

LAB Copy



DEPARTMENT OF THE NAVY
ATLANTIC DIVISION
NAVAL FACILITIES ENGINEERING COMMAND
NORFOLK, VIRGINIA 23511

TELEPHONE NO.
444-4903
AUTOVON 690-4903
IN REPLY REFER TO:

114:WLC
6280

8 SEP 1981

From: Commander, Atlantic Division, Naval Facilities Engineering Command
To: Commanding General, Marine Corps Base, Camp Lejeune (Attn: Assistant Chief of Staff for Facilities)

Subj: Monitoring data or Trihalomethanes (TTHM) in drinking water

Ref: (a) FONECON MARCORB CAMP LEJEUNE (Mr. D. Sharp)/LANTNAVFACENGCOM (Mr. W. Carter) of 21 Aug 1981
(b) LANTNAVFACENGCOM ltr 114:WLC of 26 Aug 1981

Encl: (1) TTHM Surveillance Report Form - Hadnot Point
(2) TTHM Surveillance Report Form - MCAS NEW RIVER
(3) TTHM Surveillance Report Form - Rifle Range

1. Enclosures (1) through (3) are forwarded for your information and use pursuant to references (a) and (b).

2. Questions regarding this matter should be addressed to Mr. W. Carter of this Command at AUTOVON 690-4903.

J.R. Bailey
J. R. BAILEY
By direction

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TTHM SURVEILLANCE REPORT FORM

Installation LE JEUNE - HADNOT PT.

Date Collected 22 JUL 81 PM

AVE 155 ppb

Source	Sample Number	CHCl ₃	CHCl ₂ Br	CHClBr ₂	CHBr ₃	TTHM
WTP 20	361	18.7	130	2.8	0.2	152
NH-1	362	24.8	129	3.3	0.2	157
1202	363	20.4	125	3.0	0.2	149
65	364	22.4	132	3.2	0.2	158
530	365	26.3	126	3.3	0.3	156
Reference OBS						
True						

Date Received 27 JUL 81

Date Analyzed 1 SEP 81

Remarks:

William C. Neal

WILLIAM C. NEAL, JR.
Chief, Laboratory Services

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NAV

TTHM SURVEILLANCE REPORT FORM

Installation LEJEUNE - NEW RIVER

Date Collected 23 JUL 81 PM

AVE 178 PPb

Source	Sample Number	CHCl ₃	CHCl ₂ Br	CHClBr ₂	CHBr ₃	TTHM
WTP 110	356	3.5	3.5	21.8	25.9	55
520	357	10.0	13.4	136	54.9	214
4025	358	4.9	6.7	54.1	83.6	149
710	359	4.1	5.7	40.5	55.8	106
2800	360	5.0	8.4	141	88.5	243
Reference CBS						
True						

Date Received 27 JUL 81

Date Analyzed 7 SEP 81

Remarks:

William C Neal Jr
 WILLIAM C. NEAL, JR.
 Chief, Laboratory Services

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TTHM SURVEILLANCE REPORT FORM

Installation CAMP LEJEUNE - RIFLE RANGE

Date Collected 24 JUL 81 PM

AVE 35 ppb

Source	Sample Number	CHCl ₃	CHCl ₂ Br	CHClBr ₂	CHBr ₃	TTHM
WTP	RR1	0.6	40.1	0.1	0.3	1
WTP FINISH	RR2	18.6	3.3	2.1	0.2	24
6 FIRE	RR3	27.4	5.2	3.1	0.3	36
10	RR4	29.9	5.7	3.4	0.3	39
92	RR5	30.4	5.9	3.5	0.3	40
Reference OBS						
True						

Date Received 27 JUL 81

Date Analyzed 1 SEP 81

Remarks:

William C Neal

WILLIAM C. NEAL, JR.
Chief, Laboratory Services

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ROUTINE REPLY, ENDORSEMENT, TRANSMITTAL OR INFORMATION SHEET

OPNAV 5215/158 (Rev. 7-78)
SN 0107 LF 052-1691

A WINDOW ENVELOPE MAY BE USED
Formerly NAVEXOS 3789

CLASSIFICATION (UNCLASSIFIED when detached from enclosures, unless otherwise indicated)

FROM (Show telephone number in addition to address)
Commander, Atlantic Division, Naval Facilities
Engineering Command

DATE
15 September 1981

SUBJECT
North Carolina State Safe Drinking Water Act Compliance
Information

SERIAL OR FILE NO.
114:WLC
6280

TO:
Commanding General
Marine Corps Base
Camp Lejeune, NC 28542

REFERENCE

Attention Facilities Maintenance Officer

ENCLOSURE

See remarks below

VIA: _____ ENDORSEMENT ON _____

FORWARDED RETURNED FOLLOW-UP, OR TRACER REQUEST SUBMIT CERTIFY MAIL FILE

GENERAL ADMINISTRATION	CONTRACT ADMINISTRATION	PERSONNEL
FOR APPROPRIATE ACTION UNDER YOUR COGNIZANCE	NAME & LOCATION OF SUPPLIER OF SUBJECT ITEMS	REPORTED TO THIS COMMAND:
<input checked="" type="checkbox"/> INFORMATION	SUBCONTRACT NO. OF SUBJECT ITEM:	DETACHED FROM THIS COMMAND
APPROVAL RECOMMENDED <input type="checkbox"/> YES <input type="checkbox"/> NO	APPROPRIATION SYMBOL, SUBHEAD, AND CHARGEABLE ACTIVITY	OTHER
<input type="checkbox"/> APPROVED <input type="checkbox"/> DISAPPROVED	SHIPPING AT GOVERNMENT EXPENSE <input type="checkbox"/> YES <input type="checkbox"/> NO	
COMMENT AND/OR CONCURRENCE	A CERTIFICATE, VICE BILL OF LADING	
CONCUR	COPIES OF CHANGE ORDERS, AMENDMENT OR MODIFICATION	
LOANED, RETURN BY:	CHANGE NOTICE TO SUPPLIER	
SIGN RECEIPT & RETURN	STATUS OF MATERIAL ON PURCHASE DOCUMENT	
REPLY TO THE ABOVE BY:		

REFERENCE NOT RECEIVED

SUBJECT DOCUMENT FORWARDED TO:

SUBJECT DOCUMENT RETURNED FOR:

SUBJECT DOCUMENT HAS BEEN REQUESTED, AND WILL BE FORWARDED WHEN RECEIVED

COPY OF THIS CORRESPONDENCE WITH YOUR REPLY

ENCLOSURE NOT RECEIVED

ENCLOSURE FORWARDED AS REQUESTED

ENCLOSURE RETURNED FOR CORRECTION AS INDICATED

CORRECTED ENCLOSURE AS REQUESTED

REMOVE FROM DISTRIBUTION LIST

REDUCE DISTRIBUTION AMOUNT TO

REMARKS (Continue on reverse)

Encl: (1) List of North Carolina State Commercial Labs certified to analyze drinking water for safe drinking water act compliance

(2) North Carolina State rules governing Laboratory Certification Section .0301 through .0326

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SIGNATURE & TITLE
Wallace Carter Environmental Engineer

COPY TO

CLASSIFICATION (UNCLASSIFIED when detached from enclosures, unless otherwise indicated)

NORTH CAROLINA LABS WHICH ARE CERTIFIED TO ANALYZE
DRINKING WATER FOR SAFE DRINKING WATER ACT COMPLIANCE

COMMERCIAL (Continued)

<u>Name/Address/Phone</u>	<u>Microbiological</u>	<u>Inorganic</u>	<u>Organic</u>	<u>Radiological</u>
Guilford Labs, Inc. P. O. Box 9735 Plaza Station Greensboro, N. C. 27408 Phone: (919) 274-2907	All	All	All	
Aquasystems, Inc. P. O. Box 2803 Greenville, N. C. 27834 Phone: (919) 756-5721	All			
Environment I Box 7085 Greenville, N. C. 27834 Phone: (919) 756-6208	All	Nitrate		
Glenn R. Frye Memorial Hospital Lab 420 North Center Street Hickory, N. C. 28601 Phone: (704) 322-6070	All	Nitrate		
Northampton County Health Dept. Lab P. O. Box 635 Jackson, N. C. 27845 Phone: (919) 534-5841	All			
Beacham Laboratory U. S. Highway 17, South Jacksonville, N. C. 28540 Phone: (919) 347-7036	All			
Burlington Industries, Inc. Chemical Division P. O. Box 523 Jamestown, N. C. 27282 Phone: (919) 454-3141	All	Fluoride, Mercury Nitrate, Cadmium Chromium, Silver		
Research & Analytical Labs, Inc. P. O. Box 473 106 Short Street Kernersville, N. C. 27284 Phone: (919) 996-2841	All	All		
Professional Environmental Lab of Lumberton 2401 West Fifth Street	All			

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NORTH CAROLINA LABS WHICH ARE CERTIFIED ANALYZE
DRINKING WATER FOR SAFE DRINKING WATER ACT COMPLIANCE

COMMERCIAL

<u>Name/Address/Phone</u>	<u>Microbiological</u>	<u>Inorganic</u>	<u>Organic</u>	<u>Radiological</u>	<u>TH</u>
Moore, Gardner & Associates, Inc. 110 West Walker Avenue Asheboro, N. C. 27203 Phone: (919) 625-6111	All	All	All		
Environmental Testing, Inc. 766½ Biltmore Avenue Asheville, N. C. 28803 Phone: (704) 254-7176	All				
Waste Water Services 122 Stewart Street Asheville, N. C. 28806 Phone: (704) 254-5169	All				
Biomedical Reference Lab., Inc. Industrial & Environmental Services Division P. O. Box 2230 Burlington, N. C. 27215 Phone: (919) 584-5171	All				
BAW Labs, Inc. 1215-1 Central Avenue Charlotte, N. C. 28204 Phone: (704) 376-8646	All	All			
Environmental Testing 1700 University Commercial Pl. Charlotte, N. C. 28213 Phone: (704) 525-9379	All	All			
PAR Laboratories P. O. Box 240722 Charlotte, N. C. 28224 Phone: (704) 376-2459	All	Barium, Cadmium Chromium, Lead, Selenium, Silver,			
Environmental Lab of Fayetteville P. O. Box 49 Fayetteville, N. C. 28301 Phone: (919) 864-1920	All				
Bio-Chem Laboratories P. O. Box 40 Granite Falls, N. C. 28630 Phone: (704) 396-1741	All				

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NORTH CAROLINA LABS WHICH ARE CERTIFIED TO ANALYZE
DRINKING WATER FOR SAFE DRINKING WATER ACT COMPLIANCE

COMMERCIAL (Continued)

<u>Name/Address/Phone</u>	<u>Microbiological</u>	<u>Inorganic</u>	<u>Organic</u>	<u>Radiological</u>	<u>THP</u>
Grainger Laboratories 709 West Johnson Street Raleigh, N. C. 27603 Phone: (919) 828-3360	All	All	All		All
Wake County Health Dept. Lab P. O. Box 949 Raleigh, N. C. 27602 Phone: (919) 755-6107	All				
Iredell County Health Dept. Lab P. O. Box 1268 Statesville, N. C. 28677 Phone: (704) 873-7291	All				
Statesville Water Purification Lab Water Purification Dept. P. O. Box 1111 Statesville, N. C. 28677 Phone: (704) 873-7671	All				
Waynesville Water Treatment Plant P. O. Box 61 Waynesville, N. C. 28786 Phone: (704) 456-8497	All				
Law & Company P. O. Box 629 Wilmington, N. C. 28402 Phone: (919) 762-7082	All	All	All		All

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RULES
GOVERNING LABORATORY CERTIFICATION
SECTION .0301 THROUGH .0326

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
SEPTEMBER 1, 1979

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Regulations 10 NCAC 9D .0301 - .0326; LABORATORY CERTIFICATION;
have been adopted and read as follows:

.0301 POLICY

A laboratory wishing to perform analyses of public water systems pursuant to 10 NCAC 10D .1610 - .1634 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 - .0326 of this Section for each of the particular analyses 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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.0302 NOTICE AND PROCEDURE

(a) A laboratory wishing to be certified must so notify the Department of Human Resources, Division of Health Services, Laboratory Section in writing.

(b) After such notice the laboratory premises shall be surveyed and the facilities reviewed in accordance with the rules of this Section.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.0303 CERTIFICATION

The Department of Human Resources, Division of Health Services, Laboratory Section after surveying the facilities shall certify a laboratory if it meets the minimum requirements of this Section and shall so notify the laboratory.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

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.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 (see Table 1 below for instrumentation for individual methods):
 - (a) spectrophotometer. Usable wavelength range, 400 to 700 nm. Maximum spectral bandwidth, no more than 20 nm. Wavelength accuracy, 0 ± 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, ± 0.05 units. Scale readability, ± 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 ± 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 $210^\circ \pm 0.2^\circ\text{C}$. 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

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- 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 \pm 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).

History Note: Authority G.S. 130-166.43; P.L. 93-523;
 40 C.F.R. 141;
 Eff. September 1, 1979.

.0306 CHEMISTRY GENERAL LABORATORY PRACTICES

(a) Distilled/deionized water used for chemical analyses shall have resistivity values between 0.5 to 2.0 megohms (2.0 to 0.5 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 planned intervals and documented.

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

History Note: Authority G.S. 130-166.43; P.L. 93-523;
 40 C.F.R. 141;
 Eff. September 1, 1979.

.0307 CHEMISTRY METHODOLOGY

Minimum equipment requirements, methodology and references for individual parameters of chemical analyses are shown in Table A. Equivalent equipment may be used. All other procedures are considered

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alternative analytical techniques as described in 10 NCAC 10D .1630. Application for the use of alternative methods may require acceptable comparability data.

TABLE A.-Minimum Requirements
Methodology and Required Equipment

Parameter	Methodology (unfiltered sample)	Reference (page number)		Major Equipment required (or its equivalent)
		SM ¹	EPA ²	
Arsenic ³	Atomic absorption; gaseous hydride		95-96	Atomic absorption spectrophotometer with recorder
Barium	Atomic absorption	210-215	97-98	Atomic absorption spectrophotometer with recorder
Cadmium	Atomic absorption; chelation-extrac- tion	210-215	101-103	Atomic absorption spectrophotometer with recorder
Chromium	Atomic absorption; chelation-extrac- tion	210-215	105-106	Atomic absorption spectrophotometer with recorder
Lead	Atomic absorption; chelation-extrac- tion	210-215	112-113	Atomic absorption spectrophotometer with recorder
Mercury	Flameless atomic absorption	--	118-126	Atomic absorption spectrophotometer with recorder or instrument designed specifically for measurement of mercur
Nitrate	Brucine colorimetric	461-464	--	Spectrophotometer or filter photometer
	Cadmium reduc- tion	--	201-206	Spectrophotometer
Selenium ³	Atomic absorption; gaseous hydride	--	145	Atomic absorption spectrophotometer with recorder
Silver	Atomic absorption;	210-215	146	Atomic absorption spectrophotometer with recorder
Fluoride	Electrode; distil- lation not required	172-174	65-67	Ion-selective elec- trode and expanded scale electrometer
	Colorimetric with preliminary distil- lation	171-172 174-176	59-60	Direct distillation apparatus and spec- trophotometer or filter photometer
Chlorinated hydrocarbons: Gas Chromatography		--	(⁴)	Kuderna-Danish glass- ware, gas chromato- graph equipped with

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Endrin
Lindane
Methoxychlor
Toxaphene

glass-lined injection port and electron-capture detector, and recorder
Kuderna-Danish glassware, gas chromatograph equipped with glass-lined injection port and electron-capture detector, and recorder.

Chlorophenoxy: Gas chromatography -- (4)
2,4-D
2,4,5-TP

- 1 "Standard Methods for the Examination of Water and Wastewater," 13th Edition, American Public Health Association, 1971.
- 2 "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, Office of Technology Transfer, Washington, D.C., 20460, 1974.
- 3 Complete method available in: Caldwell, J.S., Lishka, R.J., and McFarren, E.F., "Evaluation of a Low Cost Arsenic and Selenium Determination at Microgram Per Liter Levels," J. Amer. Water Works Assoc., 65:731 (November 1973).
- 4 Methods available from U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Environmental Research Center, Cincinnati, Ohio, 45268.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.0308 CHEMISTRY SAMPLES

Requirements for container types, preservatives and holding times for individual parameters of chemical analyses are shown in Table B:

Table B.-Sample collecting, handling and preservation¹

Parameter	Preservative ²	Container ³	Maximum holding time ⁴
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	38 days
		P	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	1 month
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

¹If 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.

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

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

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

⁵Well-stoppered and refrigerated extracts can be held up to 30 days.

History Note: Authority G.S. 130-166.43; P.L. 93-523; 40 C.F.R. 141; Eff. September 1, 1979.

.0509 CHEMISTRY QUALITY CONTROL

Requirements for quality control data of chemical analyses are as follows:

- (1) All quality control data shall be available for inspection.
- (2) A laboratory must 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 wishes to be certified. If problems arise, appropriate technical assistance will be provided, and a follow up performance sample should be analyzed.
- (3) The minimum daily quality control shall be as follows:
 - (a) After a standard reagent curve composed of a minimum of a reagent blank and three standards has been prepared, subsequent standard curves must be verified by use of at least a reagent blank and one standard at or near the MCL. Daily checks must be within ± 10 percent of original curve.
 - (b) If 20 or more samples per day are analyzed, working standard curve must be verified by running an additional standard at or near the MCL every 20 samples. Checks must be within ± 10 percent of original curve.
- (4) A thermometer certified by the National Bureau of Standards (or one of equivalent accuracy) shall be available to check thermometers in ovens, etc.

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History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.0310 CHEMISTRY DATA

(a) Records of chemical analyses shall 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 10 NCAC 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 sample, 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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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 + 0.1 units.

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- (2) Balances-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 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 $35.0^{\circ} \pm 0.5^{\circ}\text{C}$ 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 4.4°C (34° to 40°F).
- (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 3mm 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 μm

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- 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 glass-ware 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", 13th 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 4.4°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

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- pure water. Final pH must be 7.2 ± 0.1 .
- (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"; lactose broth is not permitted.
 - (3) Dehydrated media containers must be kept tightly closed and stored in a cool, dry location. Discolored or cake 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 over night 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 4.4°C (34° to 40°F); time must be limited to manufacturer's expiration date.

History Note: Authority G.S. 130-166.45; P.L. 523-~~01~~EW
40 C.F.R. 141;
Eff. September 1, 1979.

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.0315 MICROBIOLOGY METHODOLOGY

(a) The required procedures, which are mandatory, are described in "Standard Methods for the Examination of Water and Wastewater," 13th Edition:

- (1) standard coliform MPN tests, pp. 664-668;
- (2) single-step or enrichment standard total coliform membrane filter procedure, pp. 679-683.

(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.

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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.
- (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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.0316 MICROBIOLOGY SAMPLES

Requirements for sample collection, handling, and preservation for microbiological analyses are as follows:

- (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," 13th Edition, section 450, p. 657-660.
- (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).

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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) At least five sheen or borderline sheen colonies must be verified from each membrane containing five or more such colonies. Counts must be adjusted based on verification.

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The verification procedure must be conducted by transferring growth from colonies into lauryl tryptose broth (LTB) tubes and then transferring growth from gas-positive LTB 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 bactericidal properties for distilled water ("Standard Methods for the Examination of Water and Wastewater," 13th Edition, p. 646). 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", 13th Edition, p. 660-662. Plates must be incubated at $35^{\circ} + 0.5^{\circ}\text{C}$ for 48 hours.
- (h) Requirements for laboratory pure water are:
 - (i) pH measures 5.5-7.5;
 - (ii) conductivity measures greater than 0.2 megohms as resistivity or less than 5.0 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

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("Standard Methods for the Examination of Water and Wastewater", 13 edition, p. 646] measures 0.8-3.0,

- (v) free chlorine residual measures 0.0,
 - (vi) standard plate count measures less than 10,000/ml.
 - (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 be 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/waterbaths/aluminum block incubators must be recorded continuously or recorded daily from in-place thermometer(s) immersed in liquid and placed on shelves in use.
 - (e) Date, time, and temperature must be recorded continuously or recorded for each sterilization cycle of the autoclave.
 - (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.
 - (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 ("Standard Methods," 13th edition, p. 643).
 - (i) At least one bottle per batch of sterilized sample bottles must be checked by adding approximately 25 ml of sterile LTB broth to each bottle. It must be incubated at $35 \pm 0.5^\circ\text{C}$ 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.

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- (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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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 correct 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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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"

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(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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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, \pm 0.05 mg. Minimum scale readability, 0.1 mg.
 - (b) pH meter or specific ion meter:
 - (i) pH meter. Accuracy, \pm 0.5 units. Scale readability, \pm 0.1 units. Instrument may be either line/bench or battery/portable.
 - (ii) Specific ion meter. Expanded scale millivolt capability. Readable and accurate to \pm 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 micromhos \pm 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.

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- (f) Hot plate. Large or small units with selectable temperature control for safe heating of laboratory reagents and samples.
 - (g) Glassware. Borosilicate 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 voltage supply, amplifier, timers, and scalers). The flasks (cells) required for

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- 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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.0325 RADIOCHEMISTRY SAMPLES

The minimum requirements of sample handling for radiochemical analyses including preservation, methodology, and major instrumentation are shown in Table C:

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

Parameter	Preservative ²	Container ³	Instrumentation
Gross alpha	Concl. HCl or HNO ₃ to pH<2.5	P or G	A or B
Gross beta	Concl. HCl or HNO ₃ to pH<2.5	P or G	A
Strontium-89	Concl. HCl or HNO ₃ to pH<2	P or G	A
Strontium-90	Concl. HCl or HNO ₃ to pH<2	P or G	A
Radium-226	Concl. HCl or HNO ₃ to pH<2	P or G	A, B, or D
Radium-228	Concl. HCl or HNO ₃ to pH<2	P or G	A
Cesium-134	Concl. HCl to pH<2	P or G	A or C
Iodine-131	None	P or G	A
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

¹Federal Register, Vol. 41, No. 133, July 9, 1976.

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.

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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, 1957).

- (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 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 multichannel analyzer, in addition to appropriate electronics, must contain a memory of not less than 2000 channels and at least one readout device.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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 between 0.5 and 2.0 megohms (2.0 to 0.5 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

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

⁴A=Low background proportional system; B=Alpha scintillation system; C=Gamma spectrometer (NaI(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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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.

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

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.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.

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

.0326 RADIOCHEMISTRY ACTION RESPONSE

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

History Note: Authority G.S. 130-166.43; P.L. 93-523;
40 C.F.R. 141;
Eff. September 1, 1979.

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