for the National Pollutant Discharge Elimination System under the Clean Water Act of 1977, 33 U.S.C. 1251 et seq., in such other State, shall be considered to be certified laboratories for purposes of this chapter once they have complied with the provisions of N.J.A.C. 7:18-2.4.
- (c) Only laboratories certified pursuant to these regulations or maintained by the USEPA may be called State Certified Water Laboratories and no laboratory may adopt any name or make any oral or written statement intended or likely to mislead the public with respect to its certification status.

7:18-2.2 Categories for certification

- (a) A laboratory may apply for certification in any one or more of the following categories and shall be certified in those parameters within the category for which it demonstrates acceptable performance on proficiency samples, when available, and meets all other requirements of this chapter. The laboratory certificate shall specify the categories and the parameters within each category for which the laboratory is certified and shall be conspicuously displayed in the laboratory in a location visible to the public. The certification categories are as follows:

1. Microbiological Testing, which comprises tests for coliform bacteria conducted in accordance with the methods and procedures specified in 40 CFR 141 for compliance with the Safe Drinking Water Act and the State Primary Drinking Water Regulations, and 40 CFR 136 for compliance with the Water Pollution Control Act and the New Jersey Pollutant Discharge Elimination System Regulations.
2. Limited Chemistry, which comprises chemical tests or analyses required to determine compliance with the Safe Drinking Water Act and the State Primary and Secondary Drinking Water Regulations, and the Water Pollution Control Act and the New Jersey Pollution Discharge Elimination System Regulations, except those analyses for which the atomic absorption or gas chromatography methods are specifically required. Tests for the limited chemistry category shall be conducted in accordance with the methods and procedures specified in 40 CFR 141 for compliance with the Safe Drinking Act and the State Primary Drinking Water Regulations; N.J.A.C. 7:18-4.5 for compliance with the Safe Drinking Water Act and the State Secondary Drinking Water Regulations, and 40 CFR 136 for compliance with the Water Pollution Control Act and the New Jersey Pollutant Discharge Elimination System Regulations.
3. Atomic Absorption, which comprises tests or analyses required to determine compliance with the Safe Drinking Water Act and the State Primary and Secondary Drinking Water Regulations, and the Water Pollution Control Act and the New Jersey Pollutant Discharge Elimination System Regulations, for which the atomic absorption method is applicable or required. Tests for the atomic absorption category shall be conducted in accordance with the methods and procedures specified in 40 CFR 141 for compliance with the Safe Drinking Water Act and the State Primary Drinking Water Regulations; N.J.A.C. 7:18-4.5 for compliance with the Safe Drinking Water Act and the State Secondary Drinking Water Regulations; and 40 CFR 136 for compliance with the Water Pollution Control Act and the New Jersey Pollutant Discharge Elimination System Regulations.
4. Gas Chromatography, which comprises tests or analyses required to determine compliance with the Safe Drinking Water Act and the State Primary Drinking Water Regulations, and the Water Pollution Control Act and the New Jersey Pollutant Discharge Elimination System Regulations for which the gas

chromatography method is applicable or required. Tests for the Gas Chromatography category shall be conducted in accordance with the methods and procedures specified in 40 CFR 141 for compliance with the Safe Drinking Water Act and the State Primary Drinking Water Regulations, and 40 CFR 136 for compliance with the Water Pollution Control Act and the New Jersey Pollutant Discharge Elimination System Regulations.

5. Radiological Testing, which comprises those tests or analyses for radioactivity required to determine compliance with the Safe Drinking Water Act and the State Primary Drinking Water Regulations. Tests for the Radiological category shall be conducted in accordance with the methods and procedures specified in 40 CFR 141.
6. Bioassay Testing, which shall include any bioassay analyses required to determine compliance with the Water Pollution Control Act and the New Jersey Pollution Discharge Elimination System Regulations. Bioassay analyses shall be conducted in accordance with the methods and procedures specified in Subchapter 6 of this regulation.

7:18-2.3 Application procedures for laboratories located in New Jersey including special provisions for the phase-in of the New Jersey Pollutant Discharge Elimination System Laboratory Certification Program

- (a) The owner of a laboratory in New Jersey who wishes it to be certified in any or all of the categories and parameters thereof, described in N.J.A.C. 7:18-2.2, or, if already certified, who wishes to add a category or a parameter within a category, shall apply for certification to the New Jersey Department of Environmental Protection, Division of Fiscal and Support Services, Bureau of Collections and Licensing, P.O. Box CN 402, Trenton, New Jersey 08625; (609) 292-4071 (hereinafter "Bureau"), on forms available therefrom. The applicant shall provide all information requested and shall submit the appropriate fee.

1. Laboratories seeking certification in the Radiological category shall have participated in the USEPA's radiological proficiency testing program during the immediately preceding twelve months and shall submit copies of the USEPA's performance evaluation reports demonstrating that for each parameter in which the laboratory is seeking certification at least four performance test average values have been within the control limits established for that parameter.

2. Laboratories which intend to seek certification in the Radiological category, but which have not participated in the USEPA's radiological proficiency testing program may obtain information concerning that program from the Bureau.
- (b) If the applicant fails to submit all the information requested or fails to submit the appropriate fee, the Department shall reject the application without prejudice, and, if submitted, return the fee.
 - (c) If the applicant submits a complete application, the appropriate fee, proficiency data if required, and the information submitted meets the minimum requirements of this chapter for the category or categories for which certification is requested, the application shall be accepted. Acceptance of the application does not authorize the laboratory to perform water analyses regulated by this chapter. The applicant shall be notified of the acceptance and shall participate in the following laboratory evaluation:
 1. Microbiological and Bioassay Testing:
 - i. The Department shall contact the laboratory within three weeks after the application is accepted to arrange a mutually acceptable date for an on-site laboratory inspection;
 - ii. The laboratory shall be evaluated and inspected to determine if it is in compliance with the requirements of this chapter; and
 - iii. If the laboratory demonstrates to the Department that it is in compliance with the requirements of this chapter it shall be certified for the category and the parameters within the category for which it has requested certification.
 2. Limited Chemistry, Atomic Absorption and Gas Chromatography:
 - i. The Department shall send to the laboratory a set of proficiency samples, if available, for the parameters for which certification is requested within two weeks after acceptance of the laboratory's application by the Department;
 - ii. The laboratory shall analyze the proficiency samples and return the proficiency data, within 30 days of its receipt of the samples to the Office of Quality Assurance, Division of Water Resources, P.O. Box CN 029, Trenton, New Jersey 08625 within 30 days of its receipt of the samples;

- iii. The laboratory shall have satisfied the requirements for proficiency testing for a parameter when it acceptably analyzes both the high and low values for each parameter within a given set of proficiency samples;
- iv. Acceptable analysis for a value in all parameters excluding trihalomethane parameters occurs when the reported value falls within the 99 percent confidence interval calculated by the USEPA from available performance evaluation data of USEPA and State Laboratories;
- v. Acceptable analysis for a trihalomethane parameter occurs when the reported value falls within the acceptance limits calculated by the USEPA as ± 20 percent of the true value;
- vi. The laboratory shall have three separate opportunities to acceptably analyze one of three different sets of proficiency samples for each parameter;
- vii. If the laboratory's analytical values for the proficiency samples are acceptable, the Department shall contact the laboratory to arrange a mutually acceptable date for an on-site laboratory inspection;
- viii. If the laboratory demonstrates, during the on-site inspection, that it is in compliance with the requirements of this chapter, then it shall be certified in the category and the parameters within the category for which it has acceptably analyzed proficiency samples.
- ix. If performance evaluation samples are not available, then the evaluation of the laboratory will be based only on the on-site laboratory inspection.

3. Radiological Testing

- i. The Department shall contact the laboratory within three weeks after the application is accepted to arrange a mutually acceptable date for an on-site laboratory inspection, and the inspection will be conducted by representatives of the Department and the USEPA; and

- ii. If the laboratory demonstrates to the Department during the inspection that it is in compliance with the requirements of this chapter it shall be certified for the category and the parameters within the category for which it has requested certification.
 - iii. If the Department is unable to schedule an on-site inspection within 90 days after receiving an acceptable application from a laboratory, it may grant the laboratory an interim approval to analyze radiological samples until the laboratory is inspected provided the laboratory continues to participate in the USEPA's proficiency testing program and acceptably analyze the program's samples.
- (d) Certifications may contain conditions requiring rectification of minor deficiencies identified by the Department by a date or dates specified therein, but only if such minor deficiencies do not affect the accuracy of the analytical results.
- (e) An applicant for certification who either does not perform acceptably on the proficiency samples or does not meet the requirements of this chapter shall be notified that certification has been denied. Applicants receiving such a notification may not reapply for certification until the laboratory assures the Department in writing that all reasons for the denial of certification have been rectified.
- (f) The following special provisions are applicable to the phase-in of the New Jersey Pollutant Discharge Elimination System laboratory certification program:
- 1. The owner of a laboratory who requests certification in parameters within categories for the New Jersey Pollutant Discharge Elimination System laboratory certification program may apply to the Department for certification after July 1, 1981. The applicant shall follow the procedures and meet all the requirements of all previous subsections of this section except that:
 - i. The laboratory shall be granted an interim approval which will authorize the laboratory to perform analyses for the New Jersey Pollutant Discharge Elimination System program while the laboratory is being evaluated for certification;
 - ii. The interim approval shall be valid until the laboratory is certified or June 30, 1982, whichever is earlier, where the laboratory

- demonstrates to the satisfaction of the Department that it is making good faith efforts and diligently seeking to meet the requirements for certification;
- iii. A laboratory that fails to acceptably analyze the proficiency samples or otherwise fails to meet the requirements of this chapter for certification shall be allowed to remain in the interimly approved status until June 30, 1982 if the laboratory submits an acceptable plan to correct the deficiencies within 30 days of receiving notification of its deficiencies, to the Department's Office of Quality Assurance, Division of Water Resources, P.O. Box CN 029, Trenton, New Jersey 08625;
 - iv. On July 1, 1982 State Certified NJPDES Laboratories shall follow the normal renewal of certification procedures set forth in N.J.A.C. 7:18-2.5;
 - v. On July 1, 1982 laboratories having interim approvals to perform analyses for the New Jersey Pollutant Discharge Elimination System and any other laboratories wishing to be initially certified to perform analyses for the New Jersey Pollutant Discharge Elimination System program shall apply for certification pursuant to this section, and the following shall apply:
 - (1) Laboratories meeting the application requirements of this section shall be granted an interim approval to perform analyses for the parameters within the categories for which they are seeking certification; and
 - (2) This interim approval shall be valid until the laboratory is certified or notified that the interim approval is revoked due to the laboratory's failure to meet the certification standards set forth in this section.
 - vi. Laboratories notified that their interim approval has been revoked shall immediately cease performing analyses required to be performed in a certified laboratory for compliance with the New Jersey Pollutant Discharge Elimination System Regulations and shall comply with subsection (e) above before reapplying for certification.

7:18-2.4 Procedure for laboratories not located in New Jersey

- (a) The owner of a laboratory located in a State other than New Jersey which has been certified, under conditions no less stringent than those required by this chapter, by the agency having primary enforcement responsibility under the provisions of the Federal Safe Drinking Water Act or the Agency delegated administrative responsibility for the Federal Clean Water Act NPDES program in the State where it is located, who wishes to perform water analyses in any or all of the categories described in N.J.A.C. 7:18-2.2 for public water systems or NJPDES permitted facilities located in New Jersey or as required by the Water Pollution Control Act or the Safe Drinking Water Act, shall:
1. Annually complete the application form provided by the New Jersey Department of Environmental Protection, Division of Fiscal and Support Services, Bureau of Collections and Licensing, P.O. Box CN 402, Trenton, New Jersey 08625, (609) 292-4071;
 2. Have the form certified to by the agency having primary enforcement responsibility or delegated administrative responsibility; and
 3. Return the form with the proper fee to the Bureau.
- (b) The Bureau shall review the application and if it finds it complete and the appropriate fee has been paid, shall assign or reassign the laboratory a number to be used in all correspondence with the Department.
- (c) The receipt of the number authorizes the laboratory to perform water analyses for public water systems or NJPDES permitted facilities or as required pursuant to the Water Pollution Control Act or the Safe Drinking Water Act in New Jersey for the balance of the fiscal year which expires on June 30.
- (d) If the laboratory's certification is revoked by the agency having primary enforcement responsibility or, delegated administrative responsibility, the New Jersey authorization is thereby automatically cancelled. The laboratory manager shall notify the Bureau and all clients in New Jersey that utilize the laboratory of the revocation within 48 hours of receipt of notice of revocation by the laboratory manager.
- (e) The owner of a laboratory in a State other than New Jersey which is not certified by that State or the USEPA, or is certified under conditions less stringent than those required by this chapter, who wishes to perform water analyses in any or all of the categories

described in N.J.A.C. 7:18-2.2 for public water systems or NJPDES permitted facilities located in New Jersey, or as required by the Safe Drinking Water Act or the Water Pollution Control Act, shall apply for certification in accordance with the procedures set forth in N.J.A.C.

7:18-2.3. In addition, prior to conducting the on-site laboratory inspection, the laboratory shall submit to the Bureau as an additional fee the sum the Department determines to be sufficient to cover the travel, and room and board expenses of the certification inspectors.

7:18-2.5 Renewal of certification

Applications for renewals of certification will be accepted by the Bureau during June of each year, shall be submitted on forms provided therefor, and shall be accompanied by the appropriate fee.

7:18-2.6 Fees

- (a) Owners of laboratories applying for certification or renewal of certification, for the fiscal year commencing on July 1, 1981 and for subsequent fiscal years shall submit the appropriate fee obtained from the annual fee schedule below along with the required application materials. Fees are nonrefundable. Laboratories owned or operated by the State of New Jersey or an Agency of the Federal Government are exempt from this fee requirement, but, except for the Environmental Protection Agency, shall make appropriate application for certification in accordance with the other provisions of these regulations.

Laboratory Certification Annual Fee Schedule:

	<u>Fees</u>
Microbiological Testing	\$400.00
Chemistry	
Any one of the following categories:	
Limited chemistry, atomic absorption,	
Gas Chromatography	\$400.00
Any two of the above-mentioned	
categories	\$500.00
All three of the above-mentioned	
categories	\$600.00
Radiological Testing	
Any one or two radiological	
parameters	\$200.00
Any additional radiological	
parameter	\$50.00 per addi-
	tional parame
Bioassay Testing	\$400.00

- (b) The owner of a laboratory holding a NJPDES permit pursuant to N.J.A.C. 7:14A-1 et seq. is exempt from the laboratory fee requirement because the fee is included in the NJPDES administrative costs.
- (c) The annual fees shall not be prorated and shall apply in full to any portion of the fiscal year which remains prior to the annual renewal date, June 30.
- (d) This section is applicable to interimly approved laboratories except for those exempted by (b) above.

7:18-2.7 Required laboratory personnel policies

- (a) Every certified laboratory and laboratories seeking certification shall have sufficient properly qualified personnel commensurate with the workload and types of tests or analyses required to be performed for the parameters for which the laboratory is certified, or is seeking certification, pursuant to this chapter.
 1. One individual shall be designated as the person in responsible charge and, irrespective of any local title or designation, is herein referred to as the laboratory manager.
 2. The laboratory shall have one or more supervisors who shall be qualified in accordance with the provisions of (d) below to perform the tests or analyses required to be performed within the category or categories for which the laboratory is certified, or seeks certifications. The laboratory manager may also serve as a laboratory supervisor, depending upon the size and functions of the laboratory, provided that the laboratory manager meets the qualifications for laboratory supervisor.
 3. The laboratory shall have a sufficient number of laboratory technical personnel commensurate with the volume and diversity of the tests performed.
- (b) Current employee records shall be maintained, which shall include a resume' documenting each employee's training, experience, duties, and date or dates of relevant employment.
- (c) Work assignments shall be consistent with qualifications.
- (d) The laboratory supervisor shall possess the qualifications for the category which he/she supervises, or for laboratories applying for New Jersey Pollutant Discharge Elimination

certification in July 1, 1981, meet the requirements of paragraph seven below:

1. If the laboratory performs tests in the category of Microbiological Testing, the supervisor shall hold a bachelor's degree in a biological science or chemistry from an accredited institution with at least three credits in bacteriology and, subsequent to graduation, shall have had at least one year of laboratory training or experience in bacteriology;
2. If the laboratory performs tests or analyses in the category of Limited Chemistry, the supervisor shall hold a bachelor's degree in chemistry or in a biological science from an accredited institution and, subsequent to graduation, shall have had at least one year of laboratory training or experience in chemistry;
3. If the laboratory performs tests or analyses in the category of Atomic Absorption the supervisor shall:
 - i. Hold a bachelor's degree from an accredited institution either in chemistry or in a biological science, and
 - ii. Have subsequent to graduation at least one year of laboratory training or experience in chemistry, and
 - iii. Have either six months' experience in the operation of atomic absorption equipment or have completed a formal training course in the operation of atomic absorption equipment, and
 - iv. Demonstrate competence in the operation of atomic absorption equipment and analytical procedures during an inspection by a representative of the Department.
4. If the laboratory performs tests or analyses in the category of Gas Chromatography, the supervisor shall:
 - i. Hold a bachelor's degree from an accredited institution either in chemistry or in a biological science, and
 - ii. Have subsequent to graduation at least one year of laboratory training or experience in chemistry, and

- iii. Have either six months' experience in the operation of gas chromatography equipment or have completed a formal training course in the operation of gas chromatography equipment, and
 - iv. Demonstrate competency in the operation of gas chromatography equipment and analytical procedures during an inspection by a representative of the Department.
5. If the laboratory performs test or analyses in the category of Radiological Testing, the supervisor shall:
- i. Hold a bachelor's degree from an accredited institution in chemistry, radiochemistry, radioisotope technology, biology, physics, engineering, or any of the applied sciences, and
 - ii. Have subsequent to graduation at least five years' laboratory training or experience in any of the above, two years of which shall be in low-level radiation measurements and radiochemical procedures being considered for certification, and
 - iii. Demonstrate competency in the operation of radiological equipment and radiological procedures during an inspection by a representative of the Department.
6. If the laboratory performs tests in the category of Bioassay, the supervisor shall:
- i. Hold a bachelor's degree from an accredited institution in a biological science or chemistry which shall include two courses in any of the following subjects:
 - (1) General Zoology;
 - (2) Biological Methods and Experimental Design;
 - (3) Ichthyology;
 - (4) Comparative Physiology;
 - (5) Environmental Science; and
 - ii. Have subsequent to graduation at least one year of laboratory training or experience in the bioassay procedure being considered for certification; and

- iii. Demonstrate competency in the operation of bioassay equipment and methodologies during an inspection by a representative of the Department.
7. Prior to adoption of these regulations, the supervisor shall:
- i. Have had one year of pertinent laboratory experience working in a laboratory performing compliance analyses in a category or categories for which NJPDES certification is required in accordance with the provisions of this chapter, and
 - ii. Demonstrate the ability of complying with the testing, analytical, and quality control requirements contained in this chapter.
- (e) Experience in a certified laboratory which was gained prior to acquiring a bachelors degree may be substituted on an equivalency basis of 1.5 years of such experience for every one year of post-degree training and experience.

7:18-2.8 Duties and responsibilities of laboratory personnel

- (a) Laboratory managers shall have the following responsibilities:
- 1. The Manager shall serve the laboratory on either a full time basis or a regular part-time basis, and shall administer the operations of the laboratory including the reporting of tests and analyses. The manager shall be readily available for personal or telephone consultation, and, if the manager is to be absent, the manager shall arrange for a substitute. Where the manager is acting as laboratory supervisor the substitute shall meet the requirements of N.J.A.C. 7:18-2.7(d).
 - 2. The manager shall be responsible for the employment of an adequate number of qualified personnel, commensurate with the workload of the laboratory and the diversity of tests or analyses performed, and for the inservice training of such personnel.
 - 3. The laboratory manager shall report the discovery of an analytical error to the Department and the person requesting an analysis within 72 hours of discovering the error if the error may effect the validity of a reported analytical result.

(b) Laboratory supervisors shall have the following responsibilities:

1. Each laboratory supervisor shall provide personal and direct supervision of technical personnel and the reporting of tests and analyses within the category or categories for which the supervisor is qualified.
2. Each laboratory supervisor shall perform tests or analyses within the category or categories for which he is qualified, when such tests or analyses require special scientific skills.
3. Each laboratory supervisor shall be held responsible by the Department for the proper performance of all laboratory procedures, test and analyses, within the category or categories for which he is qualified.

7:18-2.9 Management of laboratories

- (a) A certified laboratory may offer as a service those laboratory tests, analyses, or procedures that are within the category or categories for which it is certified provided it has a qualified supervisor in accordance with the provisions of N.J.A.C. 7:18-2.7(d) and for which adequate personnel, equipment and facilities are available.
- (b) A laboratory that is certified shall accept only samples which are properly labeled, and for which there is assurance that the samples have been collected, preserved, processed, stored and transported in such a manner as to assure identity and the stability of the sample with respect to the requested tests or analyses; or if the stability of the sample has not been assured the laboratory shall refuse the sample.
- (c) This section is applicable to laboratories holding interim approvals.

7:18-2.10 Proficiency testing

- (a) Except when determined by the Department that an appropriate proficiency test is not readily available, all certified laboratories or laboratories seeking certification shall participate in a proficiency testing program covering all tests, analyses and analytical methods as made available within the category and categories in which the laboratory is certified or seeks certification.
- (b) Appropriate samples shall be distributed by the Department or its designee to such laboratories at such times and frequencies as designated by the Department.

- (c) Laboratories certified, or seeking certification, shall:
1. Receive, examine and analyze such samples;
 2. Maintain records of such proficiency testing results; and
 3. For all categories except radiological testing, submit the results of such testing, within 30 days from the date of receipt of the proficiency samples, to the Department for evaluation; or.
 4. For radiological proficiency testing, submit results in accordance with the directions of the USEPA.
- (d) The laboratory shall be informed of the results of such evaluation and if the laboratory has not analyzed the proficiency samples acceptably, the Department may require the laboratory to analyze additional proficiency samples.
- (e) The results shall be considered by the Department when making recommendations for improvement in laboratory procedures, and in evaluating whether the certification of the laboratory should be granted, denied, revoked, or suspended.
- (f) Results of proficiency testing during the preceding twelve months shall be made available by the laboratory, upon request, to any person utilizing or requesting the services of the laboratory.
- (g) Certified laboratories that desire to extend the range of tests or analyses offered shall comply with the requirements of N.J.A.C. 7:18-2.3 or 2.4 and shall demonstrate satisfactory results in at least one round of proficiency testing samples prior to the inclusion of this test or analysis in the list of tests or analyses for which proficiency has been established.

7:18-2.11 Laboratory inspections

- (a) As a condition of obtaining and maintaining certification, a laboratory shall permit and facilitate inspections by personnel of the Department.
- (b) The Department shall conduct at least one on-site inspection of a laboratory seeking certification in any parameter to determine whether or not the laboratory meets the Department's standards, as set forth in this chapter, for performing analyses for that parameter.

The on-site inspection shall be performed prior to making a decision concerning the requested certification.

- (c) Regular inspections of laboratories certified in accordance with this chapter shall be conducted during normal State business hours at intervals of not more than two years. Such inspection shall be conducted by representatives of the Department upon presentation of credentials. Laboratories that have moved to a new location shall comply with N.J.A.C. 7:18-2.14 and shall be inspected within one month of receipt of notification by the Department of such changes of location.
- (d) The Department may make an announced or unannounced inspection of any certified or interimly approved laboratory whenever it has reason to believe the laboratory is not performing water analyses in accordance with the requirements of this chapter.
- (e) During inspections, consideration will be given to competence and attitude of staff; working conditions, including adequacy of space; lighting equipment and supplies; efficient organization of the laboratory; testing or analytical methods used; quality control procedures; maintenance of all required records; and compliance with the requirements of this chapter.
- (f) Following inspections, laboratories shall be furnished with inspection reports which shall list deficiencies found, and a listing of the tests and analyses for which the laboratories have demonstrated proficiency during inspections. Such inspection reports and listings shall be deemed public records, and shall be made available to any person utilizing or requesting the services of the laboratory.
- (g) Whenever deviations from the requirements of this chapter are found, the laboratory shall be afforded not less than fifteen days, nor more than thirty days from the date the inspection report is mailed to the laboratory in which to correct such deficiencies. If deficiencies affecting the accuracy of results are found, the certification shall be immediately suspended or revoked, in accordance with the provisions of N.J.A.C. 7:8-2.12.

7:18-2.12 Cancellation, suspension, and revocation of certification

- (a) Any certified laboratory may cancel its certification in any category or parameter by notifying the Bureau in writing of the laboratory's decision to cancel its certification. The laboratory shall enclose its laboratory certificate and license with the letter of notification. This cancellation notification shall not entitle the laboratory to any refund of its certification fee.

(b) The Department may temporarily suspend a laboratory's certification in any or all categories or in any parameter when the laboratory fails to fully meet the standards of this chapter and the failure does not merit immediate decertification action. The Department shall notify the laboratory by letter of its suspension and the reason therefor. Suspensions may be invoked for, but are not limited to, the following reasons:

1. Failure to submit in a timely manner a complete renewal application or the appropriate fee;
2. Failure to submit a timely or acceptable response to the laboratory evaluation report;
3. For all categories except Radiological Testing, failure to submit results of performance evaluation samples within 30 days of receipt of such proficiency samples;
4. For the Radiological category, failure to submit results of performance evaluation samples to the USEPA in a timely fashion;
5. For all categories except Radiological Testing, failing to acceptably analyze both the high and low values for any one parameter during a proficiency test shall be grounds for suspension in the parameter; or
6. For the Radiological category, failing to acceptably analyze two performance test average values for any one parameter during any consecutive twelve month period shall be grounds for suspension in the parameter.

(c) Certification may be revoked by order of the Department for due cause, including, but not limited to:

1. Violation of a condition of the Certification;
2. Violation of a statute, regulation, or order of the Department;
3. Misrepresentations made to the Department;
4. Demonstrable nonconformance with the requirements of this chapter;
5. Substantial change in personnel, facilities or techniques without disclosure thereof to the Bureau;

6. Nonpayment of applicable fees;
7. For all categories except Radiological Testing, failure to analyze a set of proficiency testing samples within 30 days of the receipt of such proficiency samples;
8. For the Radiological category, failure to submit results of performance evaluation samples to the USEPA in a timely fashion;
9. For all categories except Radiological Testing, failing to acceptably analyze both the high and low values for any one parameter during a proficiency test shall be grounds for decertification in the parameter;
10. For the Radiological Category, failing to acceptably analyze two performance test average values for any one parameter during any consecutive twelve month period shall be grounds for decertification in the parameter; or
11. Performing and charging for additional tests or analyses that have not been requested by the customer, falsifying analyses, or engaging in other unethical or fraudulent practices.

(d) Interim approvals may be revoked for due cause by order of the Department, without right to a hearing thereon.

7:18-2.13 Effect and duration of suspension notification and revocation orders

- (a) The results of any tests or analyses performed after issuance of a suspension notification or revocation order for any category or parameter suspended or revoked shall not be accepted by the Department for compliance with the requirements of the New Jersey Safe Drinking Water Act, the New Jersey Water Pollution Control Act and Regulations adopted pursuant to those acts.
- (b) Suspension shall not be withdrawn until all bases for the suspension have been eliminated or rectified.
- (c) Revocations shall provide that reapplication for certification shall not be considered until all bases for revocation have been eliminated or rectified, and until a period of at least six months from the date of revocation has elapsed.
- (d) Recipients of certification revocation orders shall have a right to a hearing thereon, if requested in writing within 20 days of receipt of the revocation

order. Said hearing shall be held before an Administrative Law Judge and in accordance with the Administrative Procedures Act, N.J.S.A. 52:14B-1 et seq. The decision by the Commissioner, based on the hearing record and the recommendations of the Administrative Law Judge shall be the final administrative decision on the revocation.

7:18-2.14 Information to State

In the event there are any changes in the name, location, ownership, post office address or telephone number of a laboratory to which the provisions of this chapter apply, written notice thereof shall be immediately sent to the Bureau of Collections and Licensing, New Jersey Department of Environmental Protection, P.O. Box CN-402 Trenton, New Jersey 08625. In the case of change in supervisor(s) the qualifications of the new supervisor showing compliance with the requirements of N.J.A.C. 7:18-2.7(d) shall be furnished.

SUBCHAPTER 3. CRITERIA AND PROCEDURES FOR MICROBIOLOGICAL TESTING AND ANALYSIS

7:18-3.1 Scope

This chapter establishes the Department's requirements which a certified laboratory or a laboratory seeking certification shall continually meet and follow when performing microbiological analyses.

7:18-3.2 Laboratory facilities and safety

- (a) Laboratory space and facilities shall be adequate to properly carry out the services performed in, or offered by, the laboratory.
- (b) Laboratory work areas shall be arranged so as to minimize problems in transportation and communication.
- (c) Workbench space within the laboratory shall be ample for the tests or analyses to be performed, and shall be well-lighted and convenient to a sink, and such water, gas, suction and electrical outlets as are necessary to properly carry out the specific tests or analyses performed in the laboratory.
- (d) The temperature and humidity within the laboratory shall be maintained within the limits required for the proper performance of each test or analysis and for the proper operation of instruments which may be affected by temperature variations.
- (e) Each laboratory shall have available to it facilities, equipment, and instruments, including but not limited to water baths, incubators, sterilizers, and refrigerators, which shall be adequate to properly perform the tests and analyses for the parameters within this category for which the laboratory is certified or is seeking certification.
- (f) Adequate fire precautions shall be taken, including but not limited to having readily available a fire extinguisher rated for the types of fires that reasonably may be foreseen given the types of tests and analyses performed by the laboratory.
- (g) Appropriate occupational safety and health laws shall be posted and observed.

7:18-3.3 Laboratory equipment, supplies and materials

- (a) Laboratories performing microbiological tests and analyses shall have on the premises and under the

control of the laboratory supervisor the equipment and instruments listed in this section necessary for the preparation and analysis of the specific media and samples for which the laboratory is seeking certification or is certified. Such instruments, when required, shall meet the following specifications:

1. Laboratories performing pH analysis shall have available at least one pH Meter and the pH meter shall have an accuracy of ± 0.1 unit.
2. Top-loader or pan balances shall meet the following requirements:
 - i. Balances shall be clean, not corroded, and shall be provided with appropriate weights of good quality; and
 - ii. Balances shall tare out and detect a weight of 100 mg when used for general media preparation.
3. Temperature-monitoring devices shall meet the following requirements:
 - i. Glass or metal thermometers shall be graduated in 0.5°C increments for all analyses except fecal coliform analysis, in which case glass or metal thermometers shall be graduated in 0.2°C increments;
 - ii. Continuous temperature recording devices shall be sensitive and accurate to within 0.5°C ;
 - iii. The column of liquid in glass thermometers shall have no separation; and
 - iv. A certified thermometer or a thermometer of equivalent accuracy shall be available for use by the laboratory.
4. Air or water-jacketed incubators, aluminum block incubators, incubator rooms, and water baths shall meet the following requirements:
 - i. Incubators, incubator rooms, and water baths shall be of sufficient size to accommodate periods of peak work load;
 - ii. Incubators must maintain internal temperatures of $35.0 \pm 0.5^{\circ}\text{C}$ for total coliform and fecal streptococci analysis, and $44.5 \pm 0.2^{\circ}\text{C}$ for

- ii. Use of the hot air oven is recommended for sterilization of glass pipets, bottles, flasks, culture dishes, and other laboratory glassware and utensils; and
 - iii. A calibrated thermometer with its bulb placed in sand shall be placed on one of the shelves in use within the hot air oven.
7. The refrigerator shall maintain an internal temperature of 1° to 4.4°C (34° to 40°F).
8. Laboratories shall have available the following optical, counting, and lighting equipment:
- i. At least one low power magnification device, preferably a binocular microscope with 10 to 15X magnification, for use in counting fecal coliform and fecal streptococci MF colonies;
 - ii. At least one low power magnification device, preferably a binocular microscope with 10 to 15x magnification, with a fluorescent light source for use in counting total coliform MF colonies; and
 - iii. A mechanical hand tally for use in counting bacteria colonies.
9. Inoculation equipment shall meet the following requirements:
- i. The diameter of inoculation loops shall be at least 3 mm and the loops shall be constructed of 22 to 24 gauge Nichrome, chromel, or platinum-iridium wire;
 - ii. Either single-service metal inoculation loops, pre-sterilized plastic inoculation loops, or reusable metal inoculation loops shall be used; and
 - iii. Disposable dry-heat-sterilized hardwood applicator sticks may be used.
10. Membrane filtration (MF) equipment shall meet the following requirements:
- i. Units used in MF procedures shall be made of stainless steel, glass, or autoclavable plastic;
 - ii. MF equipment shall not leak and shall not be corroded; and

fecal coliform analysis, in the area of use at maximum loading;

- iii. When aluminum block incubators are used, culture dishes and tubes shall fit snugly within the block;
 - iv. Whenever an air incubator is in use, a calibrated thermometer with its bulb immersed in liquid shall be placed on one of the shelves in use within the incubator; and
 - v. The temperature within an incubator shall be recorded daily, or a recording thermometer, sensitive and accurate to temperatures of $\pm 0.5^{\circ}\text{C}$ in the case of total coliform and fecal streptococci incubators and $\pm 0.2^{\circ}\text{C}$ in the case of fecal coliform incubators, shall be used and the recording tape shall be checked daily.
5. The autoclave shall meet the following requirements:
- i. The autoclave shall be in good operating condition when observed during its operational cycle or when time-temperature charts are read, and, for most efficient operation, use of a double-walled autoclave constructed of stainless steel is suggested;
 - ii. The autoclave shall have pressure and temperature gauges on the exhaust side, and shall have a safety valve that is in good operating condition;
 - iii. The autoclave shall reach the sterilization temperature of 121°C and shall maintain that temperature throughout the sterilization cycle, and the autoclave shall complete the sterilization cycle in no more than 45 minutes; and
 - iv. During depressurization the autoclave shall not produce air bubbles in the fermentation media.
6. The hot air oven shall meet the following requirements:
- i. The hot air oven shall be constructed in a manner which shall ensure a stable sterilization temperature;

- iii. Field equipment may be used for coliform detection; however, standard laboratory MF procedures must be followed when using field equipment.
11. Membrane filters and pads shall meet the following requirements:
- i. Membrane filters shall be manufactured from cellulose ester materials, and shall be white, grid-marked, and have a 47-mm diameter and 0.45 μ m pore size; however, another pore size may be used when the performance data provided by the manufacturer show the performance of that pore size to be equal to or better than the performance of the 0.45 μ m membrane filter; and
 - ii. Membrane filters and pads shall be either autoclavable or presterilized.
12. Laboratory glassware, plastic ware, and metal utensils shall meet the following requirements:
- i. Glassware and metal utensils shall be resistant to the effects of corrosion, high temperatures, and vigorous cleaning operations;
 - ii. Flasks, beakers, dilution bottles, culture dishes, culture tubes, and other glassware shall be of borosilicate glass and free of chips, cracks, and excessive etching;
 - iii. Volumetric glassware should be Class A and need not be calibrated before use;
 - iv. Plastic items shall be of clear, inert, nontoxic materials and shall retain accurate calibration marks after repeated autoclaving; and
 - v. It is recommended that metal utensils made of stainless steel be used.
13. Sample bottles shall meet the following requirements:
- i. Either wide-mouthed hard glass and stoppered sample bottles, or plastic sample bottles with screw caps, shall be used, and all sample bottles shall have a capacity of at least 120 mL.;

- ii. Glass-stoppered bottles shall be stored so that they are protected from contamination by dust and the caps shall be covered with either aluminum foil or kraft paper;
 - iii. Screw caps shall have leakproof nontoxic liners which are capable of withstanding repeated sterilizations, at temperatures of 121°C sustained for 30 minutes per sterilization; and
 - iv. Sterile sample bottles shall contain 10 mg. of dechlorinating agent per 100 mL. of sample.
14. Pipets shall meet the following requirements:
- i. Sterile, glass or plastic pipets shall be used for measuring quantities of 10 mL or less;
 - ii. Glass pipets shall be made of borosilicate glass;
 - iii. Pipets shall deliver the required volume quickly and accurately within a 2.5 percent tolerance;
 - iv. Pipets shall not be excessively etched, mouthpiece or delivery tips shall not be chipped, and graduation marks shall be legible.
15. Pipet containers shall meet the following requirements:
- i. Open packets of disposable sterile pipets shall be resealed after each use; and
 - ii. Pipet containers shall be made of aluminum or stainless steel.
16. Culture dishes shall meet the following requirements:
- i. Sterile plastic culture dishes with tight or loose lids, or glass culture dishes with loose lids shall be used; and
 - ii. When culture dishes with loose lids are used, the relative humidity in the incubator shall not be less than 90 percent.
17. Culture dish containers shall meet the following requirements:

- i. Culture dish containers shall be made of either aluminum or stainless steel, or the culture dishes may be wrapped in heavy aluminum foil or char-resistant paper; and
 - ii. Open packs of disposable sterile culture dishes shall be resealed after each use.
18. Culture tubes and closures shall meet the following requirements:
- i. Culture tubes shall be made of borosilicate glass or other corrosion resistant glass and shall be of a sufficient size to contain both the culture medium and the sample portions to be tested, without being more than three-quarters full; furthermore, it is recommended that the fermentation vial be 10 mm x 75 mm and extend above the medium; and
 - ii. Caps should be made of snug-fitting stainless steel or plastic; however, loose-fitting aluminum caps or screw caps are also acceptable.

7:18-3.4 Sample collection, handling, and preservation

- (a) When the laboratory has been delegated responsibility for drinking water sample collection, handling, and preservation by the water purveyor, there shall be strict adherence to the required sampling procedures, complete identification of the sample, and prompt transfer of the sample to the laboratory as described in Standard Methods, 14th Edition, p. 904-907.
- (b) The minimum sampling frequency for Drinking Water analysis shall be that specified in 40 CFR 141.21.
- (c) The sample collector shall be trained by the laboratory in sampling procedures.
- (d) When collecting drinking water samples, the water tap shall flow steadily for two to three minutes before the sample is collected. Any aerator, strainer, hose attachment, or water purification device shall be removed from the tap prior to sample collection.
- (e) The sample volume shall be at least 100 mL. The sample bottle shall be filled only to the shoulder.
- (f) The sample report form shall be completed immediately after collection and shall state the sampling location, date and time of collection, chlorine residual, collectors name, and any remarks.

- (g) Sample bottles shall have a capacity of at least 120 mL and shall be made of either sterile plastic or hard glass, and shall be wide mouthed with either a plastic screw cap or glass stopper. Sample bottles shall be capable of withstanding repeated sterilization. Ten milligrams of sodium thiosulfate per one hundred mL of sample shall be added to all sample bottles during preparation of the bottles.
- (h) Immediately after the sample is delivered to the laboratory, the date and time of arrival shall be recorded on the sample report form.
- (i) Immediately after testing has begun, the date and time of initiation of testing shall be recorded on the sample report form.
- (j) The following chain of custody procedures shall be employed in collecting and handling samples.
1. Sterilized and decontaminated containers shall be used for sampling.
 2. Tie-on or affixed labels with an identification number shall be used for labeling all samples.
 3. Immediately after the container has been sterilized and decontaminated, the containers shall be labeled with the appropriate label. Such labels shall remain affixed to the container until the time of sampling.
 4. When collecting samples, if it is necessary to remove the label only one container at a time shall have its label removed. After the sample has been collected, the appropriate information as to the identity of the sample shall be written on the label. If the label has been removed it shall be reaffixed before removing the label from any other container.
 5. After collecting the sample and, if necessary, reaffixing the label, it shall remain affixed to the sample container and shall not be removed until the required analyses have been completed and the surplus sample has been discarded.
 6. Immediately upon delivery of the sample to the laboratory, the sample collector shall complete the appropriate section of a chain-of-custody form.

7. The chain-of-custody form shall list at a minimum the following information:
 - i. Sample number;
 - ii. Number of containers;
 - iii. Description of samples;
 - iv. Specific location of sample collection;
 - v. Identity of person collecting the sample;
 - vi. Date and time of sample collection;
 - vii. Date and time of custody transfer to laboratory;
 - viii. Identity of person accepting custody;
 - ix. Date and time of initiation of analyses;
 - x. Identity of person performing analyses;
 - xi. Name of laboratory performing the analyses;

8. Prior to accepting custody of the sample, the laboratory personnel who will accept custody shall be reasonably assured that the sample has met the collection, handling and preservation requirements. If the sample fails to meet those requirements, the chain of custody form and final laboratory report shall so indicate and the sample shall be rejected.

9. The laboratory personnel accepting responsibility for the sample as well as all other laboratory personnel performing analysis on that sample shall sign the chain of custody form.

10. When it is necessary to send Drinking Water samples by mail, bus, courier service, or private shipping, the chain of custody form shall be completed by the sampler prior to the shipping of the sample and shall accompany the sample during shipping. Upon receipt of the sample in the laboratory steps (j)7 thru 9 above shall be followed.

- (k) The holding time between sample collection and analysis of Drinking Water samples shall not exceed 30 hours. Samples that fail to meet this holding time shall be rejected and a new sample requested.
- (l) Fecal coliform samples collected for NJPDES compliance analysis shall be analyzed within the holding time specified in 40 CFR 136. Samples that fail to meet this holding shall be rejected and a new sample requested.
- (m) Drinking Water and NJPDES samples that cannot be analyzed within one hour following collection shall be stored in iced coolers during transit to the laboratory and refrigerated upon delivery until such analyses can be performed.
- (n) A laboratory that has received either certification or interim approval shall accept only samples that are properly labeled and for which assurance is given that the samples have been collected, preserved, processed, stored and transported in a manner that will assure both the identity of the sample and that the sample is sufficiently stable to be used in the requested tests or analyses.

7:18-3.5 Methodology

- (a) Laboratories shall use the test procedures required by 40 CFR 141 in the analysis of drinking water microbiological parameters.
- (b) Test procedures required by 40 CFR 136 shall be utilized for the analysis of NJPDES parameters.
- (c) All procedures other than those set forth in subsections (a) and (b) above are considered alternative analytical techniques as described in 40 CFR 141.27 and 40 CFR 136.4. Laboratories shall make special application to the Commissioner for the use of alternative analytical methods and such application shall include a showing of acceptable comparability data.
- (d) All laboratories which have previously been granted approval to use an alternate analytical method by the USEPA shall be allowed to continue using such method after it submits written proof of the approval to the Department.
- (e) The membrane filter (MF) procedure used for Drinking Water Analysis should show good colony development over the entire surface. The golden green metallic sheen colonies shall be counted and recorded as the total coliform density per 100 mL of water sample. The

following rules for reporting any problem with MF results shall be followed:

1. In the case of total coliform analysis, if there is confluent growth, with or without discrete sheen colonies, covering the entire filtration area of the membrane, the results shall be reported as "confluent growth per 100 mL, with (or without) total coliforms", and a new sample shall be requested.
2. In the case of fecal coliform and fecal streptococci analysis, if there is confluent growth, with or without typical discrete colonies, covering the entire filtration area of the membrane, the results shall be reported as "confluent growth per 100 mL, with (or without) fecal coliforms (or fecal streptococci)", and a new sample shall be requested.
3. When the total number of bacterial colonies on the membrane is greater than 200 total colonies, or is not sufficiently distinct, or both, the results shall be reported as "Too numerous to count (TNTC) per 100 mL, with (or without) total coliforms, (or fecal coliform, or fecal streptococci)" and a new sample shall be requested;
4. When both confluent growth and TNTC are present, a new sample shall be requested and, if the MF procedure is used, the sample volumes filtered shall be adjusted by increasing dilution of the sample; otherwise the most probable number (MPN) procedure shall be used.
5. If the laboratory has elected to use the MPN test on water supplies or discharges that have a history of confluent growth or TNTC with the MF procedure, all presumptive tubes from the MPN test that have heavy growth but no gas production should be submitted to the confirmed MPN test to check for the suppression of coliforms; the count shall be adjusted based upon confirmation and a new sample shall be requested. In addition, this procedure should be carried out on samples collected from water supplies or discharges known to have such a history, at a frequency of at least once every three months.

7:18-3.6 General laboratory practices

- (a) Laboratory sterilization procedures shall meet the following requirements:

1. The following times and temperatures shall be used for sterilization of materials by autoclaving:

Material	Temperature/Minimum Time
Membrane filter and pads	121°C/10 min.
Carbohydrate-containing media (lauryl tryptose, brilliant green lactose bile broth, etc.)	121°C/12-15 min.
Contaminated materials and discarded tests	121°C/30 min.
Membrane filter assemblies (wrapped), sample collection bottles (empty), individual glassware items	121°C/30 min.
Rinse water volumes of 500 ml to 1,000 ml	121°C/45 min.
Rinse water in excess of 1,000 ml	121°C/time adjusted for volume; check for sterility
Dilution water blanks	121°C/30 min.

2. Membrane filter assemblies shall be sterilized after each sample filtration series, the end of which is marked by the lapse of 30 minutes or more between sample filtrations;
3. At least two minutes of ultraviolet light or boiling water may be used on a membrane filter assembly to prevent bacterial carry-over between filtrations; and
4. Dried glassware shall be sterilized in a hot air oven at 170°C for a minimum of two hours.

- (b) Laboratory pure water, including distilled, deionized, or other processed waters, shall meet the following requirements:
1. An analyst shall either test the quality of the laboratory pure water or have the laboratory pure water tested by another State certified laboratory; and
 2. Only laboratory pure water meeting the requirements set forth in N.J.A.C. 7:18-3.7(a)lviii shall be used in performing bacteriological analyses.
- (c) Rinse water and dilution water used by the laboratory shall meet the following requirements:
1. Stock buffer solution shall be prepared in accordance with Standard Methods, 14th Edition or Microbiological Methods - EPA, using laboratory pure water adjusted to pH 7.2;
 2. Stock buffer shall be either autoclaved or filter-sterilized, and must be labeled, dated, and stored at 1° to 4.4°C;
 3. The stored buffer solution shall be free of turbidity; and
 4. Rinse and dilution water shall be prepared by adding 1.25 mL of stock buffer solution per liter of laboratory pure water, and the final pH shall be 7.2 ± 0.1 .
- (d) Media shall be prepared and stored in accordance with the following requirements:
1. Laboratories shall use commercial dehydrated media for routine bacteriological procedures;
 2. All media shall be prepared according to the procedures for media preparation set out in Standard Methods, 14th Edition, or Microbiological Methods - EPA, however, lactose broth shall not be used;
 3. Dehydrated media containers shall be kept tightly closed and stored in a cool, dry location, to prevent discoloration and caking; laboratories shall not use discolored or caked dehydrated media;
 4. Dissolution of the media using laboratory pure water shall be completed before dispensing to culture tubes or bottles;

5. The membrane filter broth and agar media shall be heated in a boiling water bath until completely dissolved;
6. MF broths shall be stored and refrigerated no longer than 96 hours and MF agar media shall be stored, refrigerated and used within two weeks;
7. MPN media prepared in tubes with loose-fitting caps shall be used within one week, but if MPN media are refrigerated after sterilization, they shall be incubated overnight at 35°C to confirm usability, and tubes showing growth or gas bubbles shall be discarded;
8. Media in screw cap containers may be held up to three months, provided that the media are stored in an enclosed area so that no light may enter and provided that evaporation does not exceed 0.5 mL per 10 mL total volume; in addition, commercially prepared liquid and agar media supplies may be used; and
9. Ampouled media shall be stored at 1° to 4.4°C (34° to 40°F), and storage time shall be limited to the manufacturer's expiration date.

- (e) When measuring sample volumes of more than 10 mL, graduated cylinders or graduated membrane filter funnels having an accuracy within 2.5 percent tolerance shall be used.

7:18-3.7 Quality control program

- (a) Each laboratory shall develop and have on file and available for inspection a written description of the current laboratory quality control program. Such written description shall outline the procedures which the laboratory will use in meeting the quality control requirements set forth in this section and N.J.A.C. 7:18-3.4 and 7:18-3.6. Management, supervisors, and analysts should participate in developing the quality control program. Each participant within the laboratory should have a copy of the quality control program and detailed guidelines for implementation of the participant's responsibility. A record of analytical control tests and quality control checks on media, materials, and equipment shall be prepared by the laboratory and retained for at least five years.

1. Laboratories shall perform the following analytical quality control tests to ensure that general laboratory practices and methodology are in compliance with the requirements of this subchapter:
 - i. When analyzing Drinking Water samples, the laboratory shall verify at least five sheen

or borderline sheen total coliform bacterial colonies from each membrane containing five or more such colonies. Bacteria counts shall be adjusted based on this verification. The verification procedure shall be conducted by transferring growth from the total coliform bacterial colonies into lauryl tryptose broth (hereinafter referred to as LTB) tubes and then transferring growth from gas-positive LTB cultures to brilliant green lactose bile (hereinafter referred to as BGLB) tubes. Colonies shall not be transferred exclusively to BGLB. However, colonies may be transferred to LTB and BGLB simultaneously. Negative LTB tubes shall be reincubated on the day following the verification procedure and shall be confirmed if gas is produced. It is recommended that laboratories verify all sheen colonies and borderline sheen colonies.

- ii. A start and finish MF sterile control test of rinse water, media and supplies shall be conducted for each sample filtration series. If the MF sterile control tests indicate contamination of rinse water, media, or supplies, then all data which has been generated through tests involving the use of the contaminated rinse water, media, or supplies shall be rejected and the laboratory shall request immediate resampling of those waters involved in the laboratory error.
- iii. The MPN test for Drinking Water samples shall be carried to completion on 10 percent of positive confirmed samples except that gram staining shall not be performed; but, if no positive tubes result from the tested Drinking Water samples, the complete MPN test, but not gram staining, shall be performed on a quarterly basis on at least one water source for which results have been positive;
- iv. Laboratory pure water shall be analyzed annually by the test for bactericidal properties for distilled water as set forth in Standard Methods, 14th Edition, p. 888, or Microbiological Methods - EPA, p. 200. Only satisfactorily tested laboratory pure water is permissible in preparing media, reagents, rinse, and dilution water. If the tests do not meet the requirements for laboratory pure

- water set forth in subparagraph viii. below, then corrective action, including but not limited to purchasing a fresh supply of laboratory pure water or purification of the existing supply and source of laboratory pure water, shall be taken immediately and the water shall be retested;
- v. Laboratory pure water shall be analyzed monthly for conductance, pH, chlorine residual, and standard plate count. If the test results for any of the substances exceed the standards set forth in subparagraph viii. below, then corrective action, including but not limited to purchasing a fresh supply of laboratory pure water or purification of the existing supply and source of laboratory pure water, shall be taken and the water shall be retested;
- vi. The laboratory shall ensure that laboratory pure water does not come in contact with heavy metals. Laboratory pure water shall be analyzed annually for trace metals, with particular emphasis upon analysis to detect Pb, Cd, Cr, Cu, Ni, and Zn. If the test results show that the laboratory pure water does not meet the requirements set forth in subparagraph viii of this section, then corrective action, including but not limited to purchasing a fresh supply of laboratory pure water or purification of the existing supply and source of laboratory pure water, shall be taken and the water shall be retested;
- vii. Standard plate count procedure shall be performed on laboratory pure water as described in Standard Methods, 14th Edition, or Microbiological Methods - EPA. Plates shall be incubated at $35.0 \pm 0.5^{\circ}\text{C}$ for 48 hours.
- viii. Requirements for laboratory pure water:
- | | |
|--------------|--|
| pH | 5.5 - 7.5 |
| Conductivity | Greater than 0.2 megohm-cm as resistivity or less than 5.0 micromhos/cm as conductance at 25°C |

Trace metals:	
A single metal	Not greater than 0.05 mg/L
Total metals	Equal to or less than 1.0 mg/L

Test for bactericidal properties of distilled water	
Standard Methods, 14th Edition p. 888, or Microbiological Methods - EPA, p. 200)	0.8 - 3.0
Free chlorine residual	0.0
Standard plate count	Less than 10,000 colonies/mL

- ix. Each laboratory should analyze one quality control sample per year, when available from the Department, for the parameter or parameters for which the laboratory has received certification or interim approval;
- x. Each laboratory shall satisfactorily analyze one unknown performance sample per year, when available, for the parameter or parameters within the category or categories for which the laboratory has received certification or interim approval;
- xi. Duplicate analyses should be run on known positive samples at least once per month, and the duplicates should then be run as a split sample by more than one analyst, with each split constituting a 50 mL sample;
- xii. Any laboratory associated with a public water facility shall examine a minimum of one polluted water source per month in addition to analyzing the required number of distribution samples;
- xiii. In the case of laboratories having more than one analyst, each analyst should count the total coliform sheen colonies on a membrane

from a polluted water source at least once per month, colonies on the membrane should be verified, and the analysts' counts should be compared to the verified count;

- xiv. There shall be available at all times, in the immediate bench area of laboratory personnel engaged in examining samples and performing related procedures within a category, current laboratory manuals or other complete written descriptions and instructions relating to:
 - (1) The analytical methods to be used by those personnel, properly designated and dated to reflect the most recent supervisory reviews;
 - (2) Pertinent current literature references; and
 - (3) Such written descriptions and instructions may be supplemented by, but not replaced by, textbooks relating to the particular analytical methods and procedures employed by such personnel;

- xv. Only the laboratory manager or supervisor shall make changes in laboratory procedures and those changes shall only be effective when put in writing; and

- xvi. Laboratories shall maintain an acceptable quality control program covering each method or procedure for testing and analysis performed by the laboratory in order to verify and assess accuracy, measure precision, and detect errors in the results of such tests and analyses; additionally, such quality control program shall meet the following specific requirements:
 - (1) Biological solutions, reagents, and media shall be tested and inspected on each day of use for reactivity and deterioration, and the results of such tests and inspections shall be recorded; and
 - (2) Each batch of medium for microbiological testing shall be tested with selected organisms, either before or concurrently with use of the medium, to confirm the required growth characteristics, selectivity, enrichment, and biological response, and

the results of such tests shall be recorded;

2. The following procedures shall be followed in performing quality control checks of laboratory media, equipment, and supplies:
 - i. Each pH Meter shall be cleaned immediately after each use period and calibrated prior to usage using at least two pH buffer standards that bracket the value to be measured and records of each calibration shall be maintained; buffer aliquots shall not be used more than once; and commercial buffer solutions shall be dated at the time of initial use;
 - ii. Top loader or pan balances shall be calibrated annually; calibration shall be checked monthly against class "s" weights, and a record shall be made of each calibration check;
 - iii. Glass thermometers and continuous recording devices shall be checked yearly and metal thermometers shall be checked quarterly, or at more frequent intervals if necessary, against a certified thermometer or a thermometer of equivalent accuracy, at several points throughout the entire range, including but not limited to the temperature point or range for the test, analysis or quality control measure being performed, and the results of such testing shall be recorded;
 - iv. The temperature of air or water-jacketed incubators, water baths, and incubator rooms shall be either recorded continuously or recorded daily from in-place thermometers immersed in liquid and placed on at least one of the shelves in use;
 - v. Date, time, and temperature shall be either recorded continuously or recorded individually during each sterilization cycle of the autoclave;
 - vi. Each hot air oven shall be equipped with either a thermometer calibrated in the range of 170°C, the bulb of which shall be placed in sand, or with a temperature recording device, and records shall be maintained showing the date, time and temperature of each sterilization cycle;
 - vii. Laboratories shall use only membrane filters that have been recommended by the manufacturer for use in the analysis of water;

- viii. The temperature of each refrigerator shall be either recorded continuously or recorded daily from an in-place thermometer immersed in liquid and placed on at least one of the shelves in use;
- ix. Washing processes shall be adequate to provide clean glassware with no stains or spotting, and at the time of initial use of a detergent or washing product and whenever the brand or type of washing product is changed, the rinsing process shall demonstrate that the detergent or washing product provides glassware free of toxic material by the inhibitory residue test as set forth in Standard Methods, 14th Edition, p. 885, or Microbiological Methods - EPA, p. 199;
- x. At least one sample bottle from each batch of sterilized sample bottles shall be checked by adding approximately 25 mL of sterile LTB to the bottle or bottles and then incubating the preparation at $35^{\circ} + 0.5^{\circ}\text{C}$ for 24 hours, at the end of which time the bottle or bottles shall be checked for growth;
- xii. Service contracts or internal protocols approved by the Office of Quality Assurance shall be maintained on balances, autoclave, water still, and any other equipment requiring periodic servicing, and records of actual servicing shall be entered in a log book;
- xiii. Records of preparation of each batch of sterilized media shall be made available for inspection and shall show the lot number of the batch, date of preparation, sterilization time and temperature, final pH of each batch, and the preparing technician's name;
- xiv. Both positive and negative cultures should be used, and should be tested to determine recovery and performance compared to a previous acceptable lot of medium;
- xv. Media should be ordered on the basis of estimated needs for the next 12 month period. Bottles shall be dated upon receipt by the laboratory and upon initial opening. Except for large volume uses, media should be purchased in 1/4-lb bottles. Bottles of media should be used within six months after opening; however, in no case should opened media be used after one year. Opened bottles

should be stored in a desiccator to extend storage time beyond six months. Shelf life of unopened bottles is, two years;

- xvi. Testing should be carried out on media and membranes to determine recovery and performance as compared to a previously acceptable lot of media and membranes;
- xvii. The lot number of packages of membrane filters and date of receipt by the laboratory shall be recorded;
- xviii. Heat sensitive tapes and spore strips or spore ampoules should be used during sterilization, and, it is recommended that a maximum registering thermometer be used;
- xix. All reagents and solutions shall be labelled to indicate identity and, when applicable, titer, strength or concentration, recommended storage requirements, preparation or expiration date, and other information pertinent to identification;
- xx. Materials of substandard reactivity and deteriorated materials shall not be used; and
- xxi. All outdated material shall be discarded immediately.

7:18-3.8 Records and data reporting

- (a) Each laboratory shall maintain records and report data in accordance with the requirements set forth in this section.
- (b) Records of microbiological analysis shall be kept by the laboratory for not less than five years. This requirement is equally applicable to all raw data, quality control data, chain of custody forms and laboratory reports.
- (c) The following information shall be kept by the laboratory as part of the records of all bacteriological analyses, although such information need not be included in the report to the person requesting the laboratory analysis unless otherwise required:
 - 1. Laboratory number or other form of identification of the sample;
 - 2. Date, time, and specific location of sampling, as well as the name of the person who collected the sample or the laboratory which submitted the sample;

3. For drinking water samples, identification of the sample source as a routine distribution system sample, check sample, raw or process water sample, or other applicable designation;
 4. The date and time of receipt of the sample by the laboratory; whether the sample, when received, was preserved or unpreserved; and if the sample was received unpreserved, the type of preservation measures taken;
 5. The date and time of analysis of the sample;
 6. The person or persons who performed analysis of the sample;
 7. The type of analysis performed and the specific analytical method or methods employed;
 8. The results of the analysis; and
 9. The name and address of the laboratory to which the sample was forwarded, if the analysis was not performed at the laboratory which received the sample.
- (d) The sample collector shall complete a chain of custody form immediately after each sample is taken.
- (e) A copy of the chain of custody form shall be attached to the sample report form and the results of each analysis shall be calculated and entered on the sample report form which is to be forwarded to the person requesting the analysis of the sample. A careful check shall be made to assure that each result entered on the sample report form is the same as the result entered on the bench sheet and once the check is completed the analyst shall initial the bench sheet.
- (f) The original or true duplicate of the results of the tests or analyses shall be sent promptly to the person who requested such tests or analyses, and shall be signed by the laboratory manager or a designee whose designation is in writing and has been submitted to the Department. In the case of tests or analyses performed pursuant to the Safe Drinking Water Act Regulations for Public Noncommunity Water Systems, the results of such tests or analyses shall be reported to the owner of the water system on computer input forms which will be provided by the department.
- (g) Whenever a certified or interimly approved laboratory refers samples to another laboratory for analyses, the person requesting the analyses or tests shall receive the original laboratory report or a true duplicate of

that report on the form of the laboratory that performs the tests or analyses. In the case of tests or analyses performed under the Safe Drinking Water Act Regulations for Public Noncommunity Water Systems, when use of a specific laboratory report form is required, the laboratory performing the tests shall report the results on such required forms.

- (h) All results shall be reported immediately to the person requesting the analyses.
- (i) For Drinking Water samples, positive results shall be reported as preliminary without waiting for MF verification or MPN completion. After MF verification or MPN completion, the adjusted counts shall be reported to the person requesting the analyses.
- (j) If results are entered into a computer storage system, a printout of the data should be verified with the raw data.

SUBCHAPTER 4: CRITERIA AND PROCEDURES FOR CHEMICAL TESTING AND ANALYSIS

7:18-4.1 Scope

This chapter establishes the Department's requirements which a certified laboratory or a laboratory seeking certification shall continually meet and follow when performing chemical analyses.

7:18-4.2 Laboratory facilities and safety

- (a) Laboratory space and facilities shall be adequate to properly carry out the services performed in, or offered by, the laboratory.
- (b) Laboratory work areas shall be arranged so as to minimize problems in transportation and communication.
- (c) Workbench space within the laboratory shall be ample for the tests or analyses to be performed, and shall be well-lighted and convenient to a sink, and such water, gas, suction and electrical outlets as are necessary to properly carry out the specific tests or analyses performed in, or offered by, the laboratory.
- (d) The laboratory shall be adequately ventilated to exhaust the gases produced by the tests or analyses performed by and the types of materials handled by the laboratory.
- (e) The temperature and humidity within the laboratory shall be maintained within limits required for the proper performance of each test or analysis and for the proper operation of instruments which may be affected by temperature variations.
- (f) Volatile or corrosive chemicals and flammable solvents shall be stored in accordance with the Federal Occupational Safety and Health Act and attendant Regulations.
- (g) Adequate fire precautions shall be taken, including but not limited to having readily available a fire extinguisher rated for the types of fires that may reasonably be foreseen given the types of testing and analyses performed by and the types of materials handled by the laboratory.
- (h) Appropriate occupational safety and health laws shall be posted and observed.

7:18-4.3 Specifications for laboratory equipment and instrumentation

- (a) Laboratories which have received certification or are seeking certification to perform any of the required

chemical analyses, shall have on the premises and under the control of the laboratory manager, all of the equipment and instruments necessary to analyze each parameter in which the laboratory is certified, or is seeking certification and such equipment and instruments shall meet the following specifications:

1. Analytical balance shall meet and be operated in accordance with the following specifications:
 - i. Each analytical balance shall have a sensitivity of 0.1 mg;
 - ii. The analytical balance should be mounted on a heavy, shockproof table. The balance level should be checked frequently and shall be adjusted as necessary;
 - iii. The analytical balance should be located in an area that is not near laboratory traffic and is protected from sudden drafts and humidity changes;
 - iv. The balance temperature shall be equilibrated with room temperature;
 - v. When the analytical balance is not in use, the balance beam should be raised from the knife edges, the weights should be returned to zero, and all objects, including but not limited to weighing dishes and other objects used for weighing, should be removed from the balance pan, and the slide door should be closed;
 - vi. Special precautions should be taken to avoid spillage of corrosive chemicals on the pan or inside the balance case, and the interior of the balance housing should be kept scrupulously clean.
2. The photometers shall meet and be operated in accordance with the following specifications:
 - i. Spectrophotometers:
 - (1) The maximum spectral bandwidth shall be no more than 20 nm;
 - (2) Wavelength accuracy shall be within 0 ± 2.5 nm;
 - (3) The spectrophotometer should be capable of using several sizes and shapes of absorption

cells to provide a sample path from 1 to 5 cm.

(4) , All cells shall be kept clean and free of scratches, fingerprints, smudges, and evaporated film residues; and

(5) An exterior, high-capacity, constant voltage transformer is recommended for general laboratory analyses;

ii. Filterphotometers:

(1) Isolation of relatively broad bands (10 to 75 nm) of this radiant energy is achieved by use of filters at or near the maximum absorption of the colorimetric methods; and

(2) Filterphotometers should be capable of using several sizes and shapes of absorption cells to provide a sample path from 1 to 5 cm.

3. The magnetic stirrer shall have variable speeds, and a Teflon^R coated stirring bar.

4. The pH meter shall meet and be operated in accordance with the following specifications:

i. The accuracy of the pH meter shall be within ± 0.05 units;

ii. The scale readability of the pH meter shall be ± 0.1 units;

iii. Both glass and calomel electrodes shall be rinsed well with distilled water after each reading, and shall be either rinsed or dipped several times into the next sample to be tested before the final reading is taken;

iv. Weakly buffered samples should be stirred during measurement;

v. Glass electrodes should be either immersed in distilled water or stored according to the manufacturer's recommendations during periods of inactivity.

5. Specific ion meter shall be readable and accurate to ± 5 mv.

6. The atomic absorption spectrophotometer shall meet and be operated in accordance with the following requirements and specifications:
 - i. The atomic absorption spectrophotometer shall be a single channel, single or double beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for interfacing with either a strip chart recorder or a digital printout unit;
 - ii. The lamps of the spectrophotometer should be dated when first used;
 - iii. The pressure inside the acetylene tank should always be greater than 75 psi;
 - iv. Proper ventilation shall be maintained above the burner head, and the mouth of the exhaust should be approximately 12 inches above the burner head;
 - v. A moisture trap should be incorporated into the flow system between the air source and the atomic absorption spectrophotometer itself;
 - vi. Safety goggles shall be available for use by laboratory personnel when operating the spectrophotometer;
7. Laboratories performing atomic absorption analysis shall have a strip chart recorder for use with the atomic absorption spectrophotometer. The strip chart recorder shall have a chart width of 10 inches (25 cm), a full scale response time of 0.5 second or less, 10 or 100 mv input to match the instrument, and variable chart speeds of 5 to 50 cm/min, or an equivalent chart speed. A digital printout unit may be substituted for the recorder.
8. Laboratories performing gas chromatographic analysis may use either a commercial or custom-designed gas chromatograph (with appropriate detectors), but in either case the gas chromatograph shall have a column oven capable of isothermal temperature control to within $\pm 0.2^{\circ}\text{C}$. The system should be equipped with accurate needle-valve gas flow controls and should accept 1/4 in. glass columns with the option of direct on-column injection.
9. Laboratories performing gas chromatographic analysis shall have a strip chart recorder for use with the

- gas chromatograph. The strip chart recorder shall have at a minimum a chart width of 10 in (25 cm), a full scale response time of one second or less, a 1 mv (-0.05 to 1.05) signal to match the instrument, and variable chart speeds of 5 to 50 cm/minute or an equivalent chart speed. A digital/integrator plotter may be substituted for the recorder.
10. The conductivity meter, suitable for checking laboratory pure water quality, shall be readable in ohms·cm or mhos/cm, have a range of 2 to 2,500,000 ohms·cm or equivalent mhos/cm (± 1 percent), and have a sensitivity of 0.33 percent or better. The conductivity meter should be equipped with platinum electrodes.
 11. Gravity or mechanical convection drying ovens shall have a selectable temperature control ranging from room temperature to 180°C or higher. A long stem thermometer which has been calibrated against a certified thermometer over the range of which the oven is being operated shall be inserted through a center ceiling port, and the bulb of the thermometer shall be inserted into a cylinder filled with sand.
 12. Desiccators with the appropriate indicator desiccant shall be either glass or plastic as appropriate to the particular task being performed.
 13. Hot plates shall have selectable temperature controls.
 14. For storage of aqueous reagents and samples, a standard household refrigerator may be used. However, for storage of organics and flammable materials, an explosion-proof refrigerator should be used. The refrigerator shall maintain an internal temperature of 1° to 4.4°C (34° to 40°F).
 15. Laboratory glassware should be made of borosilicate glass that is resistant to damage by heat, chemicals, and repeated use. All volumetric glassware shall be Class A and need not be calibrated before use. The following criteria and procedures apply to laboratory glassware:
 - i. Unless otherwise specified, borosilicate or bottles shall be used for the storage of reagents and standard solutions;
 - ii. Polyethylene bottles may be used instead of borosilicate bottles for the storage of reagents and standard solutions;

- iii. Serological or Mohr-type pipets are not volumetric pipets and shall not be used in tests or analyses requiring quantitative sample transfer and measurement;
16. The stirred water bath shall have a temperature range from ambient temperature to 100°C, the bath shall have a gable lid and it shall be stirred by a stirring device.
17. Temperature monitoring devices shall meet the following requirements:
 - i. Glass or metal thermometers shall be graduated in 0.5°C increments;
 - ii. Continuous temperature-monitoring, devices shall be sensitive to 0.5°C;
 - iii. The liquid column of glass thermometers shall have no separation; and
 - iv. Laboratories shall have available at least one certified thermometer or one thermometer of equivalent accuracy.

7:18-4.4 Sample collection, handling and preservation

- (a) Sample collection, handling and preservation techniques set out in the following Table I shall be required for primary drinking water parameters.

TABLE I - SAMPLE COLLECTING, HANDLING, AND PRESERVATION¹

	Preservative ²	Container ³	Maximum Holding Time ⁴
Primary Drinking Water Parameters			
Arsenic	Conc HNO ₃ to pH 2	P or G	6 months
Barium	Conc HNO ₃ to pH 2	P or G	6 months
Cadmium	Conc HNO ₃ to pH 2	P or G	6 months
Chromium	Conc HNO ₃ to pH 2	P or G	6 months
Lead	Conc HNO ₃ to pH 2	P or G	6 months
Mercury	Conc HNO ₃ to pH 2	G P	38 days 14 days
Nitrate	Conc H ₂ SO ₄ to pH 2	P or G	14 days
Selenium	Conc HNO ₃ to pH 2	P or G	6 months
Silver	Conc HNO ₃ to pH 2	P or G	6 months
Fluoride	None	P or G	6 months
Chlorinated hydrocarbons	Refrigerate at 4°C as soon as possible after collection	G with foil or Teflon-lined cap	14 days ⁵
Chlorophenoxys	Refrigerate at 4°C as soon as possible after collection	G with foil or Teflon-lined cap	7 days ⁵
Trihalomethanes	Refrigerate at 4°C as soon as possible after collection	Narrow mouth screw cap bottles with TFE fluorocarbon face silicone septa cap liners	14 days
Turbidity	None required	P or G	1 hour
Free Chlorine Residual	None required	P or G	1 hour

a laboratory has no control over these factors, the laboratory supervisor shall reject any samples not meeting these criteria and so notify the authority requesting the analyses.

Samples requiring preservation shall be preserved at time of collection. A laboratory that has interim approval or certification shall accept only samples which are properly labeled, and for which there is reasonable assurance that they have been collected, preserved, processed, stored and transported in such manner as to identity and stability of the sample with respect to the requested tests or analyses; or if the stability of the sample has not been assured, the laboratory report shall clearly state that the result may be invalid due to an unsatisfactory sample. If HNO_3 cannot be used because of shipping restrictions, sample may be initially preserved by icing and immediately shipping it to the laboratory. Upon receipt in the laboratory, the sample must be acidified with conc. HNO_3 to pH 2. At time of analysis, sample container should be thoroughly rinsed with 1:1 HNO_3 ; washings should be added to sample.

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

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

Un-stoppered and refrigerated extracts can be held up to 30 days.

(b) Sample collection, handling, and preservation techniques set out in 40 CFR 136 shall be required for NJPDES and secondary drinking water parameters and, unless stated otherwise, samples requiring preservation shall be preserved at the time of collection.

(c) Additional information on sampling for pesticide and herbicide analysis may be found in the following publications:

1. "The Manual of Analytical Methods for the Analysis of Pesticide Residues in Human and Environmental Samples", USEPA, Health Effects Research Laboratory, Research Triangle Park, N.C. 27711, 1979 (hereinafter referred to as PEST);
2. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", USEPA, EMSL, Cincinnati, Ohio 45268, EPA 600/4-79-019, 1979 (hereinafter referred to as EPAQC); and
3. Standard Methods, 14th Edition.

(d) The following chain of custody procedures shall be employed in collecting and handling samples.

1. Decontaminated containers shall be used for sampling.
2. Tie-on or affixed labels with an identification number shall be used for labeling all samples.

3. Immediately after the container has been decontaminated, the containers shall be labeled with the appropriate label. Such labels shall remain affixed to the container until the time of sampling.
4. When collecting samples, if it is necessary to remove the labels only one container at a time shall have a label removed. After the sample has been collected, the appropriate information as to identity of the sample shall be written on the label and the label shall be reattached, if necessary, before removing the label from any other container.
5. After collecting the sample and, if necessary, reattaching the label, the label shall remain affixed to the sample container and shall not be removed until the required analyses have been completed and the surplus sample has been discarded.
6. Immediately upon delivery of the sample to the laboratory, the sample collector shall complete the appropriate section of a chain of custody form.
7. The chain-of-custody form shall list at a minimum the following information:
 - i. Sample number;
 - ii. Number of containers;
 - iii. Description of samples;
 - iv. Specific location of sample collection;
 - v. Identity of person collecting the sample;
 - vi. Date and time of sample collection;
 - vii. Date and time of custody transfer to laboratory;
 - viii. Identity of person accepting custody;
 - ix. Date and time of initiation of analysis;
 - x. Identity of person(s) performing analysis; and
 - xi. Name of laboratory performing the analysis;
8. Prior to accepting custody of the sample, the laboratory personnel who will accept custody shall be reasonably assured that the sample has met the preservation requirements. If the sample fails to meet those requirements, the chain of custody form and final laboratory report shall so indicate and the sample shall be refused.

9. The laboratory personnel accepting responsibility for the sample as well as all other laboratory personnel performing analysis on that sample shall sign the chain of custody form.
10. When it is necessary to send drinking water samples by mail, bus, courier service, or private shipping, the chain of custody form shall be completed by the sampler prior to the shipping of the sample and shall accompany the sample during shipping. Upon receipt of the sample in the laboratory, steps (j)7 thru 9 above shall be followed.

7:18-4.5 Methodology

(a) Analytical methods for drinking water parameters

1. Laboratories shall use the test procedures specified in 40 CFR 141 in the analysis of primary drinking water parameters.
2. Test procedures set out in the following Table II shall be utilized for the analysis of secondary drinking water parameters.

TABLE II - METHODOLOGY FOR SECONDARY DRINKING WATER PARAMETERS

Parameter	Method Description	Method		
		S.M. 14th Ed. (1)	EPA (1979)(2)	ASTM (3)
Chloride	Potentiometric	306		
	Mercuric Nitrate	304	325.3	
	Silver Nitrate	303		
Color	Platinum-Cobalt	64-66	110.2	
Copper	Direct Aspiration (A.A)	144-147	220.1-1	
	Graphite Furnace		220.2-1	
	Neocuproine	196		
ABS/LAS	Methylene Blue (Foaming Agents)	600	425.1	
Iron	Direct Aspiration (A.A)	144-147	236.1-1	
	Graphite Furnace		236.2-1	
	Phenanthroline	208		
Manganese	Direct Aspiration (A.A.)	144-147	243.1-1	
	Persulfate	225		
	Graphite Furnace Periodate		243.2-1	
		227		
Odor	Consistent Series Method	75-82	140.1-1	
pH	Glass Electrode Method	460-465	150.1-1	D129378 A or B
Sulfate	Turbidimetric Method	496-498	375.4	
	Gravimetric	493	375.3	
Total Dis. Solids	Dried at 180 degrees C	92	160.1-1	
Zinc	Direct Aspiration (A.A)	144-147	289.1	
	Graphite Furnace		289.2	
	Dithizone	263-265		
Hardness	EDTA Test	202	130.2	D1126-67
	Automated Colorimetric		130.1	
Sodium	Direct Aspiration (A.A.)		273.1	D1428-64
	Flame Photometric	250		
	Graphite Furnace		273.2	

REFERENCES

- (1) "Standard Methods for the Examination of Water and Wastewater", American Public Health Association, 14th Edition.
- (2) "Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1979.
- (3) "Annual Book of ASTM Standards", American Society for Testing Materials, Part 31.

- (b) Test procedures identified in 40 CFR 136 shall be utilized for the analysis of National Pollutant Discharge Elimination System compliance monitoring parameters.
- (c) All procedures other than those set forth in subsections (a) and (b) are considered alternative analytical methods as described in 40 CFR 141.27 and 40 CFR 136. Laboratories shall make special application to the Commissioner for the use of alternative analytical methods and such application shall include a showing of acceptable comparability data.
- (d) All laboratories which have previously been granted approval to use an alternate analytical method by the USEPA shall be allowed to continue using such method after it submits written proof of the approval to the Department.

7:18-4.6 General Laboratory Practices

- (a) Laboratories utilizing visual comparison devices shall calibrate the standards incorporated into such devices at least once every four months. The laboratory shall make and maintain records of the date and method of each such calibration. Directions for preparing temporary and permanent type visual standards are specified in the applicable sections of Standard Methods, 14th Edition. By comparing standards of known concentrations to the sealed, permanent visual standard and plotting the comparison on graph paper, a correction factor shall be derived, documented, and applied to all future results.
- (b) Distilled and deionized water shall have at a minimum, resistivity values between 0.5 to 2.0 megohms-cm (2.0 to 0.5 micromhos/cm.) at 25°C. Preferably, distilled and deionized water should have resistivity values greater than 1.0 megohms-cm (less than 1.0 micromhos/cm) at 25°C. When purchasing distilled or deionized water, laboratories should request a list of quality specifications for the water purchased. Containers of distilled or deionized water should be capped when not in use and should be capped immediately after each use.
- (c) Analytical reagent grade (AR) chemicals should be used for most analyses. Detailed information on reagent grades is set forth in Standard Methods, 14th edition, section 102, pages 5-8. Individual analytical procedures in Standard Methods 14th Edition, and the EPA's "Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Office of Technology Transfer, Washington D.C. 20460, 1979, specify requirements for the reagents to be used. In addition, laboratory chemicals and reagents shall meet the following requirements:

1. Stock and working standard solutions shall be checked regularly for signs of decomposition, including but not limited to discoloration, formation of precipitates, and concentration change due to evaporation;
 2. All solutions shall be properly labeled with identification of the compound, concentration, solvent, date, and analyst who prepared the solution;
 3. All standards used for atomic absorption analyses shall be of spectroquality;
 4. All chemicals, solutions, and standards, shall be dated upon receipt by the laboratory; and
 5. Compressed gases used for atomic absorption analyses may be of commercial grade; and
 6. Special purity solvents and reagents may be required for specific organic analysis.
- (d) All glassware should be washed in a warm detergent solution and thoroughly rinsed first in tap water and then in distilled water. This cleaning procedure is sufficient for most analytical needs, but the individual methods should be referred to for more elaborate precautions to be taken against contamination of glassware. It has been found advantageous to maintain a separate set of glassware, suitably prepared, for the nitrate, mercury, phosphate, and lead methods due to the potential for contamination from the laboratory environment. All glassware used in pesticide and herbicide analysis should be cleaned and stored as outlined in section 3A of "The Manual of Analytical Methods for the Analysis of Pesticide Residues in Human and Environmental Samples", USEPA, Health Effects Research Laboratory, Research Triangle Park, N.C. 27711, 1979.

7:18-4.7 Quality control program

- (a) All quality control data and records required by this section shall be retained by the laboratory and shall be made available for inspection by the Office of Quality Assurance. Such retained data shall include, but shall not be limited to, the results of and the raw data generated by Proficiency Sample analyses.

- (b) Each laboratory shall develop a detailed written description of the laboratory's current quality control program, and such written description shall include, but need not be limited to, the following:
1. Procedures which the laboratory will use in meeting the quality control requirements of N.J.A.C. 7:18-4.7 pertaining to laboratory equipment and instrumentation, and the frequency with which such procedures will be performed.
 2. Procedures which the laboratory will use in meeting the quality control requirements of N.J.A.C. 7:18-4.6 pertaining to general laboratory practices and the frequency with which such procedures will be performed.
 3. Procedures which the laboratory will use in meeting the quality control requirements of subsection (e) below, and the frequency with which such procedures will be performed.
- (c) Each laboratory shall develop a written laboratory procedures manual which shall set forth, in detail, the methods which the laboratory will use in chemical analyses for all parameters for which the laboratory has received certification or for which the laboratory is seeking certification, and such methods shall comply with the criteria and procedures set forth in N.J.A.C. 7:18-4.5.
- (d) Each laboratory shall record and retain all raw data and calculations derived from analyses and quality control procedures in a manner that shall provide easy verification of the data and calculations during on-site inspections.
- (e) Laboratories shall perform the following internal quality control checks:
1. Each analytical balance shall be checked and adjusted annually by a service person employed by the laboratory or by a balance consultant. The accuracy of each analytical balance shall be checked once a month using at least two class "S" weights. The weights used, weight detected to nearest 0.1 mg, dates on which checks were performed, analyst, and other pertinent information shall be recorded in a log book.

2. The wavelength setting of the spectrophotometer shall be checked yearly by comparing the wavelength setting to the absorption maxima of colored standards or filters such as didymium glass. The wavelength observed, dates on which checks were performed, analyst and other pertinent information shall be recorded in a log book.
3. Each pH meter shall be cleaned immediately after each use period and calibrated prior to usage with at least two pH buffer standards that bracket the value to be measured and records of each calibration shall be maintained.
4. Conductivity meters equipped with conductivity cells having platinum electrodes shall be checked over the range of interest using at least five concentrations of a standard potassium chloride solution. Conductivity cells not having platinum electrodes shall be checked against a conductivity meter equipped with platinum electrodes. This check shall be performed annually and the raw data, cell constant, and results shall be recorded in a log book.
5. A daily record of the temperature of the drying oven shall be maintained for each day on which the drying oven is in use.
6. The temperature of each refrigerator and each incubator shall be either recorded continuously or recorded daily from in-place thermometers immersed in liquid and placed on one of the shelves being used.
7. The accuracy of all thermometers used to monitor temperatures shall be verified by comparing the readings of such thermometers with the readings of a certified thermometer. A record shall be made containing the identification number of each thermometer, the temperatures displayed on the certified thermometer and the thermometer being verified, correction factors when applicable, dates on which quality control checks were performed, and the name of the analyst performing such checks. Glass thermometers shall be verified yearly and metal thermometers shall be verified quarterly.

8. Standard curves used in analysis of parameters in the Limited Chemistry and Atomic Absorption categories shall be prepared as follows:
 - i. Standard curves consisting at a minimum of one reagent blank and five standards shall be prepared with each analysis; or
 - ii. A standard curve consisting at a minimum of a reagent blank and five standards shall be prepared and shall be used with each subsequent analysis provided the standard curve is verified by using at least one reagent blank and one standard at or near the maximum contaminant level (MCL) in the case of analyses under the Safe Drinking Water Program, or in the case of analyses under the NJPDES program at the concentration levels normally encountered in such analyses. A new standard curve should be prepared on a daily basis or whenever a new reagent is prepared, and shall be prepared on at least a quarterly basis. Such verifications shall be considered satisfactory if, and only if, the results are within ± 10 per cent of the original curve. All data used in drawing the curve shall be so indicated on the curve, and a record shall be made of the verification containing the dates on which such verifications were performed, the results of the verification, and the name of the analyst who performed the check.
9. Laboratories which analyze 20 or more samples per day shall verify the working standard curve by running an additional standard at or near the MCL, in the case of analyses under the Safe Drinking Water Program, or, for analyses under the NJPDES program at the concentration level normally encountered in such analyses. The frequency of such analyses shall be one verification analysis after the analysis of each set of 20 samples. Such checks shall be satisfactory only if the results are within ± 10 percent of the original documented reagent curve.
10. In all cases where possible, replicate sample analyses shall be conducted for parameters in the Limited Chemistry and Atomic Absorption categories to verify the precision of the method and such verification shall be performed at one of the two following frequencies:

- i. Laboratories which analyze twenty or more samples per month of any one parameter shall verify the precision of such analyses on at least 5 percent of the samples analyzed and shall document the result, the dates on which such verification analyses were performed, the method of verification, and the name of the analyst performing such verifications; or
 - ii. Laboratories which analyze an average of less than twenty samples per month of any one parameter shall verify the precision of the analysis once a month, and shall make a record of such verification in accordance with subparagraph i above.
11. In all cases where possible, spiked sample analyses shall be conducted to verify the accuracy of the method at the same frequency as set forth in paragraph 11 of this subsection for the applicable parameters in the Limited Chemistry and Atomic Absorption categories. Documentation shall be made in accordance with the requirements of that paragraph.
12. In all cases where possible, standard deviations shall be calculated and documented for all applicable measurements being conducted in the Limited Chemistry and Atomic Absorption categories and such calculations and documentation shall be done in accordance with the following criteria and procedures:
 - i. Standard deviations shall be calculated for control samples which have been prepared at the maximum contaminant level for the parameter of interest in the case of drinking water parameters, or at the concentration level normally encountered in the analysis for all other parameters;
 - ii. Once the standard curve has been prepared or verified, the control sample shall be analyzed;
 - iii. After 20 such determinations have been obtained, using one control sample per run, the standard deviation shall be calculated;

- iv. Standard deviations shall be obtained for all parameters;
 - v. The theoretical value, mean value, and the range of acceptable values derived from two standard deviations, shall be recorded; and
 - vi. Standard deviations shall be documented in either tabular form or, preferably on control charts.
13. Spiked reference materials (SPRM's) shall be analyzed for all organic methodologies requiring the use of a gas chromatograph at the following frequency:
- i. For laboratories ten or less samples per day, one SPRM shall be analyzed during that time of analysis and documented; or
 - ii. For laboratories analyzing more than ten samples per day, each 10th sample shall be a SPRM.

Information pertaining to SPRM may be found in section 3 of "The Manual of Analytical Methods for the Analysis of Pesticide Residues in Human and Environmental Samples", USEPA, Health Effects Laboratory, Research Triangle Park, N.C. 27711, 1979.

14. A record shall be maintained for each gas chromatograph and shall contain the following information:
 - i. Date of installation and serial number of each detector installed;
 - ii. Background current (BGC) profiles obtained at the time of installation of each detector and subsequent profiles (column identity notations should be made); and
 - iii. Date of change of pyrometer batteries, if used.

15. A record shall be maintained on each gas chromatographic column used and shall contain the following information:
 - i. Column identification number;
 - ii. Date of packing or purchase;
 - iii. Liquid phase identity and lot number of precoated column packing;
 - iv. Conditioning temperature, flow rate and number of hours;
 - v. Length and shape of column.
 - vi. Background current on newly installed column and subsequent background current profiles during the life of the column;
 - vii. Date of each silylation of column; and
 - viii. When applicable, compound conversion data; with dates monitored, and percentage of compound breakdown.

16. A record shall be maintained on the preparation of pesticide and herbicide standards and shall include, but not be limited to, the following information:
 - i. The identification number of the concentrated stock standard solution, date of preparation, chemist who prepared the solution, all chemical compounds in the solution, lot number, purity, gross weight, tare weight, net weight, adjusted net weight (corrected for purity of primary standard), dilution volume and concentrations in ng/ul;

- ii. The identification number of the intermediate concentration standard solution, date of preparation, chemist who prepared the solution, all chemical compounds in the solution, identification number of the concentrated stock, strength of concentrated stock in ng/ul, aliquot of concentrated stock, dilution volume, and final concentration in ng/ul; and
 - iii. The identification number of the working standard solution, date of preparation, chemist who prepared the solution, all chemical compounds in the solution, identification number of the intermediate concentration standards, concentration of intermediate standards, aliquot volumes, dilution volumes, and final concentrations in pg/ul.
 - iv. Additional information on preparation of standards may be found in "The Manual of Analytical Methods for the Analysis of Pesticide Residues in Human and Environmental Samples", USEPA, Health Effects Research Laboratory, Research Triangle Park, N.C. 27711, 1979, pp 59-67.
17. All quality control procedures cited in the gas chromatography methodologies shall be performed and documented.
18. All reagents and solutions shall be labelled to indicate identity and, when applicable, titer, strength, or concentration, recommended storage requirements, preparation or expiration date, and any other pertinent information. Materials of substandard reactivity and deteriorated materials shall not be used. All outdated material shall be discarded immediately.
19. There shall be available at all times, in the immediate bench area of personnel engaged in the examination of samples and related procedures within the chemical category, the most current laboratory manuals or other complete written descriptions and instructions relating to:
- i. The analytical methods to be used by such personnel, properly designated and dated to reflect the most recent supervisory reviews;

- ii. Pertinent current literature in the field for use as reference materials, including the appropriate Federal regulations; and
 - iii. Textbooks may be used to supplement such written descriptions, but may not be used in lieu thereof.
20. Only the laboratory manager or supervisor shall make changes in laboratory procedures and those changes shall only be effective when put in writing.

7:18-4.8 Records and data reporting

- (a) Records of chemical analyses, including but not limited to all raw data, calculations, quality control data, and laboratory reports, shall be kept by the laboratory for at least five years.
- (b) The following information shall be retained by the laboratory as part of the records of chemical analysis and the records of chain custody:
 - 1. The laboratory number or other form of identification of the sample;
 - 2. The date, time, specific site of sampling, and the name of the person who collected the sample or the laboratory which submitted the sample;
 - 3. In the case of drinking water samples, designation of whether the sample is a routine distribution system sample, check sample, raw or process water sample, or other applicable designation;
 - 4. The date and time when the laboratory received the sample, whether the sample was received preserved or unpreserved, and if the sample was received unpreserved, the method of preservation shall be recorded;
 - 5. The date and time of analysis of the sample;
 - 6. The person or persons who performed the analysis;
 - 7. The type of analysis performed and the analytical method employed;
 - 8. The results of the analysis and the raw data generated by the analysis; and

9. The name and address of the laboratory to which the sample was forwarded, if the analysis was not performed at the laboratory which first received the sample.
- (c) The sample collector shall complete a chain of custody form immediately after each sample is taken.
 - (d) A copy of the chain of custody form shall be attached to the sample report form and the results of each analysis shall be calculated and entered on the sample report form which is to be forwarded to the person requesting the analysis of the sample. A careful check shall be made to assure that each result entered on the sample report form is the same as the result entered on the bench sheet and once the check is completed the analyst shall initial the bench sheet.
 - (e) The original or true duplicate of the results of the test or analysis shall be sent promptly to the person who requested such tests or analysis, and shall be signed by the laboratory manager. In the case of tests or analyses performed pursuant to the Safe Drinking Water Act Regulations for Public Noncommunity Water Systems, the results of such tests or analyses shall be reported to the owner of the system on computer input forms which will be provided by the Department.
 - (f) Whenever a laboratory refers samples to another laboratory, the person ordering the examination shall receive the original laboratory report or a true duplicate of that report on the form of the laboratory that actually performed the test or analysis. In the case of tests or analyses performed under the Safe Drinking Water Act Regulations for Public Noncommunity Water Systems, when use of a specific laboratory report form is required by the Department, the laboratory performing the tests shall report the results on such required forms.

SUBCHAPTER 5. CRITERIA AND PROCEDURES FOR RADIOLOGICAL TESTING AND ANALYSIS

7:18-5.1 Scope

This chapter establishes the Department's requirements which a certified laboratory or a laboratory seeking certification shall continually meet and follow when performing radiological analyses.

7:18-5.2 Laboratory facilities

(a) Laboratory facilities shall meet the following minimum requirements:

1. The counting instruments required for measurement of those activities or specific radionuclides described in 40 CFR 141 shall be located in a room other than the one in which samples and standards are prepared or in which other types of chemical analyses are being performed. The temperature of the room in which counting instruments are located shall not exceed 27°C. Temperature variation under normal operating conditions shall not exceed 3°C.
2. All instruments shall be properly grounded, and a regulated power supply, either external or internal, shall be available for use with each instrument.
3. In areas in which radioactive standards are being prepared, care shall be taken to minimize contamination of surfaces and personnel. Either bench surfaces of an impervious material covered with absorbent paper, or trays constructed of stainless steel, plastic, or fiberglass and lined with absorbent paper may be used.
4. Laboratory space and facilities shall be adequate to properly carry out the services performed in, or offered by, the laboratory. There should be at least 100 to 150 square feet of floor space per analyst. This space should contain no less than 15 linear ft. of bench space and the following equipment:
 - i. A sink with hot and cold running water;
 - ii. Electrical outlets with a carrying capacity at 120 V a.c. and such outlets shall be grounded;
 - iii. A source of distilled or deionized water;

- iv. A supply of natural gas or liquefied petroleum, or a propane cylinder with proper attachments in the case of laboratories performing limited amounts of analytical work;
- v. A vacuum line, pump, or aspirator; and
- vi. An exhaust hood.

7:18-5.3 Specifications for laboratory equipment and instruments

(a) Laboratories performing radiological tests and analyses shall have on the premises and under the control of the laboratory manager the equipment and instruments listed in this section necessary for the preparation and analysis of the specific standards and samples for which the laboratory is seeking certification or is certified. Such instruments, when required, shall meet the following specifications:

1. The following are specifications for general instrumentation and equipment:
 - i. The analytical balance shall have a precision of ± 0.5 mg, and minimum scale readability of 0.1 mg;
 - ii. The pH meter shall have an accuracy of ± 0.1 units;
 - iii. The specific ion meter shall have an expanded scale millivolt capability, and shall be readable to 0.1 mv and accurate to ± 0.5 mv;
 - iv. The conductivity meter shall be readable in ohms or mhos, shall have a range of 2 to 2.5 million ohms-cm or micromhos/cm ± 1 percent, and shall have a sensitivity of 0.33 percent or better;
 - v. A gravity convection drying oven which shall be capable of maintaining stable temperatures;
 - vi. Either glass or plastic dessicators may be used as appropriate for the particular task being performed;
 - vii. Either a large or small hot plate may be used however hot plates shall have temperature controls;
 - viii. Laboratory glassware shall be constructed of borosilicate glass, and all volumetric glassware shall be marked Class A, denoting that it

meets Federal specifications, thereby eliminating the need for calibration prior to use;

- ix. The muffle furnace shall be automatically controlled and shall have a chamber capacity of at least 2,200 cc (10x9.5x23) and a maximum operating temperature of 1,000°C continuous and 1,100°C intermittent;
- x. A general purpose table-top centrifuge which shall have a maximum speed of at least 3,000 rpm and a loading option of 4 x 50 mL; and
- xi. The fluorometer shall be capable of detecting 0.0005 ug of uranium.

(b) The types of radiation counting systems needed to comply with measurements described in 40 CFR 141 are set forth below. Laboratories shall have on the premises and under the control of the laboratory manager those instruments needed to analyze for those activities or specific radionuclides for which the laboratory is seeking certification or for which the laboratory is certified. Such instruments shall meet the following specifications:

1. A liquid scintillation system is required if the laboratory is to be certified for measurement of tritium in drinking water samples. The system shall be such that the sensitivity shall meet or exceed the requirements of 40 CFR 141.25.
2. A gas-flow proportional counting system shall be used for the measurement of gross alpha and gross beta activities, radium-226, radium-228, strontium-89, strontium-90, cesium-134, and iodine-131 as described in the reference cited in 40 CFR 141.25 (a). The detector shall be either a "windowless" internal proportional counter or a "thin window" type. A minimum shielding equivalent to 5 cm of lead shall surround the detector. A cosmic (guard) detector should be operated in anticoincidence with the main detector. The gas-flow proportional counting system shall be such that the sensitivity of the radiological analysis of the water sample will meet or exceed the requirements of 40 CFR 141.25.
3. For measurement of gross activities and radium-226, a scintillation system designed for alpha counting may be substituted for the gas-flow proportional counter described in paragraph 2 above. When a scintillation system is used for counting, a Mylar

disc coated with a phosphor (silver-activated zinc sulfide) shall be placed either directly on the sample or on the face of a photomultiplier tube, and the disc and sample or tube shall then be enclosed within a light-tight container along with the appropriate electronics, including but not limited to a high voltage supply, amplifier, timer, and scaler.

4. For the specific measurement of radium-226 by the radon emanation method, a scintillation system designed to accept scintillation flasks ("Lucas cells") shall be used. This type of scintillation system consists of a light-tight enclosure capable of accepting the scintillation flasks, a detector (phototube), and the appropriate electronics which includes but is not limited to a high voltage supply, amplifier, timers, and scalars. The flasks (cells) required for this measurement shall be either purchased from commercial suppliers or shall be constructed by laboratory personnel in accordance with the specifications set forth in Lucas, H.F., "Improved Low-Level Alpha Scintillation Counter for Radon". Rev. Sci. Instrum., 28:680, 1967).
5. Gamma spectrometer systems shall have either a sodium iodide (NaI(Tl)) crystal detector or a solid state lithium drifted germanium (Ge(Li)) detector connected to a multichannel analyzer if the gamma spectrometer system is to be used for analyses of manmade photon emitters, and the detector shall meet the criteria and specifications set forth in either subparagraph i or subparagraph ii below:
 - i. If a sodium iodide detector is used, such detector shall meet the following criteria and specifications:
 - (1) A 10 cm x 10 cm NaI cylinder crystal should be used, but, at a minimum, a 7.5 cm x 7.5 cm crystal shall be used;
 - (2) The detector shall be shielded with at least 10 cm of iron or the equivalent thereof;
 - (3) The distance from the center of the detector to other part of the shield should be at least 30 cm;

ii. A system with a lithium drifted germanium (Ge(Li)) detector may be used for measurement of manmade photor emmitters provided the following requirements are met:

(1) The efficiency of the detector shall be such that the sensitivity of the gamma spectrometry system meets the minimum detectable activity requirements cited in 40 CFR 141.25;

(2) The detector shall be shielded with at least 10 cm of iron or the equivalent thereof; and

iii. The multichannel analyzer, in addition to appropriate electronics, shall have a memory of at least 200 channels for NaI and 2000 channels for Ge(Li) and shall have at least one readout device;

7:18-5.4 Preservation of samples, methodology, and major instrumentation

(a) Table III below gives the minimum requirements for sample handling including preservation, methodology, and major instrumentation.

Table III. - Sample handling, preservation, methodology¹ and major instrumentation (minimum requirements)

<u>Parameter</u>	<u>Preservative</u> ²	<u>Container</u> ³	<u>Instrumentation</u> ⁴
Gross alpha	Conc. HCl or HNO ₃ to pH<2 ⁵	P or G	A or B
Gross beta	Conc. HCl or HNO ₃ to pH<2 ⁵	P or G	A
Strontium-89	Conc. HCl or HNO ₃ to pH<2	P or G	A
Strontium-90	Conc. HCl or HNO ₃ to pH<2	P or G	A
Radium-226	Conc. HCl or HNO ₃ to pH<2	P or G	A, B or D
Radium-228	Conc. HCl or HNO ₃ to pH<2	P or G	A
Cesium-134	Conc. HCl to pH<2	P or G	A or C
Iodine-131	None	P or G	A
Tritium	None	G	E
Uranium	Conc. HCl or HNO ₃ to pH<2	P or G	F
Photon emitters	Conc. HCl or HNO ₃ to pH<2	P or G	C

¹40 CFR 141.

²It is recommended that the preservative be added to the sample at the time of collection unless suspended solids activity is to be measured. However, if the sample must be shipped to a laboratory or storage area, acidification of the sample (in its original container) may be delayed for a period not to exceed 5 days. A minimum of 16 hours must elapse between acidification and analysis.

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

⁴A = Low background proportional system; B = Alpha scintillation system;

C = Gamma spectrometer (Na(Tl) or Ge(Li)); D = Scintillation cell (radon) system; E = Liquid scintillation system (section C.2.a); F = Fluorometer (section C.1.i).

⁵If HCl is used to acidify samples which are to be analyzed for gross alpha or gross beta activities, the acid salts must be converted to nitrate salts before transfer of the samples to planchets.

7:18-5.5 Methodology

- (a) Laboratories shall use the analytical procedures specified in 40 CFR 141.
- (b) All procedures other than those set forth in subsection (a) of this section are considered alternative analytical methods as described in 40 CFR 141.27. Laboratories shall make special application to the Commissioner for the use of alternative analytical methods and such application shall include a showing of acceptable comparability data.

7:18-5.6 General laboratory practices

- (a) Laboratory practices shall meet the following requirements:
 1. All glassware shall be washed in a warm detergent solution and shall be thoroughly rinsed in tap water. A distilled or deionized water rinse shall follow the tap water rinse. When necessary for proper performance of specific analytical methods, more detailed procedures for ensuring cleanliness of glassware shall be employed.
 2. All water used in preparation of reagents, standards, and samples shall have resistance values that are between 0.5 to 2.0 megohms-cm (2.0 to 0.5 micromhos/cm) at 25°C. If such high quality water is not available in the laboratory, it shall be purchased from commercial suppliers; the laboratory shall request a list of quality specifications for water purchased from the supplier and the laboratory shall periodically check the actual quality of the purchased water against these specifications.
 3. Analytical reagent grade (AR) chemicals shall be used for all analyses, unless otherwise required for an individual analytical procedure.
 4. Radioactive standards and radioactive wastes shall be stored in an enclosed and properly labeled area, either within the analytical laboratory or in a separate room or facility. Standards, samples, and radioactive wastes shall be safely stored in suitable containers.
 5. Standards and samples shall be prepared in an area of the laboratory specifically designated for and exclusively used for the preparation of radioactive standards and samples. Adequate precautions shall be taken in this area to ensure against radiocative contamination. Provisions shall be made for safe storage and disposal of radioactive wastes and for monitoring of the work area for radioactivity.

6. All reagents and solutions shall be labeled with pertinent information. Materials of substandard reactivity and deteriorated materials shall not be used.

7:18-5.7 Quality control

- (a) Laboratories shall develop and implement quality control procedures meeting the following minimum requirements:

1. Each laboratory shall develop and have on file and available for inspection a written description of the current laboratory quality control program. Such quality control program shall cover all types of tests and analyses performed by the laboratory and shall provide for verification and assessment of the accuracy, measurement of precision, and detection of error. Management, supervisors, and analysts should participate in the quality control program. Each participant within the laboratory should have a copy of the quality control program and detailed guidelines for implementation of the participant's responsibility.
2. Quality control data and records shall be available for inspection.
3. There shall be available at all times, current laboratory manuals or other complete written descriptions and instructions relating to:
 - i. The analytical methods to be used by those personnel, properly designated and dated to reflect the most recent supervisory reviews;
 - ii. Operating manuals and calibration protocols for counting instruments shall be available to analysts and technicians;
 - iii. Such manuals, written descriptions, protocols, and instructions may be supplemented by, but not replaced by, textbooks relating to the particular analytical methods and procedures employed by such personnel.
4. Permanent records shall be maintained of preventive maintenance, periodic inspection, testing, and calibration for the proper operation of radiation instruments and analytical balances; validation of methods; evaluation of reagents and volumetric equipment; surveillance of results; and remedial actions taken in response to detected defects. Such records shall be kept on file by the laboratory for a period of at least five years.

5. The following minimum daily quality control measures shall be performed by the laboratory:
 - i. To verify internal laboratory precision duplicate analyses equal to ten percent of sample analyses shall be performed. The differences between duplicate measurements shall be less than twice the standard deviation of the specific analysis as described in "Environmental Radioactivity Laboratory Intercomparison Studies Program" (EPA-600/4-77-001). If the differences exceed two standard deviations, the prior measurements shall be considered to be suspect, all calculations and procedures for that day shall be examined, and all samples shall be reanalyzed when necessary.
 - ii. Laboratories performing 20 or more specific analyses each day, shall measure at least one calibration standard and at least one background sample along with each group of 20 samples.
 - iii. Laboratories performing less than 20 specific analyses in any one day, shall measure one calibration standard and one background sample along with the samples measured on that day.
 - iv. Quality control performance charts, performance records, and raw data used in the verification procedure set forth in this paragraph shall be maintained for a period of at least five years.
6. Balances shall be checked periodically using Class "S" weights; and laboratories shall have current service contracts in effect for balances.
7. Laboratories shall have an electronics technician in their employ or shall have current factory servicing contracts for repair of laboratory instruments.
8. Only the laboratory manager or supervisor shall make changes in laboratory procedures and those changes shall only be effective when put in writing.

7:18-5.8 Records and data reporting

- (a) The laboratory shall maintain records which are adequate and appropriate for the services offered.
- (b) Work records of quantitative tests shall be maintained for at least five years and shall indicate final results

together with all corresponding instrument readings and calculations. Where instrumentation produces tracings or printouts, such tracings or printouts may serve as the work record.

- (c) A record shall be maintained for at least five years of the daily receipt of samples. Each such record shall be numbered or otherwise appropriately identified and shall contain the following information:
1. The laboratory number or other identification of the sample.
 2. Identification of the sample source, water system, discharger, population served, and Public Water Supply Identification number or permit number.
 3. The name of the person or laboratory which submitted the sample.
 4. The date, time, and specific location of sample collection.
 5. The date and time of receipt of the sample by the laboratory.
 6. The type of test or analysis performed, method of testing or analysis, and date of analysis.
 7. The results of the test or analysis, including raw data, and the name of the analyst or analysts.
 8. The name and address of the laboratory to which the sample was forwarded, if the testing or analysis was not performed by the laboratory which initially received the sample.
- (d) The original or a true duplicate of the results of the tests or analyses shall be sent promptly to the person who requested such tests or analyses, and shall be signed by the laboratory manager.
- (e) Whenever a certified laboratory refers samples to another certified laboratory for analysis, the person requesting the analyses or tests shall receive the laboratory report or a true duplicate of that report on the form of the laboratory that performs the tests or analyses. In the case of tests or analyses performed under the Safe Drinking Water Act Regulations for Public Noncommunity Water Systems, where use of a specific laboratory report form is required, the laboratory performing the tests shall report the results on such required form.

- (f) Laboratories shall follow the chain of custody procedures set forth in N.J.A.C. 7:18-4.4(d), 4.8(c) and 4.8(d).
- (g) Records of radiological analyses shall be kept by the laboratory for not less than five years. This includes but is not limited to all raw data, calculations, quality control data, and reports. In addition, actual laboratory reports shall be kept for not less than five years. However, all data, with the exception of compliance check samples as detailed in 40 CFR 141.33(b) may be transferred to tabular summaries provided that the following information is included:
1. The date, specific place, and time of sampling.
 2. The name of the person who collected the sample.
 3. Identification of sample as a routine distribution sample, check sample, raw or process water sample, or other special purpose sample.
 4. The date of receipt of the sample by the laboratory and the date of analysis.
 5. The name of the laboratory and the person or persons who performed the analysis.
 6. The analytical technique or method used.
 7. The results of the test or analysis, including the raw data generated during the test or analysis.

SUBCHAPTER 6. CRITERIA AND PROCEDURES FOR
BIOASSAY TESTING AND ANALYSIS

7:18-6.1 Scope

This subchapter establishes the Department's requirements which a certified laboratory or a laboratory seeking certification shall continually meet and follow when performing bioassay analysis.

7:18-6.2 Definitions

For the purpose of this Subchapter, the following definitions in addition to those found in N.J.A.C. 7:18-1.7 are applicable.

"Acclimation" means an organism's physiological adjustment to environmental changes including, but not limited to, changes in temperature and salinity.

"Act" means the New Jersey Water Pollution Control Act, N.J.S.A. 58:10A-1 et seq.

"Acute toxicity" means causing death or severe damage to an organism by poisoning during a brief exposure period, normally 96 hours or less.

"Bioassay" means a determination of the concentration or dose of a given material necessary to affect a test organism under stated conditions.

"Biomonitoring" means all testing methods which utilize a biological system or any of its parts for assessing the presence or effects of one or more pollutants and/or environmental factors, either alone or in combination. For purposes of this subchapter, biomonitoring refers to acute toxicity bioassays.

"Composite sample" means a sample composed of several discrete samples collected at equal time intervals, or collected proportionally to the flow rate of the discharge, over the compositing period.

"Definitive test" means a full-scale bioassay consisting of a minimum of five different concentrations of effluent in a logarithmic series with each concentration and control being tested against a minimum of 20 organisms of a species designated by the Department.

"Dilution water" means the unpolluted water of desired quality to be used for preparing the different test concentrations of the effluent. Dilution water is usually collected from a point as close as possible, but not within, the mixing zone influenced by the effluent.

"Discharge" means the releasing, spilling, leaking, pumping, pouring, emitting, emptying, or dumping of a pollutant into the waters of the State or onto land or into wells from which the

pollutant might flow or drain into said waters, and shall include the release of any pollutant into a municipal treatment works.

"Effluent" means an outflow from a point source.

"Flow-through bioassay" or "Continuous flow-through bioassay" means a bioassay test technique which permits test solutions to flow into and out of the test chambers on a once through basis for the duration of the test.

"Grab sample" means an individual sample collected over a time period of less than 15 minutes.

"Incipient lethal level" or "incipient LC₅₀" means the concentration at which acute toxicity ceases, that is, the concentration at which 50 percent of the test organism's population can live for an indefinite time.

"LC₅₀" means the concentration of a toxicant which is lethal to 50 percent of the organisms of a particular species under a given set of conditions in a specified length of time (i.e., 24, 48, or 96 hours).

"Mixing zone" means a localized area of surface waters, as may be designated by the department, into which wastewater effluents may be discharged for the purpose of mixing, dispersing, or dissipating such effluents without creating nuisances or hazardous conditions in compliance with the Surface Water Quality Standards, N.J.A.C. 7-9-4.1 et seq.

"Methods for Measuring Acute Toxicity - EPA" means "Methods for Measuring Acute Toxicity of Effluents to Aquatic Organisms," U.S.E.P.A., Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, EPA-600/4-78-012.

"Modified static daily-renewal bioassay" means a test in which the aquatic organisms are exposed to a test solution which is changed once every 24 hours for the duration of the test period. Fresh samples of the effluent are obtained daily, fresh concentrations of test solution are prepared, and the test organisms are transferred daily to the new test solution.

"Permit" a NJDEP permit issued pursuant to Section 6 of the Act.

"Point source" means any discernable, confined, and discrete conveyance from a mobile or stationary source, including, but not limited to, any pipe, ditch, channel, tunnel, conduit, well, discrete fissure, container, rolling stock, concentrated animal feeding operation, vessel or other floating craft, from which pollutants are or may be discharged.

"Pollutant" means any dredged spoil, solid waste, incinerator residue, filter backwash, sewage, garbage, refuse, oil, grease, sewage sludge, munition, chemical wastes, biological materials,

radioactive materials, thermal waste, wrecked or discarded equipment, and construction waste or runoff or other residue discharged to the land, ground waters or surface waters of the state.

"Range-finding bioassay", "screening bioassay", or "exploratory bioassay" means a short-term (i.e., 8-24 hours) flow-through or, more often, static bioassay used for determining the approximate concentrations, above and below the LC50, to be used in the definitive test when the toxicity of a waste is unknown. In this test, groups of five test organisms are exposed to three to five widely-spaced effluent dilutions, usually in a logarithmic ratio, such as 0.01, 0.1, 1.0, 10 and 100 percent.

"Salinity" means the total amount of dissolved salts in sea water in parts per thousand (0/00 or ppt) by weight when all the carbonate has been converted to oxide, the bromide and iodine have been converted to chloride, and all organic matter has been completely oxidized.

"Sampling point" means a particular site whose location may be specified in a permit, or otherwise, and from which effluent samples are to be collected for testing and evaluation.

"Total length" means the straight line measurement from the tip of the snout of a fish to the extreme tip of the fish's caudal fin.

"U.S.E.P.A.-1972" means U.S. EPA, 1972. "Recommended bioassay procedure for flathead minnows, Pimephales promelas, Rafinesque.. chronic tests. National Water Quality Lab., Duluth, Minnesota.

"U.S. EPA-1978" means "Bioassay Procedures for the Ocean Disposal Permit Program", U.S. EPA, EPA-600/9-78-010, March 1978.

"Volume percent" is $(100 \times \text{volume of effluent})$ divided by $(\text{volume of effluent} + \text{volume of dilution water})$.

"Waters of the State" means the ocean and its estuaries, all springs, streams and bodies of surface or ground water, whether natural or artificial, within the boundaries of this state or subject to the jurisdiction.

"Year class" means fish which originate from the same annual brood or spawning.

7:18-6.3 Laboratory Facilities and Safety

- (a) Laboratory space and facilities shall be adequate to properly carry out the services performed in, or offered by the laboratory.
- (b) Workbench space within the laboratory shall be ample for the tests or analyses to be performed, and shall be

well-lighted and convenient to a sink, and such water, gas, suction, and electrical outlets as are necessary to properly carry out the specific tests or analyses performed in, or offered by, the laboratory.

- (c) The temperature and humidity within the laboratory shall be maintained within the limits required for the proper performance of each test or analysis and for the proper operation of instruments which may be affected by temperature variations.
- (d) Adequate fire precautions shall be taken, including but not limited to, having readily available a fire extinguisher rated for the types of fires that reasonably may be foreseen given the types of tests and analyses performed by the laboratory.
- (g) Appropriate occupational safety and health laws shall be posted and observed.

7:18-6.4 Laboratory equipment, supplies and materials

- (a) Laboratories performing bioassay tests and analyses shall have under the control of the laboratory manager the equipment and instruments listed in this section necessary for the analysis of samples for which the laboratory is seeking certification or is certified. Such instruments, when required, shall meet the following specifications:
 - 1. Air supply for aeration of tanks shall either be from compressors equipped with seals designed to prevent contamination of air lines from oil, or from compressed air or oxygen tanks.
 - 2. Bioassay testing systems may consist of temperature control units, pipes, valves and fittings, diluter, pumps, mixing equipment, tanks, and exposure chambers and shall meet the following requirements:
 - i. All components of the bioassay testing system shall be constructed of lead-free glass, No. 316 stainless steel, silicone sealant and tubing, unplasticized polyethylene or polypropylene, Teflon^R, Tygon^R, nylon, fiberglass or any other materials proven to be nontoxic in the test organisms;
 - ii. Tubing, connectors and screens made of materials known to absorb significant amounts of trace organic compounds shall be used once and discarded; and

- iii. All components that are reused shall withstand cleaning, without significant degradation, by the procedures cited in N.J.A.C. 7:18-6.7(e).
3. For flow through bioassays, a dilutor system is required for the accurate measuring, mixing, and delivery of sample and dilution water to the exposure chambers. A variety of methods may be used, however, the proportional dilutor is the preferred system for routine effluent toxicity tests. Detailed descriptions of dilutor systems may be found in Standard Methods - 14th Edition and Methods for Measuring Acute Toxicity - EPA. All dilutor systems shall meet the following requirements:
 - i. Dilutor system shall provide an adequate supply of dilution water to maintain a 24 hour continuous operation. This supply shall be provided by the usage of a large dilution water reservoir or by direct continuous pumping from the source of the water.
 - ii. The dilutor system shall be capable of metering the flow of dilution water and sample into a mixing chamber for the determination of concentrations. Metering of dilution water and sample may be accomplished by using a constant head box or metering pumps.
 - iii. Mixing chambers shall be used to ensure complete mixing of dilution water and sample prior to dispensing of solutions into the exposure chambers.
 - iv. Separate delivery tubes shall be used for transmission of the dilution water and sample from the mixing chambers to each of the duplicate exposure chambers.
 - v. The flow rate through the exposure chambers shall be sufficient to maintain adequate dissolved oxygen in the exposure chambers, according to N.J.A.C. 7:18-6.6(m)10ii. but no less than five water volume changes every 24 hours.
 - vi. The flow rate through the exposure chambers shall not vary by more than ± 10 percent between all exposure chambers or ± 5 percent within any given exposure chamber throughout the duration of the test.

- vii. The dilutor system shall maintain the test concentration in each exposure chamber within ± 5 percent of the starting concentration for the duration of the test.
 - viii. The dilutor system shall have heating and cooling equipment designed to maintain a constant temperature in the exposure chambers to within ± 2 C of the specified test temperature.
 - ix. If the supply of dilution water to the mixing chamber is interrupted, the dilutor system shall be designed to automatically curtail the delivery of the sample to the mixing chambers.
 - x. The exposure chambers shall have an overflow system designed to prevent the test organisms from entering the outlets.
 - xi. The dilutor system shall be capable of maintaining a minimum of five separate effluent dilutions and a control containing dilution water at any necessary flow rate, required by N.J.A.C. 7:18-6.4(a)3v. with duplicate exposure chambers.
 - xii. The dilutor system shall be capable of, but not limited to, providing concentrations at 10^{-1} and 10^{-2} of the logarithmic series of concentration, 5.6, 10.0, 18.0, 32.0, 56.0, and 100 percent effluent by volume.
4. Holding, acclimating, and culturing chambers shall meet the following requirements:
- i. Chambers shall be constructed of non-toxic materials and shall meet the requirements set forth in N.J.A.C. 7:18-6.4(a)2.
 - ii. Chambers shall have the necessary equipment for controlling water temperature and aeration.
 - iii. Chambers shall be constructed for ease of cleaning and the prevention of waste material build-up. It is recommended that either square or rectangular chambers with a standpipe drain at one end of the tank or round chambers with a stand pipe drain in the center of the tank be used. The standpipe drain should be installed so that upon removal of the standpipe, the drain opening is flush with the bottom of the chamber. The chamber bottoms should slope gently towards the drain.

- iv. The interior surface of the chambers shall be smooth to facilitate cleaning, reduce risk of injury to test organisms, and prevent accumulation of material in cracks or corners.
 - v. Chambers shall be shielded from outside disturbances. Materials used for shielding shall meet the requirements set forth in N.J.A.C. 7:18-6.4(a)2i.
5. Laboratories shall have available top-loader or pan balances which shall meet the following requirements:
- i. Balances shall be clean, not corroded, and shall be provided with appropriate weights of good quality; and
 - ii. Balances shall tare out and detect a weight of at least 100mg accurately.
6. Temperature monitoring devices shall meet the following requirements:
- i. Glass or metal thermometers shall be graduated in 0.5°C increments;
 - ii. Continuous temperature recording devices shall be sensitive and accurate within $\pm 0.5^\circ\text{C}$;
 - iii. The column of liquid in glass thermometers shall have no separation; and
 - iv. A certified thermometer or a thermometer of equivalent accuracy shall be available for use by the laboratory.
7. Air or water-jacketed incubators, incubator rooms, and water baths shall meet the following requirements:
- i. Incubators, incubator rooms, and water baths shall be of sufficient size to accommodate periods of peak work load;
 - ii. Incubators must maintain internal temperatures at the desired level to within $\pm 0.5^\circ\text{C}$;
 - iii. Whenever an air incubator is in use, a calibrated thermometer with its bulb immersed in liquid shall be placed on one of the shelves in use within the incubator; and

- iv. The temperature within an incubator shall be recorded daily, or a recording thermometer, sensitive to temperature of $\pm 0.5^{\circ}\text{C}$ shall be used and the recording tape shall be checked daily.
8. The autoclave shall meet the following requirements:
- i. It shall be in good operating condition when observed during its operational cycle or when time-temperature charts are read, and, for most efficient operation, use of a double-walled autoclave constructed of stainless steel is suggested;
 - ii. It shall have pressure and temperature gauges on the exhaust side, and shall have a safety-valve that is in good operating condition;
 - iii. It shall reach the sterilization temperature of 121°C and pressure of 1.1 lb/cm^2 (15 psi) and shall maintain that temperature and pressure throughout the sterilization cycle. The sterilization cycle shall be for 15 minutes or 15 minutes per liter of sample, whichever is longer;
 - iv. After the sample has cooled, it shall be allowed to equilibrate either in an air or carbon dioxide atmosphere so that the lost carbon dioxide is replaced.
9. The refrigerator shall be of sufficient size to accept the required sample volumes, and shall maintain an internal temperature of 1° to 4.4°C .
10. Laboratories shall have at least one low power magnification device, preferably a binocular microscope with up to 10x magnification, for working with invertebrates.
11. Laboratory glassware, plastic-ware, and metal utensils not previously specified, shall meet the following requirements:
- i. Glassware and metal utensils shall be resistant to the effects of corrosion, high temperatures, and vigorous cleaning operations;
 - ii. Volumetric containers should be Class A and need not be calibrated before use;
 - iii. Plastic items shall be of inert, nontoxic materials;

- iv. Metal utensils shall be made of stainless steel.
12. Dilution water sample containers shall meet the following requirements:
- i. Either wide-mouthed lead-free glass and stoppered containers or plasticized plastic containers with screw caps shall be used and stored so that they are protected from contamination.
 - ii. Screw caps shall have leakproof nontoxic liners.
13. Effluent sample containers shall meet the following requirements:
- i. Either wide-mouthed lead-free glass and stoppered containers, or disposable unplasticized plastic containers with screw caps shall be used.
 - (1) After each use, containers shall be cleaned in accordance with procedures set forth in N.J.A.C. 7:18-6.7(e).
 - ii. Glass-stoppered containers shall be stored to prevent contamination.
 - iii. Screw caps shall have leakproof non-toxic liners.

7:18-6.5 Sample collection, handling and preservation

(a) Dilution water samples shall be collected, handled and preserved as follows:

- 1. Dilution water shall be deemed acceptable for use in a bioassay provided healthy test organisms survive in it through the acclimation period without showing any signs of stress, including but not limited to, abnormal behavior or discoloration.
- 2. Dilution water samples shall be representative of the receiving water system which the effluent is discharged into. Samples shall be collected in the following manner:
 - i. In non-tidal waters, dilution water samples shall be collected from a location as close as possible to, but upstream of, the effluent mixing zone;

- ii. In tidal waters, dilution water samples shall be collected from a location as close as possible to, but upstream of, the effluent mixing zone. Samples shall also be collected during the outgoing tide up to and during low slack tide;
 - iii. The sampling location shall be such that the salinity shall be within ± 3 ppt., to that of the receiving water at the effluent outfall.
 - iv. When samples are collected from streams or rivers, it is recommended that an integrated sample be collected. This is a sample that is collected from bottom to top of the water column so that the sample collected is proportional to flow. If only a grab sample can be taken then it should be collected at mid-depth in midstream.
 - v. When samples are collected from reservoirs or lakes, the effects of seasonal stratification, runoff, and previous rainfall upon the chemical/physical characteristics of the water should be considered.
3. If the receiving water is influenced immediately upstream of the effluent outfall by other point sources of pollution so as to disqualify its use as dilution water, then the dilution water sample(s) shall be obtained from a location just above the other point sources.
4. If acceptable dilution water cannot be obtained from the receiving water at any location because of upstream pollution, then some other unpolluted water, meeting the following requirements, shall be used:
 - i. Another surface water or groundwater having a natural quality similar to that of the receiving water prior to its pollution may be used; or
 - ii. Reconstituted or artificial freshwater or saltwater having a natural quality similar to that of the receiving water prior to its pollution may be used;
 - iii. A substitute dilution water shall have a total hardness, total alkalinity, and specific conductance within 25 percent and a pH within 0.2 units of the receiving water prior to its pollution.

iv. Modification of a substitute dilution water or the preparation of reconstituted fresh or saltwater shall follow the procedures given in Standard Methods, 14th Edition, pp. 461 and 462 and Methods for Measuring Acute Toxicity - EPA, pp. 14-16.

5. Except for the procedures described in N.J.A.C. 7:18-6.5(a)4iv., the only other permissible treatment of the dilution water shall be filtration through screening made of a non-toxic material as specified in N.J.A.C. 7:18-6.4(a)2. This screening shall have a mesh of 2mm or larger for freshwater or a 20 micron filter shall be used for saltwater.
6. Sample collection and transport containers shall meet the requirements listed in N.J.A.C. 7:18-6.4(a) 12. Prior to sample collection, containers shall be pre-rinsed with the dilution water and then filled so that there is little or no air in the container neck or cap.
7. Dilution water sample storage shall be in covered containers constructed of non-toxic materials as specified in N.J.A.C. 7:18-6.4(a)2.
8. Samples shall not be stored for more than 96 hours and should be collected as close as possible to the time of testing.

(b) Effluent samples shall be collected, handled and preserved as follows:

1. Unless otherwise specified by the Department, the effluent sampling location shall be the same as the specified in the NJPDES permit. An alternate sampling location may be specified when the following conditions prevail:
 - i. When there is better access to the effluent at a point located between the final treatment and the discharge outfall; or
 - ii. When the processed waste is chlorinated prior to discharge and the purpose of the test is to determine the toxicity levels of the unchlorinated effluent. In this case, the sampling point shall be located prior to the effluent's contact with the chlorine.

2. Samples shall be representative of the discharge, taking into account the plant operating conditions and the retention time of the effluent in the wastewater treatment plant.
3. For flow-through bioassays the following sampling procedures shall be adhered to in order to insure a representative effluent sample:
 - i. If the facility discharges continuously, the effluent shall be pumped directly and continuously from the discharge line to the diluter system for the duration of the test; or
 - ii. If the facility discharges continuously but the effluent cannot be pumped directly and continuously to the diluter system, then the following procedure shall be employed:
 - (1) When the minimum retention time of the wastewater in the treatment plant is less than 96 hours as determined from flow metering devices or dye studies, a six hour composite sample consisting of equal volumes taken once every 30 minutes shall be collected and transported to the diluter every six hours for the duration of the test.
 - (2) When the minimum retention time of the wastewater in the treatment plant is between 96 hours (four days) and 14 days, as determined from flow-metering devices or dye studies, then a 24 hour composite sample consisting of equal volumes taken once every hour shall be collected and transported to the diluter daily for the duration of the test.
 - (3) When the minimum retention time of the wastewater in the treatment plant is greater than 14 days as determined from flow-metering devices or dye studies, a single grab sample shall be collected and transported to the diluter daily for the duration of the test.
 - iii. If the facility discharges intermittently, a composite sample consisting of equal volumes collected once every 30 minutes shall be taken for the duration of the plant's operating schedule or a single grab sample shall be taken in the case of a short-term batch discharge.

4. In order to insure the collection of a representative effluent sample for a static or modified static bioassay, the following sampling procedures shall be followed:
 - i. When the minimum retention time of the wastewater in the treatment plant is less than 96 hours as determined from flow-metering devices or dye studies, four consecutive six hour composite samples, each consisting of an equal volume taken once every 30 minutes, shall be collected and used in setting up four separate static or modified static bioassay tests. This procedure is repeated for the duration of the test.
 - ii. When the minimum retention time of the wastewater in the treatment plant is between 96 hours (four days) and 14 days as determined from flow-metering devices or dye studies, a single grab sample shall be collected and used to set-up a single test. This procedure is repeated for the duration of the test.
 - iii. When the minimum retention time of the wastewater in the treatment plant is greater than 24 days, as determined from flow-metering devices or dye studies, a single grab sample shall be collected and used to set-up a single test. This procedure is repeated for the duration of the test.
5. Samples shall not be altered in any way except that the effluent may be filtered through Teflon^R or No. 316 austenitic stainless steel screening having a mesh of 2mm or larger. Screening constructed of unplasticized polyethylene or polypropylene may be substituted provided the screens are discarded upon the completion of a bioassay.
6. Composite or grab sample collection and handling containers shall meet the requirements listed in N.J.A.C. 7:18-6.4(a)13. Prior to sample collection, containers shall be pre-rinsed with the effluent and then filled so that there is no air space in either the neck or cap.
7. Effluent samples shall be stored in covered, unsealed containers constructed of non-toxic materials as specified in N.J.A.C. 7:18-6.4(a)2.
8. Unless the purpose of the bioassay is to ascertain the persistence of the toxicity of an effluent, testing shall begin within 24 hours of the collection of an effluent.

9. If samples are collected for offsite testing, the samples should be stored so that they are maintained at the test temperature specified in N.J.A.C. 7:18-6.6(1)4 by holding in either in a constant-temperature bath or a controlled-temperature room. Samples that are to be tested two or more hours after collection should be kept chilled at between 0° - 4°C.

(c) The following chain of custody procedures shall be employed in collecting and handling composite or grab samples:

1. Only clean or previously unused containers, as specified in N.J.A.C. 7:18-6.4(a)12 and 13, shall be used for taking composite or grab samples.
2. Tie-on affixed labels with an identification number shall be used for labeling all samples.
3. After a sample has been collected, the appropriate information as to identity of the sample shall be written on the label and the label affixed. The label shall remain affixed until the test has begun and the surplus sample has been discarded.
4. Immediately upon delivery of a sample to the laboratory, the sample collector shall complete the appropriate section of a chain of custody form.
5. The chain of custody form shall list at a minimum the following information:
 - i. Sample number;
 - ii. Number of containers;
 - iii. Description of samples;
 - iv. Specific location of sample collection;
 - v. Identity of person collecting the sample;
 - vi. Date and time of sample collection;
 - vii. Date and time of custody transfer to laboratory;
 - viii. Identity of person accepting custody;
 - ix. Date and time of initiation of analyses;
 - x. Identity of person performing analysis; and
 - xi. Name of laboratory performing the analyses.
6. Prior to accepting custody of a sample, the laboratory personnel accepting custody shall be assured that the sample has met the collection and handling requirements. If the sample fails to meet those requirements, the chain of custody form and final laboratory report shall so indicate and the sample shall be refused.

7. The laboratory personnel accepting responsibility for the sample, as well as all other laboratory personnel performing the analysis on that sample shall sign the chain of custody form.

7:18-6.6 Methodology

- (a) The following aquatic organisms shall be used for bioassay testing:
 1. The fathead minnow, *Pimephales promelas*, shall be used as the bioassay test organism when the effluent's receiving waters has a salinity of less than or equal to five ppt.
 2. The mysid shrimp, *Mysidopsis bahia*, shall be used as the bioassay test organism when the effluent's receiving waters has a salinity of greater than five ppt., such as estuarine and ocean waters.
- (b) Bioassay test organisms should either be cultured in the laboratory or obtained from commercial or Federal government hatcheries. Collecting bioassay test organisms from the field is not recommended.
- (c) Culturing of fathead minnows in the laboratory shall be in accordance with the methods contained in U.S. EPA-1972.
- (d) Culturing of the mysid shrimp in the laboratory shall be in accordance with the methods contained in U.S. EPA-1978.
- (e) Fathead minnows to be used as test organisms shall meet the following requirements:
 1. All fish shall be actively feeding young of the year which have not been used previously in any bioassay or other test procedure;
 2. All fish used in a bioassay shall be from the same source;
 3. The total length of the longest fish of a group shall not be more than 1 1/2 times that of the shortest fish of the same group to be used for any test; and
 4. The wet weight of each fish should be within 0.1 to 0.5 grams.
- (f) Mysid shrimp to be used as test organisms shall meet the following requirements.
 1. Only newly hatched juvenile mysids less than or equal to 24 hours old shall be used; and

2. All adult mysid shrimp from which the juveniles are obtained shall be from the same source.
- (g) The holding and handling of the organisms shall be as follows:
1. While being transported to the laboratory, organisms shall not be overcrowded, the dissolved oxygen shall be maintained at or above 60 percent of saturation, and the temperature shall not change by more than 3°C in any 12 hour period;
 2. When first brought into the laboratory, the organisms shall be quarantined for at least 10 days in order to observe them for parasites and diseases. If more than 10 percent of the quarantined organisms die after the second day or are heavily diseased or parasitized, and the problem cannot be controlled, that lot shall be destroyed, the holding tanks and equipment cleaned and sterilized, and another batch of organisms obtained;
 3. After the quarantine period is over and the healthy organisms have been transferred to the stock holding tanks, the organisms shall be gradually acclimated to the laboratory holding conditions. The organisms shall not be subjected to water temperature changes of more than 3°C or salinity changes of more than three ppt., in any 12 hour period, nor to a total change of more than 6°C or six ppt. salinity during the entire transportation, quarantine, and acclimation period;
 4. Organisms that touch dry surfaces, are dropped, or are injured during handling shall be discarded;
 5. Organisms shall be handled as little as possible. When handling is required, the following equipment shall be used:
 - i. Large organisms shall be handled with dipnets of the appropriate size and mesh; and
 - ii. Small organisms, such as juvenile invertebrates and fish fry, shall be handled by pipeting with smooth glass tubes of approximately 9mm I.D.;
 6. The preferred type of holding tank is one of flow through design which allows for a flow rate of at least two tank volumes per day. If flow-through tanks are not feasible, then holding tanks with a closed-recirculating water system, where the water is filtered through charcoal, shall be used;

7. Only laboratory grade water as specified in N.J.A.C. 7:18-6.7(a) shall be used to hold organisms in the laboratory;
 8. Dissolved oxygen levels in holding tanks shall be kept above 60 percent of saturation at all times. If necessary, the use of aeration is acceptable;
 9. Photoperiods and light intensities favorable to the organisms as specified in Standard Methods, 14th Edition, pp. 719 - 721 should be used;
 10. The environment the organisms are maintained in should follow the natural seasonal variations. Temperature shall be as follows:
 - i. Depending upon the season, fathead minnows shall be held at temperatures between 10° - 25°C; and
 - ii. Depending upon the season, mysid shrimp shall be held at temperatures between 18° - 28°C; and
 11. Organisms shall not be fed for at least 24 hours prior to a test, except for mysid shrimp which shall be fed ad libitum up to and during a test.
- (h) Test organisms shall be taken from groups whose mortality while held was less than 10 percent for the seven day period prior to the bioassay.
- (i) The test organisms shall be acclimated to the test dilution water and the test temperature in accordance with the following requirements:
1. The test dilution water for mysid shrimp shall be the same type as they were cultured or held in, be it filtered natural seawater or artificial seawater.
 2. Fathead minnows shall be acclimated by gradually changing from 100 percent laboratory grade holding water to 100 percent dilution water over a 24 hour period.
 3. All test organisms shall be exposed to 100 percent dilution water for a minimum of 24 hours before they are used in the bioassay.
 4. All test organisms shall be held for at least 24 hours at the required test temperature of 22°C ± 2°C.

5. If more than five percent of the test organisms die during the 48 hour acclimation period, immediately preceding the test, the organisms shall not be used and the following procedures shall be used:
 - i. The entire group of test organisms shall be discarded and a new group obtained;
 - ii. The new group of organisms shall be transported, held, and acclimated in accordance with the procedures contained in N.J.A.C. 7:18-6.6 (a) through (i); and
 - iii. If more than five percent of the second group of test organisms die within 48 hours, an alternate source of dilution water shall be used.
- (j) The following procedures shall be used for tests conducted with mysid shrimp:
 1. The dilution water used shall be adjusted or prepared to a salinity of greater than or equal to 15 ppt. The preferred salinity range is 22 - 26 ppt.; and
 2. If the effluent sample is of a salinity less than 15 ppt., the salinity shall be adjusted to the proper level by the addition of dry, autoclaved artificial sea salts. This is best accomplished by using a magnetic stirrer in the sample while the dry salts are added and dissolved.
- (k) The dissolved oxygen levels in the exposure chambers shall be maintained at or above 40 percent of saturation for the fathead minnow and 60 percent of saturation for the mysid shrimp by the following methods:
 1. In static and modified static bioassay tests, a depression of dissolved oxygen shall necessitate aerating of all exposure chambers.
 2. In flow-through bioassay tests, if the dissolved oxygen becomes depressed, first increase the flow rate to the maximum, if necessary. If the increased flow does not sufficiently increase the dissolved oxygen then, aerate the dilution water prior to the addition of the effluent and aerate all exposure chambers along with using the increased flow rate.
- (l) Short-term range-finding or screening bioassays shall be used to determine the approximate range of effluent concentrations that should be used in a subsequent short-term definitive test. The range-finding methodology shall meet the following requirements:

1. Test duration shall be 24 hours;
2. Test type shall be either static or flow-through with the following specifications;
 - i. Exposure chamber loading for the fathead minnows shall not exceed 2.5 grams per liter in flow-through tests and 0.4 grams per liter for static tests.
 - ii. Exposure chamber loading for the bay mysid shall not exceed 10 mysids per liter for static tests and 12 mysids per liter for flow-through tests;
3. Test organisms shall be exposed to:
 - i. At least five widely spaced effluent concentrations based either on a logarithmic ratio, such as 0.01, 0.1, 1.0, 10 and 100 percent; or
 - ii. Progressive bisections of intervals on the logarithmic scale as described in Standard Methods, 14th Edition, pp. 715 - 716; and
 - iii. A control.
4. Effluent concentrations shall be expressed as percent effluent by volume;
5. Five test organisms shall be exposed to each effluent concentration and the control;
6. Water temperature in the exposure chambers shall be maintained for the duration of the test, to within $\pm 2^{\circ}\text{C}$ of the specified test temperature;
7. If the lowest concentration used kills all the test organisms, another test shall be set-up using a series of concentrations which starts at the lowest concentration previously tested;
8. All effluent solutions for a concentration series shall be prepared from the same sample of effluent; and
9. Any undissolved material in the effluent sample shall be dispersed uniformly by gentle agitation prior to withdrawal and aliquots of both effluent and dilution water shall be mixed well in the exposure chambers. Test organisms shall be added within 30 minutes to begin test.

- (m) Short-term definitive bioassay tests shall be used to determine the acute toxicity of an effluent. The test methodology shall meet the following requirements:
1. Test duration shall be at least 96 hours, but if required by the Department the test shall be continued until the toxicity curve shows that the threshold toxicity called the Incipient LC_{50} has been reached;
 2. Test type shall be either modified static (daily renewal) or flow-through with the same specifications on test organism loading as listed in N.J.A.C. 7:18-6.6(1)2;
 3. Test organisms shall be exposed to at least five effluent concentrations, the range of which will have been determined previously by a range-finding bioassay based on progressive bisections of intervals on the logarithmic scale as described in Standard Methods, 14th Edition, pp. 715 - 716, and a control. Concentrations shall be expressed as percent effluent by volume;
 4. The test shall be conducted in replicate with at least twenty organisms exposed to each effluent concentration and the control. Replicates shall be true replicates with no direct water connections between them;
 5. Exposure chambers shall be randomly assigned to either an effluent concentration or the control, and the test organisms shall be randomly assigned to the exposure chambers;
 6. Water temperature maintenance shall be as specified in N.J.A.C. 7:18-6.6(1)6;
 7. Test organisms shall be acclimated to the dilution water in accordance with the procedures listed in N.J.A.C. 7:18-6.6(i) prior to their use in a test;
 8. All effluent solutions for a concentration series shall be prepared from the same sample of effluent;
 9. The following required methods apply only to conducting modified static tests:
 - i. The test organisms shall be exposed to fresh solution of the same concentration of effluent every 24 hours either by transferring the test organisms from one test chamber to another or by replacing the effluent solutions in the exposure chambers;

- ii. The modified static test procedure used for mysid shrimp should follow the method described in U.S.E.P.A.-1978, pp. 61-63;
 - iii. The procedure described in N.J.A.C. 7:18-6.6(1) 9 shall be followed for setting-up and beginning a modified static test;
10. The following required methods apply only to conducting flow-through tests:
- i. The diluter system of the flow-through apparatus shall be in operation for at least 24 hours prior to the addition of the organisms and the beginning of the test. During this time, the water temperature, flow rate through the exposure chamber, and the effluent concentrations in the exposure chambers shall be adjusted to the test requirements.
 - ii. After adjustments but prior to beginning the test, there shall be at least one tank volume exchange. Flow rate through the exposure chambers shall be sufficient to maintain a minimum dissolved oxygen concentration of 40 percent of saturation for the fathead minnow and 60 percent of saturation for the mysid shrimp and provide no less than five tank water volume changes every 24 hours.
- (n) Observations of test organisms in the exposure chambers shall be made at least once every 24 hours for the duration of the test. It is suggested that observations be made of each exposure chamber at 1.5, 3, 6, 12, and 24 hours after the beginning of the test and twice a day thereafter.
- (o) In short-term acute toxicity bioassays, death is the adverse effect which shall be quantified. The criterion for death shall be no movement which will include respiratory movement in fish, no movement of antenna, mouth parts or other organs in invertebrates, and reaction to gentle prodding.
- (p) Effects such as erratic swimming, loss of reflex, hyperventilation, curved spine, hemorrhaging, discoloration, changes in behavior, excessive mucus production, molting and cannibalism shall be reported.
- (q) During short term tests, deaths in the controls should be virtually absent. For the fathead minnows, a control mortality of greater than or equal to 10 percent shall invalidate the test. When using mysid shrimp, control mortalities of greater than or equal to 15 percent shall invalidate the test.

- (r) Dissolved oxygen, pH, specific conductivity, total alkalinity, total hardness, and, when applicable, salinity shall be measured in the exposure chambers and recorded initially and at least once every day thereafter for the duration of the test.
- (s) The lengths and weights of the test organisms (fish) shall be determined by sacrificing and measuring a representative sample of the stock organisms before the test and by measuring all of the test organisms (fish) after the completion of the test. This may be accomplished by preserving both the surviving and dead fish. An acceptable alternative shall be to measure a representative sample of the test organisms, consisting of both surviving and dead fish, instead of measuring all of the test organisms.
- (t) The calculation and reporting of the results of any bioassay shall meet the following requirements:
1. The results of all bioassays are to be expressed in terms of their median lethal concentration, or LC_{50} for a specified time period.
 2. Range-finding bioassays shall be analyzed by using the graphical interpolation method for estimating the LC_{50} presented in Standard Methods, 14th Edition, pp. 731 - 733, Methods for Measuring Acute Toxicity - EPA, pp. 37-38.
 3. Definitive bioassays shall be analyzed by any of the following methods and with the following requirements:
 - i. The LC_{50} values for the 24, 48, 72, and 96 hour exposure times, depending upon the duration of exposure, shall be estimated by Litchfield-Wilcoxon method as described in Methods for Measuring Acute Toxicity - EPA, pp. 29-36, or by probit analysis or Finney's method of formal probit analysis as described in Standard Methods, 14th Edition, pp. 733 - 735.
 - ii. The 95 percent confidence or fiducial limits for the 96 hour or Incipient LC_{50} 's shall be calculated. The simplified nomographic methods of Litchfield and Wilcoxon, Methods for Measuring Acute Toxicity-EPA, are acceptable.
 - iii. In order to estimate an LC_{50} for a definitive test by any of the aforementioned methods, one concentration shall have killed more than

65 percent of the test organisms exposed to the effluent and one concentration, other than the control, shall have killed less than 35 percent of the test organisms. If these conditions are not met, the LC₅₀ shall be estimated by the graphical interpolation method referenced in N.J.A.C. 7:18-6.6(t)2.

- iv. If the highest effluent concentration does not kill more than 65 percent of the test organisms exposed to it, the percentage of organisms killed by various concentrations of the effluent shall be reported.
 - v. A toxicity curve shall be plotted using the LC₅₀'s for each of the observation times according to the methodology presented in Standard Methods, 14th Edition, section 801 F. 26. The presence or absence of an Incipient LC₅₀, as estimated from the toxicity curve, shall be reported along with its value.
4. If the responses from two or more exposure chambers deviate from the expected trend in such a manner that a lower effluent concentration shows a more toxic response than a higher effluent concentration, then the test should be considered invalid, no estimation of the LC₅₀ made, and the test should be repeated.
5. LC₅₀ values shall not be estimated for any bioassay if a test is invalid under the definition given in N.J.A.C. 7:18-6.6(q).
- (u) The analysis of all parameters, excluding salinity, as required by this subchapter shall be conducted in accordance with the requirements set forth in 40 CFR 136 and subchapter 4.
 - (v) The determination of salinity as required in this subchapter shall be computed from chlorinity, electrical conductivity, refractive index, or some other property whose relationship is well established.

7:18-6.7 General laboratory practices

- (a) Laboratory grade water shall meet the following requirements:
 - 1. Natural or artificial sources of water may be used, but natural sources are preferred.
 - 2. Natural sources shall be free of pollution, low in turbidity, high in dissolved oxygen, low in B.O.D., and the pH shall be favorable to the maintenance of the organisms.

3. Fresh water shall meet the following requirements:
 - i. Fresh water shall be constant in quality and shall not contain more than the designated amounts of the following:
 - (1) 20 mg/l of suspended solids;
 - (2) 10 mg/l of total organic carbon or chemical oxygen demand;
 - (3) 20 ug/l of un-ionized ammonia;
 - (4) 50 ug/l of residual chlorine;
 - (5) 50 ng/l of total organophosphorus pesticides;
 - (6) 50 ng/l of total organochloride pesticides plus PCB's; and
 - (7) Water shall be considered of constant quality if the monthly ranges of total alkalinity, total hardness, specific conductivity, TOC or COD, and salinity are less than 10 percent of the respective averages.
 - ii. Municipal water supplies are not recommended since they often contain unacceptable concentrations of heavy metals and fluoride. If municipal water must be used as a source it shall be determined that the concentrations of these materials are less than 1 ug/l each. Residual chlorine can be removed by passing the water thru activated carbon filters.
4. Saltwater shall meet the following requirements:
 - i. Natural saltwater shall be from a source free of pollution, and having a pH and salinity favorable to the organism. A salinity between 10 to 27 ppt. shall be used for mysid shrimp;
 - ii. If adjustments to the salinity of natural saltwater are necessary they shall be made by adding either deionized water or dry seasalts only. Prior to use, the saltwater shall be filtered through a 20 micron filter; and
 - iii. Artificial saltwater may be substituted for natural saltwater if an acceptable supply of the latter is unavailable. It shall be prepared according to the methods listed in

Standard Methods, 14th Edition, pp. 696 - 697
Methods for Measuring Acute Toxicity-EPA, or
obtained from a commercial source.

- (b) The food and feeding of the test organisms shall be as follows:
1. Fathead minnows shall be fed at least once a day a combination of natural foods, either live or frozen, and any of several prepared dried foods.
 2. Mysid shrimp shall be fed ad libitum live 48 hour old *Artemia nauplii*.
- (c) Treatment of diseased or parasitized organisms shall be in accordance with the procedures given in Standard Methods, 14th Edition, pp. 703 - 704, and Methods for Measuring Acute Toxicity-EPA.
- (d) Organisms treated for disease or parasites shall not be used in bioassays for at least 10 days after treatment.
- (e) Cleaning of all chambers and equipment shall be in accordance with the following procedure:
1. First, soak and wash with a synthetic detergent/laboratory grade fresh warm water solution, and then rinse with 50°C or warmer water;
 2. Secondly rinse with a fresh, 5 percent hydrochloric or nitric acid solution for the removal of metals and bases, and rinse again with 50°C or warmer laboratory grade fresh water; and
 3. Finally, rinse with acetone to remove organic compounds, and rinse twice with laboratory grade freshwater.
- (f) When measuring sample volumes of more than 10 ml, graduate cylinders having an accuracy within 2.5 percent tolerance shall be used.
- (g) A laboratory that has received either certification or interim approval shall accept only samples that are properly labeled and for which reasonable assurance is given that the samples have been collected, preserved, processed, stored and transported in a manner that will assure both the identify of the sample and that the sample is sufficiently stable to be used in the requested tests or analyses. If the identity or stability of the sample has not been assured, both the chain of custody form and the laboratory report shall clearly state that the result may be invalid due to the possible misidentification or instability of the sample.

7:18-6.8 Quality control program

(a) An acceptable degree of precision for definitive bioassays shall be that the 95 percent confidence or fiducial intervals be within less than ± 30 percent of the 96 hour or incipient LC_{50} value.

(b) Each laboratory shall develop and have on file and available for inspection a written description of the current laboratory quality control program. The written description shall outline the procedures which the laboratory will use in meeting the quality control requirements set forth in N.J.A.C. 7:18-4.6 and 7:18-4.7. A record of analytical control tests and quality control checks on equipment and materials shall be prepared by the laboratory and retained for at least five years.

1. Laboratories shall perform the following analytical quality control tests to ensure that general laboratory practices and methodology are in compliance with the requirements of this subchapter:

i. Laboratory pure water shall be analyzed for and meet the following requirements:

pH	5.5 - 7.5
Conductivity	Greater than 0.2 megohm-cm as resistivity or less than 5.0 microhomo ^s cm as conductance at 25°C
Trace metals:	
A single metal	Not greater than 0.05 mg/l
Total metals	Equal to or less than 1.0 mg
Test for bactericidal properties of distilled water (Standard Methods, 14th Edition, pg. 888, or Microbiological Methods - EPA, pp 200)	0.8 - 3.0
Free chlorine residual	0.0

ii. Laboratory pure water checks for pH, conductivity, and free chlorine residual shall be performed monthly and documented.

iii. Laboratory pure water checks for trace metals, bactericidal properties, and standard plate count shall be performed annually and documented.

- iv. Laboratory grade water shall be analyzed monthly for pH, D.O., chlorine residual, and specific conductance;
- v. Laboratory grade fresh water shall be analyzed at least twice annually for the materials specified in N.J.A.C. 7:18-6.7(a)3;
- vi. There shall be available at all times, in the immediate bench area of laboratory personnel engaged in examining samples and performing related procedures within a category, current laboratory manuals or other complete written descriptions and instructions relating to:
 - (1) The analytical methods to be used by those personnel, properly designated and dated to reflect the most recent supervisory reviews;
 - (2) Pertinent current literature references; and
 - (3) Such written descriptions and instructions may be supplemented by, but not replaced by, textbooks relating to the particular analytical methods and procedures employed by such personnel;
- vii. Only the laboratory manager or supervisor shall make changes in laboratory procedure and those changes shall only be effective when put in writing.
- viii. The following procedures shall be followed in performing quality control checks of laboratory media, equipment, and supplies:
 - (1) Each pH meter shall be cleaned immediately after each use period and calibrated prior to usage using at least two pH buffer standards that bracket the value to be measured and records of each calibration shall be maintained; buffer aliquots shall not be used more than once; and commercial buffer solutions shall be dated at the time of initial use;
 - (2) Top loader or pan balances shall be calibrated annually, calibration shall be checked monthly against class "s" weights, and a record shall be made of each calibration check;

- (3) Glass thermometers and continuous recording devices shall be checked yearly and metal thermometers shall be checked quarterly, or at more frequent intervals if necessary, against a certified thermometer of equivalent accuracy, at several points throughout the entire range, including but not limited to the temperature point or range for the test, analysis or quality control measure being performed, and the results of such testing shall be recorded;
- (4) The temperature of air or water-jacketed incubators, aluminum block incubators, water baths, and incubator rooms shall be either recorded continuously or recorded daily from in-place thermometers immersed in liquid and placed on at least one of the shelves in use.
- (5) Date, time and temperature shall be either recorded continuously, or recorded individually during each sterilization cycle of the autoclave;
- (6) Each hot air oven shall be equipped with either a thermometer calibrated in the range of 170°C, the bulb of which shall be placed in sand, or with a temperature recording device, and records shall be maintained showing the date, time and temperature of each sterilization cycle;
- (7) The temperature of each refrigerator shall be either recorded continuously or recorded daily from an in-place thermometer immersed in liquid and placed on at least one of the shelves in use;
- (8) All reagents and solutions shall be labelled to indicate identity and, when applicable, titer, strength or concentration, recommended storage requirements, preparation or expiration date, and other information pertinent to identification;
- (9) Materials of substandard reactivity and deteriorated materials shall not be used; and
- (10) All outdated material shall be discarded immediately.

- (c) The temperature in, flow rate through the exposure chambers and the maintainance of effluent concentrations, shall be checked initially, daily during the duration of the test, and upon completion of the test, adjusted as necessary, and documentation of these adjustments and measurements shall be made.

7:18-6.9 Records and data reporting

- (a) Each laboratory shall maintain records and report data in accordance with the requirements set out in this section.
- (b) Records of bioassay analysis shall be kept by the laboratory for not less than five years. This requirement is equally applicable to all raw data, quality control data, chain of custody forms and laboratory reports.
- (c) The following information shall be kept by the laboratory as part of the daily log of feeding, behavioral observations, and mortality of organisms during holding and acclimation:
 1. Water temperature of holding tanks;
 2. Air temperature in culturing/holding room;
 3. Mortalities of organisms per holding tank;
 4. Analysis of laboratory grade water as specified in N.J.A.C. 7:18-6.6(b);
 5. Food and feeding schedule; and
 6. General observations of behavior and condition.
- (d) A sample report form shall be completed immediately after collection of either dilution water or effluent composite or grab samples and shall state the sampling location, date and time of collection, chlorine residual, collectors name, and any remarks.
- (e) Immediately after the sample is delivered to the laboratory, the date and time shall be recorded on the sample report form.
- (f) Immediately after testing has begun, the data and time of initiation of testing shall be recorded on the sample report form.
- (g) The bioassay experimental results shall be reported in accordance with the specifications given in Methods for Measuring Acute Toxicity-EPA, pp. 24-25, and;
 1. The incipient LC_{50} shall be reported if applicable.
 2. A figure showing the toxicity curve shall be included.

(h) The original or true duplicate of the results of the bioassay shall be sent promptly to the person who requested such test and shall be signed by the laboratory manager.



State of New Jersey

DEPARTMENT OF ENVIRONMENTAL PROTECTION
DIVISION OF WATER RESOURCES

Arnold Schiffman
Director

~~XXXXXXXXXX~~ CN-029
TRENTON, NEW JERSEY 08625

April 8, 1981

To All Interested Parties:

The Department is pleased to submit for your review and comment Proposed Regulations Governing Laboratory Certification and Standards of Performance pursuant to the authority of N.J.S.A. 13:1D et seq., the Water Pollution Control Act, N.J.S.A. 58:10A-1 et seq., and the Safe Drinking Water Act, N.J.S.A. 58:12A-1 et seq.

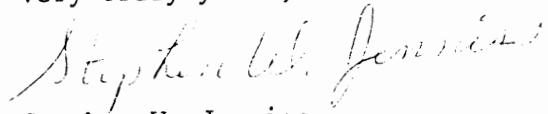
This proposed regulation will require that all water analyses performed for compliance with regulations adopted or orders issued pursuant to the Water Pollution Control Act and the Safe Drinking Water Act be performed in a certified laboratory. The Department will offer certification in the Microbiological Testing, Limited Chemistry, Atomic Absorption, Gas Chromatography, Radiological Testing, and Bioassay Testing categories. The regulation establishes the program administrative procedures which laboratories shall follow to obtain and maintain certification. It also establishes minimum standards for laboratory instrumentation, analytical methodologies, laboratory practices, analytical quality control, and data handling procedures.

The Department will accept written comments on this proposal until June 22, 1981. It will also hold two public hearings to receive comments on this proposal. They will be held on May 28, 1981, in Room 106, Rutgers University Law Center, Fifth and Penn Streets, Camden and on May 29, 1981 in the Seminar Room, New Jersey Institute of Technology Alumni Center, 150 Bleaker Street, Newark. Comments will be welcomed at the meeting or can be mailed to:

Stephen W. Jenniss
Quality Assurance Coordinator
Monitoring and Planning Element
Division of Water Resources
P.O. Box CN-029
Trenton, New Jersey 08625

I hope you will be able to attend these hearings and provide us with your comments.

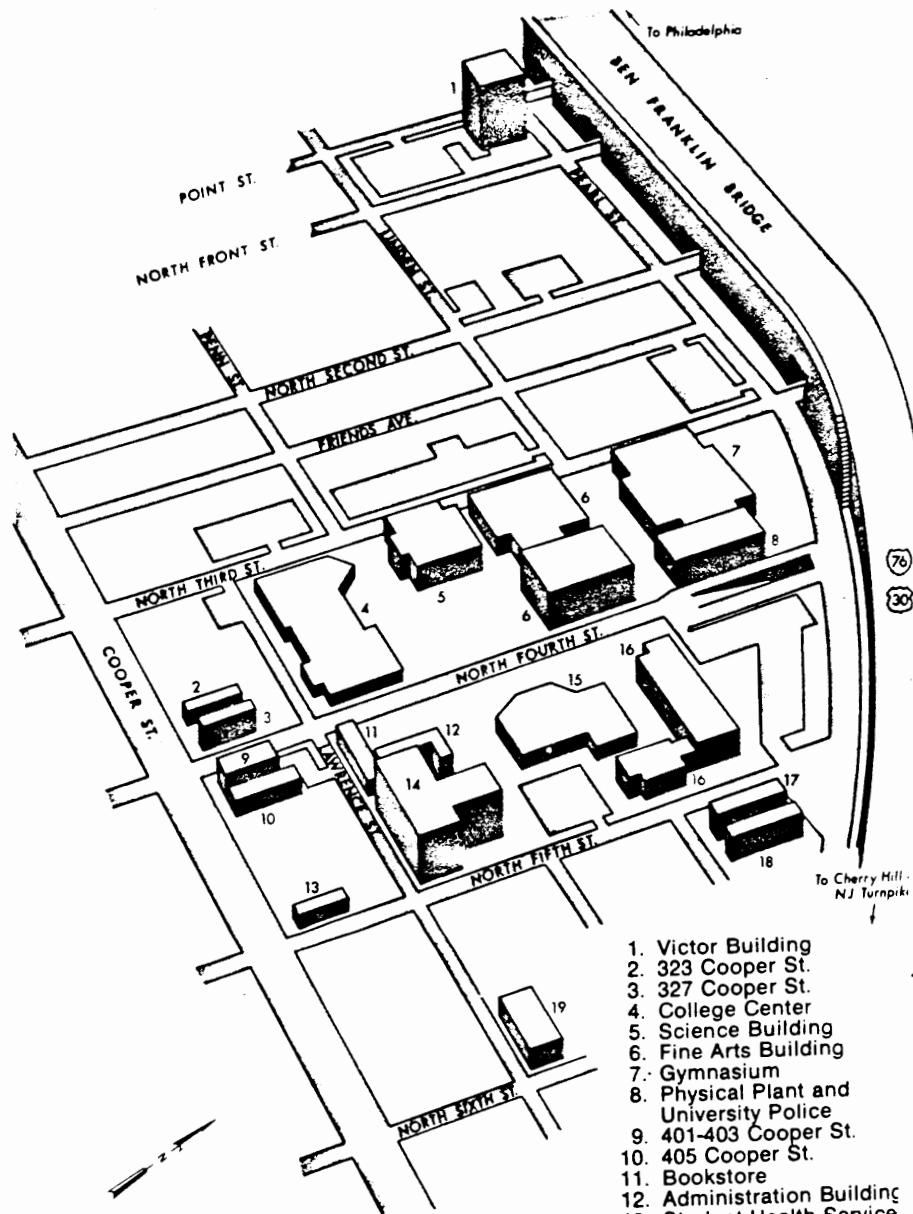
Very truly yours,



Stephen W. Jenniss
Quality Assurance Coordinator

SWJ:mas

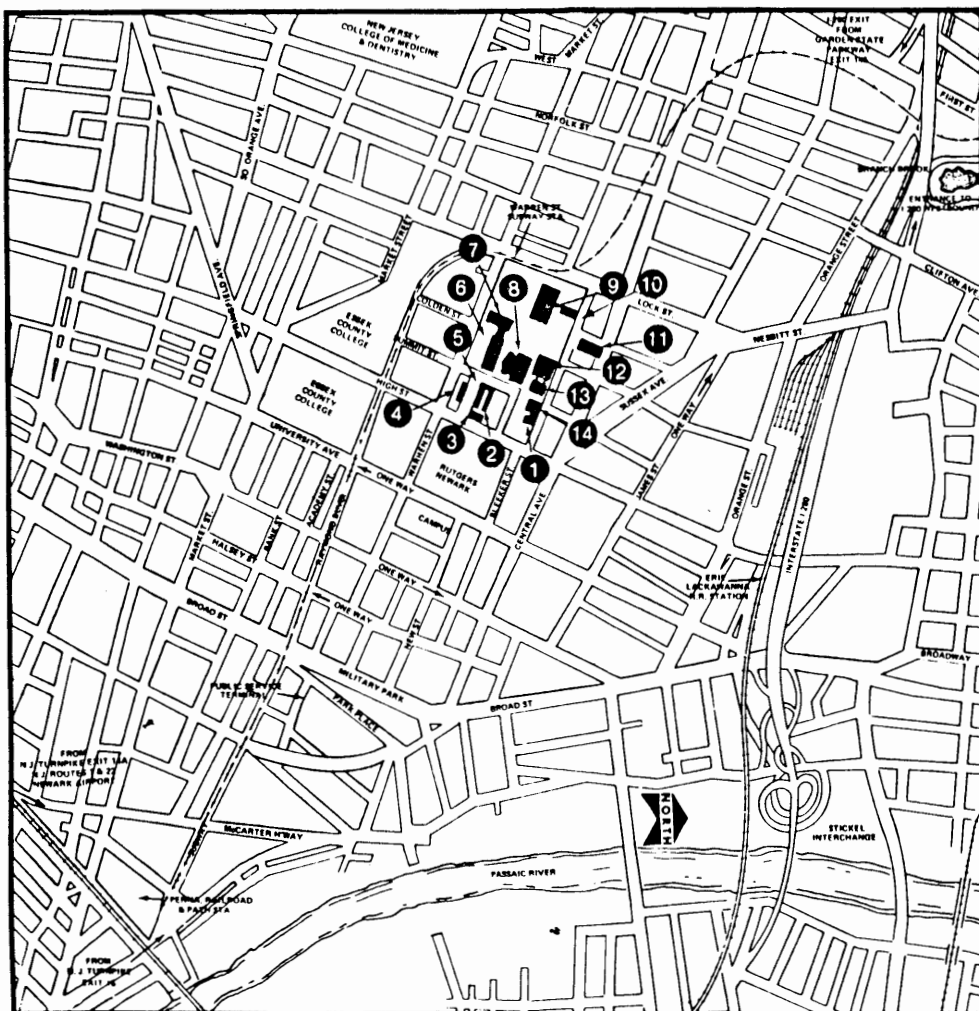
CAMDEN CAMPUS



Map prepared by Robert W. Kirwan
Cartography Laboratory
Department of Geography, May 1977
Revised by Chuck Ogrosky

1. Victor Building
2. 323 Cooper St.
3. 327 Cooper St.
4. College Center
5. Science Building
6. Fine Arts Building
7. Gymnasium
8. Physical Plant and University Police
9. 401-403 Cooper St.
10. 405 Cooper St.
11. Bookstore
12. Administration Building
13. Student Health Service
14. Law Center
15. Library
16. Armitage Hall
17. College Hall
18. 502 Linden St.
19. 217 N. Sixth St.

Downtown Newark and the Campus of New Jersey Institute of Technology



Transportation to NJIT

Seven major highways and public transportation make NJIT easily accessible.

By Car: Garden State Parkway (Exit 145); New Jersey Turnpike (Exit 15E); Interstate 280 (Newark exit); U.S. Route 1, Route 9, and Route 22; and N.J. Route 21.

†By Rail: PATH rapid transit, to Penn Station in Newark — from New York, Harrison, Jersey City and Hoboken.

*Penn Central**, to Penn Station in Newark — from New York and southern section of N.J.

*Erie-Lackawana** — from suburban communities north and west of Newark. E-L station is within walking distance of campus.

*Central Railroad of New Jersey** — serves Union and Somerset Counties.

*New York and Long Branch RR**, to Penn Station in Newark — from coastal sector of N.J.

*ALL Divisions of CONRAIL

†By Bus: Transport of New Jersey (TNJ) operates more than 70 commuter routes to and from six northern counties.

DeCamp Lines — No. 145 & 146 serves Greater Morristown area and the Oranges.

Somerset Bus Company — No. 140 & 141 serves Union and Somerset Counties.

Mountain Coach — No. 144 serves Caldwell, Roseland and West Orange.

Greyhound and Trailways — direct bus service to Newark from all points.

By City Subway: The Newark City Subway provides quick service between the Belleville border and Penn Station, Newark. It makes 11 stops in route, including a stop on Warren Street at the SW corner of the campus.

By Taxi: A large fleet of taxis serves Newark.

By Air: Every major air line serves Newark International Airport. Cabs, limousine service and the No. 21 bus provide easy access from the airport to downtown Newark.

†Penn Station is a major point of transfer in Newark for busses and trains and is within walking distance of the campus.

Parking: There is limited amount of parking available on campus. Commercial parking areas are located adjacent to the campus on Warren Street (See campus map).

- | | | |
|--|--|---|
| 1. EBERHARDT HALL (E)
323 High Street | 6. FACULTY MEMORIAL HALL (F)
111 Summit Street | 11. MECHANICAL ENGINEERING (B)
200 Central Avenue |
| 2. CAMPBELL HALL (C)
110 Summit Street | 7. TIERNAN HALL (T)
161 Warren Street | 12. THE CENTER
150 Bleeker Street |
| 3. WESTON HALL (W)
367 High Street | 8. ROBERT W. VAN HOUTEN LIBRARY
99 Summit Street | 13. ALUMNI CENTER FOR CONTINUING ENGINEERING STUDIES
150 Bleeker Street |
| 4. SPECHT MAINTENANCE BUILDING
120 Summit Street | 9. ENTWISLE PHYSICAL EDUCATION BUILDING
80 Lock Street | 14. CULLIMORE HALL (M)
70 Summit Street |
| 5. COLTON HALL (L)
Summit Place | 10. DORMITORY
186 Bleeker Street | |

the Administrator's affirmative determination pursuant to section 312(f)(3) of the Act. Upon receipt of an application under section 312(f)(3) of the Act, the Administrator will determine within 90 days whether adequate facilities for the safe and sanitary removal and treatment of sewage from all vessels using such waters are reasonably available. Applications made by States pursuant to section 312(f)(3) of the Act shall include: (1) A certification that the protection and enhancement of the waters described in the petition require greater environmental protection than the applicable Federal standard; (2) a map showing the location of commercial and recreational pump-out facilities; (3) a description of the location of pump-out facilities within waters designated for no discharge; (4) the general schedule of operating hours of the pump-out facilities; (5) the draught requirements on vessels that may be excluded because of insufficient water depth adjacent to the facility; (6) information indicating that treatment of wastes from such pump-out facilities is in conformance with Federal law; and (7) information on vessel population and vessel usage of the subject waters.

(b) A State may make a written application to the Administrator, Environmental Protection Agency, under section 312(f)(4) of the Act, for the issuance of a regulation completely prohibiting discharge from a vessel of any sewage, whether treated or not, into particular waters of the United States or specified portions thereof, which waters are located within the boundaries of such State. Such application shall specify with particularity the waters, or portions thereof, for which a complete prohibition is desired. The application shall include identification of water recreational areas, drinking water intakes, aquatic sanctuaries, identifiable fish-spawning and nursery areas, and areas of intensive boating activities. If, on the basis of the State's application and any other information available to him, the Administrator is unable to make a finding that the waters listed in the application require a complete prohibition of any discharge in the waters or portions thereof covered by the applica-

tion, he shall state the reasons why he cannot make such a finding, and shall deny the application. If the Administrator makes a finding that the waters listed in the application require a complete prohibition of any discharge in all or any part of the waters or portions thereof covered by the State's application, he shall publish notice of such findings together with a notice of proposed rule making, and then shall proceed in accordance with 5 U.S.C. 553. If the Administrator's finding is that applicable water quality standards require a complete prohibition covering a more restricted or more expanded area than that applied for by the State, he shall state the reasons why his finding differs in scope from that requested in the State's application.

(1) For the following waters the discharge from a vessel of any sewage (whether treated or not) is completely prohibited:

Boundary Waters Canoe Area, formerly designated as the Superior, Little Indian Sioux, and Caribou Roadless Areas, in the Superior National Forest, Minnesota, as described in 16 U.S.C. 577-577d1.

[41 FR 4453, Jan. 29, 1976, as amended at 42 FR 43837, Aug. 31, 1977]

§ 140.5 Analytical procedures.

In determining the composition and quality of effluent discharge from marine sanitation devices, the procedures contained in 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants," or subsequent revisions or amendments thereto, shall be employed.

PART 141—NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS

Subpart A—General

- Sec.
- 141.1 Applicability.
 - 141.2 Definitions.
 - 141.3 Coverage.
 - 141.4 Variances and exemptions.
 - 141.5 Siting requirements.
 - 141.6 Effective date.

Subpart B—Maximum Contaminant Levels

- 141.11 Maximum contaminant levels for inorganic chemicals.

§ 141.9

Sec.

- 141.12 Maximum contaminant levels for organic chemicals.
- 141.13 Maximum contaminant levels for turbidity.
- 141.14 Maximum microbiological contaminant levels.
- 141.15 Maximum contaminant levels for radium-226, radium-228, and gross alpha particle radioactivity in community water systems.
- 141.16 Maximum contaminant levels for beta particle and photon radioactivity from man-made radionuclides in community water systems.

Subpart C—Monitoring and Analytical Requirements

- 141.21 Microbiological contaminant sampling and analytical requirements.
- 141.22 Turbidity sampling and analytical requirements.
- 141.23 Inorganic chemical sampling and analytical requirements.
- 141.24 Organic chemical sampling and analytical requirements.
- 141.25 Analytical Methods for Radioactivity.
- 141.26 Monitoring Frequency for Radioactivity in Community Water Systems.
- 141.27 Alternative analytical techniques.
- 141.28 Approved laboratories.
- 141.29 Monitoring of consecutive public water systems.
- 141.30 Total trihalomethanes sampling, analytical and other requirements.

Subpart D—Reporting, Public Notification and Record Keeping

- 141.31 Reporting requirements.
- 141.32 Public notification.
- 141.33 Record maintenance.

Subpart E—Special Monitoring Regulations for Organic Chemicals

- 141.40 Special monitoring for organic chemicals.

APPENDIX A—SUMMARY OF PUBLIC COMMENTS AND EPA RESPONSES ON PROPOSED AMENDMENTS TO THE NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS FOR CONTROL OF TRIHALOMETHANES IN DRINKING WATER

APPENDIX B—SUMMARY OF MAJOR COMMENT (FOR RESPONSES SEE APPENDIX A)

APPENDIX C—ANALYSIS OF TRIHALOMETHANES

AUTHORITY: Secs. 1412, 1414, 1445, and 1450 of the Public Health Service Act, 88 Stat. 1660 (42 U.S.C. 300g-1, 300g-3, 300j-4, and 300j-9).

SOURCE: 40 FR 59570, Dec. 24, 1975, unless otherwise noted.

Title 40—Protection of Environment

EFFECTIVE DATE NOTE: For community water systems serving 75,000 or more persons, monitoring must begin 1 year following promulgation and the effective date of the MCL is 2 years following promulgation. For community water systems serving 10,000 to 75,000 persons, monitoring must begin within 3 years from the date of promulgation and the effective date of the MCL is 4 years from the date of promulgation. Effective immediately, systems that plan to make significant modifications to their treatment processes for the purpose of complying with the TTHM MCL are required to seek and obtain State approval of their treatment modification plans. This note affects §§ 141.2, 141.6, 141.12, 141.24 and 141.30. For additional information see 44 FR 68641, Nov. 29, 1979.

Subpart A—General

§ 141.1 Applicability.

This part establishes primary drinking water regulations pursuant to section 1412 of the Public Health Service Act, as amended by the Safe Drinking Water Act (Pub. L. 93-523); and related regulations applicable to public water systems.

§ 141.2 Definitions.

As used in this part, the term:

(a) "Act" means the Public Health Service Act, as amended by the Safe Drinking Water Act, Pub. L. 93-523.

(b) "Contaminant" means any physical, chemical, biological, or radiological substance or matter in water.

(c) "Maximum contaminant level" means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of a public water system, except in the case of turbidity where the maximum permissible level is measured at the point of entry to the distribution system. Contaminants added to the water under circumstances controlled by the user, except those resulting from corrosion of piping and plumbing caused by water quality, are excluded from this definition.

(d) "Person" means an individual, corporation, company, association, partnership, State, municipality, or Federal agency.

(e) "Public water system" means a system for the provision to the public of piped water for human consump-

tion, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A public water system is either a "community water system" or a "noncommunity water system."

(i) "Community water system" means a public water system which serves at least 15 service connections used by year-round residents or regularly serves at least 25 year-round residents.

(ii) "Non-community water system" means a public water system that is not a community water system.

(f) "Sanitary survey" means an onsite review of the water source, facilities, equipment, operation and maintenance of a public water system for the purpose of evaluating the adequacy of such source, facilities, equipment, operation and maintenance for producing and distributing safe drinking water.

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

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

(i) "Supplier of water" means any person who owns or operates a public water system.

(j) "Dose equivalent" means the product of the absorbed dose from ionizing radiation and such factors as account for differences in biological effectiveness due to the type of radiation and its distribution in the body as specified by the International Commission on Radiological Units and Measurements (ICRU).

(k) "Rem" means the unit of dose equivalent from ionizing radiation to the total body or any internal organ or organ system. A "millirem (mrem)" is 1/1000 of a rem.

(l) "Pecurie (pCi)" means the quantity of radioactive material producing 2.22 nuclear transformations per minute.

(m) "Gross alpha particle activity" means the total radioactivity due to alpha particle emission as inferred from measurements on a dry sample.

(n) "Man-made beta particle and photon emitters" means all radionuclides emitting beta particles and/or photons listed in Maximum Permissible Body Burdens and Maximum Permissible Concentration of Radionuclides in Air or Water for Occupational Exposure, NBS Handbook 69, except the daughter products of thorium-232, uranium-235 and uranium-238.

(o) "Gross beta particle activity" means the total radioactivity due to beta particle emission as inferred from measurements on a dry sample.

(p) "Halogen" means one of the chemical elements chlorine, bromine or iodine.

(q) "Trihalomethane" (THM) means one of the family of organic compounds, named as derivatives of methane, wherein three of the four hydrogen atoms in methane are each substituted by a halogen atom in the molecular structure.

(r) "Total trihalomethanes" (TTHM) means the sum of the concentration in milligrams per liter of the trihalomethane compounds (trichloromethane [chloroform], dibromochloromethane, bromodichloromethane and tribromomethane [bromoform]), rounded to two significant figures.

(s) "Maximum Total Trihalomethane Potential (MTP)" means the maximum concentration of total trihalomethanes produced in a given water containing a disinfectant residual after 7 days at a temperature of 25° C or above.

(t) "Disinfectant" means any oxidant, including but not limited to chlorine, chlorine dioxide, chloramines, and ozone added to water in any part of the treatment or distribution proc-

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ess, that is intended to kill or inactivate pathogenic microorganisms.

[40 FR 59570, Dec. 24, 1975, as amended at 41 FR 28403, July 9, 1976; 44 FR 68641, Nov. 29, 1979]

§ 141.3 Coverage.

This part shall apply to each public water system, unless the public water system meets all of the following conditions:

(a) Consists only of distribution and storage facilities (and does not have any collection and treatment facilities);

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

(c) Does not sell water to any person; and

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

§ 141.4 Variances and exemptions.

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

§ 141.5 Siting requirements.

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

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

(b) Except for intake structures, is within the floodplain of a 100-year flood or is lower than any recorded high tide where appropriate records exist. The U.S. Environmental Protection Agency will not seek to override land use decisions affecting public

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water systems siting which are made at the State or local government levels.

§ 141.6 Effective dates.

(a) Except as provided in paragraph (b) of this section, the regulations set forth in this part shall take effect on June 24, 1977.

(b) The regulations for total trihalomethanes set forth in § 141.12(c) shall take effect 2 years after the date of promulgation of these regulations for community water systems serving 75,000 or more individuals, and 4 years after the date of promulgation for communities serving 10,000 to 74,999 individuals.

[44 FR 68641, Nov. 29, 1979]

Subpart B—Maximum Contaminant levels

§ 141.11 Maximum contaminant levels for inorganic chemicals.

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

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

Contaminant	Level, milligrams per liter
Arsenic.....	0.05
Barium.....	1.
Cadmium.....	0.010
Chromium.....	0.05
Lead.....	0.05
Mercury.....	0.002
Nitrate (as N).....	10
Selenium.....	0.01
Silver.....	0.05

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

Temperature Degrees Fahrenheit	Degrees Celsius	Level, milligrams per liter
53.7 and below	12.0 and below	2.4
53.8 to 58.3	12.1 to 14.6	2.2
58.4 to 63.8	14.7 to 17.6	2.0
63.9 to 70.6	17.7 to 21.4	1.8
70.7 to 79.2	21.5 to 26.2	1.6
79.3 to 90.5	26.3 to 32.5	1.4

§ 141.12 Maximum contaminant levels for organic chemicals.

The following are the maximum contaminant levels for organic chemicals. The maximum contaminant levels for organic chemicals in paragraphs (a) and (b) of this section apply to all community water systems. Compliance with the maximum contaminant levels in paragraphs (a) and (b) is calculated pursuant to § 141.24. The maximum contaminant level for total trihalomethanes in paragraph (c) of this section applies only to community water systems which serve a population of 10,000 or more individuals and which add a disinfectant (oxidant) to the water in any part of the drinking water treatment process. Compliance with the maximum contaminant level for total trihalomethanes is calculated pursuant to § 141.30.

	Level, milligrams per liter
(a) Chlorinated hydrocarbons:	
Endrin (1,2,3,4,10, 10-hexachloro-6, 7-epoxy- 1,4, 4a,5,6,7,8,81-octahydro-1,4-endo, endo-5,8-dimethano naphthalene).	0.0002
Lindane (1,2,3,4,5,6-hexachlorocyclo- hexane, gamma isomer).	0.004
Methoxychlor (1,1,1-Trichloro-2, 2-bis [p- methoxyphenyl] ethane).	0.1
Toxaphene (C ₁₂ H ₁₀ Cl ₇ -Technical chlorinated camphene, 67-69 percent chlorine).	0.005
(b) Chlorophenoxy:	
2,4-D, (2,4-Dichlorophenoxyacetic acid)	0.1
2,4,5-TP Silvex (2,4,5-Trichlorophenoxypropionic acid).	0.01
(c) Total trihalomethanes (the sum of the concentrations of bromodichloromethane, dibromochloromethane, tribromomethane (bromofom) and trichloromethane (chlorofom))	
	0.10 mg/l.

[40 FR 59570, Dec. 24, 1975, as amended at 44 FR 68641, Nov. 29, 1979]

§ 141.13 Maximum contaminant levels for turbidity.

The maximum contaminant levels for turbidity are applicable to both community water systems and non-community water systems using surface water sources in whole or in part. The maximum contaminant levels for turbidity in drinking water, measured at a representative entry point(s) to the distribution system, are:

(a) One turbidity unit (TU), as determined by a monthly average pursuant to § 141.22, except that five or fewer turbidity units may be allowed if the supplier of water can demonstrate to the State that the higher turbidity does not do any of the following:

- (1) Interfere with disinfection;
- (2) Prevent maintenance of an effective disinfectant agent throughout the distribution system; or
- (3) Interfere with microbiological determinations.

(b) Five turbidity units based on an average for two consecutive days pursuant to § 141.22.

§ 141.14 Maximum microbiological contaminant levels.

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

(a) When the membrane filter technique pursuant to § 141.21(a) is used, the number of coliform bacteria shall not exceed any of the following:

(1) One per 100 milliliters as the arithmetic mean of all samples examined per month pursuant to § 141.21 (b) or (c);

(2) Four per 100 milliliters in more than one sample when less than 20 are examined per month; or

(3) Four per 100 milliliters in more than five percent of the samples when 20 or more are examined per month.

(b) (1) When the fermentation tube method and 10 milliliter standard portions pursuant to § 141.21(a) are used, coliform bacteria shall not be present in any of the following:

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(i) More than 10 percent of the portions in any month pursuant to § 141.21 (b) or (c);

(ii) Three or more portions in more than one sample when less than 20 samples are examined per month; or

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

(2) When the fermentation tube method and 100 milliliter standard portions pursuant to § 141.21(a) are used, coliform bacteria shall not be present in any of the following:

(i) More than 60 percent of the portions in any month pursuant to § 141.21 (b) or (c);

(ii) Five portions in more than one sample when less than five samples are examined per month; or

(iii) Five portions in more than 20 percent of the samples when five or more samples are examined per month.

(c) For community or non-community systems that are required to sample at a rate of less than 4 per month, compliance with paragraph (a), (b)(1), or (b)(2) of this section shall be based upon sampling during a 3 month period, except that, at the discretion of the State, compliance may be based upon sampling during a one-month period.

§ 141.15 Maximum contaminant levels for radium-226, radium-228, and gross alpha particle radioactivity in community water systems.

The following are the maximum contaminant levels for radium-226, radium-228, and gross alpha particle radioactivity:

(a) Combined radium-226 and radium-228—5 pCi/1.

(b) Gross alpha particle activity (including radium-226 but excluding radon and uranium)—15 pCi/1.

[41 FR 28404, July 9, 1976]

§ 141.16 Maximum contaminant levels for beta particle and photon radioactivity from man-made radionuclides in community water systems.

(a) The average annual concentration of beta particle and photon radioactivity from man-made radionuclides in drinking water shall not produce an

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annual dose equivalent to the total body or any internal organ greater than 4 millirem/year.

(b) Except for the radionuclides listed in Table A, the concentration of man-made radionuclides causing 4 mrem total body or organ dose equivalents shall be calculated on the basis of a 2 liter per day drinking water intake using the 168 hour data listed in "Maximum Permissible Body Burdens and Maximum Permissible Concentration of Radionuclides in Air or Water for Occupational Exposure," NBS Handbook 69 as amended August 1963, U.S. Department of Commerce. If two or more radionuclides are present, the sum of their annual dose equivalent to the total body or to any organ shall not exceed 4 millirem/year.

TABLE A.—Average annual concentrations assumed to produce a total body or organ dose of 4 mrem/yr

Radionuclide	Critical organ	pCi per liter
Tritium.....	Total body.....	20,000
Strontium-90.....	Bone marrow.....	8

[41 FR 28404, July 9, 1976]

Subpart C—Monitoring and Analytical Requirements

§ 141.21 Microbiological contaminant sampling and analytical requirements.

(a) Suppliers of water for community water systems and non-community water systems shall analyze for coliform bacteria for the purpose of determining compliance with § 141.14. Analyses shall be conducted in accordance with the analytical recommendations set forth in "Standard Methods for the Examination of Water and Wastewater," American Public Health Association, 13th Edition, pp. 662-688, except that a standard sample size shall be employed. The standard sample used in the membrane filter procedure shall be 100 milliliters. The standard sample used in the 5 tube most probable number (MPN) procedure (fermentation tube method) shall be 5 times the standard portion. The standard portion is either 10 milliliters

or 100 milliliters as described in § 141.14(b) and (c). The samples shall be taken at points which are representative of the conditions within the distribution system.

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

Population served	Minimum number of samples per month
25 to 1,000.....	1
1,001 to 2,500.....	2
2,501 to 3,300.....	3
3,301 to 4,100.....	4
4,101 to 4,900.....	5
4,901 to 5,800.....	6
5,801 to 6,700.....	7
6,701 to 7,600.....	8
7,601 to 8,500.....	9
8,501 to 9,400.....	10
9,401 to 10,300.....	11
10,301 to 11,100.....	12
11,101 to 12,000.....	13
12,001 to 12,900.....	14
12,901 to 13,700.....	15
13,701 to 14,600.....	16
14,601 to 15,500.....	17
15,501 to 16,300.....	18
16,301 to 17,200.....	19
17,201 to 18,100.....	20
18,101 to 18,900.....	21
18,901 to 19,800.....	22
19,801 to 20,700.....	23
20,701 to 21,500.....	24
21,501 to 22,300.....	25
22,301 to 23,200.....	26
23,201 to 24,000.....	27
24,001 to 24,900.....	28
24,901 to 25,000.....	29
25,001 to 28,000.....	30
28,001 to 33,000.....	35
33,001 to 37,000.....	40
37,001 to 41,000.....	45
41,001 to 46,000.....	50
46,001 to 50,000.....	55
50,001 to 54,000.....	60
54,001 to 59,000.....	65
59,001 to 64,000.....	70
64,001 to 70,000.....	75
70,001 to 76,000.....	80
76,001 to 83,000.....	85
83,001 to 90,000.....	90
90,001 to 96,000.....	95
96,001 to 111,000.....	100
111,001 to 130,000.....	110
130,001 to 160,000.....	120
160,001 to 190,000.....	130
190,001 to 220,000.....	140
220,001 to 250,000.....	150
250,001 to 290,000.....	160
290,001 to 320,000.....	170
320,001 to 360,000.....	180
360,001 to 410,000.....	190
410,001 to 450,000.....	200
450,001 to 500,000.....	210
500,001 to 550,000.....	220
550,001 to 600,000.....	230
600,001 to 660,000.....	240

Population served	Minimum number of samples per month
660,001 to 720,000.....	250
720,001 to 780,000.....	260
780,001 to 840,000.....	270
840,001 to 910,000.....	280
910,001 to 970,000.....	290
970,001 to 1,050,000.....	300
1,050,001 to 1,140,000.....	310
1,140,001 to 1,230,000.....	320
1,230,001 to 1,320,000.....	330
1,320,001 to 1,420,000.....	340
1,420,001 to 1,520,000.....	350
1,520,001 to 1,630,000.....	360
1,630,001 to 1,730,000.....	370
1,730,001 to 1,850,000.....	380
1,850,001 to 1,970,000.....	390
1,970,001 to 2,060,000.....	400
2,060,001 to 2,270,000.....	410
2,270,001 to 2,510,000.....	420
2,510,001 to 2,750,000.....	430
2,750,001 to 3,020,000.....	440
3,020,001 to 3,320,000.....	450
3,320,001 to 3,620,000.....	460
3,620,001 to 3,960,000.....	470
3,960,001 to 4,310,000.....	480
4,310,001 to 4,690,000.....	490
4,690,001 or more.....	500

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

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

(d) (1) When the coliform bacteria in a single sample exceed four per 100 milliliters (§ 141.14(a)), at least two consecutive daily check samples shall be collected and examined from the same sampling point. Additional check samples shall be collected daily, or at a frequency established by the State, until the results obtained from at least two consecutive check samples show

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less than one coliform bacterium per 100 milliliters.

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

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

(4) The location at which the check samples were taken pursuant to paragraph (d) (1), (2), or (3) of this section shall not be eliminated from future sampling without approval of the State. The results from all coliform bacterial analyses performed pursuant to this subpart, except those obtained from check samples and special purpose samples, shall be used to determine compliance with the maximum contaminant level for coliform bacteria as established in § 141.14. Check samples shall not be included in calculating the total number of samples taken each month to determine compliance with § 141.21 (b) or (c).

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

(f) When a maximum contaminant level set forth in paragraph (a), (b) or (c) of § 141.14 is exceeded, the supplier of water shall report to the State and notify the public as prescribed in § 141.31 and § 141.32.

(g) Special purpose samples, such as those taken to determine whether disinfection practices following pipe placement, replacement, or repair

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have been sufficient, shall not be used to determine compliance with § 141.14 or § 141.21 (b) or (c).

(h) A supplier of water of a community water system or a non-community water system may, with the approval of the State and based upon a sanitary survey, substitute the use of chlorine residual monitoring for not more than 75 percent of the samples required to be taken by paragraph (b) of this section, *Provided*, That the supplier of water takes chlorine residual samples at points which are representative of the conditions within the distribution system at the frequency of at least four for each substituted microbiological sample. There shall be at least daily determinations of chlorine residual. When the supplier of water exercises the option provided in this paragraph (h) of this section, he shall maintain no less than 0.2 mg/l free chlorine throughout the public water distribution system. When a particular sampling point has been shown to have a free chlorine residual less than 0.2 mg/l, the water at that location shall be retested as soon as practicable and in any event within one hour. If the original analysis is confirmed, this fact shall be reported to the State within 48 hours. Also, if the analysis is confirmed, a sample for coliform bacterial analysis must be collected from that sampling point as soon as practicable and preferably within one hour, and the results of such analysis reported to the State within 48 hours after the results are known to the supplier of water. Analyses for residual chlorine shall be made in accordance with "Standard Methods for the Examination of Water and Wastewater," 13th Ed., pp. 129-132. Compliance with the maximum contaminant levels for coliform bacteria shall be determined on the monthly mean or quarterly mean basis specified in § 141.14, including those samples taken as a result of failure to maintain the required chlorine residual level. The State may withdraw its approval of the use of chlorine residual substitution at any time.

§ 141.22 Turbidity sampling and analytical requirements.

(a) Samples shall be taken by suppliers of water for both community water systems and non-community water systems at a representative entry point(s) to the water distribution system at least once per day, for the purpose of making turbidity measurements to determine compliance with § 141.13. The measurement shall be made by the Nephelometric Method in accordance with the recommendations set forth in "Standard Methods for the Examination of Water and Wastewater," American Public Health Association, 13th Edition, pp. 350-353, or "Methods for Chemical Analysis of Water and Wastes," pp. 295-298, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

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

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

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

§ 141.23 Inorganic chemical sampling and analytical requirements.

(a) Analyses for the purpose of determining compliance with § 141.11 are required as follows:

(1) Analyses for all community water systems utilizing surface water sources shall be completed within one year fol-

lowing the effective date of this part. These analyses shall be repeated at yearly intervals.

(2) Analyses for all community water systems utilizing only ground water sources shall be completed within two years following the effective date of this part. These analyses shall be repeated at three-year intervals.

(3) For non-community water systems, whether supplied by surface or ground water sources, analyses for nitrate shall be completed within two years following the effective date of this part. These analyses shall be repeated at intervals determined by the State.

(b) If the result of an analysis made pursuant to paragraph (a) of this section indicates that the level of any contaminant listed in § 141.11 exceeds the maximum contaminant level, the supplier of water shall report to the State within 7 days and initiate three additional analyses at the same sampling point within one month.

(c) When the average of four analyses made pursuant to paragraph (b) of this section, rounded to the same number of significant figures as the maximum contaminant level for the substance in question, exceeds the maximum contaminant level, the supplier of water shall notify the State pursuant to § 141.31 and give notice to the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until the maximum contaminant level has not been exceeded in two successive samples or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(d) The provisions of paragraphs (b) and (c) of this section notwithstanding, compliance with the maximum contaminant level for nitrate shall be determined on the basis of the mean of two analyses. When a level exceeding the maximum contaminant level for nitrate is found, a second analysis shall be initiated within 24 hours, and if the mean of the two analyses exceeds the maximum contaminant level, the supplier of water shall report his findings to the State pursu-

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ant to § 141.31 and shall notify the public pursuant to § 141.32.

(e) For the initial analyses required by paragraph (a)(1), (2) or (3) of this section, data for surface waters acquired within one year prior to the effective date and data for ground waters acquired within 3 years prior to the effective date of this part may be substituted at the discretion of the State.

(f) Analyses conducted to determine compliance with § 141.11 shall be made in accordance with the following methods:

(1) Arsenic—Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," pp. 95-96, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(2) Barium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes," pp. 97-98, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(3) Cadmium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes," pp. 101-103, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(4) Chromium—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes," pp. 105-106, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(5) Lead—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes," pp. 112-113, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(6) Mercury—Flameless Atomic Absorption Method, "Methods for

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Chemical Analysis of Water and Wastes," pp. 118-126, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(7) Nitrate—Brucine Colorimetric Method, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 461-464, or Cadmium Reduction Method, "Methods for Chemical Analysis of Water and Wastes," pp. 201-206, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(8) Selenium—Atomic Absorption Method, "Methods for Chemical Analysis of Water and Wastes," p. 145, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(9) Silver—Atomic Absorption Method, "Standard Methods for the Examination of Water and Wastewater", 13th Edition, pp. 210-215, or "Methods for Chemical Analysis of Water and Wastes", p. 146, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

(10) Fluoride—Electrode Method, "Standard Methods for the Examination of Water and Wastewater", 13th Edition, pp. 172-174, or "Methods for Chemical Analysis of Water and Wastes," pp. 65-67, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974, or Colorimetric Method with Preliminary Distillation, "Standard Methods for the Examination of Water and Wastewater," 13th Edition, pp. 171-172 and 174-176, or "Methods for Chemical Analysis of Water and Wastes," pp. 59-60, Environmental Protection Agency, Office of Technology Transfer, Washington, D.C. 20460, 1974.

§ 141.24 Organic chemicals other than total trihalomethanes, sampling and analytical requirements.

(a) An analysis of substances for the purpose of determining compliance with § 141.12(a) and § 141.12(b) shall be made as follows:

(1) For all community water systems utilizing surface water sources, analyses shall be completed within one year

following the effective date of this part. Samples analyzed shall be collected during the period of the year designated by the State as the period when contamination by pesticides is most likely to occur. These analyses shall be repeated at intervals specified by the State but in no event less frequently than at three year intervals.

(2) For community water systems utilizing only ground water sources, analyses shall be completed by those systems specified by the State.

(b) If the result of an analysis made pursuant to paragraph (a) of this section indicates that the level of any contaminant listed in § 141.24 (a) and (b) exceeds the maximum contaminant level, the supplier of water shall report to the State within 7 days and initiate three additional analyses within one month.

(c) When the average of four analyses made pursuant to paragraph (b) of this section, rounded to the same number of significant figures as the maximum contaminant level for the substance in question, exceeds the maximum contaminant level, the supplier of water shall report to the State pursuant to § 141.31 and give notice to the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until the maximum contaminant level has not been exceeded in two successive samples or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(d) For the initial analysis required by paragraphs (a) (1) and (2) of this section, data for surface water acquired within one year prior to the effective date of this part and data for ground water acquired within three years prior to the effective date of this part may be substituted at the discretion of the State.

(e) Analyses made to determine compliance with § 141.12(a) shall be made in accordance with "Method for Organochlorine Pesticides in Industrial Effluents," MDQARL, Environmental Protection Agency, Cincinnati, Ohio, November 28, 1973.

(f) Analyses made to determine compliance with § 141.12(b) shall be con-

ducted in accordance with "Methods for Chlorinated Phenoxy Acid Herbicides in Industrial Effluents," MDQARL, Environmental Protection Agency, Cincinnati, Ohio, November 28, 1973.

[40 FR 59570, Dec. 24, 1975, as amended at 44 FR 68641, Nov. 29, 1979]

§ 141.25 Analytical Methods for Radioactivity.

(a) The methods specified in *Interim Radiochemical Methodology for Drinking Water*, Environmental Monitoring and Support Laboratory, EPA-600/4-75-008, USEPA, Cincinnati, Ohio 45268, or those listed below, are to be used to determine compliance with §§ 141.15 and 141.16 (radioactivity) except in cases where alternative methods have been approved in accordance with § 141.27.

(1) Gross Alpha and Beta—Method 302 "Gross Alpha and Beta Radioactivity in Water" *Standard Methods for the Examination of Water and Wastewater*, 13th Edition, American Public Health Association, New York, N.Y., 1971.

(2) Total Radium—Method 304 "Radium in Water by Precipitation" Ibid.

(3) Radium-226—Method 305 "Radium-226 by Radon in Water" Ibid.

(4) Strontium-89,90 — Method 303 "Total Strontium and Strontium-90 in Water" Ibid.

(5) Tritium—Method 306 "Tritium in Water" Ibid.

(6) Cesium-134 — ASTM D-2459 "Gamma Spectrometry in Water," 1975 *Annual Book of ASTM Standards, Water and Atmospheric Analysis*, Part 31, American Society for Testing and Materials, Philadelphia, PA. (1975).

(7) Uranium—ASTM D-2907 "Microquantities of Uranium in Water by Fluorometry," Ibid.

(b) When the identification and measurement of radionuclides other than those listed in paragraph (a) of this section is required, the following references are to be used, except in cases where alternative methods have been approved in accordance with § 141.27.

(1) *Procedures for Radiochemical Analysis of Nuclear Reactor Aqueous*

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Solutions, H. L. Krieger and S. Gold, EPA-R4-73-014. USEPA, Cincinnati, Ohio, May 1973.

(2) *HASL Procedure Manual*, Edited by John H. Harley. HASL 300, ERDA Health and Safety Laboratory, New York, N.Y., 1973.

(c) For the purpose of monitoring radioactivity concentrations in drinking water, the required sensitivity of the radioanalysis is defined in terms of a detection limit. The detection limit shall be that concentration which can be counted with a precision of plus or minus 100 percent at the 95 percent confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).

(1) To determine compliance with § 141.15(a) the detection limit shall not exceed 1 pCi/l. To determine compliance with § 141.15(b) the detection limit shall not exceed 3 pCi/l.

(2) To determine compliance with § 141.16 the detection limits shall not exceed the concentrations listed in Table B.

TABLE B.—DETECTION LIMITS FOR MAN-MADE BETA PARTICLE AND PHOTON EMITTERS

Radionuclide	Detection limit
Tritium	1,000 pCi/l.
Strontium-89	10 pCi/l.
Strontium-90	2 pCi/l.
Iodine-131	1 pCi/l.
Cesium-134	10 pCi/l.
Gross beta	4 pCi/l.
Other radionuclides	1/10 of the applicable limit.

(d) To judge compliance with the maximum contaminant levels listed in §§ 141.15 and 141.16, averages of data shall be used and shall be rounded to the same number of significant figures as the maximum contaminant level for the substance in question.

[41 FR 28404, July 9, 1976]

§ 141.26 Monitoring Frequency for Radioactivity in Community Water Systems.

(a) Monitoring requirements for gross alpha particle activity, radium-226 and radium-228.

(1) Initial sampling to determine compliance with § 141.15 shall begin within two years of the effective date of these regulations and the analysis shall be completed within three years of the effective date of these regulations. Compliance shall be based on the analysis of an annual composite of

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four consecutive quarterly samples or the average of the analyses of four samples obtained at quarterly intervals.

(i) A gross alpha particle activity measurement may be substituted for the required radium-226 and radium-228 analysis *Provided*, That the measured gross alpha particle activity does not exceed 5 pCi/l at a confidence level of 95 percent (1.65σ where σ is the standard deviation of the net counting rate of the sample). In localities where radium-228 may be present in drinking water, it is recommended that the State require radium-226 and/or radium-228 analyses when the gross alpha particle activity exceeds 2 pCi/l.

(ii) When the gross alpha particle activity exceeds 5 pCi/l, the same or an equivalent sample shall be analyzed for radium-226. If the concentration of radium-226 exceeds 3 pCi/l the same or an equivalent sample shall be analyzed for radium-228.

(2) For the initial analysis required by paragraph (a)(1) of this section, data acquired within one year prior to the effective date of this part may be substituted at the discretion of the State.

(3) Suppliers of water shall monitor at least once every four years following the procedure required by paragraph (a)(1) of this section. At the discretion of the State, when an annual record taken in conformance with paragraph (a)(1) of this section has established that the average annual concentration is less than half the maximum contaminant levels established by § 141.15, analysis of a single sample may be substituted for the quarterly sampling procedure required by paragraph (a)(1) of this section.

(i) More frequent monitoring shall be conducted when ordered by the State in the vicinity of mining or other operations which may contribute alpha particle radioactivity to either surface or ground water sources of drinking water.

(ii) A supplier of water shall monitor in conformance with paragraph (a)(1) of this section within one year of the introduction of a new water source for a community water system. More frequent monitoring shall be conducted

when ordered by the State in the event of possible contamination or when changes in the distribution system or treatment processing occur which may increase the concentration of radioactivity in finished water.

(iii) A community water system using two or more sources having different concentrations of radioactivity shall monitor source water, in addition to water from a free-flowing tap, when ordered by the State.

(iv) Monitoring for compliance with § 141.15 after the initial period need not include radium-228 *except when* required by the State, *Provided*, That the average annual concentration of radium-228 has been assayed at least once using the quarterly sampling procedure required by paragraph (a)(1) of this section.

(v) Suppliers of water shall conduct annual monitoring of any community water system in which the radium-226 concentration exceeds 3 pCi/l, when ordered by the State.

(4) If the average annual maximum contaminant level for gross alpha particle activity or total radium as set forth in § 141.15 is exceeded, the supplier of a community water system shall give notice to the State pursuant to § 141.31 and notify the public as required by § 141.32. Monitoring at quarterly intervals shall be continued until the annual average concentration no longer exceeds the maximum contaminant level or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(b) Monitoring requirements for manmade radioactivity in community water systems.

(1) Within two years of the effective date of this part, systems using surface water sources and serving more than 100,000 persons and such other community water systems as are designated by the State shall be monitored for compliance with § 141.16 by analysis of a composite of four consecutive quarterly samples or analysis of four quarterly samples. Compliance with § 141.16 may be assumed without further analysis if the average annual concentration of gross beta particle activity is less than 50 pCi/l and if the average annual concentrations of tri-

tium and strontium-90 are less than those listed in Table A, *Provided*, That if both radionuclides are present the sum of their annual dose equivalents to bone marrow shall not exceed 4 millirem/year.

(i) If the gross beta particle activity exceeds 50 pCi/l, an analysis of the sample must be performed to identify the major radioactive constituents present and the appropriate organ and total body doses shall be calculated to determine compliance with § 141.16.

(ii) Suppliers of water shall conduct additional monitoring, as ordered by the State, to determine the concentration of man-made radioactivity in principal watersheds designated by the State.

(iii) At the discretion of the State, suppliers of water utilizing only ground waters may be required to monitor for man-made radioactivity.

(2) For the initial analysis required by paragraph (b)(1) of this section data acquired within one year prior to the effective date of this part may be substituted at the discretion of the State.

(3) After the initial analysis required by paragraph (b)(1) of this section suppliers of water shall monitor at least every four years following the procedure given in paragraph (b)(1) of this section.

(4) Within two years of the effective date of these regulations the supplier of any community water system designated by the State as utilizing waters contaminated by effluents from nuclear facilities shall initiate quarterly monitoring for gross beta particle and iodine-131 radioactivity and annual monitoring for strontium-90 and tritium.

(i) Quarterly monitoring for gross beta particle activity shall be based on the analysis of monthly samples or the analysis of a composite of three monthly samples. The former is recommended. If the gross beta particle activity in a sample exceeds 15 pCi/l, the same or an equivalent sample shall be analyzed for strontium-89 and cesium-134. If the gross beta particle activity exceeds 50 pCi/l, an analysis of the sample must be performed to identify the major radioactive constituents present and the appropriate

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organ and total body doses shall be calculated to determine compliance with § 141.16.

(ii) For iodine-131, a composite of five consecutive daily samples shall be analyzed once each quarter. As ordered by the State, more frequent monitoring shall be conducted when iodine-131 is identified in the finished water.

(iii) Annual monitoring for strontium-90 and tritium shall be conducted by means of the analysis of a composite of four consecutive quarterly samples or analysis of four quarterly samples. The latter procedure is recommended.

(iv) The State may allow the substitution of environmental surveillance data taken in conjunction with a nuclear facility for direct monitoring of manmade radioactivity by the supplier of water where the State determines such data is applicable to a particular community water system.

(5) If the average annual maximum contaminant level for man-made radioactivity set forth in § 141.16 is exceeded, the operator of a community water system shall give notice to the State pursuant to § 141.31 and to the public as required by § 141.32. Monitoring at monthly intervals shall be continued until the concentration no longer exceeds the maximum contaminant level or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

[41 FR 28404, July 9, 1976]

§ 141.27 Alternative analytical techniques.

With the written permission of the State, concurred in by the Administrator of the U.S. Environmental Protection Agency, an alternative analytical technique may be employed. An alternative technique shall be acceptable only if it is substantially equivalent to the prescribed test in both precision and accuracy as it relates to the determination of compliance with any maximum contaminant level. The use of the alternative analytical technique shall not decrease the frequency of monitoring required by this part.

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§ 141.28 Approved laboratories.

For the purpose of determining compliance with § 141.21 through § 141.27, samples may be considered only if they have been analyzed by a laboratory approved by the State except that measurements for turbidity and free chlorine residual may be performed by any person acceptable to the State.

§ 141.29 Monitoring of consecutive public water systems.

When a public water system supplies water to one or more other public water systems, the State may modify the monitoring requirements imposed by this part to the extent that the interconnection of the systems justifies treating them as a single system for monitoring purposes. Any modified monitoring shall be conducted pursuant to a schedule specified by the State and concurred in by the Administrator of the U.S. Environmental Protection Agency.

§ 141.30 Total trihalomethanes sampling, analytical and other requirements.

(a) Community water system which serve a population of 10,000 or more individuals and which add a disinfectant (oxidant) to the water in any part of the drinking water treatment process shall analyze for total trihalomethanes in accordance with this section. For systems serving 75,000 or more individuals, sampling and analyses shall begin not later than 1 year after the date of promulgation of this regulation. For systems serving 10,000 to 74,999 individuals, sampling and analyses shall begin not later than 3 years after the date of promulgation of this regulation. For the purpose of this section, the minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples. All samples taken within an established frequency shall be collected within a 24-hour period.

(b)(1) For all community water systems utilizing surface water sources in whole or in part, and for all community water systems utilizing only ground water sources that have not been determined by the State to qualify for the monitoring requirements of paragraph (c) of this section, analyses for total trihalomethanes shall be performed at quarterly intervals on at least four water samples for each treatment plant used by the system. At least 25 percent of the samples shall be taken at locations within the distribution system reflecting the maximum residence time of the water in the system. The remaining 75 percent shall be taken at representative locations in the distribution system, taking into account number of persons served, different sources of water and different treatment methods employed. The results of all analyses per quarter shall be arithmetically averaged and reported to the State within 30 days of the system's receipt of such results. Results shall also be reported to EPA until such monitoring requirements have been adopted by the State. All samples collected shall be used in the computation of the average, unless the analytical results are invalidated for technical reasons. Sampling and analyses shall be conducted in accordance with the methods listed in paragraph (e) of this section.

(2) Upon the written request of a community water system, the monitoring frequency required by paragraph (b)(1) of this section may be reduced by the State to a minimum of one sample analyzed for TTHMs per quarter taken at a point in the distribution system reflecting the maximum residence time of the water in the system, upon a written determination by the State that the data from at least 1 year of monitoring in accordance with paragraph (b)(1) of this section and local conditions demonstrate that total trihalomethane concentrations will be consistently below the maximum contaminant level.

(3) If at any time during which the reduced monitoring frequency prescribed under this paragraph applies, the results from any analysis exceed 0.10 mg/l of TTHMs and such results are confirmed by at least one check

sample taken promptly after such results are received, or if the system makes any significant change to its source of water or treatment program, the system shall immediately begin monitoring in accordance with the requirements of paragraph (b)(1) of this section, which monitoring shall continue for at least 1 year before the frequency may be reduced again. At the option of the State, a system's monitoring frequency may and should be increased above the minimum in those cases where it is necessary to detect variations of TTHM levels within the distribution system.

(c)(1) Upon written request to the State, a community water system utilizing only ground water sources may seek to have the monitoring frequency required by subparagraph (1) of paragraph (b) of this section reduced to a minimum of one sample for maximum TTHM potential per year for each treatment plant used by the system taken at a point in the distribution system reflecting maximum residence time of the water in the system. The system shall submit to the State the results of at least one sample analyzed for maximum TTHM potential for each treatment plant used by the system taken at a point in the distribution system reflecting the maximum residence time of the water in the system. The system's monitoring frequency may only be reduced upon a written determination by the State that, based upon the data submitted by the system, the system has a maximum TTHM potential of less than 0.10 mg/l and that, based upon an assessment of the local conditions of the system, the system is not likely to approach or exceed the maximum contaminant level for total TTHMs. The results of all analyses shall be reported to the State within 30 days of the system's receipt of such results. Results shall also be reported to EPA until such monitoring requirements have been adopted by the State. All samples collected shall be used for determining whether the system must comply with the monitoring requirements of paragraph (b) of this section, unless the analytical results are invalidated for technical reasons. Sampling and analyses shall be conducted in ac-

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cordance with the methods listed in paragraph (e) of this section.

(2) If at any time during which the reduced monitoring frequency prescribed under paragraph (c)(1) of this section applies, the results from any analysis taken by the system for maximum TTHM potential are equal to or greater than 0.10 mg/l, and such results are confirmed by at least one check sample taken promptly after such results are received, the system shall immediately begin monitoring in accordance with the requirements of paragraph (b) of this section and such monitoring shall continue for at least one year before the frequency may be reduced again. In the event of any significant change to the system's raw water or treatment program, the system shall immediately analyze an additional sample for maximum TTHM potential taken at a point in the distribution system reflecting maximum residence time of the water in the system for the purpose of determining whether the system must comply with the monitoring requirements of paragraph (b) of this section. At the option of the State, monitoring frequencies may and should be increased above the minimum in those cases where this is necessary to detect variation of TTHM levels within the distribution system.

(d) Compliance with § 141.12(c) shall be determined based on a running annual average of quarterly samples collected by the system as prescribed in subparagraphs (1) or (2) of paragraph (b) of this section. If the average of samples covering any 12 month period exceeds the Maximum Contaminant Level, the supplier of water shall report to the State pursuant to § 141.31 and notify the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(e) Sampling and analyses made pursuant to this section shall be conducted by one of the following EPA approved methods:

(1) "The Analysis of Trihalomethanes in Drinking Waters by the Purge

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and Trap Method," Method 501.1, EMSL, EPA Cincinnati, Ohio.

(2) "The Analysis of Trihalomethanes in Drinking Water by Liquid/Liquid Extraction," Method 501.2, EMSL, EPA Cincinnati, Ohio.

Samples for TTHM shall be dechlorinated upon collection to prevent further production of Trihalomethanes, according to the procedures described in the above two methods. Samples for maximum TTHM potential should not be dechlorinated, and should be held for seven days at 25° C (or above) prior to analysis, according to the procedures described in the above two methods.

(f) Before a community water system makes any significant modifications to its existing treatment process for the purposes of achieving compliance with § 141.12(c), such system must submit and obtain State approval of a detailed plan setting forth its proposed modification and those safeguards that it will implement to ensure that the bacteriological quality of the drinking water served by such system will not be adversely affected by such modification. Each system shall comply with the provisions set forth in the State-approved plan. At a minimum, a State approved plan shall require the system modifying its disinfection practice to:

(1) Evaluate the water system for sanitary defects and evaluate the source water for biological quality;

(2) Evaluate its existing treatment practices and consider improvements that will minimize disinfectant demand and optimize finished water quality throughout the distribution system;

(3) Provide baseline water quality survey data of the distribution system. Such data should include the results from monitoring for coliform and fecal coliform bacteria, fecal streptococci, standard plate counts at 35° C and 20° C, phosphate, ammonia nitrogen and total organic carbon. Virus studies should be required where source waters are heavily contaminated with sewage effluent;

(4) Conduct additional monitoring to assure continued maintenance of optimal biological quality in finished water, for example, when chloramines

are introduced as disinfectants or when pre-chlorination is being discontinued. Additional monitoring should also be required by the State for chlorate, chlorite and chlorine dioxide when chlorine dioxide is used. Standard plate count analyses should also be required by the State as appropriate before and after any modifications;

(5) Consider inclusion in the plan of provisions to maintain an active disinfectant residual throughout the distribution system at all times during and after the modification.

[44 FR 68641, Nov. 29, 1979, as amended at 45 FR 15545, 15547, Mar. 11, 1980]

APPENDIX A—SUMMARY OF PUBLIC COMMENTS AND EPA RESPONSES ON PROPOSED AMENDMENTS TO THE NATIONAL INTERIM PRIMARY DRINKING WATER REGULATIONS FOR CONTROL OF TRIHALOMETHANES IN DRINKING WATER

[44 FR 68642, Nov. 29, 1979]

APPENDIX B—SUMMARY OF MAJOR COMMENTS (FOR RESPONSES, SEE APPENDIX A)

[44 FR 68666, Nov. 29, 1979]

EDITORIAL NOTE: At 44 FR 68642 and 68666, Appendices A and B were published in the FEDERAL REGISTER but are not being codified in the Code of Federal Regulations.

APPENDIX C—ANALYSIS OF TRIHALOMETHANES

PART I: THE ANALYSIS OF TRIHALOMETHANES IN DRINKING WATER BY THE PURGE AND TRAP METHOD

1. Scope

1.1 This method (1) is applicable in the determination of four trihalomethanes, i.e. chloroform, dichlorobromomethane, dibromochloromethane, and bromoform in finished drinking water, raw source water, or drinking water in any stage of treatment. The concentration of these four compounds is totaled to determine total trihalomethanes (TTHM).

1.2 For compounds other than the above-mentioned trihalomethanes, or for other sample sources, the analyst must demonstrate the usefulness of the method by collecting precision and accuracy data on actual samples as described (2).

1.3 Although the actual detection limits are highly dependent upon the

gas chromatographic column and detector employed, the method can be used over a concentration range of approximately 0.5 to 1500 micrograms per liter.

1.4 Well in excess of 100 different water supplies have been analyzed using this method. Supplementary analyses using gas chromatography mass spectrometry (GC/MS) have shown that there is no evidence of interference in the determination of trihalomethanes (3). For this reason, it is not necessary to analyze the raw source water as is required with the Liquid/Liquid Extraction Method (4).

2. Summary

2.2 Trihalomethanes are extracted by an inert gas which is bubbled through the aqueous sample. The trihalomethanes, along with other organic constituents which exhibit low water solubility and a vapor pressure significantly greater than water, are efficiently transferred from the aqueous phase to the gaseous phase. These compounds are swept from the purging device and are trapped in a short column containing a suitable sorbent. After a predetermined period of time, the trapped components are thermally desorbed and backflushed onto the head of a gas chromatographic column and separated under programmed conditions. Measurement is accomplished with a halogen specific detector such as electrolytic conductivity or microcoulometric titration.

2.3 Confirmatory analyses are performed using dissimilar columns, or by mass spectrometry (5).

2.4 Aqueous standards and unknowns are extracted and analyzed under identical conditions in order to compensate for extraction losses.

2.5 The total analysis time, assuming the absence of other organohalides, is approximately 35 minutes per sample.

3. Interferences

3.1 Impurities contained in the purge gas and organic compounds outgassing from the plumbing ahead of the trap usually account for the majority of contamination problems. The presence of such interferences are easily monitored as a part of the quality control program. Sample blanks are normally run between each set of

samples. When a positive trihalomethane response is noted in the sample blank, the analyst should analyze a method blank. Method blanks are run by charging the purging device with organic-free water and analyzing in the normal manner.

If any trihalomethane is noted in the method blank in excess of $0.4 \mu\text{g/l}$, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. *Subtracting the blank values is not recommended.* The use of non-TFE plastic tubing, non-TFE thread sealants, or flow controllers with rubber components should be avoided since such materials generally out-gas organic compounds which will be concentrated in the trap during the purge operation. Such out-gasing problems are common whenever new equipment is put into service; as time progresses, minor out-gasing problems generally cure themselves.

3.2 Several instances of accidental sample contamination have been noted and attributed to diffusion of volatile organics through the septum seal and into the sample during shipment and storage. The sample blank is used as a monitor for this problem.

3.3 For compounds that are not efficiently purged, such as bromoform, small variations in sample volume, purge time, purge flow rate, or purge temperature can affect the analytical result. Therefore, samples and standards must be analyzed under identical conditions.

3.4 Cross-contamination can occur whenever high-level and low-level samples are sequentially analyzed. To reduce this likelihood, the purging device and sample syringe should be rinsed twice between samples with organic-free water. Whenever an unusually concentrated sample is encountered, it is highly recommended that it be followed by a sample blank analysis to ensure that sample cross contamination does not occur. For samples containing large amounts of water soluble materials, it may be necessary to wash out the purging device with a soap solution, rinse with distilled water, and then dry in a 105°C oven between analyses.

3.5 Qualitative misidentifications are a problem in using gas chromatographic analysis.

Whenever samples whose qualitative nature is unknown are analyzed, the following precautionary measures should be incorporated into the analysis.

3.5.1 Perform duplicate analyses using the two recommended columns (4.2.1 and 4.2.2) which provide different retention order and retention times for the trihalomethanes and other organohalides.

3.5.2 Whenever possible, use GC/MS techniques which provide unequivocal qualitative identifications (5).

4. Apparatus

4.1 The purge and trap equipment consists of three separate pieces of apparatus: the purging device, trap, and desorber. Construction details for a purging device and an easily automated trap-desorber hybrid which has proven to be exceptionally efficient and reproducible are shown in Figures 1 through 4 and described in 4.1.1. through 4.1.3. An earlier acceptable version of the above-mentioned equipment is described in (1).

4.1.1 Purging Device—Construction details are given in Figure 1 for an all-glass 5 ml purging device. The glass frit installed at the base of the sample chamber allows finely divided gas bubbles to pass through the sample while the sample is restrained above the frit. Gaseous volumes above the sample are kept to a minimum to eliminate dead volume effects, yet allowing sufficient space for most foams to disperse. The inlet and exit ports are constructed from heavy-walled $\frac{1}{4}$ -inch glass tubing so that leak-free removable connections can be made using "finger-tight" compression fittings containing Teflon ferrules. The removable foam trap is used to control samples that foam.

4.1.2 Trapping Device—The trap (Figure 2) is a short gas chromatographic column which at $<35^\circ\text{C}$ retards the flow of the compounds of interest while venting the purge gas and, depending on which sorbent is used, much of the water vapor. The trap should be constructed with a low thermal mass so that it can be heated to 180°C in less than 1 minute for efficient desorption, then rapidly cooled to room temperature for recycling. Variations in the trap ID, wall thick-

ness, sorbents, sorbent packing order, and sorbent mass could adversely affect the trapping and desorption efficiencies for compounds discussed in this text. For this reason, it is important to faithfully reproduce the trap configurations recommended in Figure 2. Traps containing Tenax only, or combinations of Tenax and other sorbents are acceptable for this analysis.

4.1.3 Desorb assembly—Details for the desorb are shown in Figures 3, and 4. With the 6-port valve in the Purge Sorb position (Figure 3), the effluent from the purging device passes through the trap where the flow rate of the organics is retarded. The GC carrier gas also passes through the 6-port valve and is returned to the GC. With the 6-port valve in the Purge-Sorb position, the operation of the GC is in no way impaired; therefore, routine liquid injection analyses can be performed using the gas chromatograph. After the sample has been purged, the 6-port valve is turned to the desorb position (Figure 4). In this configuration the trap is coupled in series with the gas chromatographic column allowing the carrier gas to backflush the trapped materials into the analytical column. Just as the valve is actuated, the power is turned on to the resistance wire wrapped around the trap. The power is supplied by an electronic temperature controller. Using this device, the trap is rapidly heated to 180° C and then maintained at 180° C with minimal temperature overshoot. The trapped compounds are released as a "plug" to the gas chromatograph. Normally, packed columns with theoretical efficiencies near 500 plates/foot under programmed temperature conditions can accept such desorb injections without altering peak geometry. Substituting a non-controlled power supply, such as a manually-operated variable transformer, will provide nonreproducible retention times and poor quantitative data unless Injection Procedure (8.9.2) is used.

4.1.4 Several Purge and Trap Devices are now commercially available. It is recommended that the following be taken into consideration if a unit is to be purchased:

a. Be sure that the unit is completely compatible with the gas chromatograph to be used for the analysis.

b. Use a 5-ml purging device similar to that shown in Figure 1.

c. Be sure the Tenax portion of the trap meets or exceeds the dimensions shown in Figure 2.

d. With the exception of sample introduction, select a unit that has as many of the purge trap functions automated as possible.

4.2 Gas chromatograph—The chromatograph must be temperature programmable and equipped with a halide specific detector.

4.2.1 Column I is an unusually efficient column which provides outstanding separations for a wide variety of organic compounds. Because of its ability to resolve trihalomethanes from other organochlorine compounds, column I should be used as the primary analytical column (see Table 1 for retention data using this column).

4.2.1.1 Column I parameters: Dimensions—8 feet long x 0.1 inch ID stainless steel or glass tubing. Packing—1% SP-1000 on Carbopack-B (60/80) mesh. Carrier Gas—helium at 40 ml/minute. Temperature program sequence: 45° C isothermal for 3 minutes, program at 8° C/minute to 220° C then hold for 15 minutes or until all compounds have eluted.

NOTE.—It has been found that during handling, packing, and programming, active sites are exposed on the Carbopack-B packing. This results in tailing peak geometry and poor resolution of many constituents. To correct this, pack the first 5 cm of the column with 3% SP-1000 on Chromosorb-W 60/80 followed by the Carbopack-B packing. Condition the precolumn and the Carbopack columns with carrier gas flow at 220° C overnight. Pneumatic shocks and rough treatment of packed columns will cause excessive fracturing of the Carbopack. If pressure in excess of 60 psi is required to obtain 40 ml/minute carrier flow, then the column should be repacked.

4.2.1.2 Acceptable column equivalent to Column I: Dimensions—8 feet long x 0.1 inch ID stainless steel or glass tubing. Packing—0.2% Carbowax 1500 on Carbopack-C (80/100) mesh. Carrier Gas—helium at 40 ml/minute. Temperature program sequence—60° C isothermal for 3 minutes, program at

App. C

8° C /minute to 160° C, then hold for 2 minutes or until all compounds have eluted.

NOTE.—It has been found that during handling, packing, and programming, active sites are exposed on the Carbopack-C packing. This results in poor resolution of constituents and poor peak geometry. To correct this, place a 1 ft. 0.125 in. OD x 0.1 in. ID stainless steel column packed with 3% Carbowax 1500 on Chromosorb-W 60/80 mesh in series before the Carbopack-C column. Condition the precolumn and the Carbopack columns with carrier gas flow at 190° C overnight. The two columns may be retained in series for routine analyses. Trihalomethane retention times are listed in Table 1.

4.2.2 Column II provides unique organohalide-trihalomethane separations when compared to those obtained from Column I (see Figures 5 and 6). However, since the resolution between various compounds is generally not as good as those with Column I, it is recommended that Column II be used as a qualitative confirmatory column for unknown samples when GC/MS confirmation is not possible.

4.2.2.1 Column II parameters: Dimensions—6 feet long x 0.1 inch ID stainless steel or glass. Packing—n-octane on Porisil-C (100/120 mesh). Carrier Gas—helium at 40 cc/minute. Temperature program sequence—50° C isothermal for 3 minutes, program at 6°/minute to 170° C, then hold for 4 minutes or until all compounds have eluted. Trihalomethane retention times are listed in Table 1.

5.8 Organic-free water is defined as water free of interference when employed in the purge and trap analysis.

5.8.1 Organic-free water is generated by passing tap water through a carbon filter bed containing about 1 lb. of activated carbon. Change the activated carbon bed whenever the concentration of any trihalomethane exceeds 0.4 µg/l.

5.8.2 A Millipore Super-Q Water System or its equivalent may be used to generate organic-free water.

5.8.3 Organic-free water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90° C, bubble a contaminant-free inert gas through the water for one hour. While still hot, transfer the water to a

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narrow-mouth screw-cap bottle with a Teflon seal.

5.8.4 Test organic free water each day it is used by analyzing according to Section 8.

5.9 Standards.*

5.9.1 Bromoform—96%—available from Aldrich Chemical Company.

5.9.2 Bromodichloromethane 97%—available from Aldrich Chemical Company.

5.9.3 Chlorodibromomethane—available from Columbia Chemical Inc., Columbia, S.C.

5.9.4 Chloroform—99%—available from Aldrich Chemical Company.

5.10 Standard Stock Solutions

5.10.1 Place about 9.8 ml of methyl alcohol into a ground glass stoppered 10 ml volumetric flask.

5.10.2 Allow the flask to stand unstoppered about 10 minutes or until all alcohol wetted surfaces have dried.

5.10.3 Weigh the flask to the nearest 0.1 mg.

5.10.4 Using a 100 µl syringe, immediately add 2 drops of the reference standard to the flask, then reweigh. *Be sure that the 2 drops fall directly into the alcohol without contacting the neck of the flask.*

5.10.5 Dilute to volume, stopper, then mix by inverting the flask several times.

5.10.6 Transfer the solution to a dated and labeled 15 ml screw cap bottle with a Teflon cap liner.

NOTE.—Because of the toxicity of trihalomethanes, it is necessary to prepare primary dilutions in a hood. It is further recommended that a NIOSH/MESA approved toxic gas respirator be used when the analyst handles high concentrations of such materials.

5.10.7 Calculate the concentration in micrograms per microliter from the net gain in weight.

5.10.8 Store the solution at 4° C.

NOTE.—All standard solutions prepared in methyl alcohol are stable up to 4 weeks when stored under these conditions. They should be discarded after that time has elapsed.

5.11 Aqueous Calibration Standard Precautions.

*As a precautionary measure, all standards must be checked for purity by boiling point determinations or GC/MS assays (5).

5.11.1 In order to prepare accurate aqueous standard solutions, the following precautions must be observed.

a. Do not inject more than 20 μ l of alcoholic standards into 100 ml of organic-free water.

b. Use of 25 μ l Hamilton 702N microsyringe or equivalent. (Variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water.)

c. Rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as fast as possible after injection.

d. Mix aqueous standards by inverting the flask three times only.

e. Discard the contents contained in the neck of the flask. Fill the sample syringe from the standard solution contained in the expanded area of the flask as directed in Section 3.5.

f. Never use pipets to dilute or transfer samples or aqueous standards.

g. Aqueous standards when stored with a headspace are not stable and should be discarded after one hour.

h. Aqueous standards can be stored according to Sections 6.4 and 8.6.

5.11.2 Prepare, from the standard stock solutions, secondary dilution mixtures in methyl alcohol so that a 20 μ l injection into 100 ml of organic-free water will generate a calibration standard which produces a response close ($\pm 10\%$) to that of the sample (See 9.1).

5.11.3 Purge and analyze the aqueous calibration standards in the same manner as the samples.

5.11.4 Other calibration procedures (3) which require the delivery of less than 20 μ l of a methanolic standard into a 5.0 ml volume of water already contained in the sample syringe are acceptable only if the methanolic standard is delivered by the solvent flush technique (6).

5.12 Quality Check Standard (2.0 μ g/l)

5.12.1 From the standard stock solutions, prepare a secondary dilution in methyl alcohol containing 10 ng/ μ l of each trihalomethane (See Section 5.10.8 Note).

5.12.2 Daily, inject 20.0 μ l of this mixture into 100.0 ml of organic-free

water and analyze according to Section 8.

6. Sample Collection and Handling

6.1. The sample containers should have a total volume of at least 25 ml.

6.1.1 Narrow mouth screw cap bottles with the TFE fluorocarbon face silicone septa cap liners are strongly recommended.

6.2 Sample Bottle Preparation

6.2.1 Wash all sample bottles and TFE seals in detergent. Rinse with tap water and finally with distilled water.

6.2.2 Allow the bottles and seals to air dry at room temperature, then place in a 105° C oven for one hour, then allow to cool in a area known to be free of organics.

NOTE.—Do not heat the TFE seals for extended period of time (>1 hour) because the silicone layer slowly degrades at 105° C.

6.2.3 When cool, seal the bottles using the TFE seals that will be used for sealing the samples.

6.3 Sample Stabilization—A chemical reducing agent (Section 5.6) is added to the sample in order to arrest the formation of trihalo-methanes after sample collection (3, 7). *Do not add the reducing agent to samples when data on maximum trihalomethane formation is desired.* If chemical stabilization is employed, the reagent is also added to the blanks. The chemical agent (2.5 to 3 mg/40 ml) is added to the empty sample bottles just prior to shipping to the sampling site.

6.4 Sample Collection

6.4.1 Collect all samples in duplicate.

6.4.2 Fill the sample bottles in such a manner that no air bubbles pass through the sample as the bottle is filled.

6.4.3 Seal the bottles so that no air bubbles are entrapped in it.

6.4.4 Maintain the hermetic seal on the sample bottle until analysis.

6.4.5 Sampling from a water tap.

6.4.5.1 Turn on water and allow the system to flush until the temperature of the water has stabilized. Adjust the flow to about 500 ml/minute and collect duplicate samples from the flowing stream.

6.4.6 Sampling from an open body of water.

6.4.6.1 Fill a 1-quart wide-mouth bottle with sample from a representa-

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tive area. Carefully fill duplicate sample bottles from the 1-quart bottle as noted in 6.4.2.

6.4.7 If a chemical reducing agent has been added to the sample bottles, fill with sample just to overflowing, seal the bottle, and shake vigorously for 1 minute.

6.4.8 Sealing practice for septum seal screw cap bottles.

6.4.8.1 Open the bottle and fill to overflowing, place on a level surface, position the TFE side of the septum seal upon the convex sample meniscus and seal the bottle by screwing the cap on tightly.

6.4.8.2 Invert the sample and lightly tap the cap on a solid surface. The absence of entrapped air indicates a successful seal. If bubbles are present, open the bottle, add a few additional drops of sample and reseal the bottle as above.

6.4.9 Blanks.

6.4.9.1 Prepare blanks in duplicate at the laboratory by filling and sealing sample bottles with organic-free water just prior to shipping the sample bottles to the sampling site.

6.4.9.2 If the sample is to be stabilized, add an identical amount of stabilization reagent to the blanks.

6.4.9.3 Ship the blanks to and from the sampling site along with the sample bottles.

6.4.9.4 Store the blanks and the samples collected at a given site (sample set) together. A sample set is defined as all the samples collected at a given site (i.e., at a water treatment plant, the duplicate raw source waters, the duplicate finished waters and the duplicate blank samples comprise the sample set).

6.5 When samples have been collected according to Section 6, no measurable loss of trihalomethanes has been detected over extended periods of storage time (3). It is recommended that all samples be analyzed within 14 days of collection.

7. Conditioning Traps

7.1 Condition newly packed traps overnight at 180° C with an inert gas flow of at least 20 ml/min.

7.1.1 Vent the trap effluent to the room, not to the analytical column.

7.2 Prior to daily use, condition traps 10 minutes while backflushing at

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180° C. It may be beneficial to routinely condition traps overnight while backflushing at 180° C.

7.2.1 The trap may be vented to the analytical column; however, after conditioning, the column must be programmed prior to use.

8. Extraction and Analysis

8.1 Adjust the purge gas (nitrogen or helium) flow rate to 40 ml/min.

8.2 Attach the trap inlet to the purging device. Turn the valve to the purge-sorb position (Figure 3).

8.3 Open the syringe valve located on the purging device sample introduction needle.

8.4 Remove the plungers from two 5 ml syringes and attach a closed syringe valve to each.

8.5 Open the sample bottle and carefully pour the sample into one of the syringe barrels until it overflows. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 ml. Close the valve.

8.6 Fill the second syringe in an identical manner from the same sample bottle. This second syringe is reserved for a duplicate analysis, if necessary (See Sections 9.3 and 9.4).

8.7 Attach the syringe-valve assembly to the syringe valve on the purging device.

8.8 Open the syringe valve and inject the sample into the purging chamber. Close both valves. Purge the sample for $11.0 \pm .05$ minutes.

8.9 After the 11-minute purge time, attach the trap to the chromatograph (turn the valve to the desorb position) and introduce the trapped materials to the GC column by rapidly heating the trap to 180°C while backflushing the trap with an inert gas between 20 and 60 ml/min for 4 minutes.

8.9.1 If the trap can be rapidly heated to 180°C and maintained at this temperature, the GC analysis can begin as the sample is desorbed, i.e., the column is at the initial 45°C operating temperature. The equipment described in Figure 4 will perform accordingly.

8.9.2 With other types of equipment (see Section 4.1.4 and Reference 1) where the trap is not rapidly heated or is not heated in a reproducible

manner, it may be necessary to transfer the contents of the trap into the analytical column at $<30^{\circ}\text{C}$ where it is once again trapped. Once the transfer is complete (4 minutes), the column is rapidly heated to the initial operating temperature for analysis.

8.9.3 If injection procedure 8.9.1 is used and the early eluting peaks in the resulting chromatogram have poor geometry or variable retention times, then Section 8.9.2 should be used.

8.10 After the extracted sample is introduced into the gas chromatograph, empty the gas purging device using the sample introduction syringe, followed by two 5-ml flushes of organic-free water. When the purging device is emptied, leave the syringe valve open allowing the purge gas to vent through the sample introduction needle.

8.11 Analyze each sample and sample blank from the sample set in an identical manner (see Section 6.4.9.4) on the same day.

8.12 Prepare calibration standards from the standard stock solutions (Section 5.10) in organic-free water that are close to the unknown in trihalomethane composition and concentration (Section 9.1). The concentrations should be such that only 20 μl or less of the secondary dilution need be added to 100 ml of organic-free water to produce a standard at the same level as the unknown.

8.13 As an alternative to Section 8.12, prepare a calibration curve for each trihalomethane containing at least 3 points, two of which must bracket the unknown.

9. Analytical Quality Control

9.1 Analyze the 2 $\mu\text{g/l}$ check sample daily before any samples are analyzed. Instrument status checks and lower limit of detection estimations based upon response factor calculations at five times the noise level are obtained from these data. In addition, response factor data obtained from the 2 $\mu\text{g/l}$ check standard can be used to estimate the concentration of the unknowns. From this information, the appropriate standard dilutions can be determined.

9.2 Analyze the sample blank to monitor for potential interferences as described in Sections 3.1, 3.2, and 3.4.

9.3 Spiked Samples

9.3.1 For laboratories analyzing more than 10 samples a day, each 10th sample should be a laboratory generated spike which closely duplicates the average finished drinking water in trihalomethane composition and concentration. Prepare the spiked sample in organic-free water as described in Section 5.11.

9.3.2 For laboratories analyzing less than 10 samples daily, each time the analysis is performed, analyze at least 1 laboratory generated spike sample which closely duplicates the average finished drinking water in trihalomethane composition and concentration. Prepare the spiked sample in organic-free water as described in Section 5.11.

9.4 Randomly select and analyze 10% of all samples in duplicate.

9.4.1 Analyze all samples in duplicate which appear to deviate more than 30% from any established norm.

9.5 Maintain an up-to-date log on the accuracy and precision data collected in Sections 9.3 and 9.4. If results are significantly different than those cited in Section 11.1, the analyst should check out the entire analyses scheme to determine why the laboratory's precision and accuracy limits are greater.

9.6 Quarterly, spike an EMSL-Cincinnati trihalomethane quality control sample into organic-free water and analyze.

9.6.1 The results of the EMSL trihalomethane quality control sample should agree within 20% of the true value for each trihalomethane. If they do not then the analyst must check each step in the standard generation procedure to solve the problem (Section 5.9, 5.10, and 5.11).

9.7 Maintain a record of the retention times for each trihalomethane using data gathered from spiked samples and standards.

9.7.1 Daily calculate the average retention time for each trihalomethane and the variance encountered for the analyses.

9.7.2 If individual trihalomethane retention time varies by more than 10% over an eight hour period or does not fall with 10% of an established norm, the system is "out of control."

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The source of retention data variation must be corrected before acceptable data can be generated.

10. Calculations

10.1 Locate each trihalomethane in the sample chromatogram by comparing the retention time of the suspect peak to the data gathered in 9.7.1. The retention time of the suspect peak must fall within the limits established in 9.7.1 for single column identification.

10.2 Calculate the concentration of the samples by comparing the peak height or peak areas of the samples to the standard peak height (8.12). Round off the data to the nearest $\mu\text{g/l}$ or two significant figures.

$$\mu\text{g/l} = \frac{\text{peak height sample}}{\text{peak height standard}} \times (\text{conc. std. } \mu\text{g/l})$$

10.3 Report the results obtained from the lower limit of detection estimates along with the data for the samples.

10.4 Calculate the total trihalomethane concentration (TTHM) by summing the 4 individual trihalomethane concentrations in $\mu\text{g/l}$. TTHM ($\mu\text{g/l}$) = (Conc. CHCl_3) + (Conc. CHBrCl_2) + (Conc. CHBr_2Cl) + (Conc. CHBr_3).

10.5 Calculate the limit of detection (LOD) for each trihalomethane not detected using the following criteria:

$$\text{LOD } (\mu\text{g/l}) = \frac{A \times \text{ATT}}{B \times \text{ATT}} \quad (2 \mu\text{g/l})$$

where B = peak height (mm) of 2 $\mu\text{g/l}$ quality check standard

A = 5 times the noise level in (mm) at the exact retention time of the trihalomethane or the baseline displacement in (mm) from the theoretical zero at the exact retention time of the trihalomethane.

ATT = Attenuation factor

11. Accuracy and Precision

11.1 One liter of organic-free water was spiked with the trihalomethanes and used to fill septum seal vials which were stored under ambient conditions. The spiked samples were ran-

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domly analyzed over a 2-week period of time. The single laboratory data listed in Table II reflect the errors due to the analytical procedure and storage.

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TABLE I—Retention Data for Trihalomethanes

Trihalomethane	Retention time minutes		
	Column I 1% sp1000 Carbopack B	Acceptable Alternative	Column II n-octane Porasil-C
		to column I 0.4% Carbowax Carbopack	
Chloroform	10.7	8.2	12.2
Bromodichloromethane	13.7	10.8	14.7
Chlorodibromomethane (Dibromochloromethane)	16.5	13.2	16.6
Bromoform	19.2	15.7	19.2

TABLE II—Single Laboratory Accuracy and Precision for Trihalomethanes

Spike μg/l	Number samples	Mean μg/l	Precision standard deviation	Accuracy percent recovery
Chloroform				
1.2	12	1.2	0.14	100
12.0	8	11.	0.16	92
119.0	11	105	7.9	88
Bromodichloromethane				
1.6	12	1.5	0.05	94
16.0	8	15.	0.39	94
160.0	11	145.	10.2	91

TABLE II—Single Laboratory Accuracy and Precision for Trihalomethanes—Continued

Spike μg/l	Number samples	Mean μg/l	Precision standard deviation	Accuracy percent recovery
Chlorodibromomethane				
2.0	12	1.9	0.09	95
20.0	8	19.	0.70	95
196.0	11	185.	10.6	94
Bromoform				
2.3	12	2.3	0.16	100
23.0	8	23.	1.38	100
231.0	11	223	16.3	97

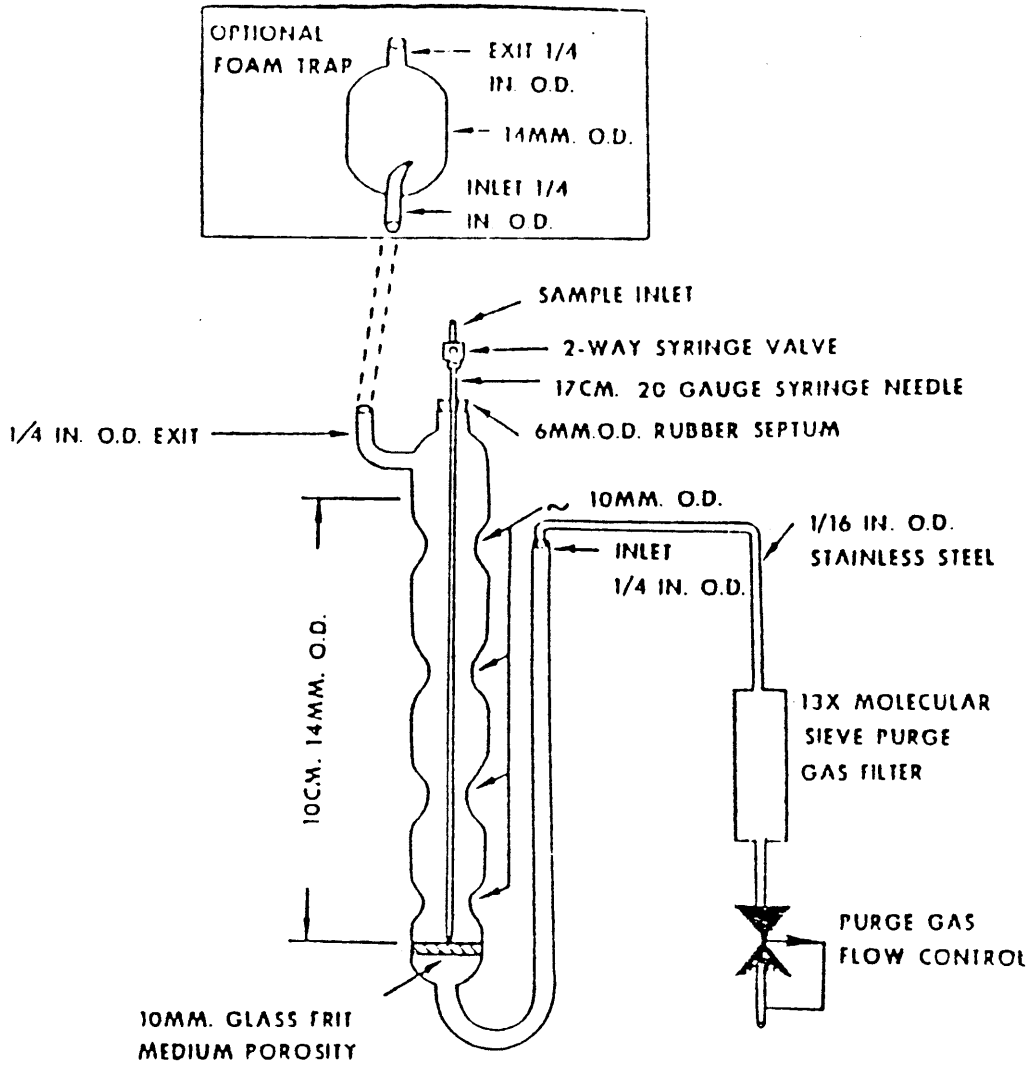


FIGURE 1. PURGING DEVICE

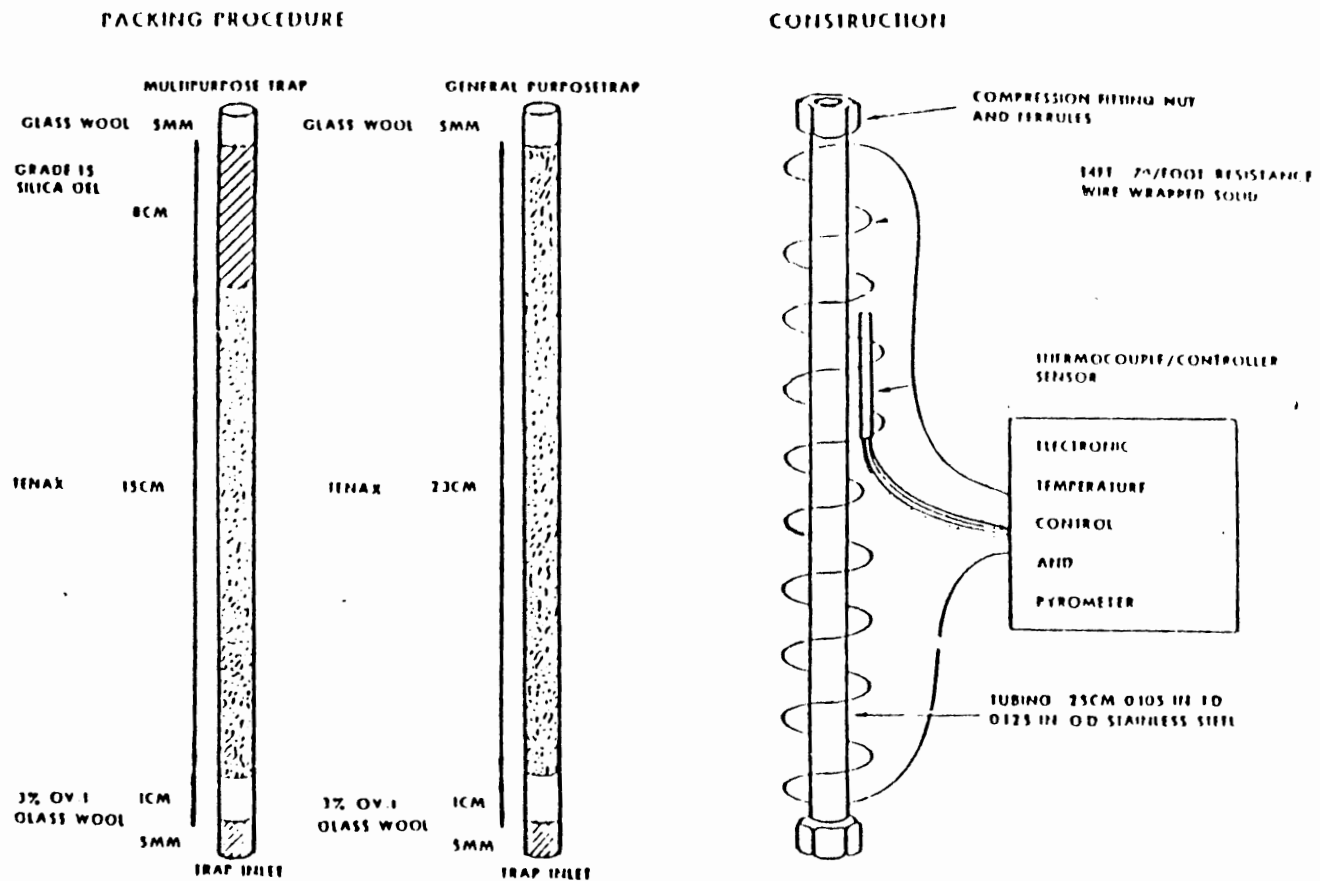


FIGURE 2 TRAP

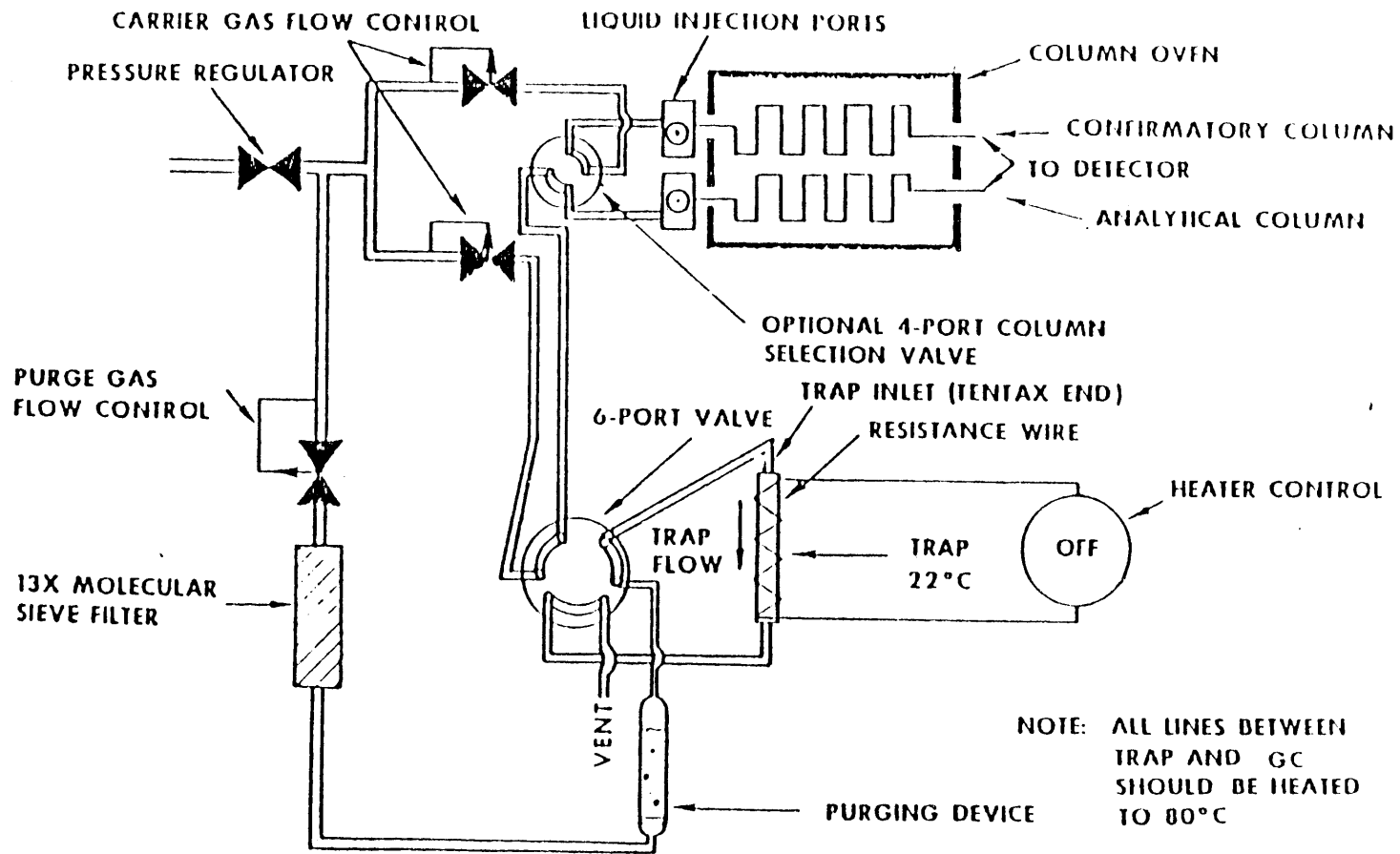


FIGURE 3 PURGE-TRAP SYSTEM (PURGE-SORB MODE)

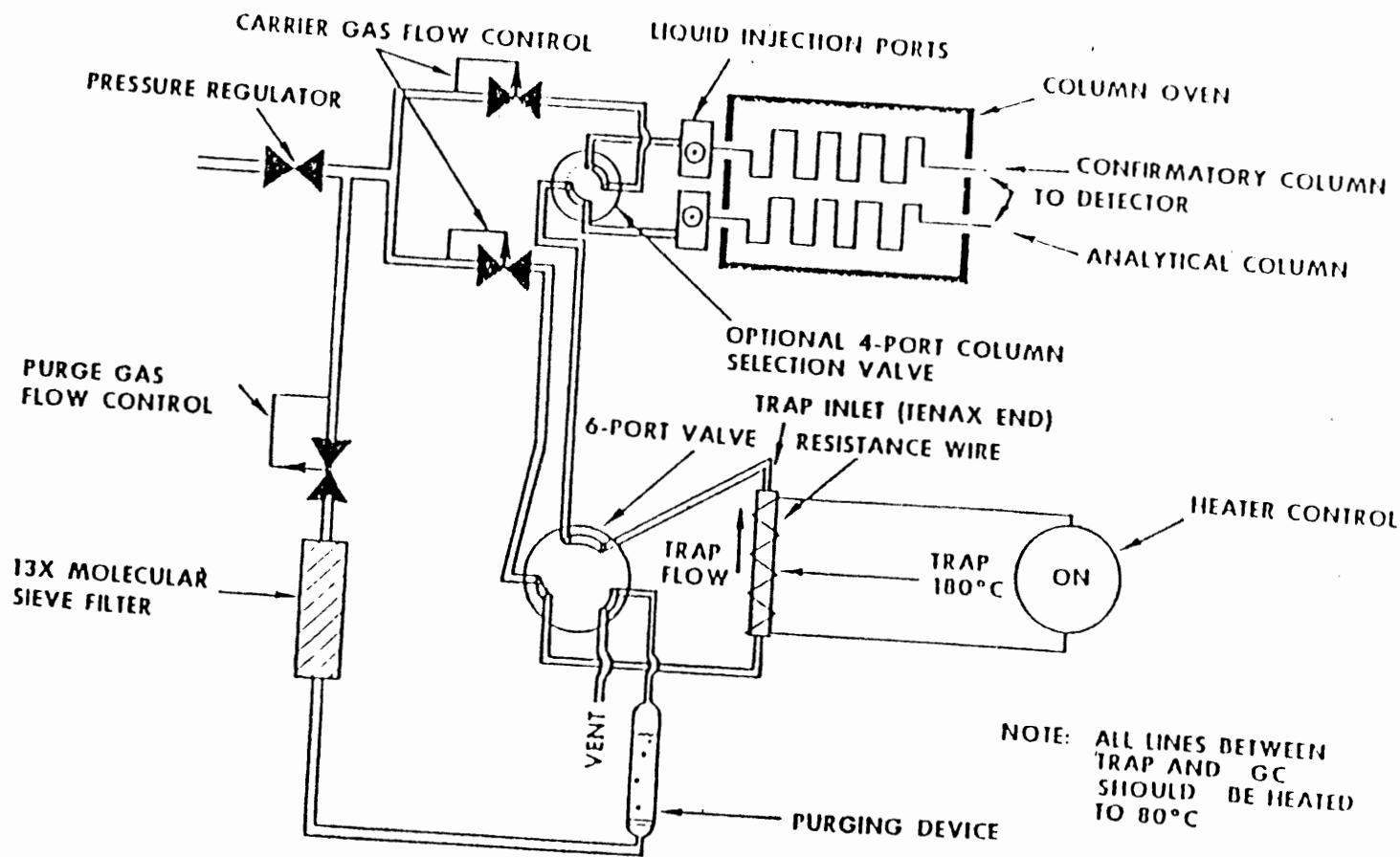


FIGURE 4 PURGE-TRAP SYSTEM (DESORB MODE)

PART II: ANALYSIS OF
TRIHALOMETHANES IN DRINKING WATER
BY LIQUID/LIQUID EXTRACTION

1. Scope.

1.1 This method (1,2) is applicable only to the determination of four trihalomethanes, i.e., chloroform, bromodichloromethane, chlorodibromomethane, and bromoform in finished drinking water, drinking water during intermediate stages of treatment, and the raw source water.

1.2 For compounds other than the above-mentioned trihalomethanes, or for other sample sources, the analyst must demonstrate the usefulness of the method by collecting precision and accuracy data on actual samples as described in (3) and provide qualitative confirmation of results by Gas Chromatography/Mass Spectrometry (GC/MS) (4).

1.3 Qualitative analyses using GC/MS or the purge and trap method (5) must be performed to characterize each raw source water if peaks appear as interferences in the raw source analysis.

1.4 The method has been shown to be useful for the trihalomethanes over a concentration range from approximately 0.5 to 200 $\mu\text{g/l}$. Actual detection limits are highly dependent upon the characteristics of the gas chromatographic system used.

2. Summary

2.1 Ten milliliters of sample are extracted one time with 2 ml of solvent. Three μl of the extract are then injected into a gas chromatograph equipped with a linearized electron capture detector for separation and analysis.

2.2 The extraction and analysis time is 10 to 50 minutes per sample depending upon the analytical conditions chosen. (See Table 1 and Figures 1, 2, and 3.)

2.3 Confirmatory evidence is obtained using dissimilar columns and temperature programming. When component concentrations are sufficiently high ($>50 \mu\text{g/l}$), halogen specific detectors may be employed for improved specificity.

2.4 Unequivocal confirmatory analyses at high levels ($>50 \mu\text{g/l}$) can be

performed using GC/MS in place of the electron capture detector. At levels below 50 $\mu\text{g/l}$, unequivocal confirmation can only be performed by the purge and trap technique using GC/MS (4, 5).

2.5 Standards dosed into organic free water and the samples are extracted and analyzed in an identical manner in order to compensate for possible extraction losses.

2.6 The concentration of each trihalomethane is summed and reported as total trihalomethanes in $\mu\text{g/l}$.

3. Interferences

3.1 Impurities contained in the extracting solvent usually account for the majority of the analytical problems. Solvent blanks should be analyzed before a new bottle of solvent is used to extract samples. Indirect daily checks on the extracting solvent are obtained by monitoring the sample blanks (6.4.10). Whenever an interference is noted in the sample blank, the analyst should reanalyze the extracting solvent. The extraction solvent should be discarded whenever a high level ($>10 \mu\text{g/l}$) of interfering compounds are traced to it. Low level interferences generally can be removed by distillation or column chromatography (6); however, it is generally more economical to obtain a new source of solvent or select one of the approved alternative solvents listed in Section 5.1. Interference free solvent is defined as a solvent containing less than 0.4 $\mu\text{g/l}$ individual trihalomethane interference. Protect interference-free solvents by storing in a non-laboratory area known to be free of organochlorine solvents. *Subtracting blank values is not recommended.*

3.2 Several instances of accidental sample contamination have been attributed to diffusion of volatile organics through the septum seal on the sample bottle during shipment and storage. The sample blank (6.4.10) is used to monitor for this problem.

3.3 This liquid/liquid extraction technique efficiently extracts a wide boiling range of non-polar organic compounds and, in addition, extracts the polar organic components of the sample with varying efficiencies. In order to perform the trihalomethane analysis as rapidly as possible with

sensitivities in the low $\mu\text{g/l}$ range, it is necessary to use the semi-specific electron capture detector and chromatographic columns which have relatively poor resolving power. Because of these concessions, the probability of experiencing chromatographic interferences is high. Trihalomethanes are primarily products of the chlorination process and generally do not appear in the raw source water. The absence of peaks in the raw source water analysis with retention times similar to the trihalomethanes is generally adequate evidence of an interference-free finished drinking water analysis. Because of these possible interferences in addition to each finished drinking water analysis, a representative raw source water (6.4.5) must be analyzed. When potential interferences are noted in the raw source water analysis, the alternate chromatographic columns must be used to reanalyze the sample set. If interferences are still noted, qualitative identifications should be performed according to Sections 2.3 and 2.4. If the peaks are confirmed to be other than trihalomethanes and add significantly to the total trihalomethane value in the finished drinking water analysis, then the sample set must be analyzed by the purge and trap method (5).

4. Apparatus

4.1 Extraction vessel—A 15 ml total volume glass vessel with a Teflon lined screw-cap is required to efficiently extract the samples.

4.1.1 For samples that do not form emulsions 10 ml screw-cap flasks with a Teflon faced septum (total volume is ml) are recommended. Flasks and caps—Pierce—#13310 or equivalent. Septa—Teflon silicone—Pierce #12718 or equivalent.

4.1.2 For samples that form emulsions (turbid source water) 15 ml screw cap centrifuge tubes with a Teflon cap liner are recommended. Centrifuge tube—Corning 8062-15 or equivalent.

4.2 Sampling containers—40 ml screw cap sealed with Teflon faced silicone septa. Vials and caps—Pierce #13075 or equivalent. Septa—Pierce #12722 or equivalent.

4.3 Micro syringes—10, 100 μl .

4.4 Micro syringe—25 μl with a 2-inch by 0.006-inch needle—Hamilton 702N or equivalent.

4.5 Syringes—10 ml glass hypodermic with luerlok tip (2 each).

4.6 Syringe valve—2-way with luer ends (2 each)—Hamilton #86570—1FM1 or equivalent.

4.7 Pipette—2.0 ml transfer.

4.8 Glass stoppered volumetric flasks—10 and 100 ml.

4.9 Gas chromatograph with linearized electron capture detector. (Recommended option—temperature programmable. See Section 4.12.)

4.10 Column A—4 mm ID x 2m long glass packed with 3% SP-1000 on Supelcoport (100/120 mesh) operated at 50°C with 60 ml/min flow. (See Figure 1 for a sample chromatogram and Table 1 for retention data.)

4.11 Column B—2 mm ID x 2m long glass packed with 10% squalane on Chromosorb WAW (80/100 mesh) operated at 67°C with 25 ml/min flow. This column is recommended as the primary analytical column. Trichloroethylene, a common raw source water contaminate, coelutes with bromodichloromethane. (See Figure 2 for a sample chromatogram and Table 1 for retention data.)

4.12 Column C—2 mm ID x 3m long glass packed with 6% OV-11/4% SP-2100 on Supelcoport (100/120 mesh) temperature program 45°C for 12 minutes, then program at 1°/minute to 70°C with a 25 ml/min flow. (See Figure 3 for a sample chromatogram and Table I for retention data.)

4.13 Standard storage containers—15 ml amber screw-cap septum bottles with Teflon faced silicone septa. Bottles and caps—Pierce #19830 or equivalent. Septa—Pierce #12716 or equivalent.

5. Reagents

5.1 Extraction solvent—(See 3.1). Recommended—Pentane*. Alternates—

* Pentane has been selected as the best solvent for this analysis because it elutes, on all of the columns, well before any of the trihalomethanes. High altitudes or laboratory temperatures in excess of 75°F may make the use of this solvent impractical. For these reasons, alternative solvents are acceptable; however, the analyst may expect

Footnotes continued on next page

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tive—hexane, methylcyclohexane or 2,2,4-trimethylpentane.

5.2 Methyl alcohol—ACS Reagent Grade.

5.3 Free and combined chlorine reducing agents—Sodium thiosulfate ACS Reagent Grade—sodium sulfite ACS Reagent Grade.

5.4 Activated carbon—Filtrisorb—200, available from Calgon Corporation, Pittsburgh, PA, or equivalent.

5.5 Standards.^b

5.5.1 Bromoform 96%—available from Aldrich Chemical Company.

5.5.2 Bromodichloromethane 97%—available from Aldrich Chemical Company.

5.5.3 Chlorodibromomethane—available from Columbia Chemical, Incorporated, Columbia, S.C.

5.5.4 Chloroform 99%—available from Aldrich Chemical Company.

5.6 Organic-free water—Organic-free water is defined as water free of interference when employed in the procedure described herein.

5.6.1 Organic-free water is generated by passing tap water through a carbon filter bed containing carbon. Change the activated carbon whenever the concentration of any trihalomethane exceeds 0.4 µg/l.

5.6.2 A Millipore Super-Q Water System or its equivalent may be used to generate organic-free deionized water.

5.6.3 Organic-free water may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the temperature at 90° C, bubble a contaminant free inert gas through the water at 100 ml/minute for one hour. While still hot, transfer the water to a narrow mouth screw cap bottle with a Teflon seal.

5.6.4 Test organic free water each day it is used by analyzing it according to Section 7.

Footnotes continued from last page
rience baseline variances in the elution areas of the trihalomethanes due to coelution of these solvents. The degree of difficulty appears to be dependent upon the design and condition of the electron capture detector. Such problems should be insignificant when concentrations of the coeluting trihalomethane are in excess of 5 µg/l.

^bAs a precautionary measure, all standards must be checked for purity by boiling point determinations or GC/MS assays.

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5.7 Standard stock solutions.

5.7.1 Fill a 10.0 ml ground glass stoppered volumetric flask with approximately 9.8 ml of methyl alcohol.

5.7.2 Allow the flask to stand unstoppered about 10 minutes or until all alcohol wetted surfaces dry.

5.7.3 Weigh the unstoppered flask to the nearest 0.1 mg.

5.7.4 Using a 100 µl syringe, immediately add 2 to 3 drops of the reference standard to the flask, then reweigh. *Be sure that the reference standard falls directly into the alcohol without contacting the neck of the flask.*

5.7.5 Dilute to volume, stopper, then mix by inverting the flask several times.

5.7.6 Transfer the standard solution to a dated and labeled 15 ml screw-cap bottle with a Teflon cap liner.

NOTE.—Because of the toxicity of trihalomethanes, it is necessary to prepare primary dilutions in a hood. It is further recommended that a NIOSH/MESA-approved toxic gas respirator be used when the analyst handles high concentrations of such materials.

5.7.7 Calculate the concentration in micrograms per microliter from the net gain in weight.

5.7.8 Store the solution at 4° C.

NOTE.—All standard solutions prepared in methyl alcohol are stable up to 4 weeks when stored under these conditions. They should be discarded after that time has elapsed.

5.8 Aqueous calibration standard precautions.

5.8.1 In order to prepare accurate aqueous standard solutions, the following precautions must be observed:

a. Do not inject more than 20 µl of alcoholic standards into 100 ml of organic-free water.

b. Use a 25 µl Hamilton 702N microsyringe, or equivalent. (Variations in needle geometry will adversely affect the ability to deliver reproducible volumes of methanolic standards into water.)

c. Rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as fast as possible after injection.

d. Mix aqueous standards by inverting the flask three times only.

e. Discard the contents contained in the neck of the flask. Fill the sample syringe from the standard solution contained in the expanded area of the flask as directed in Section 7.

f. Never use pipets to dilute or transfer samples and aqueous standards.

g. Aqueous standards, when stored with a headspace, are not stable and should be discarded after one hour. Aqueous standards can be stored according to Sections 6.4.9 and 7.2.

5.9 Calibration standards.

5.9.1 Prepare, from the standard stock solutions, a multicomponent secondary dilution mixture in methyl alcohol so that a 20 μ l injection into 100 ml of organic-free water will generate a calibration standard which produces a response close ($\pm 25\%$) to that of the unknown. (See 8.1.)

5.9.2 Alternative calibration procedure.

5.9.2.1 Construct a calibration curve for each trihalomethane containing a minimum of 3 different concentrations. Two of the concentrations must bracket each unknown.

5.9.3 Extract and analyze the aqueous calibration standards in the same manner as the unknowns.

5.9.4 Other calibration procedures (7) which require the delivery of less than 20 μ l of methanolic standards to 10.0 ml volumes of water contained in the sample syringe are acceptable only if the methanolic standard is delivered by the solvent flush technique (8).

5.10 Quality Check Standard Mixture.

5.10.1 Prepare, from the standard stock solutions, a secondary dilution mixture in methyl alcohol that contains 10.0 ng/ μ l of each compound. (See 5.7.6 and 5.7.8.)

5.10.2 Daily, prepare and analyze a 2.0 μ g/l aqueous dilution from this mixture by dosing 20.0 μ l into 100 ml of organic-free water (See Section 8.1).

6. Sample Collection and Handling.

6.1 The sample containers should have a total volume of at least 25 ml.

6.1.1 Narrow-mouth screw-cap bottles with the TFE fluorocarbon faced silicone septa cap liners are strongly recommended.

6.2 Glassware Preparation.

6.2.1 Wash all sample bottles, TFE seals, and extraction flasks in detergent. Rinse with tap water and finally with distilled water.

6.2.2 Allow the bottles and seals to air dry, then place in an 105° C oven for 1 hour, then allow to cool in an area known to be free of organics.

NOTE.—Do not heat the TFE seals for extended periods of time (>1 hour) because the silicone layer slowly degrades at 105° C.

6.2.3 When cool, seal the bottles using the TFE seals that will be used for sealing the samples.

6.3 Sample stabilization—A chemical reducing agent (Section 5.3) is added to all samples in order to arrest the formation of additional trihalomethanes after sample collection (7.9) and to eliminate the possibility of free chlorine reacting with impurities in the extraction solvent to form interfering organohalides. **DO NOT ADD THE REDUCING AGENT TO SAMPLES AT COLLECTION TIME WHEN DATA FOR MAXIMUM TRIHALOMETHANE FORMATION IS DESIRED.** If chemical stabilization is employed, then the reagent is also added to the blanks. The chemical agent (2.5 to 3 mg/40 ml) is added in crystalline form to the empty sample bottle just prior to shipping to the sampling site. If chemical stabilization is not employed at sampling time then the reducing agent is added just before extraction.

6.4 Sample Collection.

6.4.1 Collect all samples in duplicate.

6.4.2 Fill the sample bottles in such a manner that no air bubbles pass through the sample as the bottle is filled.

6.4.3 Seal the bottle so that no air bubbles are entrapped in it.

6.4.4 Maintain the hermetic seal on the sample bottle until analysis.

6.4.5 The raw source water sample history should resemble the finished drinking water. The average retention time of the finished drinking water within the water plant should be taken into account when sampling the raw source water.

6.4.6 Sampling from a water tap.

6.4.6.1 Turn on the water and allow the system to flush until the temperature of the water has stabilized.

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Adjust the flow to about 500 ml/minute and collect duplicate samples from the flowing stream.

6.4.7 Sampling from an open body of water.

6.4.7.1 Fill a 1-quart wide-mouth bottle with sample from a representative area. Carefully fill duplicate sample bottles from the 1-quart bottle as in 6.4.

6.4.8 If a chemical reducing agent has been added to the sample bottles, fill with sample just to overflowing, seal the bottle, and shake vigorously for 1 minute.

6.4.9 Sealing practice for septum seal screw cap bottles.

6.4.9.1 Open the bottle and fill to overflowing. Place on a level surface. Position the TFE side of the septum seal upon the convex sample meniscus and seal the bottle by screwing the cap on tightly.

6.4.9.2 Invert the sample and lightly tap the cap on a solid surface. The absence of entrapped air indicates a successful seal. If bubbles are present, open the bottle, add a few additional drops of sample, then reseal bottle as above.

6.4.10 Sample blanks.

6.4.10.1 Prepare blanks in duplicate at the laboratory by filling and sealing sample bottles with organic-free water just prior to shipping the sample bottles to the sampling site.

6.4.10.2 If the sample is to be stabilized, add an identical amount of reducing agent to the blanks.

6.4.10.3 Ship the blanks to and from the sampling site along with the sample bottles.

6.4.10.4 Store the blanks and the samples, collected at a given site (sample set), together in a protected area known to be free from contamination. A sample set is defined as all the samples collected at a given site (i.e., at a water treatment plant, duplicate raw source water, duplicate finished water and the duplicate sample blanks comprise the sample set).

6.5 When samples are collected and stored under these conditions, no measurable loss of trihalomethanes has been detected over extended periods of time (7). It is recommended that the samples be analyzed within 14 days of collection.

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7. Extraction and Analysis.

7.1 Remove the plungers from two 10-ml syringes and attach a closed syringe valve to each.

7.2 Open the sample bottle (or standard) and carefully pour the sample into one of the syringe barrels until it overflows. Replace the plunger and compress the sample. Open the syringe valve and vent any residue air while adjusting the sample volume to 10.0 ml. Close the valve.

7.3 Fill the second syringe in an identical manner from the same sample bottle. This syringe is reserved for a replicate analysis (see 8.3 and 8.4).

7.4 Pipette 2.0 ml of extraction solvent into a clean extraction flask.

7.5 Carefully inject the contents of the syringe into the extraction flask.

7.6 Seal with a Teflon faced septum.

7.7 Shake vigorously for 1 minute.

7.8 Let stand until the phases separate (60 seconds).

7.8.1 If the phases do not separate on standing then centrifugation can be used to facilitate separation.

7.9 Analyze the sample by injecting 3.0 μ l (solvent flush technique, (8)) of the upper (organic) phase into the gas chromatograph.

8. Analytical Quality Control.

8.1 A 2 μ g/l quality check standard (See 5.10) should be extracted and analyzed each day before any samples are analyzed. Instrument status checks and lower limit of detection estimations based upon response factor calculations at 5 times the noise level are obtained from these data. In addition, the data obtained from the quality check standard can be used to estimate the concentration of the unknowns. From this information the appropriate standards can be determined.

8.2 Analyze the sample blank and the raw source water to monitor for potential interferences as described in Sections 3.1, 3.2, and 3.3.

8.3 Spiked samples.

* If for any reason the chemical reducing agent has not been added to the sample, then it must be added just prior to analyses at the rate of 2.5 to 3 mg/40 ml or by adding 1 mg directly to the sample in the extraction flask.

8.3.1 For those laboratories analyzing more than 10 samples a day, each 10th sample analyzed should be a laboratory-generated spike which closely duplicates the average finished drinking water in trihalomethane composition and concentration. Prepare the spiked sample in organic-free water as described in section 5.9.

8.3.2 In those laboratories analyzing less than 10 samples daily, each time the analysis is performed, analyze at least one laboratory generated spike sample which closely duplicates the average finished drinking water in trihalomethane composition and concentration. Prepare the spiked sample in organic-free water as described in section 5.9.

8.3.3 Maintain an up-to-date log on the accuracy and precision data collected in Sections 8.3 and 8.4. If results are significantly different than those cited in Section 10.1, the analyst should check out the entire analysis scheme to determine why the laboratory's precision and accuracy limits are greater.

8.4 Randomly select and analyze 10% of all samples in duplicate.

8.5 Analyze all samples in duplicate which appear to deviate more than 30% from any established norm.

8.6 Quarterly, spike an EMSL-Cincinnati trihalomethane quality control sample into organic-free water and analyze.

8.6.1 The results of the EMSL trihalomethane quality control sample should agree within 20% of the true value for each trihalomethane. If they do not, the analyst must check each step in the standard generation procedure to solve the problem.

8.7 It is important that the analyst be aware of the linear response characteristics of the electron capture system that is utilized. Calibration curves should be generated and rechecked quarterly for each trihalomethane over the concentration range encountered in the samples in order to confirm the linear response range of the system. Quantitative data cannot be calculated from non-linear responses. Whenever non-linear responses are noted; the analyst must dilute the sample for reanalysis.

8.8 Maintain a record of the retention times for each trihalomethane using data gathered from spiked samples and standards.

8.8.1 Daily calculate the average retention time for each trihalomethane and the variance encountered for the analyses.

8.8.2 If individual trihalomethane retention time varies by more than 10% over an eight hour period or does not fall within 10% of an established norm, the system is "out of control." The source of retention data variation must be corrected before acceptable data can be generated.

9. Calculations.

9.1 Locate each trihalomethane in the sample chromatogram by comparing the retention time of the suspect peak to the data gathered in 8.8.1. The retention time of the suspect peak must fall within the limits established in 8.8.1 for a single column identification.

9.2 Calculate the concentration of each trihalomethane by comparing the peak heights or peak areas of the samples to those of the standards. Round off the data to the nearest $\mu\text{g}/\text{l}$ or two significant Figures.

Concentration, $\mu\text{g}/\text{l}$ = sample peak height / standard peak height \times standard concentration, $\mu\text{g}/\text{l}$.

9.3 Calculate the total trihalomethane concentration (TTHM) by summing the 4 individual trihalomethane concentrations in $\mu\text{g}/\text{l}$: TTHM ($\mu\text{g}/\text{l}$) = (conc. CHCl_3) + (conc. CHBrCl_2) + (conc. CHBr_2Cl) + (conc. CHBr_3)

9.4 Calculate the limit of detection (LOD) for each trihalomethane not detected using the following criteria:

$$\text{LOD } (\mu\text{g}/\text{l}) = \frac{(\text{AXATT})}{(\text{BXATT}) \times (2 \mu\text{g}/\text{l})}$$

Where:

B = peak height (mm) of 2 $\mu\text{g}/\text{l}$ quality check standard

A = 5 times the noise level in mm at the exact retention time of the trihalomethane or the base line displacement in mm from theoretical zero at the exact retention time for the trihalomethane.

ATT = attenuation factor.

9.5 Report the results obtained from the lower limit of detection esti-

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mates along with the data for the samples.

10. Precision and Accuracy

10.1 Single lab precision and accuracy. The data in Table II were generated by spiking organic-free water with trihalomethanes as described in 5.9. The mixtures were analyzed by the analyst as true unknowns.

TABLE 1.—Retention Times for Trihalomethanes

Trihalomethane	Retention time minutes		
	Column A	Column B	Column C
Chloroform.....	1.0	1.3	4.9
Bromodichloromethane.....	1.5	2.5	11.0
Chlorodibromomethane.....	2.6	5.6	23.1
(Dibromochloromethane) bromoform.....	5.5	10.9	39.4

*On this column, trichloroethylene, a common raw source water contaminant, coelutes with bromodichloromethane.

Table II.—Single Laboratory Accuracy and Precision

Compound:	Dose level µg/l	Number of samples	Mean µg/l	Precision relative standard deviation, percent	Accuracy percent recovery
CHCl ₃	9.1	5	10	11	110
CHCl ₃	69	3	73	5.3	106
CHBrCl ₂	1.2	5	1.3	9.8	108
CHBrCl ₂	12	2	15	1.4	125
CHBr ₂ Cl.....	2.7	5	2.0	17	74
CHBr ₂ Cl.....	17	3	16	9.9	94
CHBr ₃	2.9	5	2.2	10	76
CHBr ₃	14	3	16	12	114

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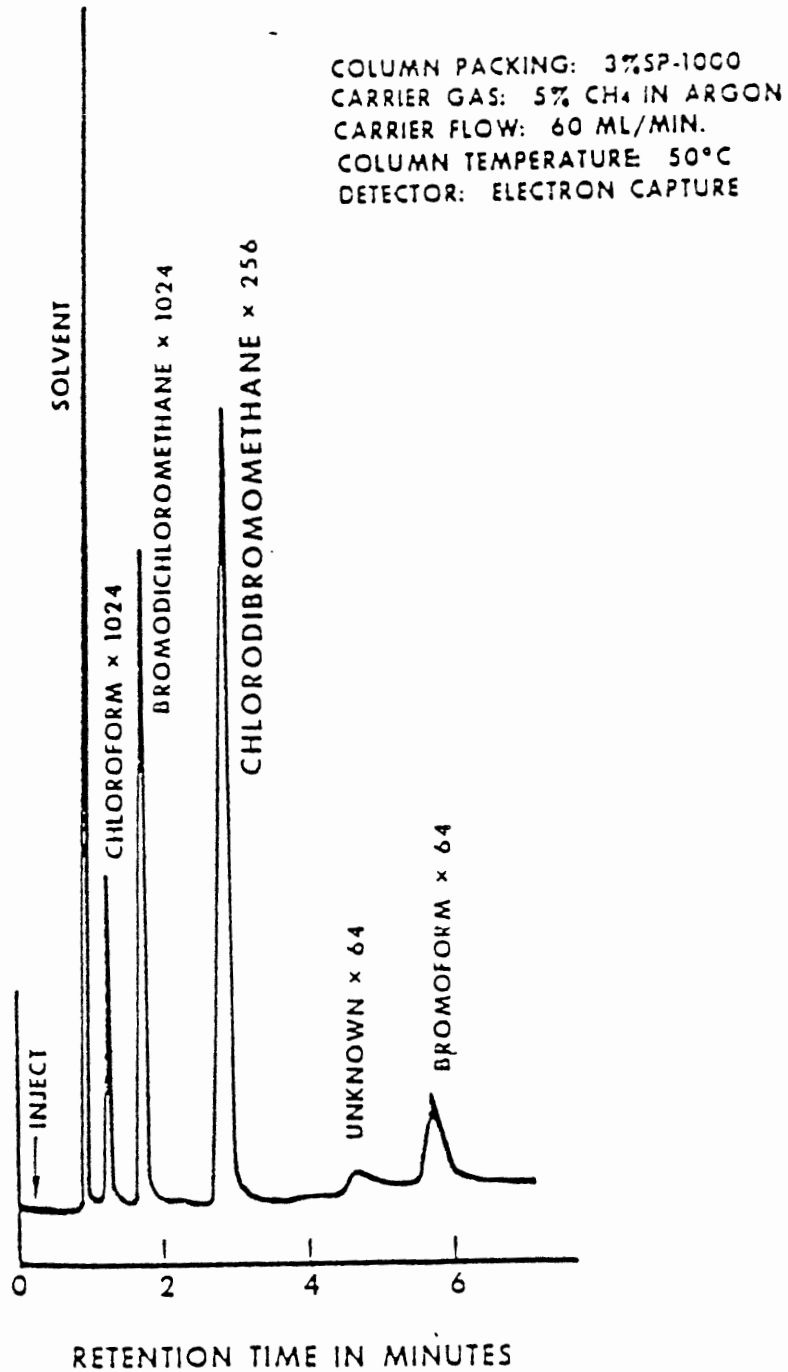


FIGURE 1. FINISHED WATER EXTRACT

COLUMN PACKING: 10%
SQUALANE CARRIER
FLOW: 25ml/min COLUMN
TEMPERATURE: 67

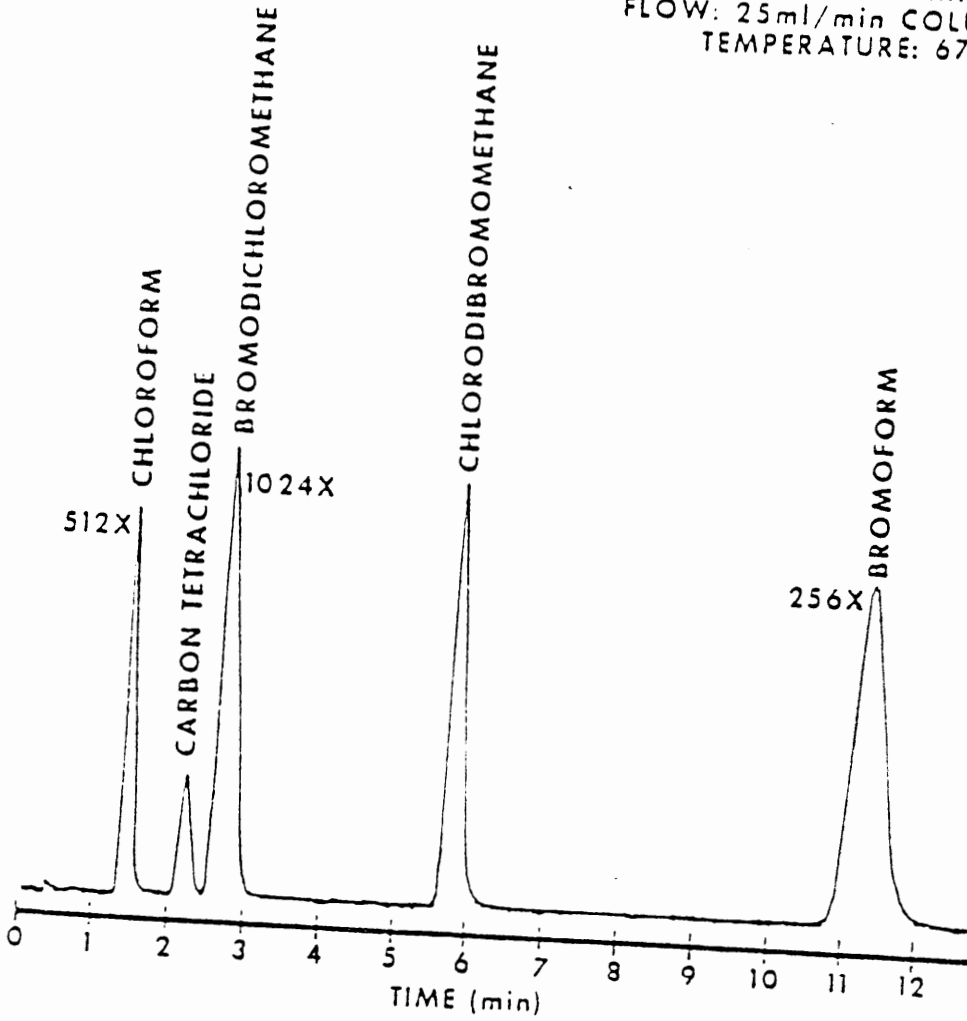


FIGURE 2. EXTRACT OF STANDARD

COLUMN PACKING: 6% OV-11+4% SP-2100
CARRIER FLOW: 25 ml/min
TEMPERATURE PROGRAM: 45°C-12 MINUTES
1°/MINUTE TO 70°C

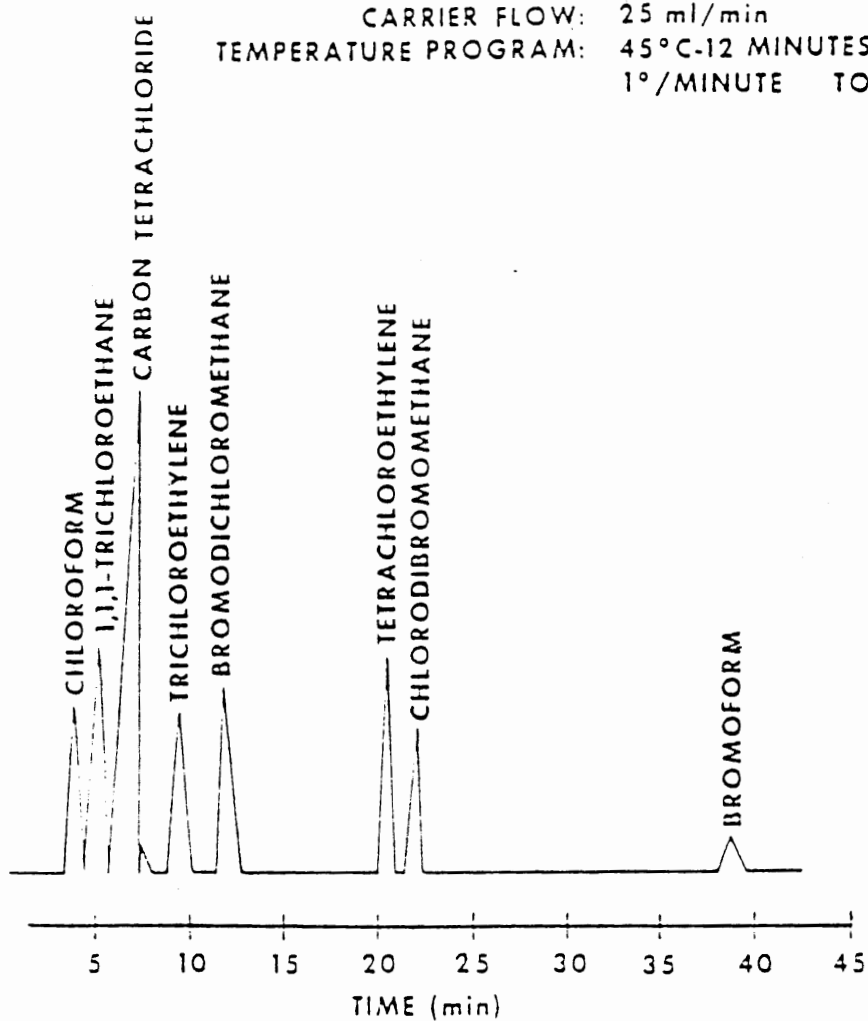


FIGURE 3. EXTRACT OF STANDARD

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**PART III—DETERMINATION OF MAXIMUM
TOTAL TRIHALOMETHANE POTENTIAL
(MTP)**

The water sample used for this determination is taken from a point in the distribution system that reflects maximum residence time. Procedures for sample collection and handling are given in EMSL Methods 501.1 and 501.2. No reducing agent is added to "quench" the chemical reaction producing THMs at the time of sample collection. The intent is to permit the level of THM precursors to be depleted and the concentration of the THMs to be maximized for the supply being tested.

Four experimental parameters affecting maximum THM production are pH, temperature, reaction time and the presence of a disinfectant residual. These parameters are dealt with as follows:

Measure the disinfectant residual at the selected sampling point. Proceed only if a measurable disinfectant residual is present. Collect triplicate 40 ml water samples at the pH prevailing at the time of sampling, and prepare a method blank according to the EMSL methods. Seal and store these samples together for 7 days at 25°C or above. After this time period, open one of the sample containers and check for disinfectant residual. Absence of a disinfectant residual invalidates the sample for further analyses. Once a disinfectant residual has been demonstrated, open another of the sealed samples and determine total THM concentration using either of the EMSL analytical methods.

[45 FR 68672, Nov. 29, 1979]

**Subpart D—Reporting, Public
Notification and Record Keeping**

§ 141.31 Reporting requirements.

(a) Except where a shorter reporting period is specified in this part, the supplier of water shall report to the State within 40 days following a test, measurement or analysis required to be made by this part, the results of that test, measurement or analysis.

(b) The supplier of water shall report to the State within 48 hours

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the failure to comply with any primary drinking water regulation (including failure to comply with monitoring requirements) set forth in this part.

(c) The supplier of water is not required to report analytical results to the State in cases where a State laboratory performs the analysis and reports the results to the State office which would normally receive such notification from the supplier.

§ 141.32 Public notification.

(a) If a community water system fails to comply with an applicable maximum contaminant level established in Subpart B, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable maximum contaminant level, fails to comply with the requirements of any schedule prescribed pursuant to a variance or exemption, or fails to perform any monitoring required pursuant to Section 1445 (a) of the Act, the supplier of water shall notify persons served by the system of the failure or grant by inclusion of a notice in the first set of water bills of the system issued after the failure or grant and in any event by written notice within three months. Such notice shall be repeated at least once every three months so long as the system's failure continues or the variance or exemption remains in effect. If the system issues water bills less frequently than quarterly, or does not issue water bills, the notice shall be made by or supplemented by another form of direct mail.

(b) If a community water system has failed to comply with an applicable maximum contaminant level, the supplier of water shall notify the public of such failure, in addition to the notification required by paragraph (a) of this section, as follows:

(1) By publication on not less than three consecutive days in a newspaper or newspapers of general circulation in the area served by the system. Such notice shall be completed within fourteen days after the supplier of water learns of the failure.

(2) By furnishing a copy of the notice to the radio and television stations serving the area served by the

system. Such notice shall be furnished within seven days after the supplier of water learns of the failure.

(c) If the area served by a community water system is not served by a daily newspaper of general circulation, notification by newspaper required by paragraph (b) of this section shall instead be given by publication on three consecutive weeks in a weekly newspaper of general circulation serving the area. If no weekly or daily newspaper of general circulation serves the area, notice shall be given by posting the notice in post offices within the area served by the system.

(d) If a non-community water system fails to comply with an applicable maximum contaminant level established in Subpart B of this part, fails to comply with an applicable testing procedure established in Subpart C of this part, is granted a variance or an exemption from an applicable maximum contaminant level, fails to comply with the requirement of any schedule prescribed pursuant to a variance or exemption or fails to perform any monitoring required pursuant to Section 1445(a) of the Act, the supplier of water shall give notice of such failure or grant to the persons served by the system. The form and manner of such notice shall be prescribed by the State, and shall insure that the public using the system is adequately informed of the failure or grant.

(e) Notices given pursuant to this section shall be written in a manner reasonably designed to inform fully the users of the system. The notice shall be conspicuous and shall not use unduly technical language, unduly small print or other methods which would frustrate the purpose of the notice. The notice shall disclose all material facts regarding the subject including the nature of the problem and, when appropriate, a clear statement that a primary drinking water regulation has been violated and any preventive measures that should be taken by the public. Where appropriate, or where designated by the State, bilingual notice shall be given. Notices may include a balanced explanation of the significance or seriousness to the public health of the subject of the notice, a fair explanation of steps

taken by the system to correct any problem and the results of any additional sampling.

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

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

§ 141.33 Record maintenance.

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

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

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

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

(3) Date of analysis;

(4) Laboratory and person responsible for performing analysis;

(5) The analytical technique/method used; and

(6) The results of the analysis.

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

(c) Copies of any written reports, summaries or communications relating to sanitary surveys of the system con-

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ducted by the system itself, by a private consultant, or by any local, State or Federal agency, shall be kept for a period not less than 10 years after completion of the sanitary survey involved.

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

evaluation of analytical results, including records of previous monitoring and analyses, information on possible sources of contamination and treatment techniques used by the system.

[40 FR 59588, Dec. 24, 1975]

**Subpart E—Special Monitoring
Regulations for Organic Chemicals**

§ 141.40 Special monitoring for organic chemicals.

(a) The Administrator may designate, by publication in the FEDERAL REGISTER, public water systems which are required to take water samples, provide information, and in appropriate cases analyze water samples for the purpose of providing information on contamination of drinking water sources and of treated water by organic chemicals.¹

(b) The Administrator shall provide to each public system designated pursuant to paragraph (a) of this section a written schedule for the sampling of source water or treated water by the system, with written instructions for the sampling methods and for handling of samples. The schedule may designate the locations or types of locations to be sampled.

(c) In cases where the public water system has a laboratory capable of analyzing samples for constituents specified by the Administrator, the Administrator may require analyses to be made by the public water system for submission to EPA. If the Administrator requires the analyses to be made by the public water system, he shall provide the system with written instructions as to the analytical procedures to be followed, or with references to technical documents describing the analytical procedures.

(d) Public water systems designated by the Administrator pursuant to paragraph (a) of this section shall provide to the Administrator, upon request, information to be used in the

¹A list of designated public water systems was published at 41 FR 5281, Jan. 5, 1976.

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and the Regional Administrator of the Environmental Protection Agency for the region in which such violation is alleged to have occurred.

(3) If the alleged violator is a Federal agency, service of notice shall be accomplished by certified mail addressed to, or by personal service upon, the head of such agency. A copy of such notice shall be mailed to the Administrator of the Environmental Protection Agency, the Regional Administrator of the Environmental Protection Agency for the region in which such violation is alleged to have occurred, the Attorney General of the United States, and the Chief administrative officer of the water pollution control agency for the State in which the violation is alleged to have occurred.

(b) Service of notice of intent to file suit pursuant to section 505(a)(2) of the Act shall be accomplished by certified mail addressed to, or by personal service upon, the Administrator, Environmental Protection Agency, Washington, D.C. 20460. A copy of such notice shall be mailed to the Attorney General of the United States.

(c) Notice given in accordance with the provisions of this part shall be deemed to have been served on the postmark date if mailed, or on the date of receipt if served personally.

§ 135.3 Contents of notice.

(a) *Violation of standard, limitation or order.* Notice regarding an alleged violation of an effluent standard or limitation or of an order with respect thereto, shall include sufficient information to permit the recipient to identify the specific standard, limitation, or order alleged to have been violated, the activity alleged to constitute a violation, the person or persons responsible for the alleged violation, the location of the alleged violation, the date or dates of such violation, and the full name, address, and telephone number of the person giving notice.

(b) *Failure to act.* Notice regarding

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an alleged failure of the Administrator to perform any act or duty under the Act which is not discretionary with the Administrator shall identify the provision of the Act which requires such act or creates such duty, shall describe with reasonable specificity the action taken or not taken by the Administrator which is alleged to constitute a failure to perform such act or duty, and shall state the full name, address and telephone number of the person giving the notice.

(c) *Identification of counsel.* The notice shall state the name, address, and telephone number of the legal counsel, if any, representing the person giving the notice.

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Sec.

- 136.1 Applicability.
- 136.2 Definitions.
- 136.3 Identification of test procedures.
- 136.4 Application for alternate test procedures.
- 136.5 Approval of alternate test procedures.

AUTHORITY: Sec. 304(g) of Federal Water Pollution Control Act Amendments of 1972, 86 Stat. 816, et seq., Pub. L. 92-500.

§ 136.1 Applicability.

The procedures prescribed herein shall, except as noted in § 136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(a) An application submitted to the Administrator, or to a State having an approved NPDES program, for a permit under section 402 of the Federal Water Pollution Control Act as amended (FWPCA), and,

(b) Reports required to be submitted by discharges under the NPDES established by Parts 124 and 125 of this chapter, and,

(c) Certifications issued by States pursuant to section 401 of the FWPCA, as amended.

[38 FR 28758, Oct. 16, 1973]

§ 136.2 Definitions.

As used in this part, the term:

(a) "Act" means the Federal Water Pollution Control Act, as amended, 33 U.S.C. 1314, et seq.

(b) "Administrator" means the Administrator of the U.S. Environmental Protection Agency.

(c) "Regional Administrator" means one of the EPA Regional Administrators.

(d) "Director" means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) "National Pollutant Discharge Elimination System (NPDES)" means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) "Standard Methods" means *Standard Methods for the Examination of Water and Waste Water*, 14th Edition, 1976. This publication is available from the American Public Health Association, 1015 18th Street, N.W., Washington, D.C. 20036.

(g) "ASTM" means *Annual Book of Standards, Part 31, Water*, 1975. This publication is available from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

(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 Trans-

fer, Industrial Environmental Research Laboratory, Cincinnati, Ohio 45268. This publication is available from the Office of Technology Transfer.

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976]

§ 136.3 Identification of test procedures.

(a) Every parameter or pollutant for which an effluent limitation is now specified pursuant to sections 401 and 402 of the Act is named together with test descriptions and references in Table I. The discharge parameter values for which reports are required must be determined by one of the standard analytical methods cited and described in Table I, or under certain circumstances by other methods that may be more advantageous to use when such other methods have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing that the Director of the State in which such discharge will occur does not object to the use of such alternate test procedures.

(b) Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such 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.

TABLE I.—List of approved test procedures ¹

Parameter and units	Method	1974 EPA meth- ods	14th ed. standard meth- ods	References (page nos.)		Other ap- proved meth- ods
				Pt. 31 1975 ASTM	USGS meth- ods ¹	
1. Acidity, as CaCO ₃ , milligrams per liter.	Electrometric end point (pH of 8.2) or phenol-phthalein end point.	1	273(4d)	116	40	³ (607)
2. Alkalinity, as CaCO ₃ , milligrams per liter.	Electrometric titration (only to pH 4.5) manual or automated, or equivalent automated methods.	3 5	278	111	41	³ (607)
3. Ammonia (as N), milligrams per liter.	Manual distillation ⁴ (at pH 9.5) followed by nesslerization, titration, electrode, Automated phenolate.	159 165 168	410 412 616	237	116	³ (614)
BACTERIA						
4. Coliform (fecal) ⁵ , number per 100 ml.	MPN; ⁶ membrane filter		922 937			¹ (45)
5. Coliform (fecal) ⁵ in presence of chlorine, number per 100 ml.	do. ⁶ ⁷		922			
6. Coliform (total), ⁵ number per 100 ml	do. ⁶		928,937 916 928			¹ (35)
7. Coliform (total) ⁵ in presence of chlorine, number per 100 ml.	MPN; ⁶ membrane filter with enrichment.		916 933			
8. Fecal streptococci, ⁵ number per 100 ml.	MPN; ⁶ membrane filter; plate count.		943 944 947			¹ (50)
9. Benzidine, milligrams per liter	Oxidation—colorimetric ⁸					
10. Biochemical oxygen demand, 5-d (BOD ₅), milligrams per liter.	Winkler (Azide modification) or electrode method.		543			¹⁰ (17)
11. Bromide, milligrams per liter	Titrimetric, iodine-iodate	14		323	58	
12. Chemical oxygen demand (COD), milligrams per liter.	Dichromate reflux	20	550	472	124	³ (610) ¹⁰ (17)
13. Chloride, milligrams per liter	Silver nitrate; mercuric nitrate; or automated colorimetric-ferricyanide.	29 31	303 304 613	267 265		³ (615)
14. Chlorinated organic compounds (except pesticides), milligrams per liter.	Gas chromatography ¹²					
15. Chlorine—total residual, milligrams per liter.	Iodometric titration, amperometric or starch-iodine end-point; DPD colorimetric or Titrimetric methods (these last 2 are interim methods pending laboratory testing).	35	318 322 332 329	278		
16. Color, platinum cobalt units or dominant wave length, hue, luminance, purity.	Colorimetric; spectrophotometric; or ADMI procedure. ¹³	36 39	64 66		82	
17. Cyanide, total. ¹⁴ milligrams per liter	Distillation followed by silver nitrate titration or pyridine pyrazolone (or barbituric acid) colorimetric.	40	361	503	85	¹⁰ (22)
18. Cyanide amenable to chlorination, milligrams per liter.	do.	49	376	505		
19. Dissolved oxygen, milligrams per liter	Winkler (Azide modification) or electrode method.	51 56	443 450	368	126	³ (609)
20. Fluoride, milligrams per liter	Distillation ⁴ followed by ion electrode; SPADNS; or automated complexone.	65 59 61	389 391 393 614	307 305	93	

See footnotes at end of table

TABLE I.—List of approved test procedures ¹—Continued

Parameter and units	Method	1974 EPA meth- ods	14th ed. standard meth- ods	References (page nos.)		Other ap- proved meth- ods
				Pt. 31 1975 ASTM	USGS meth- ods	
21. Hardness—Total, as CaCO ₃ , milli-grams per liter.	EDTA titration; automated colorimetric; or atomic absorption (sum of Ca and Mg as their respective carbonates).	68 70	202	161	94	³ (617)
22. Hydrogen ion (pH), pH units.....	Electrometric measurement	239	460	178	129	³ (506)
23. Kjeldahl nitrogen (as N), milligrams per liter.	Digestion and distillation followed by nesslerization, titration, or electrode; automated digestion automated phenolate.	175 185 182	437		122	³ (612)
METALS						
24. Aluminum—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption ¹⁶ or by colorimetric (Enochrome Cyanine R).	92	152 171		¹¹ (19)	
25. Aluminum—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced methods for total aluminum.					
26. Antimony—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption. ¹⁶	94				
27. Antimony—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total antimony.					
28. Arsenic—Total, milligrams per liter..	Digestion followed by silver diethyldithio- carbamate; or atomic absorption. ¹⁶ ¹⁸	9 95	285 283 159		¹¹ (31) ¹¹ (37)	
29. Arsenic—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total arsenic.					
30. Barium—Total, milligrams per liter ..	Digestion ¹⁵ followed by atomic absorption. ¹⁶	97	152		52	
31. Barium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total barium.					
32. Beryllium—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption ¹⁶ or by colorimetric (Aluminon).	99	152 177		53	
33. Beryllium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total beryllium.					
34. Boron—Total, milligrams per liter	Colorimetric (Curcumin)	13	287			
35. Boron—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total boron.					
36. Cadmium—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption ¹⁶ or by colorimetric (Dithzone).	101	148 182	345	62	³ (619) ¹⁶ (37)
37. Cadmium—Dissolved, milligrams per liter	0.45 micron filtration ¹⁷ followed by referenced method for total cadmium.					
39. Calcium—Total, milligrams per liter.	Digestion ¹⁵ followed by atomic absorption; or EDTA titration.	103	148 189	345	66	
39. Calcium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total calcium.					
40. Chromium VI, milligrams per liter.....	Extraction and atomic absorption; colorimetric (Diphenylcarbazide).	89, 105	192		76 75	
41. Chromium VI—Dissolved, milli-grams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for chromium VI.					

¹ See footnotes at end of table

TABLE I.—List of approved test procedures ¹—Continued

Parameter and units	Method	1974 EPA meth- ods	14th ed. standard meth- ods	References (page nos.)		Other ap- proved meth- ods
				Pt. 31 1975 ASTM	USGS meth- ods	
42. Chromium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Diphenylcarbazide).	105	148 192	345 286	78 77	³ (619)
43. Chromium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total chromium.					
4. Cobalt—Total, milligrams per liter ...	Digestion ¹³ followed by atomic absorption. ¹⁴	107	148	345	80	¹⁶ (37)
45. Cobalt—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total cobalt.					
46. Copper—Total, milligrams per liter..	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Neocuproine).	108	148 196	345 293	83	³ (619) ¹⁶ (37)
47. Copper—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total copper.					
48. Gold—Total, milligrams per liter	Digestion ¹³ followed by atomic absorption. ¹⁹					
49. Iridium—Total, milligrams per liter ...	Digestion ¹³ followed by atomic absorption. ¹⁹					
50. Iron—Total, milligrams per liter.....	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Phenanthroline).	110	148 208	345 326	102	³ (619)
51. Iron—Dissolved, milligrams per liter	0.45 micron filtration ¹⁷ followed by referenced method for total iron.					
52. Lead—Total, milligrams per liter.....	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Dithizone).	112	148 215	345	105	³ (619)
53. Lead—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total lead.					
54. Magnesium—Total, milligrams per liter.	Digestion ¹³ followed by atomic absorption; or gravimetric.	114	148 221	345	109	³ (619)
55. Magnesium—Dissolved milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total magnesium.					
56. Manganese—Total milligrams per liter.	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Persulfate or periodate).	116	148 225, 227	345	111	³ (619)
57. Manganese—Dissolved milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total manganese.					
58. Mercury—Total, milligrams per liter.	Flameless atomic absorption.	118	156	338	¹¹ (51)	
59. Mercury—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total mercury.					
60. Molybdenum—Total, milligrams per liter	Digestion ¹³ followed by atomic absorption. ¹⁴	139		350		⁷
61. Molybdenum—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total molybdenum.					
62. Nickel—Total, milligrams per liter....	Digestion ¹³ followed by atomic absorption ¹⁴ or by colorimetric (Heptoxime).	141 223	148	345	115	
63. Nickel—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total nickel.					
64. Osmium—Total, milligrams per liter	Digestion ¹³ followed by atomic absorption. ¹⁹					

See footnotes at end of table.

TABLE I.—List of approved test procedures¹—Continued

Parameter and units	Method	1974 EPA meth- ods	14th ed. standard meth- ods	References (page nos.)		Other ap- proved meth- ods
				Pt. 31 1975 ASTM	USGS meth- ods	
65. Palladium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption. ¹⁹
66. Platinum—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption. ¹⁹
67. Potassium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption, colorimetric (Cobaltinitrite), or by flame photometric.	143	235 234	134	³ (620)
68. Potassium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total potassium.
69. Rhodium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption. ¹⁹
70. Ruthenium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption. ¹⁹
71. Selenium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption. ^{18, 19}	145	159
72. Selenium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total selenium.
73. Silica—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by colorimetric (Molybdosilicate).	274	487	398	139
74. Silver—Total, ²⁰ milligrams per liter.	Digestion ¹⁸ followed by atomic absorption ¹⁸ or by colorimetric (Dithizone).	146	148 243	142	³ (619) ¹⁸ (37)
75. Silver—Dissolved, ²⁰ milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total silver.
76. Sodium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption or by flame photometric.	147	250	403	143	³ (621)
77. Sodium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total sodium.
78. Thallium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption. ¹⁸	149
79. Thallium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total thallium.
80. Tin—Total, milligrams per liter.....	Digestion ¹⁸ followed by atomic absorption. ¹⁸	150	¹¹ (65)
81. Tin—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total tin.
82. Titanium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption. ¹⁸	151
83. Titanium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total titanium.
84. Vanadium—Total, milligrams per liter.	Digestion ¹⁸ followed by atomic absorption ¹⁸ or by colorimetric (Gallic acid).	153	152 260	441	¹¹ (67)
85. Vanadium—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total vanadium.
86. Zinc—Total, milligrams per liter.....	Digestion ¹⁸ followed by atomic absorption ¹⁸ or by colorimetric (Dithizone).	155	148 265	345	159	³ (619) ¹⁸ (37)
87. Zinc—Dissolved, milligrams per liter.	0.45 micron filtration ¹⁷ followed by referenced method for total zinc.

¹ See footnotes at end of table

TABLE I.—List of approved test procedures ¹—Continued

Parameter and units	Method	1974 EPA methods	14th ed. standard methods	References (page nos.)		Other approved methods
				Pt. 31 1975 ASTM	USGS methods	
88. Nitrate (as N), milligrams per liter....	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction. ¹¹	201 197 207	423 427 620	358	119	³ (814) ²⁶ (28)
89. Nitrite (as N), milligrams per liter....	Manual or automated colorimetric (Diazotization).	215	434		121	
90. Oil and grease, milligrams per liter..	Liquid-liquid extraction with trichloro-trifluoro-ethane-gravimetric.	229	515			
91. Organic carbon; total (TOC), milligrams per liter.	Combustion—Infrared method. ²²	236	532	467	²² (4)	
92. Organic nitrogen (as N), milligrams per liter.	Kjeldahl nitrogen minus ammonia nitrogen.	175, 159	437		122	³ (612, 814)
93. Orthophosphate (as P), milligrams per liter.	Manual or automated ascorbic acid reduction.	249 256	481 624	364	131	³ (621)
94. Pentachlorophenol, milligrams per liter.	Gas chromatography ¹³					
95. Pesticides, milligrams per liter.....	do. ¹²		555	529	²² (24)	
96. Phenols, milligrams per liter.....	Distillation followed by Colorimetric. (4AAP)	241	574	545		
97. Phosphorus (elemental), milligrams per liter.	Gas chromatography ²⁴					
98. Phosphorus; total (as P), milligrams per liter.	Persulfate digestion followed by manual or automated ascorbic acid reduction.	249 256	476, 481 624	384	133	³ (621)
RADIOLOGICAL						
99. Alpha—Total, pCi per liter.....	Proportional or scintillation counter.		648	591	¹¹ ²⁸ (75+78)	
100. Alpha—Counting error, pCi per liter.	do.....		648	594	¹¹ (79)	
101. Beta—Total, pCi per liter.....	Proportional counter		648	601	¹¹ ²⁸ (75+78)	
102. Beta—Counting error, pCi per liter	do.....		648	606	¹¹ (79)	
103. (a) Radium—Total, pCi per liter....	do.....		661	661		
(b) ²²⁶ Ra, pCi per liter.....	Scintillation counter		667		¹¹ (81)	
RESIDUE						
104. Total, milligrams per liter.....	Gravimetric, 103 to 105° C.....	270	91			
105. Total dissolved (filterable), milligrams per liter.	Glass fiber filtration, 180° C.....	266	92			
106. Total suspended residue.....	Glass fiber filtration, 103 to 105° C., post-washing of residue.	268	94			²⁷ (537)
107. Settleable, milliliters per liter or milligrams per liter.	Volumetric or gravimetric.....		95			
108. Total volatile, milligrams per liter...	Gravimetric, 550° C.....	272	95			
109. Specific conductance, micromhos per centimeter at 25° C.	Wheatstone bridge conductmetry.	275	71	120	148	³ (806)
110. Sulfate (as SO ₄), milligrams per liter.	Gravimetric; turbidimetric; or automated colorimetric (barium chloranilate).	277 279	493 496	424 425		³ (624) ³ (623)
111. Sulfide (as S), milligrams per liter..	Titrimetric—Iodine for levels greater than 1 mg per liter; Methylene blue photometric.	284	505 503		154	
112. Sulfite (as SO ₃), milligrams per liter.	Titrimetric, iodine-iodate	285	508	435		
113. Surfactants, milligrams per liter.....	Colorimetric (Methylene blue).	157	600	494	²² (11)	
114. Temperature, degrees C.....	Calibrated glass or electrometric thermometer.	286	125		²⁸ (31)	
115. Turbidity, NTU.....	Nephelometric.....	295	132	223	156	

¹ Recommendations for sampling and preservation of samples according to parameter measured may be found in "Methods for Chemical Analysis of Water and Wastes, 1974" U.S. Environmental Protection Agency, table 2, pp. viii-xii.

Footnotes continued on next page.

¹All page references for USGS methods, unless otherwise noted, are to Brown, E., Skougstad, M.W., and Fishman, M.J., "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A1. (1970).

²EPA comparable method may be found on indicated page of "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 12th ed. (1975).

³Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

⁴The method used must be specified.

⁵The 5 tube MPN is used.

⁶Slack, K.V. and others, "Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples: U.S. Geological Survey Techniques of Water-Resources Inv. book 5, ch. A4 (1973)."

⁷Since the membrane filter technique usually yields low and variable recovery from chlorinated wastewaters, the MPN method will be required to resolve any controversies.

⁸Adequately tested methods for benzidine are not available. Until approved methods are available, the following interim method can be used for the estimation of benzidine: (1) "Method for Benzidine and Its Salts in Wastewaters," available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

⁹American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, N.Y. 10018.

¹⁰Fishman, M.J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey for Analysis of Wastewaters," (1976) open-file report 76-177.

¹¹Procedures for pentachlorophenol, chlorinated organic compounds and pesticides can be obtained from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

¹²Color method (ADMI procedure) available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

¹³For samples suspected of having thiocyanate interference, magnesium chloride is used as the digestion catalyst. In the approved test procedure for cyanides, the recommended catalysts are replaced with 20 ml of a solution of 510 g/l magnesium chloride ($MgCl_2 \cdot 6H_2O$). This substitution will eliminate thiocyanate interference for both total cyanide and cyanide amenable to chlorination measurements.

¹⁴For the determination of total metals the sample is not filtered before processing. Because vigorous digestion procedures may result in a loss of certain metals through precipitation, a less vigorous treatment is recommended as given on p. 83 (4.1.4) of "Methods for Chemical Analysis of Water and Wastes" (1974). In those instances where a more vigorous digestion is desired the procedure on p. 82 (4.1.3) should be followed. For the measurement of the noble metal series (gold, indium, osmium, palladium, platinum, rhodium and ruthenium), an aqua regia digestion is to be substituted as follows: Transfer a representative aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated redistilled HNO_3 . Place the beaker on a steam bath and evaporate to dryness. Cool the beaker and cautiously add a 5 ml portion of aqua regia. (Aqua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO_3 .) Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 50 min. Remove cover and evaporate to dryness. Cool and take up the residue in a small quantity of 1:1 HCl. Wash down the beaker walls and wash glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

¹⁵As the various furnace devices (flameless AA) are essentially atomic absorption techniques, they are considered to be approved test methods. Methods of standard addition are to be followed as noted in p. 78 of "Methods for Chemical Analysis of Water and Wastes," 1974.

¹⁶Dissolved metals are defined as those constituents which will pass through a 0.45 μm membrane filter. A prefiltration is permissible to free the sample from larger suspended solids. Filter the sample as soon as practical after collection using the first 50 to 100 ml to rinse the filter flask. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Discard the portion used to rinse the flask and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO_3 to a pH of 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the samples.

¹⁷See "Atomic Absorption Newsletter," vol. 13, 75 (1974). Available from Perkin-Elmer Corp., Main Ave., Norwalk, Conn. 06852.

¹⁸Method available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

¹⁹Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/l and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/l 20 ml of sample should be diluted to 100 ml by adding 40 ml each of 2M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/l the recommended method is satisfactory.

²⁰An automated hydrazine reduction method is available from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

²¹A number of such systems manufactured by various companies are considered to be comparable in their performance. In addition, another technique, based on combustion-methane detection is also acceptable.

²²Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water": U.S. Geological Survey Techniques of Water-Resources Inv., book 5, ch. A3 (1972).

²³R.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," "Journal of Chromatography," vol. 47, No. 3, pp. 421-426, 1970.

²⁴The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only suspended. Therefore, the 2 results must be added together to obtain "total."

²⁵Stevens, H. H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation: U.S. Geological Survey Techniques of Water Resources Inv., book 1 (1975)."

²⁶Standard Methods for the Examination of Water and Wastewater, 13th Edition, (1971).

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976; 42 FR 3306, Jan. 18, 1977; 42 FR 37205, July 20, 1977]

§ 136.4

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an application for an alternate test procedure may be made by letter in triplicate. Any application for an alternate test procedure under this paragraph (c) shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

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(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976]

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will occur has final responsibility for approval of any alternate test procedure proposed by the responsible person or firm making the discharge.

(b) Within thirty days of receipt of an application, the Director will forward such application proposed by the responsible person or firm making the discharge, together with his recommendations, to the Regional Administrator. Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application, and shall forward a copy of the rejected application and his decision to the Director of the State Permit Program and to the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(c) Before approving any application for an alternate test procedure proposed by the responsible person or firm making the discharge, the Regional Administrator shall forward a copy of the application to the Director of the Environmental Monitoring and Support Laboratory, Cincinnati.

(d) Within ninety days of receipt by the Regional Administrator of an application for an alternate test procedure, proposed by the responsible person or firm making the discharge, the Regional Administrator shall notify the applicant and the appropriate State agency of approval or rejection, or shall specify the additional in-

formation which is required to determine whether to approve the proposed test procedure. Prior to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Director of the Environmental Monitoring and Support Laboratory, Cincinnati. A copy of all approval and rejection notifications will be forwarded to the Director, Environmental Monitoring and Support Laboratory, Cincinnati, for the purposes of national coordination.

(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.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976]

PART 140—MARINE SANITATION DEVICE STANDARD

Sec.

- 140.1 Definitions.
- 140.2 Scope of standard.
- 140.3 Standard.
- 140.4 Complete prohibition.
- 140.5 Analytical procedures.

AUTHORITY: Sec. 312, as added October 18, 1972, Pub. L. 92-500, sec. 2, 86 Stat. 871. Interpret or apply sec. 312(b)(1), 33 U.S.C. 1322 (b)(1).

SOURCE: 41 FR 4453, Jan. 29, 1976, unless otherwise noted.

§ 140.1 Definitions.

For the purpose of these standards the following definitions shall apply:

(a) "Sewage" means human body wastes and the wastes from toilets and other receptacles intended to receive or retain body wastes;

(b) "Discharge" includes, but is not limited to, any spilling, leaking, pumping, pouring, emitting, emptying, or dumping;

(c) "Marine sanitation device" includes any equipment for installation onboard a vessel and which is designed to receive, retain, treat, or discharge sewage and any process to treat such sewage;

(d) "Vessel" includes every description of watercraft or other artificial contrivance used, or capable of being used, as a means of transportation on waters of the United States;

(e) "New vessel" refers to any vessel on which construction was initiated on or after January 30, 1975;

(f) "Existing vessel" refers to any vessel on which construction was initiated before January 30, 1975;

(g) "Fecal coliform bacteria" are those organisms associated with the intestines of warm-blooded animals that are commonly used to indicate the presence of fecal material and the potential presence of organisms capable of causing human disease.

§ 140.2 Scope of standard.

The standard adopted herein applies only to vessels on which a marine sanitation device has been installed. The standard does not require the installation of a marine sanitation device on any vessel that is not so equipped. The standard applies to vessels owned and operated by the United States unless the Secretary of Defense finds that compliance would not be in the interest of national security.

§ 140.3 Standard.

(a) (1) In freshwater lakes, freshwater reservoirs or other freshwater impoundments whose inlets or outlets are such as to prevent the ingress or egress by vessel traffic subject to this regulation, or in rivers not capable of

NOTAC 7.18

BASIS AND BACKGROUND

I. Overview

The Department is in the process of developing a laboratory certification program which will require the use of a certified laboratory for all water compliance analyses performed pursuant to Departmental Statutes and regulations.

In 1979, the Department satisfied the mandate of the Safe Drinking Water Act, N.J.S.A. 58:12A-1 et seq. by establishing a Drinking Water Laboratory Certification program as part of the Safe Drinking Water Act regulations. After operating this program for over two years, the Department is ready to take the next step by proposing this regulation to revise the current Drinking Water Laboratory Certification program, establish a drinking water radiological certification program, establish a laboratory certification program for the Water Pollution Control Act, N.J.S.A. 58:10A-1 et seq. and the New Jersey Pollution Discharge Elimination System regulations (N.J.P.D.E.S.) and consolidate the two programs into one regulation. This regulation will eventually address the certification of all water analyses being performed for the Department including solid waste, hazardous waste, and sludge analysis.

During the development of this proposal, the proposed regulation was reviewed by the United States Environmental Protection Agency's (U.S. EPA) Region II Quality Assurance Coordinator, a task force made up of 17 representatives of municipal, industrial, and private laboratories, and the appropriate Departmental personnel.

II. History

Section 4 of the New Jersey Safe Drinking Water Act, P.L. 1977, c. 224; N.J.S.A. 58:12A-1 et seq. requires the Department to "establish and maintain a program for the certification of laboratories conducting analytical measurements of drinking water contaminants specified in the State primary and secondary drinking water regulations". In July of 1979, the Department adopted the Safe Drinking Water Act regulations, N.J.A.C. 7:10-1 et seq. Subchapter 8 of these regulations established a program for the certification of microbiological and chemical drinking water laboratories.

The current Drinking Water Laboratory Certification program requires certified laboratories and laboratories seeking certification to acceptably analyze laboratory performance evaluation samples each year for the categories in which the laboratory is certified or is seeking certification. In addition, each laboratory must pass an on-site inspection by Departmental personnel at least once every two years. This inspection evaluates the laboratory against the standards set forth in the United States Environmental Protection Agency's "Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies".

After reviewing the results of the proficiency samples analyzed by the certified laboratories and the information gathered during the on-site inspections, the Department determined that the quality control requirements of the program were not sufficient to ensure that the data being generated by the certified laboratories is valid. In addition, the results of a quality assurance pilot study the Department conducted jointly with the United States Environmental Protection Agency convinced the Department of the need for a laboratory certification program for laboratories performing all types of water and wastewater analyses.

Since the requirements set forth in the USEPA's "Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies" are not easily enforceable, inadequately address chemical testing and analysis of drinking water parameters, and since it does not address wastewater analysis, the Department decided to propose this regulation which will establish and combine the Department's requirements for both water and wastewater analysis. The requirements set forth in this proposed regulation were established by building upon the requirements in the USEPA's manual and adding requirements necessary to make the regulation applicable to wastewater analysis. In addition, the Department made the quality control requirements in the chemistry category much more stringent, as well as included all current Federal requirements for N.J.P.D.E.S. microbiological and chemical compliance analysis.

This proposed regulation will establish a certification program for drinking water radiological testing and N.J.P.D.E.S. bioassay testing. Currently, all drinking water radiological analyses are performed by the Department. Due to recent laboratory requests for radiological certification, the Department is establishing a drinking water radiological category with specific criteria and procedures that must be followed by the laboratory when applying for and maintaining certification. Also, an increasing number of permitted dischargers are now being required to perform bioassay testing as part of their discharge monitoring requirements. In the past, approval of bioassay techniques have been on a case-by-case basis. This procedure has resulted in an inconsistency between laboratories in methodology, data analysis and quality control procedures. To correct this problem, the Department is proposing standardized procedures and criteria for bioassay certification.

This proposed regulation is the Department's second step in developing a comprehensive laboratory certification program. In the future, the Department intends to expand the certification program to cover all water analyses and at the same time amend this regulation by incorporating changes that will improve the program based upon the knowledge gained while operating the program.

III. Description of the Proposed Regulation Governing Laboratory Certification and Standards of Performance

A. Subchapters 1 and 2: Administration of Program

Subchapters 1 and 2 contain the administrative procedures and requirements for laboratories to follow to obtain and maintain certifications, and the criteria and procedures laboratories must follow in analyzing water samples. The proposed regulation requires all water sample analyses performed for the purpose of determining compliance with the Safe Drinking Water Act, N.J.S.A. 58:12A-1 et seq., the Water Pollution Control Act, N.J.S.A. 58:10A-1 et seq., the Safe Drinking Water Act Regulations, N.J.A.C. 7:10-1 et seq., the N.J.P.D.E.S. regulations N.J.A.C. 7:14A-1 et seq., and when ordered by the Department be performed in State certified laboratories.

The basic program in these two subchapters was derived from the current Drinking Water Laboratory Certification program. However, program changes were incorporated to enable the Department to administer and enforce the program more effectively.

One such change is the proposed certification of laboratories by parameter rather than by category. This will enable the Department to address the parameter in question whenever a laboratory demonstrates poor performance in the analysis of the specific parameter. In addition, this change will make it clear that certified laboratories are only allowed to analyze those parameters in a category for which the laboratory is certified.

Another change being proposed is the addition of a suspension provision as a means of addressing non-complying laboratories. Suspension of a laboratory's certification would temporarily prohibit the laboratory from performing compliance analyses. Past experience has found laboratories to be slow in submitting renewal applications, results of performance evaluation samples, and responses to laboratory evaluation reports. This information is essential for evaluating the performance of the laboratories, as well as, managing the program. Also, laboratories failing to acceptably analyze both the high and low values of a parameter in any given set of proficiency samples would immediately have its certification suspended for that specific parameter until the laboratory can again demonstrate proficiency in that parameter. This proposed change will allow the Department to temporarily suspend the certification of non-complying laboratories until they comply with the program requirements or are decertified.

Another change is the elimination of the interim approval procedure from the drinking water certification program. The interim approval procedure was established in the original laboratory certification program primarily as a tool for starting up the program. This procedure allowed a large number of laboratories to enter the program and begin performing compliance analyses in a relatively short amount of time. The problems associated with the interim approval procedure are that it allows laboratories to enter the program and begin performing analyses before the Department is certain the laboratory is in compliance with the program requirements and is capable of properly analyzing samples. Now that most laboratories desiring drinking water certification are within the program and because of the above problems, the Department has decided to abolish the interim approvals for the drinking water program. Laboratories seeking certification for drinking water parameters will be required to acceptably analyze proficiency samples and pass an on-site

inspection before being certified and allowed to analyze samples for compliance with the Safe Drinking Water Act program.

Although the interim approval procedure will be eliminated from the drinking water certification program, it will, however, be used to phase-in the N.J.P.D.E.S. laboratory certification program. This phasing-in of the N.J.P.D.E.S. laboratory certification program using interim approvals will begin July 1, 1981 and continue until January 1, 1983. During this period, the Department's goal is to upgrade and certify the laboratories performing N.J.P.D.E.S. compliance analysis. Interim approvals will no longer be offered after January 1, 1983. As with the drinking water certification program, laboratories requesting certification for N.J.P.D.E.S. analyses after January 1, 1983, will be required to demonstrate acceptable proficiency and compliance with the required criteria and procedures prior to being allowed to perform N.J.P.D.E.S. compliance analysis.

This regulation also addresses the source of funding for the program. Since the Department has determined that this program be totally self-supporting, a fee schedule sufficient to cover the cost of the program has been included in this regulation. All certified and interimly approved laboratories, and laboratories seeking certification, must pay the appropriate fee, except for laboratories owned by an entity paying a N.J.P.D.E.S. permit fee. Laboratories owned by an entity paying a N.J.P.D.E.S. permit fee are exempt from the laboratory fee requirement because their fee is paid from the funds collected by the N.J.P.D.E.S. program.

Subchapter 2 also establishes a radiological certification category for drinking water analysis and a bioassay certification for wastewater analysis. The administrative procedures are similar to the procedures established for the microbiology and chemistry categories.

B. Subchapter 3: Microbiological Testing

Subchapter 3 establishes the Department's requirements which a certified laboratory or a laboratory seeking certification shall continually meet and follow when performing microbiological analyses for both Drinking Water and N.J.P.D.E.S. compliance analyses. This subchapter was modeled after the USEPA's "Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies" and establishes the standards for laboratory facilities and safety; laboratory equipment, supplies and materials; sample collection, handling and preservation; methodologies; general laboratory practices; quality control; and records and data reporting. Subchapters 4, 5, and 6 follow the same sequence and covers the same topics as this subchapter.

Section 3.4 address the sample collection, handling, and preservation procedures to be used for water and wastewater samples. The Department has adopted the current Federal drinking water and N.P.D.E.S. requirements, as well as, included the requirement that laboratories implement a chain of custody procedure is also being required in subchapters 4, 5, and 6. This means that all water and wastewater samples being collected for microbiological, chemical, radiological or bioassay testing must follow the required chain of custody procedures. Through the use of chain of custody procedures the Department will be able to track all responsible parties who handled the sample, determine if the sample was preserved properly and determine if the sample was analyzed within the required holding time.

In this subchapter, the Department adopts current Federal drinking water and N.J.P.D.E.S. analytical methodology requirements as mandatory for this program. The Department also adopts the Federal drinking water quality control requirements and, where applicable, applies these requirements to wastewater analysis.

C. Subchapter 4: Chemical Testing

Subchapter 4 establishes the Department's requirements which a certified laboratory or a laboratory seeking certification shall continually meet and follow when performing chemical analyses.

Section 4.4 adopts USEPA's mandatory sample collection, handling and preservation requirements for primary drinking water parameters and N.P.D.E.S. monitoring parameters. Currently, the USEPA does not have mandatory sample collection, handling, and preservation requirements for the secondary drinking water parameters. The Department decided that such requirements are necessary and has applied the USEPA's N.P.D.E.S. requirements to the secondary drinking water parameters. Analytical methodologies were addressed in a similar way. The primary drinking water parameter methodologies and the N.J.P.D.E.S. parameter methodologies are the same as the USEPA's mandatory requirements. However, the secondary drinking water parameter methodologies were derived from both EPA and State recommendations.

As in Subchapter 3, the Department is requiring that drinking water and N.J.P.D.E.S. samples follow a chain of custody procedure which will identify all responsible parties that handled the sample, as well as, sample preservation steps taken and sample holding times.

The proposed quality control program for drinking water analysis is more stringent than that required by the USEPA. These quality control requirements apply to both water and wastewater analysis and require laboratories to have a written quality control program which will indicate all quality control procedures implemented by the laboratory and a written

laboratory procedures manual which will document the actual laboratory procedures being used. This subchapter also requires laboratories to conduct and document quality control checks on the analytical instruments to ensure proper instrumental operation. Another requirement of this subchapter is the analysis of replicate and spiked samples on at least 5% of all samples for all applicable parameters. This requirement is to establish the laboratory's precision and accuracy in the analysis of those parameters.

Section 4.7 sets mandatory quality control requirements for the analysis of organic compounds. Laboratories will be required to analyze spiked reference materials when performing pesticide and herbicide analyses. Also, all quality control requirements listed in the USEPA approved gas chromatographic methodologies will be a mandatory part of the quality control program and documentation of all quality control checks will be required.

Subchapter 5: Radiological Testing

Subchapter 5 establishes the Department's requirements for certified laboratories and laboratories seeking certification to follow when performing radiological analyses on drinking water samples within the new radiological category. To ensure the quality of the radiological data being generated by certified laboratories, the Department decided that the requirements and recommendations listed in USEPA's "Manual for the Interim Certification of Laboratories Involved in Analyzing Public Drinking Water Supplies" will all be mandatory requirements for certification. As in subchapters 3 and 4, all drinking water samples collected for radiological analysis must follow chain of custody procedures. These procedures will make it possible to identify all responsible parties who handled the sample, as well as, the sample preservation steps taken and the holding time used.

Subchapter 6 establishes the Department's requirements which a certified laboratory or a laboratory seeking certification shall continually meet and follow when performing bioassay analyses. This subchapter was modeled after the USEPA's "Interim NPDES Compliance Biomonitoring Inspection Manual", MCD-62, with specific references to Standard Methods for the Examination of Water and Wastewater, American Public Health Association, 14th Edition, USEPA's "Bioassay Procedures for the Ocean Disposal Permit Program", EPA-600/9-78-010, and "Recommended Bioassay Procedure for Fathead Minnow, Pimephales promelas Rafinesque, Chronic Tests", USEPA National Water Quality Laboratory, January 1979.

As with subchapters 3, 4, and 5, samples collected for bioassay analysis will be required to follow chain of custody procedures that will identify all responsible parties who handled the sample, as well as, the sample holding time. The sampling procedures specified in sections 6.4(b) 3 and 4 are procedures that are used by the USEPA for obtaining representative effluent samples. It is the Department's intent to adopt these sampling procedures as mandatory requirements.

In writing subchapter 6, it was decided that the specific test organisms used in the bioassay be standardized. The fathead minnow, Pimephales promelas, and the mysid shrimp, Mysidopsis bahia, were selected based upon the Department's past experience with the bioassay test procedure and consultation with the USEPA, Region II. The USEPA also recommends that twenty test organisms be used per effluent concentration and that all tests be performed in replicate. The Department agrees with this recommendation and is incorporating it as a mandatory requirement for certification. Section 4.5 also states the procedures to be used in analyzing the data generated from the bioassay. These procedures were selected to provide the Department with a clear and complete description of the level of acute toxicity measured.

IV Program Goal

The goal of this proposed regulation is to determine the quality of all water data presently being submitted to the Department and establish standards and criteria which will ensure the Department that the data submitted in the future will be qualitatively and quantitatively satisfactory. This will be accomplished by adopting and enforcing more stringent quality control requirements for drinking water certification and adopting and enforcing a wastewater certification program. In the future the Department intends to expand this regulation to cover solid waste, hazardous waste and sludge analysis, and at the same time make changes to the water and wastewater regulation based on the knowledge gained while operating the program.

