RULES

GOVERNING LABORATORY CERTIFICATION

SECTION .0001 THROUGH .0330

OF THE

NORTH CAROLINA ADMINISTRATIVE CODE

TITLE 10

DEPARTMENT OF HUMAN RESOURCES

CHAPTER 9

HEALTH SERVICES: LABORATORY SECTION

SUBCHAPTER 9D

LABORATORY CERTIFICATION

NORTH CAROLINA
DEPARTMENT OF HUMAN RESOURCES
DIVISION OF HEALTH SERVICES
LABORATORY SECTION
EFFECTIVE
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.0301 POLICY

A laboratory wishing to perform analyses of public water systems pursuant to 10NCAC 10D .1610-.1635 must be certified by the Department of Human Resources, Division of Health Services, Laboratory Section and shall meet the minimum requirements for certification contained in .0302-.0330 of this section for each particular test category it wishes to perform. A laboratory may also be acceptable if certified by the Environmental Protection Agency or by the certification program of another state with primary enforcement responsibility.

.0302 NOTICE AND PROCEDURE

(a) A laboratory seeking certification must request in writing an application for certification from the Department of Human Resources, Division of Health Services, Laboratory Section, 306 North Wilmington Street, Raleigh, North Carolina, 27611. The application for certification shall include:

(1) Name and address of the laboratory and its owner(s) or directors,
(2) Names and qualifications of the laboratory personnel,
(3) Test categories for which certification is requested,
(4) Description of facilities, equipment, and methodologies,
(5) Such other information as the Department of Human Resources deems necessary for certification purposes.

(b) Upon review of the application by a state laboratory certification evaluator, an on-site visit shall be scheduled to evaluate the laboratory premises.

(c) A written report describing deviations from minimum requirements will be prepared by the laboratory certification evaluator and submitted to the laboratory director or other responsible person. Within 30 days of receiving the written report, the laboratory shall submit a letter with supporting documents (records, reports, data, purchase orders, etc.) showing the action taken to comply with the minimum requirements. The letter shall be sent to the laboratory certification evaluator for review.

.0303 CERTIFICATION AND CERTIFICATION RENEWAL

(a) The Department of Human Resources shall grant certification for the test categories requested within 30 days of finding that a laboratory meets the minimum requirements set forth in this section.

(b) A laboratory shall renew its certification every two years. The renewal shall be based upon an on-site evaluation by a laboratory certification evaluator and compliance with the minimum requirements of this section.

(c) The certificate shall remain the property of the Department of Human Resources and shall be surrendered upon revocation pursuant to .0327 of this section.

.0327 REVOCATION, DOWNGRADING OR DENIAL

(a) The Department of Human Resources or its delegate may revoke or deny laboratory certificaton when there is substantial evidence that a laboratory or its employees have done any of the following:

(1) Knowingly made false statements on any documents associated with certification,
(2) Falsified results of analyses,
(3) Demonstrated incompetence or made consistent errors in analyses,

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(4) Failed to employ approved laboratory methodology in the performance of the analyses required by 10 NCAC 10D .1610-.1635,
(5) Failed to correctly analyze performance evaluation samples or failed to report the results within 45 days of receipt of the performance evaluation samples,
(6) Failed to report analytical results or to maintain records as required by this section,
(7) Failed to maintain facilities and equipment in accordance with the minimum requirements of this section,
(8) Violated or aided and abetted in the violation of any provision of this section.

(b) The Department of Human Resources or its delegate may downgrade a laboratory certificate to provisional status for the reasons set forth in (a) rather than revoke certification. A laboratory may continue to perform analyses with a provisional certificate. The provisional status shall continue for six months. At the end of six months the laboratory certification shall be reinstated if the laboratory has made corrections and is in compliance with the minimum requirements of this section. If no corrections have been made the laboratory certificate may be revoked.

(c) The Department of Human Resources or its delegate shall notify a laboratory of its intent to revoke, downgrade to provisional status or deny certification. The notice shall be in writing and include reasons for the decision and shall be delivered by certified mail. A laboratory may request a formal hearing on the decision by submitting a written request for an administrative hearing within 30 days of receipt of the notice. When a hearing is requested it shall be held in accordance with the rules contained in 10NCAC 1B .0200.

(d) This rule shall not preclude informal conferences concerning a decision to revoke, downgrade to provisional status or deny certification.

.0328 RECERTIFICATION

(a) A laboratory is eligible for recertification six months after revocation of its certificate, except in the following instances:
(1) A laboratory which lost certification because of false statements on documents or falsified analytical results is eligible for recertification one year after revocation, or when the person responsible for false statements or results is no longer associated with the laboratory.
(2) A laboratory which lost certification for failure to correctly analyze performance evaluation samples is eligible for recertification after correctly analyzing a performance evaluation sample.

(b) Application for recertification shall be made in the same way as application for certification as contained in .0302 of this section.

.0329 CONTRACT LABORATORIES

(a) A laboratory may sub-contract analytical work to another laboratory if the sub-contracting laboratory has been certified by the Department of Human Resources, by another state's certification program or by the EPA for the appropriate parameters.

(b) Laboratory reports must indicate the laboratory which performs the analysis, including the name of the sub-contracting laboratory.

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.0330 RECIPROCITY

Laboratories which have been certified under equivalent programs in other states or by the EPA are eligible for reciprocal certification in North Carolina. A notarized copy of the certificate and a copy of the program, if requested, must be received by the Department of Human Resources with the application for certification.

.0304 CHEMISTRY FACILITIES

A laboratory seeking certification for chemical analyses of public water supplies shall have the following facilities:

(1) sink with hot and cold running water,
(2) electricity,
(3) source of distilled and/or deionized water (depending on parameters measured),
(4) exhaust hood or equivalent for analysis of organic chemicals and trace metals,
(5) 200 square feet of space per person and 6 linear feet of usable bench space per analyst.

.0305 CHEMISTRY EQUIPMENT

The only instruments required are those needed to perform the chemical analyses for which the laboratory is being certified, but those instruments should meet the following specifications:

(1) Analytical balance. Should provide sensitivity of at least 0.1 mg.
(2) Photometer
   (a) Spectrophotometer. Usable wavelength range, 400 to 700 nm. Maximum spectral bandwidth, no more than 20 nm. Wavelength accuracy, 0 $\pm$ 2.5 nm. Photometer should be capable of using several sizes and shapes of absorption cells providing a sample path length varying from approximately 1 to 5 cm.
   (b) Filter photometer (abridged spectrophotometer). Capable of measuring radiant energy in range of 400 to 700 nm. Relatively broad bands (10 to 75 nm) of this radiant energy are isolated by use of filters at or near the maximum absorption of the colorimetric methods. Photometer should be capable of using several sizes and shapes of absorption cells providing a sample path length varying from approximately 1 to 5 cm.
(3) Magnetic stirrer. Variable speed, 120 V, with Teflon-coated stirring bar.
(4) pH meter. Accuracy, $\pm$ 0.05 units. Scale readability, $\pm$ 0.1 units. Laboratories purchasing a new pH meter are strongly advised to purchase one capable of functioning with specific ion electrodes (see following item). Unit may be line/bench or battery/portable operated.
(5) Specific ion meter. Readable and accurate to $\pm$ 5 mV. Unit may be line/bench or battery/portable operated.
(6) Atomic absorption spectrophotometer. 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 a strip chart recorder.

(7) Recorder for atomic absorption. Strip chart recorder having a chart width of 10 in or 25 cm, a full scale response time of 0.5 s or less, 10- or 100-mV input to match the instrument, and variable chart speeds of 5 to 50 cm/min, or equivalent.

(8) Gas chromatograph (equipped with an electron-capture detector). A commercial or custom-designed gas chromatograph with a column oven capable of isothermal temperature control to at least 220°C ± 0.2°C. System should be equipped with accurate needle-valve gas-flow controls, accept 1/4-in glass columns with the option of direct on-column injection. System must be demonstrated to be suitable for chlorinated hydrocarbon pesticides, with a minimum of decomposition and loss of compounds of interest.

(9) Recorder for gas chromatograph. Strip chart recorder having a chart width of 10 in or 25 cm, a full scale response time of 1 s or less, 1-mV (-0.05 to 1.05) signal to match the instrument, and variable chart speeds of 5 to 50 cm/min or equivalent.

(10) Conductivity meter. Suitable for checking distilled water quality. Should be readable in ohms or mhos, have a range of 2 to 2.5 million ohms or equivalent micromhos ± 1 percent, and have a sensitivity of 0.33 percent or better. Unit may be line/bench or battery/portable operated.

(11) Drying oven. Gravity and mechanical convection units with selectable temperature control from room temperature to 170° C or higher.

(12) Desiccator. Glass or plastic models, depending on particular application.

(13) Hot plate. Large or small units with selectable temperature controls for safe heating of laboratory reagents.

(14) Refrigerator. A standard kitchen type domestic refrigerator for storage of aqueous reagents and samples. For storing organics and flammable materials, an "explosion-proof" type of refrigerator should be used. When refrigeration is not required, an explosion-proof cabinet may be used.

(15) Glassware. Should be of Pyrex or Kimax type glass, which is more resistant than regular soft glass to damage by heat, chemicals, and abuse. All volumetric glassware should be marked Class A, denoting that it meets Federal Specifications and need not be calibrated before used.

(16) Stirred boiling water bath. For ambient temperature to 100° C (with gable lid).

(17) Thermometer. Instrument shall be that required by the individual method of analysis.

(18) Automatic analysis system. Equipment shall be that required by the individual method of analysis.

(19) Laboratories performing analyses for trihalomethanes must have the following equipment:

(a) TTHM by purge and trap. The instrument must be temperature programmable from 45° to 220° C at about 8°C/min and equipped with either a microcoulometric titration or electrolytic conductivity detector.

(b) TTHM by liquid/liquid extraction. The instrument must be equipped with a linearized (frequency modulated) electron capture detector.

(c) TTHM by gas chromatography/mass spectrometry. The gas chromatograph which must be temperature programmable, should be interfaced to the mass spectrometer with an all-glass enrichment device and an all-glass transfer line. Mass spectral data are to be obtained with electron-impact ionization at a nominal electron energy of 70 eV. The mass spectrometer must produce a spectrum that meets all criteria in Table I (Appendix, page a) when 50 ng or less of p-bromofluorobenzene is introducted into the gas chromatograph. An interfaced data system is required to acquire, store, reduce and output mass spectral data. The data system must be equipped with software to acquire and manipulate data for only a few ions that were detected as characteristic of trihalomethanes and the internal standard (or surrogate compound).

(d) Purge and trap system must be a commercial or custom-designed system containing three separate elements. When used with a compatible gas chromatograph, the assembly must be able to detect .05 ug/l of each of the individual trihalomethanes and measure them with a reproducibility not to exceed 8% relative standard deviations at 20 ug/l. The system shall also meet the following requirements:

(i) Purging device. Must be designed for a 5 ml sample volume with a minimum of headspace. Gas inlet must disperse finely divided gas bubbles through the sample.

(ii) Trapping device. Must be capable of retaining purged trihalomethanes at room temperatures.

(iii) Desorber assembly. Must be capable of heating the trapping-device to 180°C in one minute with less than 40°C overshoot.

(e) Sample container. Total trihalomethane sample bottle is required.

(f) Supplemental equipment. Must have constant temperature storage container, water bath or incubator, 25°C or above.

.0306 CHEMISTRY GENERAL LABORATORY PRACTICES

(a) Distilled/deionized water used for chemical analyses shall have resistivity values greater than 0.5 megohms (less than 2.0 micromhos)/cm at 25°C. Megohms are related to micromhos in the following manner:

$$\frac{1}{\text{megohms}} = \text{micromhos}, \quad \frac{1}{\text{micromhos}} = \text{megohms}$$

Quality of distilled/deionized water shall be maintained by sealing from the atmosphere. Quality checks shall be made at monthly intervals and documented.

(b) Analytical reagent grade (AR) chemicals shall be used for chemical analyses.

.0307 CHEMISTRY METHODOLOGY

(a) Minimum equipment requirements and methodology for individual parameters of chemical analyses shall be in accordance with methods adopted in 10 NCAC 10D .1624; 10NCAC 10D .1625; and 10 NCAC 10D .1635.

(b) Sodium, free chlorine residual, turbidity and the several tests required for the calculation of corrosivity do not need to be performed in certified laboratories. Approved methodology for these tests and the required

equipment shall be in accordance with methods adopted in 10 NCAC 10D .1623 for turbidity; 10 NCAC 10D .1636 for sodium; and 10 NCAC 10D .1621 for corrosivity. The test for free chlorine residual shall be made in accordance with the colorimetric or titrimetric DPD methods found in "Standard Methods for the Examination of Water or Wastewater", Method 409E or F, 14th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1975.

(c) A list of these methods may be obtained from the Laboratory Section, Division of Health Services, North Carolina Department of Human Resources, 306 North Wilmington Street, Raleigh, North Carolina (Appendix).

(d) All other procedures are considered alternative analytical techniques as described in 10 NCAC 10D .1630. Application for use of alternative methods may require acceptable comparability data.

.0308 CHEMISTRY SAMPLES

(a) Requirements for container types, preservatives and holding times for individual parameters of inorganic chemical analyses are shown in Table II (Appendix, page b).

(b) Requirements for container types, preservatives and holding times for individual parameters of organic chemical analyses are shown in Table III (Appendix, page c).

.0309 CHEMISTRY QUALITY CONTROL

(1) All quality control data shall be available for inspection.

(2) A laboratory shall analyze an unknown performance sample (when available) once per year for parameters measured. Results must be within the control limits, established by EPA for each analysis for which the laboratory is or wishes to be certified. If results are not within the control limits, a follow-up performance sample shall be analyzed and results must be within control limits established by EPA.

(3) The minimum daily quality control shall be as follows:

(a) Inorganic contaminants:

(1) A standard reagent curve composed of a minimum of a reagent blank and three standards covering the concentration range of the samples must be prepared.

(2) For each day on which analyses are performed, the standard curve must be verified by use of at least a laboratory method blank and one standard at or below the MCL. Daily checks must be within $\pm$ 10 percent of the original curve.

(3) If 20 or more samples per day are analyzed, the working standard curve must be verified by running an additional standard at or near the MCL every 20 samples. Each check must be within $\pm$ 10 percent of the original curve.

(b) Organic Contaminants:

(1) For each day on which analyses are initiated, a laboratory method blank must be analyzed with the same procedures used to analyze samples.

(2) A minimum of three calibration standards must be analyzed each day to calibrate the gas chromatographic system. If the laboratory can thereby demonstrate that the instrument response

is linear through the origin, this requirement can be reduced to one standard at or near the MCL; providing the response of the standard is within ± 10 percent of previous calibrations.

(3) If 5 or more samples per day are analyzed, an additional calibration standard must be run at or near the average sample concentration found. Checks must be within ± 10 percent of the original calibration standard.

(4) Each quarter, the laboratory must analyze a certified TTHM laboratory control standard (USEPA Quality Control Sample or equivalent). If errors exceed 20 percent of the true value, corrective action must be taken and documented.

(5) The laboratory must analyze 10 percent of all samples for TTHM in duplicate. A continuing record of all calibration checks (accuracy) and duplicates (precision) must be maintained.

(6) The laboratory must maintain records of retention times for each trihalomethane and of actions taken when they vary more than 10 percent from an established norm.

(7) If a mass spectrometer detector is used for TTHM analysis, the mass spectrometer performance tests described under equipment specifications using BFB must be conducted once during each 8-hour work shift, and records of satisfactory performance and corrective action must be maintained. Criteria for mass spectrometer performance tests are contained in Table I (Appendix, page a).

(8) The laboratory must analyze a known laboratory control standard each day and calculate detection limits based upon the responses found.

(9) Laboratories that analyze TTHM by liquid-liquid extraction must demonstrate access to a purge-and-trap instrument when necessary to confirm electron capture identifications.

(4) A thermometer certified by the National Bureau of Standards (or one of equivalent accuracy) shall be available to check thermometers in ovens, etc.

.0310 CHEMISTRY DATA

(a) Records of chemical analyses should be kept by the laboratory for not less than one year. Enforcement data shall be kept for three years. This includes all raw data, calculations, quality control data, and reports.

(b) Actual laboratory reports may be kept. However, data, with the exception of compliance check samples as detailed in 10NCAC 10D .1632(2), may be transferred to tabular summaries. The following information should be included:

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

(2) identification of sample as to whether it is a routine distribution sample, check sample, raw or process water samples, or other special purpose sample;

(3) date of receipt of sample and analysis;

(4) laboratory and persons responsible for performing analysis;

(5) analytical techniques/method used;

(6) results of analysis.

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.0311 CHEMISTRY ACTION RESPONSE

When action response is a designated laboratory responsibility, the proper authority shall be promptly notified of unsatisfactory samples, and a request shall be made for resampling from the same sampling point.

.0312 MICROBIOLOGY FACILITIES

A laboratory seeking certification for microbiological analyses of public water supplies shall have 200 square feet of space per person and 6 linear feet of bench space per analyst.

.0313 MICROBIOLOGY EQUIPMENT

A laboratory seeking certification for microbiological analyses of public water supplies shall have available or access to the items required for the total coliform membrane filter or most probable number procedures as listed below:

(1) pH Meter. Accuracy must be $\pm$ 0.1 units.

(2) Balance-top loader or pan. Balance must be clean, not corroded, and be provided with appropriate weights of good quality. Balance must tare out and detect 50-mg weight accurately: this sensitivity is required for use in general media preparation of 2g or larger quantities.

(3) Temperature-monitoring devices:
   (a) Glass or metal thermometers must be graduated in 0.5°C or smaller increments.
   (b) Continuous temperature recording devices must be sensitive to within 0.5°C.
   (c) Liquid column of glass thermometers must have no separation.
   (d) A certified thermometer or one of equivalent accuracy must be available.

(4) Air (or water jacketed) incubator/incubator rooms/waterbaths/aluminum block incubators:
   (a) Unit must maintain internal temperature of 35.0° $\pm$ 0.5°C in area of use at maximum loading.
   (b) When aluminum block incubators are used, culture dishes and tubes must be snug-fitting in block.

(5) Autoclave:
   (a) Autoclave must be in good operating condition when observed during operational cycle or when time-temperature charts are read.
   (b) Autoclave must have pressure and temperature gauges on exhaust side and an operating safety valve.
   (c) Autoclave must reach sterilization temperature (121°C) and be maintained during sterilization cycle: no more than 45 minutes is required for a complete cycle.
   (d) Depressurization must not produce air bubbles in fermentation media.

(6) Hot-air oven. Oven must be constructed to ensure a stable sterilization temperature.

(7) Refrigerator. Refrigerator must hold temperature at 1° to 5°C.

(8) Optical/counting/lighting equipment: Low power magnification device (preferably binocular microscope with 10 to 15x) with fluorescent light source must be available for counting MF colonies.

(9) Inoculation equipment: Loop diameter must be at least 3 mm and of 22 to 24 gauge Nichrome, chromel, or platinum-iridium wire. Single-service metal loops, disposable dry heat-sterilized hardwood applicator sticks, pre-sterilized plastic, or metal loops may be used (optional).

(10) Membrane filtration equipment:
   (a) Units must be made of stainless steel, glass, or autoclavable plastic. Equipment must not leak and must be uncorroded.
   (b) Field equipment is acceptable for coliform detection only when standard laboratory MF procedures are followed.

(11) Membrane filters and pads:
   (a) Membrane filters must be manufactured from cellulose ester materials, white, grid-marked, 47-mm diameter, 0.45-um pore size. Another pore size may be used if the manufacturer gives performance data equal to or better than the 0.45-um membrane filter.
   (b) Membranes and pads must be autoclavable or presterilized.

(12) Laboratory glassware, plastic ware, and metal utensils:
   (a) Except for disposable plastic ware, items must be resistant to effects of corrosion, high temperature, and vigorous cleaning operations.
   (b) Flasks, beakers, pipets, dilution bottles, culture dishes, culture tubes, and other glassware must be of borosilicate glass and free of chips, cracks, or excessive etching. Volumetric glassware should be Class A, denoting that it meets Federal specifications and need not be calibrated before use.
   (c) Plastic items must be of clear, inert, nontoxic material and must retain accurate calibration marks after repeated autoclaving.

(13) Culture dishes:
   (a) Sterile tight or loose-lid plastic culture dishes or loose-lid glass culture dishes must be used.
   (b) For loose-lid culture dishes, relative humidity in the incubator must be at least 90 percent.
   (c) Culture dish containers must be aluminum or stainless steel; or dishes may be wrapped in heavy aluminum foil or char-resistant paper.
   (d) Open packs of disposable sterile culture dishes must be resealed between uses.

(14) Culture tubes and closures:
   (a) Culture tubes must be made of borosilicate glass or other corrosion resistant glass and must be of a sufficient size to contain the culture medium, as well as the sample portions employed, without being more than 3/4 full.
   (b) Caps must be snug-fitting stainless steel or plastic; loose-fitting aluminum caps or screw caps are also acceptable.

(15) Measuring equipment:
   (a) Sterile, glass or plastic pipets must be used for measuring 10 ml or less.
   (b) Pipets must deliver the required volume quickly and accurately within a 2.5 percent tolerance.
   (c) Pipets must not be badly etched: mouthpiece or delivery tips must not be chipped; graduation marks must be legible.
   (d) Open packs of disposable sterile pipets must be resealed between uses.

(e) Pipet containers must be aluminum or stainless steel.

(f) Graduated cylinders must be used for samples larger than 10 ml; calibrated membrane filter funnel markings are permissible provided accuracy is within 2.5 percent tolerance.

.0314 MICROBIOLOGY GENERAL LABORATORY PRACTICES

(a) The following sterilization procedures shall be used for microbiological analyses:

(1) The following times and temperatures shall be used for autoclaving materials:

Material	Temperature/Minimum Time
Membrane filters 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 blank	121°C/30 min.

(2) Membrane filter assemblies must be sterilized between sample filtration series. A filtration series ends when 30 minutes or longer elapse between sample filtrations. At least 2 minutes of UV light or boiling water may be used on membrane filter assembly to prevent bacterial carry-over between filtrations.

(3) Dried glassware must be sterilized at a minimum of 170°C for 2 hours.

(b) Laboratory pure water (distilled, deionized, or other processed waters) used for microbiological analyses must meet the following requirements:

(1) An analyst must test the quality of the laboratory pure water or have it tested by the State or by a State-authorized laboratory.

(2) Only water determined as laboratory pure water (see .0317 of this Section) can be used for performing bacteriological analyses.

(c) Rinse and dilution water used for microbiological analyses shall meet the following standards:

(1) Stock buffer solution must be prepared according to "Standard Methods for the Examination of Water and Wastewater", 14th Edition, using laboratory pure water adjusted to pH 7.2. Stock buffer must be autoclaved or filter-sterilized, labeled, dated, and stored at 1° to 5°C. The stored buffer solution must be free of turbidity.

(2) Rinse and dilution water must be prepared by adding 1.25 ml of stock buffer solution per liter of laboratory pure water. Final pH must be 7.2 ± 0.1.

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(d) The following are minimum requirements for storing and preparing media used for microbiological analyses:

(1) Laboratories must use commercial dehydrated media for routine bacteriological procedures as quality control measures.

(2) Lauryl tryptose and brilliant green lactose bile broths must be prepared according to "Standard Methods";

(3) Dehydrated media containers must be kept tightly closed and stored in a cool, dry location. Discolored or caked dehydrated media cannot be used.

(4) Laboratory pure water must be used; dissolution of the media must be completed before dispensing to culture tubes or bottles.

(5) The membrane filter broth and agar media must be heated in a boiling water bath until completely dissolved.

(6) Membrane filter (MF) broths must be stored and refrigerated no longer than 96 hours. MF agar media must be stored, refrigerated and used within two weeks.

(7) Most probable number (MPN) media prepared in tubes with loose-fitting caps must be used within 1 week. If MPN media are refrigerated after sterilization, they must be incubated overnight at 35°C to confirm usability. Tubes showing growth or gas bubbles must be discarded.

(8) Media in screw-cap containers shall only be held for three months. The media shall be stored in the dark and evaporation shall not be excessive (0.5 ml per 10 ml total volume). Commercially prepared liquid and agar media supplies may be used.

(9) Ampouled media must be stored at 1° to 5°C; time must be limited to manufacturer's expiration date.

.0315 MICROBIOLOGY METHODOLOGY

(a) Minimum equipment requirements and methodology for microbiological analyses shall be in accordance with methods adopted in 10 NCAC 10D .1622. A list of these methods is available from the Laboratory Section, Division of Health Services, Department of Human Resources, 306 North Wilmington Street, Raleigh, North Carolina (Appendix).

(b) Tentative methods are not acceptable. All other procedures are considered alternative analytical techniques as described in 10 NCAC 10D .1630. Application for the use of alternative methods may require acceptable comparability data.

(c) The membrane filter procedure is preferred because it permits analysis of large sample volumes in reduced analysis time. The membranes should show good colony development over the entire surface. The golden green metallic sheen colonies should be counted and recorded as the coliform density per 100 ml of water sample. The following rules for reporting any problem with MF results shall be observed:

(1) Confluent growth: Growth (with or without discrete sheen colonies) covering the entire filtration area of the membrane. Results are reported as "confluent growth per 100 ml, with (or without) coliforms," and a new sample requested.

(2) TNTC (Too numerous to count): The total number of bacterial colonies on the membrane is too numerous (usually greater than 200 total colonies), not sufficiently distinct, or both. An accurate count cannot be made. Results are reported as "TNTC per 100 ml, with (or without) coliforms," and a new sample requested.

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(3) Confluent growth and TNTC: A new sample must be requested, and the sample volumes filtered must be adjusted to apply the MF procedure; otherwise the MPN procedure must be used.

(4) Confirmed MPN test on problem supplies: If the laboratory has elected to use the MPN test on water supplies that have a continued history of confluent growth or TNTC with the MF procedure, all presumptive tubes with heavy growth without gas production should be submitted to the confirmed MPN test to check for the suppression of coliforms. A count is adjusted based upon confirmation and a new sample requested. This procedure should be carried out on one sample from each problem water supply once every three months.

.0316 MICROBIOLOGY SAMPLES

(1) When the laboratory has been delegated responsibility for sample collecting, handling, and preservation, there must be strict adherence to correct sampling procedures, complete identification of the sample, and prompt transfer of the sample to the laboratory as described in "Standard Methods for the Examination of Water and Wastewater", 14th Edition, pp. 38-44.

(2) The sample must be representative of the potable water system. The sampling program must include examination of the finished water at selected sites that systematically cover the distribution network.

(3) Minimum sample frequency must be that specified in 10 NCAC 10D .1622.

(4) The collector must be trained in sampling procedures and approved by the State regulatory authority or its delegated representative.

(5) The water tap must be sampled after maintaining a steady flow for two or three minutes to clear service line. The tap is free of aerator, strainer, hose attachment, or water purification devices.

(6) The sample volume must be a minimum of 100 ml. The sample bottle must be filled only to the shoulder to provide space for mixing.

(7) The sample report form must be completed immediately after collection with location, date and time of collection, chlorine residual, collector's name, and remarks.

(8) Sample bottles must be of at least 120 ml-capacity, sterile plastic or hard glass, wide-mouthed with stopper or plastic screw-cap, and capable of withstanding repeated sterilization. Sodium thiosulfate (100 mg/l) is added to all sample bottles during preparation. As an example, 0.1 ml of a 10 percent solution is required in a 4-oz. (120-ml) bottle.

(9) **Date and time of sample arrival must be added to the sample report form when sample is received in the laboratory.**

(10) **State regulations relating to chain-of-custody, if required, must be followed in the field and in the laboratory.**

(11) Samples delivered by collectors to the laboratory must be analyzed on the day of collection.

(12) Where it is necessary to send water samples by mail, bus, United Parcel Service, courier service, or private shipping, holding/transit time between sampling and analyses must not exceed 30 hours.

(13) If the laboratory is required by State regulation to examine samples after 30 hours and up to 48 hours, the laboratory must indicate that the data may be invalid because of excessive delay before sample

processing. Samples arriving after 48 hours shall be refused without exception and a new sample requested. (The problem of holding time is under investigation by EPA).

.0317 MICROBIOLOGY QUALITY CONTROL

Requirements for quality control of microbiological analyses are as follows:

(1) A written description for current laboratory quality control program must be available for review. Management, supervisors, and analysts participate in setting up the quality control program. Each participant should have a copy of the quality control program and a detailed guide of his own portion. A record on analytical quality control tests and quality control checks on media materials, and equipment shall be prepared and retained for three years.

(2) The minimum requirements for analytical quality control tests for general practices and methodology are:

(a) All sheen or borderline sheen colonies, or at least five sheen or borderline sheen colonies must be verified from each membrane containing such colonies. Counts must be adjusted based on verification. The verification procedure must be conducted by transferring growth from colonies into lauryl tryptose broth (LTB) or lactose broth (LB) tubes and then transferring growth from gas-positive LTB or LB cultures to brilliant green lactose bile (BGLB)tubes. Colonies must not be transferred exclusively to BGLB because of the lower recovery of stressed coliforms in this more selective medium. However, colonies may be transferred to LTB and BGLB simultaneously. Negative LTB tubes must be reincubated a second day and confirmed if gas is produced. It is desirable to verify all sheen and borderline sheen colonies.

(b) A start and finish MF control test (rinse water, medium, and supplies) must be conducted for each filtration series. If sterile controls indicate contamination, all data on samples affected must be rejected and a request made for immediate resampling of those waters involved in the laboratory error.

(c) The MPN test must be carried to completion, except for gram staining, on 10 percent of positive confirmed samples. If no positive tubes result from potable water samples, the completed test except for gram staining must be performed quarterly on at least one positive source water.

(d) Laboratory pure water must be analyzed annually by the test for bacteridical properties for distilled water as described in "Standard Methods for the Examination of Water and Wastewater", 14th Edition, pp. 888-892. Only satisfactorily tested water is permissible in preparing media, reagents, rinse, and dilution water. If the tests do not meet requirements, corrective action must be taken and the water retested.

(e) Laboratory pure water must be analyzed monthly for conductance, pH, chlorine residual, and standard plate count. If tests exceed requirements, corrective action must be taken and the water retested.

(f) Laboratory pure water must not be in contact with heavy metals. It must be analyzed initially and annually thereafter for trace metals (especially Pb, Cd, Cr, Cu, Ni, and Zn). If tests do not meet the requirements, corrective action must be taken and the water retested.

(g) Standard plate count procedure must be performed as described in"Standard Methods for the Examination of Water and Wastewater", 14th Edition, pp. 908-913.
Plates must be incubated at 35°$\pm$ 0.5°C for 48 hours.

(h) Requirements for laboratory pure water are:
  (i) pH measures 5.5-7.5;
  (ii) conductivity measures greater than 0.5 megohms as resistivity or less than 2 micromhos/cm at 25°C,
  (iii) trace metals:
    (A) a single metal measures not greater than 0.05 mg/l,
    (B) total metals measure equal to or less than 1.0 mg/l,
  (iv) test for bactericidal properties of distilled water measures 0.8-3.0, as described in "Standard Methods for the Examination of Water and Wastewater", 14th Edition, pp. 888-892.
  (v) free chlorine residual measures less than 0.1 mg/l
  (vi) standard plate count measures less than 1000 (freshly distilled or ultra pure) and less than 10,000/ml (stored or deionized).

(i) Laboratory must analyze one quality control sample per year (when available) for parameter(s) measured.

(j) Laboratory must satisfactorily analyze one unknown performance sample per year (when available) for parameter(s) measured.

(3) The minimum requirements for quality control checks of laboratory media, equipment, and supplies are:

(a) pH meter must clean and calibrated each use period with pH 7.0 standard buffer. Buffer aliquot must be used only once. Commercial buffer solutions must be dated on initial use.

(b) Balances (top loader or pan) must be calibrated annually.

(c) Glass thermometers or continuous recording devices for incubators must be checked yearly and metal thermometers quarterly (or at more frequent intervals when necessary) against a certified thermometer or one of equivalent accuracy.

(d) Temperature in air (or water jacketed) incubator/incubator room/water-baths/aluminum block incubators must be recorded continuously or recorded daily from in-place thermometer(s) immersed in liquid and placed on top and bottom shelves.

(e) The autoclave must be checked at least weekly with a maximum registering thermometer calibrated in 1°C divisions.

(f) Hot air oven must be equipped with a thermometer calibrated in the range of 170°C or with a temperature recording device. Records must be maintained showing date, time, and temperature of each sterilization cycle.

(g) Membrane filters used must be those recommended by the manufacturer for water analysis. The recommendation must be based on data relating to ink toxicity, recovery, retention, and absence of growth-promoting substances.

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(h) Washing processes must provide clean glassware with no stains or spotting. With initial use of a detergent or washing product and whenever a different washing product is used, the rinsing process must demonstrate that it provides glassware free of toxic material by the inhibitory residue test as described in "Standard Methods for the Examination of Water and Wastewater", 14th Edition, p. 885.

(i) At least one bottle per batch of sterilized sample bottles must be checked by adding approximately 25 ml of sterile non-selective broth to each bottle. It must be incubated at $35^{o}\pm$ 0.5°C for 24 hours and checked for growth.

(j) Service contracts or approved internal protocols must be maintained on balances, autoclave, water still, etc., and the service records entered in a log book.

(k) Records must be available for inspection on batches of sterilized media showing lot numbers, date, sterilization time-temperature, final pH, and technician's name.

.0318 MICROBIOLOGY DATA

(a) Where the laboratory has the responsibility for microbiological sample collections, the sample collector should complete a sample report form immediately after each sample is taken. The information on the form includes sample identification number, sample collector's name, time and date of collection, arrival time and date in the laboratory, direct count, MF verified count, MPN confirmed count, analyst's name, and other special information.

(b) Results of microbiological analyses should be calculated and entered on the sample report form to be forwarded. A careful check should be made to verify that each result was entered correctly from the bench sheet and initialed by the analyst.

(c) All results of microbiological analyses should be reported immediately to the proper authority.

(d) Positive results from microbiological analyses are reported as preliminary without waiting for MF verification or MPN confirmation. After MF verification and/or MPN confirmation, the adjusted counts should be reported.

(e) A copy of the microbiological sample report form should be retained either by the laboratory or State program for three years. If results are entered into a computer storage system, a printout of the data should be returned to the laboratory for verification with bench sheets.

.0319 MICROBIOLOGY ACTION RESPONSE

When action response is a designated laboratory responsibility, the proper authorities shall be promptly notified on unsatisfactory microbiological sample results, and a request shall be made for resampling from the same sampling point.

.0320 RADIOCHEMISTRY FACILITIES

A laboratory seeking certification for performance of radiochemical analyses of public water supplies shall meet the following requirements:

(1) The counting instrument(s) required for measurement of those radionuclides described in the "Federal Register" (Vol. 41, No. 133, July 9, 1976) must be located in a room other than the one in which samples and standards are being prepared or in which other types of

chemical analyses are being performed. Temperature of this room must not exceed 27°C. Temperature variation under normal operating conditions must not exceed 3°C.

(2) All instruments must be properly grounded, and a regulated power supply, either external or internal, shall be available to each instrument.

(3) In areas where radioactive standards are being prepared, care must be taken to minimize contamination of surfaces and personnel. Either bench surfaces of an impervious material covered with absorbent paper, or trays (stainless steel, plastic, or fiberglass) lined with absorbent paper are acceptable.

(4) Laboratory space shall be 200 square feet per person and must contain no less than 6 linear feet of bench space per analyst and include the following:

(a) sink with hot and cold running water;
(b) electrical outlets (120 V a.c. grounded);
(c) source of distilled or deionized water;
(d) gas supply (natural gas or liquefied petroleum), a propane cylinder with proper attachments may be adequate in laboratories doing limited amounts of analytical work;
(e) vacuum line, pump, or aspirator;
(f) exhaust hood.

.0321 RADIOCHEMISTRY EQUIPMENT

The only instruments required are those needed to perform the specific radiochemical analyses for which the laboratory is being certified, but those instruments should meet the following specifications:

(1) General instrumentation and equipment specifications are:

(a) Analytical balance. Precision, ± 0.05 mg. Minimum scale readability, 0.1 mg

(b) pH meter or specific ion meter:

(i) pH meter. Accuracy, ± 0.5 units. Scale readability, ± 0.1 units. Instrument may be either line/bench or battery/portable.

(ii) Specific ion meter. Expanded scale millivolt capability. Readable and accurate to ± 0.1 mV. Instrument may be either line/bench or battery/portable.

(c) Conductivity Meter. Readable in ohms or mhos, a range of 2 to 2.5 million ohms or equivalent micromhos ± 1 percent, and a sensitivity of 0.33 percent or better. Meter may be either line/bench or battery/portable.

(d) Drying oven. Gravity convection type.

(e) Desiccator. Glass or plastic models, depending on particular application.

(f) Hot plate. Large or small units with selectable temperature control for safe heating of laboratory reagents and samples.

(g) Glassware. Borosilcate type glass. All volumetric glassware should be marked Class A, denoting that it meets Federal specifications and need not be calibrated before use.

(h) Muffle furnace. Automatically controlled with a chamber capacity of at least 2,200 cc (10 x 9.5 x 23) and a maximum operating temperature of 1,000°C continuous and 1,100°C intermittent.

(i) Centrifuge. General purpose table-top model with a maximum speed of at least 3,000 rpm and a loading option of 4 x 50 ml.

(j) Fluorometer. Capable of detecting 0.0005 ug of uranium.

(2) Radiation instrument specifications are:

(a) Liquid scintillation system. 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 will meet or exceed the requirements of 10 NCAC 10D .16.

(b) Gas-flow proportional counting system.

(i) A gas-flow proportional counting system may 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 section 141.25 (a). The detector may be either a "windowless" (internal proportional counter) or a "thin window" type. A minimum shielding equivalent to 5 cm of lead must surround the detector. A cosmic (guard) detector should be operated in anticoincidence with the main detector. The system shall be such that the sensitivity of the radioanalysis of water samples will meet or exceed the requirements of 10 NCAC 10D .16.

(ii) For measurement of gross alpha activities and radium-226, a scintillation system designed for alpha counting may be substituted for the gas-flow proportional counter described. In such a system, a Mylar disc coated with a phosphor (silver-activated zinc sulfide) is either placed directly on the sample or on the face of a photomultiplier tube, enclosed within a light-tight container, along with the appropriate electronics (high voltage supply, amplifier, timer, and scaler).

(c) Scintillation cell system. 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. The system consists of a light-tight enclosure capable of accepting the scintillation flasks, a detector (phototube), and the appropriate electronics (high volage supply, amplifier, timers, and scalers). The flasks (cells) required for this measurement may either be purchased from commercial suppliers or constructed according to published specifications (Lucas, H.,F., "Improved Low-Level Alpha Scintillation Counter for Radon," Rev.Sci. Instrum., 28:680, 1967).

(d) Gamma spectrometer systems.

(i) Either a sodium iodide (NaI(Tl)) crystal or a solid state lithium drifted germanium (Ge(Li)) detector connected to a multichannel analyzer is required if the laboratory is to be certified for analyses of manmade photon emitters.

(ii) If a sodium iodide detector is used, a 7.5 cm x 7.5 cm NaI cylindrical crystal is satisfactory. However, a 10 cm x 10 cm crystal is recommended. The detector must be shielded with a minimum of 10 cm of iron or equivalent. It is recommended that the distance from the center of the detector to any part of the shield should not be less than 30 cm. The multichannel analyzer, in addition to appropriate electronics, must contain a

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memory of not less than 200 channels and at least one readout device.

(iii) A system with a lithium drifted germanium (Ge(Li)) detector may be used for measurement of manmade photon emitters if the efficiency of the detector is such that the sensitivity of the system meets the minimum detectable activity requirements cited in 10 NCAC 10D .16. The Ge(Li) detector must be shielded with a minimum of 10 cm of iron or equivalent. The multi-channel analyzer, in addition to appropriate electronics, must contain a memory of not less than 2000 channels and at least one readout device.

.0322 RADIOCHEMISTRY GENERAL LABORATORY PRACTICES

A laboratory seeking certification for performing radiochemical analyses shall meet the following requirements:

(1) Glassware preparations. All glassware shall be washed in a warm detergent solution and thoroughly rinsed in tap water. A distilled water rinse shall follow the tap water rinse. This cleaning procedure is sufficient for most analytical needs. However, specific analytical methods may dictate the need for more elaborate procedures for ensuring cleanliness of glassware.

(2) Water Quality. All water used in preparation of reagents, standards, and samples shall have resistance values greater than 0.5 megohms (less than 2.0 micromhos)/cm at 25°C.

(3) Chemicals and reagents. "Analytical reagent grade" (AR) chemicals shall be used for most analyses.

(4) Storage of radioactive standards and radioactive wastes. There shall be an enclosed and properly labeled area, either within the analytical laboratory or in a separate room, for the safe storage (in suitable containers) of standards, samples, and radioactive wastes.

(5) Standards and sample preparation. There shall be a designated area within the laboratory for preparation of radioactive standards and samples. Adequate precautions shall be taken in this area to ensure against radioactive contamination. Provisions shall be made for safe storage and disposal of radioactive wastes and for monitoring of the work area.

.0323 RADIOCHEMISTRY METHODS AND SAMPLING

(a) Minimum requirements and methods for radiochemical analyses shall be made in accordance with methods adopted in 10 NCAC 10D .1626. A list of these methods may be obtained from the Laboratory Section, Division of Health Services, Department of Human Resources, 306 North Wilmington Street, Raleigh, North Carolina, Table IV (Appendix, page d).

(b) The minimum requirements of sample handling for radiochemical analyses, including preservation, and major instrumentation are shown in Table IV (Appendix, page d)

.0324 RADIOCHEMISTRY QUALITY CONTROL

Requirements for quality control of radiochemical analyses are as follows:

(1) Quality control data and records must be available for inspection.

(2) Laboratory must participate at least twice each year in those EPA laboratory intercomparison studies that include each of the analyses

for which the laboratory is or wants to be, certified. Analytical results must be within control limits described in "Environmental Radioactivity Laboratory Intercomparison Studies Program-FY-1977" (EPA-600/4-77-001), or in subsequent revisions.

(3) Laboratory must participate once each year in an appropriate unknown performance study administered by EPA. Analytical results must be within control limits established by EPA for each analysis for which the Laboratory is, or wants to be, certified.

(4) Operating manuals and calibration protocols for counting instruments must be available to analyst(s) and technician(s).

(5) Calibration data and maintenance records on all radiation instruments and analytical balances must be maintained in a permanent record.

(6) The following specifications are included in minimum daily quality control:

(a) To verify internal laboratory precision for a specific analysis, a minimum of 10-percent duplicate analyses must be performed. The difference between duplicate measurements must be less than two times the standard deviation of the specific analysis as described in EPA-600/4-77-001. If difference exceeds two standard deviations, prior measurements are suspect, calculations and procedures must be examined, and samples should be reanalyzed when necessary.

(b) When 20 or more specific analyses are performed each day, a performance standard and a background sample must be measured with each 20 samples. If less than 20 specific analyses are performed in any 1 day, a performance standard and a background sample must be measured along with the samples.

(c) Quality control performance charts, or performance records, must be maintained.

.0325 RADIOCHEMISTRY DATA

(a) Records of radiochemical analyses shall be kept for not less than three years. This includes all raw data, calculations, quality control data, and reports.

(b) Actual laboratory reports may be kept. However, all data with the exception of compliance check samples as detailed in 10 NCAC 10D .1632(2), may be transferred to tabular summaries provided that the following information is included:

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

(2) identification of sample as to whether it is a routine distribution system sample, check sample, raw or process water sample, surface or ground water sample, or other special purpose sample

(3) date of receipt of sample and analysis.

(4) laboratory and persons responsible for performing analysis.

(5) analytical technique/method used.

(6) results of analysis.

.0326 RADIOCHEMISTRY ACTION RESPONSE

When action response is a designated laboratory responsiblity, the water plant operator and state engineer shall be promptly notified of unsatisfactory sample results, and a request shall be made for resampling from same sampling point.

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APPENDIX

TABLE I. pBROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	*Ion Abundance Criteria*
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	96 to 100% of mass 174
177	5 to 9% of mass 176

TABLE II. SAMPLE COLLECTING, HANDLING, AND PRESERVATION FOR INORGANIC CONTAMINANTS[1]

Contaminant	Preservative[2]	Container[3]	Maximum Holding Time[4]
Alkalinity	Cool, 4°C	P or G	14 days
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
Calcium hardness	Cool, 4°C, Conc HNO_3 to pH <2	P or G	6 months
Chloride	None	P or G	7 days
Chromium	Conc HNO_3 to pH <2	P or G	6 months
Fluoride	None	P or G	1 month
Free chlorine residual	None	P or G	1 hour
Lead	Conc HNO_3 to pH <2	P or G	6 months
Mercury	Conc HNO_3 to pH <2	G	38 days
		P	14 days
Nitrate	Conc H_2SO_4 to pH <2	P or G	14 days
pH	None	P or G	6 hours
Selenium	Conc HNO_3 to pH <2	P or G	6 months
Silver	Conc HNO_3 to pH <2	P or G	6 months
Sodium	Conc HNO_3 to pH <2	P or G	6 months
Sulfate	Cool, 4°C	P or G	7 days
Temperature	None	P or G	None
Total filterable residue	Cool, 4°C	P or G	7 days
Turbidity	None	P or G	1 hour

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

[2]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.

[3]P = plastic, hard or soft; G = Glass, hard or soft.

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

TABLE III. SAMPLE COLLECTING, HANDLING AND PRESERVATION FOR ORGANIC CONTAMINANTS[1]

Contaminant	Preservative	Container	Maximum Holding Time[2]
Chlorinated hydrocarbons	Refrigerate at 4°C as soon as possible after collection	Glass with foil or Teflon-lined cap	14 days[3]
Chlorophenoxys	Refrigerate at 4°C as soon as possible after collection	Glass with foil	7 days[3]
TTHM	Sodium thiosulfate or sodium sulfite	Glass with Teflon-lined septum	14 days

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

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

[3]Well-stoppered and refrigerated extracts can be held up to 30 days.

TABLE IV. SAMPLE HANDLING, PRESERVATION, METHODOLOGY,[1] AND MAJOR INSTRUMENTATION (MINIMUM REQUIREMENTS)

Parameter	Preservative[2]	Container[3]	Instrumentation[4]
Gross alpha	Concl. HCl or HNO_3 to pH <2[5]	P or G	A, B or G
Gross beta	Concl. HCl or HNO_3 to pH <2[5]	P or G	A or G
Strontium-89	Concl. HCl or HNO_3 to pH <2	P or G	A or G
Strontium-90	Concl. HCl or HNO_3 to pH <2	P or G	A or G
Radium-226	Concl. HCl or HNO_3 to pH <2	P or G	A, B, or D or G
Radium-228	Concl. HCl or HNO_3 to pH <2	P or G	A or G
Cesium-134	Concl. HCl to pH <2	P or G	A, C, or G
Iodine-131	None	P or G	A or G
Tritium	None	G	E
Uranium	Concl. HCl or HNO_3 to pH <2	P or G	F
Photon emitters	Concl. HCl or HNO_3 to pH <2	P or G	C

[1]Federal Register, Volume 41, No. 133, July 9, 1976.

[2]It 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.

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

[4]A = Low background proportional system; B = Alpha scintillation system; C = Gamma spectrometer (NaI (TI)) or Ge (Li); D = Scintillation cell (radon) system; E = Liquid-scintillation system (section C.2.a); F = Fluorometer (section C.1.i); G = Low background alpha and beta counting system other than gas-flow proportional.

[5]If 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.

APPROVED METHODOLOGY FOR INORGANIC CONTAMINANTS

Contaminant	Methodology	Reference (Method Number) EPA[1]	ASTM[2]	SM[3]	Other
Alkalinity	Methyl orange titrimetric or Potentiometric	310.1	D1067-70B	403	-
Arsenic	Atomic absorption; furnace technique	206.2	-	-	-
	Atomic absorption; gaseous hydride	206.3	D2972-78B	301A-VII	I-1062-78[4]
	Spectrophotometric, silver diethyldithiocarbamate	206.4	D2972-78A	404A & 404B(4a)	-
Barium	Atomic absorption; direct aspiration	208.1	-	301A-IV	-
	Atomic absorption; furnace technique	208.2	-	-	-
Cadmium	Atomic absorption; direct aspiration	213.1	D3557-78A or B	301A-II or III	-
	Atomic absorption; furnace technique	213.2	-	-	-
Calcium Hardness	EDTA titrimetric	-	D1126-27	306C	-
	Atomic absorption; direct aspiration	215.1	-	301A-II & 309	-
Chloride	Potentiometric	-	-	408C	-
Chromium	Atomic absorption; direct aspiration	218.1	D1687-77D	301A-II or III	-
	Atomic absorption; furnace technique	218.2	-	-	-
Corrosivity	Langelier index	-	-	203	-
	Aggressive index	-	-	-	C400-77[5]
Fluoride	Colorimetric SPADNS; with distillation	340.1	D1179-72A	414 A and C	-
	Potentiometric ion selective electrode	340.2	D1179-72B	414B	-
	Automated Alizarin fluoride blue; with distillation	-	-	603	129-71W[6]
	Automated ion selective electrode	-	-	-	380-75WE[7]
	Zirconium eriochrome cyanine R; with distillation	-	-	-	I-3325-78[4]
Free chlorine residual	Colorimetric DPD	-	-	409F	-
Lead	Atomic absorption; direct aspiration	239.1	D3559-78A or B	301A-II or III	-
	Atomic absorption; furnace technique	239.2	-	-	-
Mercury	Manual cold vapor technique	245.1	D3223-79	301A-VI	-
	Automated cold vapor technique	245.2	-	-	-
Nitrate	Colorimetric brucine	352.1	D992-71	419D	-
	Spectrometric; cadmium reduction	353.3	D3867-79B	419C	-
	Automated hydrazine reduction	353.1	-	-	-
	Automated cadmium reduction	353.2	D3867-79A	605	-
	Potentiometric, Ion Selective Electrode	-	-	-	Orion Res. Inc.

APPROVED METHODOLOGY FOR INORGANIC CONTAMINANTS

Contaminant	Methodology	Reference (Method Number) EPA[1]	ASTM[2]	SM[3]	Other
pH	Potentiometric	150.1	D1293-78A or B	424	-
Selenium	Atomic absorption; furnace technique	270.2	-	-	-
	Atomic absorption; gaseous hydride	270.3	D3859-79	301A-VII	I-1667-78[4]
Silver	Atomic absorption; direct aspiration	272.1	-	301A-II	-
	Atomic absorption; furnace technique	272.2	-	-	-
Sodium	Atomic absorption; direct aspiration	273.1	D1428-64A	320A	-
	Atomic absorption; furnace technique	273.2	-	-	-
Sulfate	Turbidimetric	375.4	-	427C	-
Temperature	Thermometer	-	-	212	-
Total filterable residue	Gravimetric	160.1	-	208B	-
Turbidity	Nephelometric	180.1	-	214A	-

[1]"Methods of Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268 (EPA-600/4-79-020), March 1979. Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. For approved analytical procedures for metals, the technique applicable to total metals must be used.

[2]Annual Book of ASTM Standards, Part 31 Water, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

[3]"Standard Methods for the Examination of Water and Wastewater," 14th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1975.

[4]Techniques of Water-Resources Investigation of the United States Geological Survey, Chapter A-1, "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," Book 5 (1979, Stock #024-001-03177-9. Available from Superintendent of Documents, U. S. Government Printing Office, Washington, D. C. 20402.

[5]"AWWA Standard for Asbestos - Cement Pipe, 4 in. through 24 in. for Water and Other Liquids," AWWA C400-77, Revision of C400-75, AWWA, Denver, Colorado.

[6]"Fluoride in Water and Wastewater. Industrial Method #129-71 W." Technicon Industrial Systems. Tarrytown, New York 10591, December 1972.

[7]"Fluoride in Water and Wastewater," Technicon Industrial Systems. Tarrytown, New York 10591, February 1976.

APPROVED METHODOLOGY FOR ORGANIC CONTAMINANTS

Contaminant	Methodology	Reference (Method Number or page numbers) EPA	ASTM[3]	SM[4]	USGS[5]
Chlorinated hydrocarbons: endrin, lindane, methoxychlor, toxaphene	Solvent extraction, gas chromatography	1	D3086-79	509A	pp. 24-39
Chlorophenoxys: 2,4-D, 2,4,5-T	Solvent extraction, derivatization, gas chromatography	2	D3478-79	509B	pp. 24-39

[1]"Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. (pp. 1-19)

[2]Ibid. (pp. 20-35)

[3]Annual Book of ASTM Standards, Part 31 Water, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.

[4]"Standard Methods for the Examination of Water and Wastewater," 14th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1975.

[5]Techniques of Water-Resources Investigation of the United States Geological Survey, Chapter A-3, "Methods for Analysis of Organic Substances in Water, Book 5, 1972. Available from Superintendent of Documents, U. S. Government Printing Office, Washington, D. C. 20402.

APPROVED METHODOLOGY FOR THM

Contaminant	Methodology	Reference EPA
Total Trihalomethanes (TTHM)	Purge and trap, gas chromatography	1
	Solvent extraction, gas chromatography	2
	Gas chromatography/mass spectrometry	3

[1]"The Analysis of Trihalomethanes in Finished Waters by the Purge and Trap Method," Method 501.1, EMSL, EPA, Cincinnati, Ohio 45268.

[2]"The Analysis of Trihalomethanes in Drinking Water by Liquid/Liquid Extraction," Method 501.2, EMSL, EPA, Cincinnati, Ohio 45268.

[3]"The Analysis of Trihalomethanes in Drinking Water by Gas Chromatography/Mass Spectrometry," Method 501.3, EMSL, EPA, Cincinnati, Ohio 45268.

APPROVED METHODOLOGY FOR MICROBIOLOGICAL CONTAMINANTS

Contaminant	Methodology	Reference (Method Numbers) EPA[1]	SM[2]	Other
Coliforms	Multiple Tube Technique	pp. 114-119	908A & 908D	-
	Membrane Filter Technique	pp. 108-114	909A	-

[1]"Microbiological Methods for Monitoring the Environment, Water and Waste", EPA 600/8-78-017, U.S. EPA, EMSL, Cincinnati, Ohio 45268.

[2]"Standard Methods for the Examination of Water and Wastewater", 14th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1975.

MODEL STATE INFORMATION SYSTEM
(MSIS)

Contaminant	Contaminant Code	Methodology	Method Code
Alkalinity	1927	Methyl orange titrimetric or Potentiometric	142
Arsenic	1005	Atomic absorption; furnace technique	125
		Atomic absorption; gaseous hydride	123
		Spectrophotometric, silver diethyldithiocarbamate	113
Barium	1010	Atomic absorption; direct aspiration	101
		Atomic absorption; furnace technique	125
Cadmium	1015	Atomic absorption; direct aspiration	101
		Atomic absorption; furnace technique	125
Calcium Hardness	1919	EDTA titrimetric	141
		Atomic absorption; direct aspiration	
Chloride	1017	Potentiometric	127
Chromium	1020	Atomic abosrption; direct aspiration	101
		Atomic absorption; furnace technique	125
Corrosivity	1910	Langelier index	138
		Aggressive index	140
Fluoride	1025	Colorimetric SPADNS; with distillation	111
		Potentiometric ion selective electrode	107
		Automated Alizarin fluoride blue; with distillation	115
		Automated ion selective electrode	118
		Zirconium eriochrome cyanine R; with distillation	117
Free chlorine residual	1012	Colorimetric DPD	301
Lead	1030	Atomic abosrption; direct aspiration	101
		Atomic absorption; furnace technique	125
Mercury	1035	Manual cold vapor technique	103
		Automated cold vapor technique	119
Nitrate	1040	Colorimetric brucine	105
		Spectrometric; cadmium reduction	109
		Automated hydrazine reduction	121
		Automated cadmium reduction	163
		Potentiometric, Iselective Electrode	107

MODEL STATE INFORMATION SYSTEM
(MSIS)

Contaminant	Contaminant Code	Methodology	Method Code
pH	1925	Potentiometric	135
Selenium	1045	Atomic absorption; furnace technique	125
		Atomic absorption; gaseous hydride	123
Silver	1050	Atomic absorption; direct aspiration	101
		Atomic absorption; furnace technique	125
Sodium	1052	Atomic absorption; direct aspiration	101
		Atomic absorption: furnace technique	125
Sulfate	1055	Turbidimetric	137
Temperature	1996	Thermometer	130
Total filterable residue	1930	Gravimetric	139
Turbidity	0100	Nephelometric	001
Chlorinated hydrocarbons:		Solvent extraction, gas chromatography	
endrin	2005		201
lindane	2010		201
methoxychlor	2015		201
toxaphene	2020		201
Chlorophenoxys:		Solvent extraction, derivatization, gas chromatography	203
2,4-D	2105		203
2,4,5-T	2110		
Total Trihalomethanes (TTHM)	2950	Purge and trap, gas chromatography	213
		Solvent extraction, gas chromatography	215
		Gas chromatography/mass spectrometry	217
Coliforms	3000	Multiple Tube Technique	305
		Membrane Filter Technique	303