STATE OF NEBRASKA

DEPARTMENT OF ENVIRONMENTAL CONTROL
Randolph Wood
Director
301 Centennial Mall South
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Lincoln, Nebraska 68509-8922
Phone (402) 471-2186

E. Benjamin Nelson
Governor

Thomas T. Holloway, Chief
Emergency Planning and Response Branch
Environmental Services Division
U. S. EPA Region VII
25 Funston Road
Kansas City, KS 66115

RE: ARARs for Removal Action at Gladstone and Tomora

Dear Mr. Holloway:

For your information we have enclosed a list of Applicable or Relevant and Appropriate Requirements (ARARs) which may affect the proposed Superfund activities within Nebraska.

In response to your letter dated May 27, 1992, and received by us on June 8, 1992, ARARs specifically identified for the proposed Removal Action at Gladstone and Tomora groundwater contamination sites are as follows:

* Title 179, Chapter 2, Section 007.02 (The Nebraska Department of Health: Regulations Governing Public Water Supply System) related to connection of 3 private wells at Gladstone to a community water system.

* Bottled water Requirements (The Nebraska Department of Agriculture) regarding providing bottled water to Tomora, Nebraska.

Copies of the above mentioned documents are enclosed. Please contact me or Emily Chen at (402) 471-3388 if you have any questions.

Sincerely,

Rich Schlenker

Rich Schlenker
Superfund Unit Supervisor
Hazardous Waste Section
Land Quality Division

RS/EC/ls

REV 5/92

POTENTIAL STATE OF NEBRASKA
APPLICABLE OR RELEVANT AND APPROPRIATE REQUIREMENTS (ARARS)

		CITATION
I.	Nebraska Environmental Protection Act	Neb. Rev. Stat. Ch. 81 Article 15
A.	Rules and Regulations Governing the Nebraska Pretreatment Program	Neb. Adm. Rules & Regs Title 127
B.	Effluent Guidelines and Standards	Neb. Adm. Rules & Regs Title 121
C.	Rules and Regulations Pertaining to the Issuance of Permits Under the National Pollutant Discharge Elimination System	Neb. Adm. Rules & Regs Title 119
D.	Rules and Regulations for Underground Injection and Mineral Production Wells	Neb. Adm. Rules & Regs Title 122
E.	Air Pollution Control Rules and Regulations	Neb. Adm. Rules & Regs Title 129
F.	Nebraska Surface Water Quality Standards	Neb. Adm. Rules & Regs Title 117
G.	Ground Water Quality Standards and Use Classification	Neb. Adm. Rules & Regs Title 118
H.	Rules and Regulations Pertaining to Solid Waste Management	Neb. Adm. Rules & Regs Title 132
I.	Rules and Regulations Governing Hazardous Waste Management in Nebraska	Neb. Adm. Rules & Regs Title 128
J.	Rules and Regulations Pertaining to the Management of Wastes	Neb. Adm. Rules & Regs Title 126
II.	Water Well Standards and Contractors' Licensing Act	Neb. Rev. Sta. Ch. 46 Article 12
A.	Regulations Governing Licensure of Water Well and Pump Installation Contractors and Certification of Water Well Drilling and Pump Installation Supervisors	Neb. Adm. Rules & Regs Title 178
III.	Statutes Relating to Ground Water	Neb. Rev. Stat. Ch. 46 Article 6

REV 5/92
(Cont'd)

POTENTIAL STATE OF NEBRASKA APPLICABLE OR RELEVANT AND APPROPRIATE REQUIREMENTS (ARARS)	CITATION
IV. Nebraska Safe Drinking Water Act	Neb. Rev. Stat. Ch. 71 Article 53
A. Regulations Governing Public Water Supply Systems	Neb. Adm. Rules & Regs Title 179
V. Flood Plain Management	Neb. Rev. Stat. Ch. 31 Article 10
A. Flood Plain Rules	Neb. Adm. Rules & Regs Title 455
B. Rules Governing Flood Plain Management	Neb. Adm. Rules & Regs Title 258
VI. Statutes Relating to Disposal Sites	Neb. Rev. Stat. Ch. 19 Articles 21 and 41
VII. Nebraska Nongame and Endangered Species Conservation Act	Neb. Rev. Stat. Ch. 37-430 to Ch. 37-438
A. Nebraska Game and Parks Commission Rules and Regulations Concerning Wildlife	Neb. Adm. Rules & Regs Title 163, Chapter 6

STATE OF NEBRASKA

DEPARTMENT OF AGRICULTURE
Larry E. Sitzman
Director

E. Benjamin Nelson
Governor

RECEIVED
JUN 19 1992
DEPARTMENT OF
ENVIRONMENTAL CONTROL

June 19, 1992

MEMO TO: Emily Chen
Nebraska Department of Environmental Control

FROM: George H. Hanssen GHH
Food Division Manager

SUBJECT: Bottled Water Requirements

The Nebraska Department of Agriculture, Bureau of Dairies & Foods, regulates bottled waters in Nebraska.

The Foods Division inspects and licenses firms that bottle water in Nebraska, including bottled water vending machines found in retail stores. The State Health Department requires quarterly water samples for these community systems.

We request firms shipping bottled waters into Nebraska send us water sample results. We follow the Code of Federal Regulations, Title 21, Parts 103 and 129 and compare the results with the standards in the CFR.

Please call me if you have further questions.

GHH

DEPARTMENT OF AGRICULTURE, 301 CENTENNIAL MALL SOUTH

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P.O. Box 94947
Lincoln, NE 68509-4947
(402) 471-2341
FAX: (402) 471-2759

Agriculture Laboratories
3703 South 14th Street
Lincoln, NE 68502-5399
(402) 471-2176
FAX: (402) 479-4172

Bureau of Animal Industry
P.O. Box 94787
Lincoln, NE 68509-4787
(402) 471-2351
FAX: (402) 471-3252

Bureau of Dairies & Foods
P.O. Box 95064
Lincoln, NE 68509-5064
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FAX: (402) 471-3252

Bureau of Plant Industry
P.O. Box 94756
Lincoln, NE 68509-4756
(402) 471-2394
FAX: (402) 471-3252

Weights and Measures
P.O. Box 94757
Lincoln, NE 68509-4757
(402) 471-4292
FAX: (402) 47[illegible]

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NEBRASKA DEPARTMENT OF HEALTH

REGULATIONS GOVERNING PUBLIC WATER SUPPLY SYSTEMS

TITLE 179 NEBRASKA ADMINISTRATIVE CODE,
CHAPTER 2

INCLUDING AMENDMENTS MADE THROUGH MARCH 30, 1992

printed on recycled paper

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TITLE 179 - NEBRASKA DEPARTMENT OF HEALTH
CHAPTER 2 - REGULATIONS GOVERNING PUBLIC WATER SUPPLY SYSTEMS

001 GENERAL (INCLUDES AMENDMENTS MADE THROUGH MARCH 30, 1992)

001.01 Definitions. As used in these regulations, unless the context to be intelligible or prevent absurdity otherwise requires:

001.01A "Air Gap" means the unobstructed vertical distance through the free atmosphere between the lowest opening from any pipe or faucet supplying water to a tank, plumbing fixture or other device and the flood rim of the receptor.

001.01B Backflow" means the undesirable reversal of flow in a potable water distribution system as a result of a cross connection.

001.01C "Backflow Preventer" means an assembly or means that prohibits the backflow of water into the potable water supply.

001.01D "Backpressure" means a pressure, higher than the supply pressure, caused by a pump, elevated tank, boiler, air/steam pressure, or any other means, which may cause backflow.

001.01E "Backsiphonage" means backflow caused by negative or reduced pressure in the supply piping.

001.01F "Coagulation" means a process using coagulant chemicals and mixing by which colloidal and suspended materials are destabilized and agglomerated into flocs.

001.0G "Community Water Supply System" means a public water supply system which serves at least fifteen service connections used by year round residents or regularly serves twenty-five year round residents.

001.01H "Confluent Growth" means a continuous bacterial growth covering the entire filtration area of a membrane filter, or a portion thereof, in which bacterial colonies are not discrete.

001.01I "Contamination" means an impairment of a potable water supply by the introduction or admission of any foreign substance that degrades the quality and creates a health hazard.

001.01J "Conventional Filtration Treatment" means a series of processes including coagulation, flocculation, sedimentation, and filtration resulting in substantial particulate removal.

001.01K "Cross Connection" means any physical arrangement whereby a potable water supply is connected, directly or indirectly, with any other water supply system, sewer, drain, conduit, tank, pump, plumbing fixture, heat exchanger, or other mechanical equipment or device which contains, or may contain, contaminated water, sewage or other waste, liquid or gas of unknown or unsafe quality which may be capable of imparting contamination or pollution to the potable water supply as a result of backflow (due to either backpressure or backsiphonage). Bypass arrangements, jumper connections, hose connections, removable spools, swivel or changeover devices, four-way valve connections and other temporary or permanent devices through which, or because of which, backflow (or backsiphonage) could occur are considered to be cross-connections. Protecting a public water supply system against potential contamination from cross-connections is possible by containment and/or isolation.

001.01L "Cross Connection Control" means the enforcement of an ordinance or other instrument regarding cross connections.

001.01M "CT or CTcalc" is the product of "residual disinfectant concentration" (C) in mg per liter determined before or at the first customer, and the corresponding "disinfectant contact time" (T) in minutes, i.e., "C" x "T".

001.01N "CT(99.9)" means the CT value required for 99.9 percent (3-log) inactivation of Giardia lamblia cysts. CT(99.9) for a variety of disinfectants and conditions appear in Tables 1.1-1.6, 2.1, and 3.1 of subsection 002.04.

001.01O "Council" means the Advisory Council on Public Water Supply.

001.01P "Diatomaceous Earth Filtration" means a process resulting in substantial particulate removal in which (1) a precoat cake of diatomaceous earth filter media is deposited on a support membrane (septum), and (2) while the water is filtered by passing through the cake on the septum, additional filter media known as body feed is continuously added to the feed water to maintain the permeability of the filter cake.

001.01Q "Direct Filtration" means a series of processes including coagulation and filtration but excluding sedimentation resulting in substantial particulate removal.

001.01R "Director" means the Director of Health or his authorized representative.

001.01S "Disinfection Contact Time" ("T" in CT calculations) means the time in minutes that it takes for water to move from the point of disinfectant application or the previous point of disinfectant residual measurement to a point before or at the point where residual disinfectant concentration ("C") is measured.

001.01T "Disinfection" means a process which inactivates pathogenic organisms in water by chemical oxidants or equivalent agents.

001.01U "Domestic or Other Non-Distribution System Plumbing Problem" means a coliform contamination problem in a public water system with more than one service connection, that is limited to the specific service connection from which the coliform-positive sample was taken.

001.01V "Dose Equivalent" means the product of the absorbed dose from ionizing radiation and such factors as account for differences in biological effectiveness due to the type of radiation and its distribution in the body as specified by the International Commission on Radiological Units and Measurements (ICRU).

001.01W "Double Check-Valve Assembly" means a backflow prevention device consisting of two independently acting check valves, internally force loaded to a normally closed position between two tightly closing shut-off valves, and with means for testing for tightness.

001.01X "Drinking Water Standards" means the rules and regulations adopted pursuant to NEB. REV. STAT. §71-5302 (Reissue 1981), which establish maximum levels for harmful materials which, in the judgement of the Director, may have an adverse effect on the health of persons and which apply only to public water supply systems.

001.01Y "Effective Opening" means the minimum cross-sectional area at the point of water supply discharge, measured or expressed in terms of the diameter of a circle, or if the opening is not circular, the diameter of a circle of equivalent cross-sectional area.

001.01Z "Filtration" means a process for removing particulate matter from water by passage through porous media.

001.01AA "Flocculation" means a process to enhance agglomeration or collection of smaller floc particles into larger, more easily settleable particles through hydraulic or mechanical means.

001.01BB "Gross Alpha Particle Activity" means the total radioactivity due to alpha particle emission as inferred from measurements on a dry sample.

001.01CC "Gross Beta Particle Activity" means the total radioactivity due to beta particle emission as inferred from measurements on a dry sample.

001.01DD "Groundwater System" means a water supply system utilizing wells as the source of water.

001.01EE "Ground Water Under the Direct Influence of Surface Water" means any water beneath the surface of the ground with (1) significant occurrence of insects or other macroorganisms, algae, or large-diameter pathogens such as Giardia lamblia, or (2) significant and relatively rapid shifts in water characteristics such as turbidity, temperature, conductivity, or pH which closely correlate to climatological or surface water conditions.

001.01FF "Halogen" means one of the chemical elements chlorine, bromine, or iodine.

001.01GG "Inactivation Ratio" means the ratio of CT to CT(99.9), i.e., CT divided by CT(99.9).

001.01HH "Lead Free"

001.01HH1 When used with respect to solders and flux, means solders and flux containing not more than two-tenths percent lead and

001.01HH2 When used with respect to pipe and pipe fittings, means pipe and pipe fittings containing not more than eight percent lead.

001.01II "Legionella" means a genus of bacteria, some species of which have caused a type of pneumonia called Legionnaires Disease.

001.01JJ "Major Construction, Extension, or Alteration" means those structural changes that affect the source of supply, treatment processes, or transmission of water to service areas, but shall not include the extension of service mains within established service areas.

001.01KK "Man-Made Beta Particle and Photon Emitters" means all radionuclides emitting beta particles and/or photons listed in "Maximum Permissible Body Burdens and Maximum Permissible Concentration of Radionuclides in Air or Water for Occupational Exposure", NBS Handbook 69, except the daughter products of Thorium-232, Uranium-235, and Uranium-238.

001.01LL "Maximum Contaminant Level" (MCL) means the maximum permissible level of a contaminant in water as measured at the point of entry to the distribution system or at the free flowing outlet of the ultimate user of the public water supply system. Contaminants added to the water under circumstances controlled by the user, except those resulting from corrosion of piping and plumbing caused by water quality, are excluded from this definition.

001.01MM "Maximum Total Trihalomethane Potential" (MTP) means the maximum concentration of total trihalomethanes produced in a given water containing disinfectant residual after 7 days at a temperature of 25° C or above.

001.01NN "Near the First Service Connection" means at one of the 20 percent of all service connections in the entire system that are nearest the water supply treatment facility, as measured by water transport time within the distribution system.

001.01OO "Non-Community Water Supply System" means any public water supply system that is not a community water supply system.

001.01PP "Non-Transient, Non-Community Water Supply System" means a public water supply system that is not a community water supply system and that regularly serves at least 25 of the same persons over six (6) months per year.

001.01QQ "Operator" means the individual or individuals responsible for the continued performance of the water supply system or any part of such system, during assigned duty hours.

001.01RR "Owner" means any person owning or operating a public water supply system.

001.01SS "Person" means any individual, firm, partnership, association, company, corporation, political subdivision, or other entity.

001.01TT "Picocurie" (pCi) means that quantity of radioactive material producing 2.22 nuclear transformations per minute.

001.01UU "Point of Disinfectant Application" is the point where the disinfectant is applied and water downstream of that point is not subject to recontamination by surface water runoff.

001.01VV "Pollution" means the presence of any foreign substance in water that tends to degrade its quality so as to constitute a nonhealth hazard or impair the usefulness of the water.

001.01WW "Potable Water" means water that is safe for human consumption as set forth in section 002 of these regulations.

001.01XX "Protection by Containment" (i.e., containing the potential contamination source within the water customer's building, factory, facility, property distribution system, or trailer court, etc.) shall mean the installation of an approved backflow prevention device or method on the water service line(s) serving any premises, location, facility or area.

001.01YY "Protection by Isolation" means control of cross-connections within a building's plumbing system by the installation of approved backflow prevention devices or methods at or near the potential sources of pollution or contamination.

001.01ZZ "Public Water Supply System" means a water supply system designed to provide the public piped water fit for human consumption, if such system has at least fifteen service connections or regularly serves at least twenty-five individuals daily, at least sixty days out of the year. A public water supply system is either a community public water supply system, a non-community water supply system or a non-transient, non-community water supply system. This definition shall include:

001.01ZZ1 Any collection, treatment, storage, or distribution facilities under control of the operator of such system and used primarily in connection with such system, and

001.01ZZ2 Any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system.

001.01AAA "Reduced Pressure Principle Backflow Preventer" means a backflow prevention device consisting of two independently acting check valves, internally force loaded to a normally closed position and separated by an intermediate chamber (or zone) in which there is an automatic relief means of venting to atmosphere internally loaded to a normally open position between two tightly closing shut-off valves and with means for testing for tightness of the checks and opening of relief means.

001.01BBB "Rem" means the unit of dose equivalent from ionizing radiation to the total body or any internal organ or organ system. A "millirem" (mrem) is 1/1000 of a rem.

001.01CCC "Residual Disinfectant Concentration" ("C" in CT calculations) means the concentration of disinfectant measured in mg per liter in a representative sample of water.

001.01DDD "Sanitary Survey" means an on-site review of the water source, facilities, equipment, operation and maintenance of a public water system for the purpose of evaluating the adequacy of such source, facilities, equipment, operation and maintenance for producing and distributing safe drinking water.

001.01EEE "Sedimentation" means a process for removal of solids before filtration by gravity or separation.

001.01FFF "Service Area" means the land area which the owner of a public water supply system has legal franchise or authority to remain the sole distributor of piped drinking water.

001.01GGG "Slow Sand Filtration" means a process involving passage of raw water through a bed of sand at low velocity (generally less than 0.4 m/h) resulting in substantial particulate removal by physical and biological mechanisms.

001.01HHH "Surface Water" means all water which is open to the atmosphere and subject to surface runoff.

001.01III "Surface Water System" means a water supply system utilizing surface water as the source of water.

001.01JJJ "System with a Single Service Connection" means a system which supplies drinking water to consumers via a single service line.

001.01KKK "Too Numerous to Count" means that the total number of bacterial colonies exceeds 200 on a 47-mm diameter membrane filter used for coliform detection.

001.01LLL "Treatment Technique" means the use of aeration, settling, filtration, or other physical process and/or the addition of any chemical or chemicals for the purpose of removing, deactivation, or adjusting the level of one or more contaminants present in the raw water supply source.

001.01MMM "Trihalomethane" (THM) means one of a family of organic compounds, named as derivatives of methane, wherein three of the four hydrogen atoms in methane are each substituted by a halogen atom in the molecular structure.

001.01NNN "Total Trihalomethanes" (TTHM) means the sum of the concentration in milligrams per liter of the trihalomethane compounds [trichloromethane (chloroform), dibromochloromethane, bromodichloromethane, and tribromomethane (bromoform)], rounded to two significant figures.

001.01OOO "Vacuum Breaker, Atmospheric Type" means a vacuum breaker which is not designed to be subject to static line pressure.

001.01PPP "Vacuum Breaker, Pressure Type" means a vacuum breaker which is designed to be subject to static line pressure.

001.01QQQ "Virus" means a virus of fecal origin which is infectious to humans by waterborne transmission.

001.01RRR "Waterborne Disease Outbreak" means the significant occurrence of acute infectious illness, epidemiologically associated with the ingestion of water from a public water supply system which is deficient in treatment as determined by the Director.

001.01SSS "Water Supply System" means all sources of water and their surroundings under control of one owner, and shall include all structures, conduits, and appurtenances by means of which such water is collected, treated, stored, or delivered, except service pipes between street mains and buildings and the plumbing within or in connection with the buildings served.

001.02 Coverage. This chapter shall apply to each public water supply system, unless the public water supply system meets all of the following conditions:

001.02A Which consists only of distribution and storage facilities and does not have any collection and treatment facilities

001.02B Which obtains all of its water from, but is not owned operated by, a public water supply stem to which such standards apply;

001.02C Which does not sell water to any person; and

001.02D Which is not a carrier which conveys passengers in interstate commerce.

001.03 Responsibility. The owner of each public water supply, as defined in these regulation, shall designate an individual, or individuals, who shall be responsible for contact and communications with the Director in matters relating to system alteration and construction, monitoring and sampling, maintenance, operation, record keeping, and reporting, as required by law and these regulation. Any change in assigned responsibilities or designated individuals shall be promptly reported to the Director.

002 DRINKING WATER STANDARDS
(INCLUDES AMENDMENTS MADE THROUGH SEPTEMBER 15, 1991)

002.01 Applicability. The basis for the establishment of maximum contaminant levels is based either upon potential acute health effects over a short length of time of exposure or chronic health effects over a long length of time of exposure.

002.01A Standards Based upon Acute Health Effects. Standards based upon acute health effects over a short length of time of exposure shall apply to all public water supply systems covered by this Chapter. Contaminants governed by these standards are:

002.01A1 Nitrates

002.01A2 Turbidity

002.01A3 Microbiological

002.01B Standards Based upon Chronic Health Effects. Standards based upon chronic health effects over a long length of time of exposure shall apply to community and non-transient, non-community water supply systems. Contaminants governed by these standards are:

002.01B1 inorganic chemicals except for nitrate

002.01B2 organic chemicals

002.01B3 radium-226, radium-228 and gross alpha particle activity

002.01B4 beta particle and photon radioactivity from man-made radionuclides.

002.02 Maximum Contaminant Levels.

002.02A Inorganic Chemicals. The maximum contaminant levels for inorganic chemical contaminants are as follows:

CONTAMINANT	LEVEL MILLIGRAMS PER LITER
Arsenic	0.05
Barium	1.0
Cadmium	0.010
Chromium total	0.05
Fluoride*	4.0
Lead	0.05
Mercury	0.002
Nitrate (as N)	10
Selenium	0.01
Silver	0.05
Sodium	500.0

*Community water supply systems experiencing fluoride levels above 2.0 milligrams per liter must notify the public as required in Section 004.07

002.02B Organic Chemicals. The maximum contaminant levels for organic chemical contaminants, are as follows:

CONTAMINANT	LEVEL,MILLIGRAMS PER LITER
002.02B1 Chlorinated Hydrocarbons:	
Endrin (1,2,3,4,10,10,-hexachloro-6, 7-epoxy-1,4,4a,5,6,7,8,8a-octa-hydro-1, 4-endo, endo-5, 8-dimethanonaphthalene).	0.0002
Lindane (1,2,3,4,5,6-hexachloro-cyclohexane, gamma isomer)	0.004
Methoxychlor (1,1,1-Trichloro-2,2-bis (p-methoxyphenyl ethane)	0.1
Toxaphene ($C^{1}_{0}H_{10}Cl_{8}$) Technical chlorinated camphene, 67-69 percent chlorine	0.005
002.02B2 Chlorophenoxys:	
2,4-D (2,4-Dichlorophenoxyacetic acid)	0.1
2,4,5-TP Silvex (2,4,5-Trichloro-phenoxypropionic acid)	0.01
002.02B3 Trihalomethanes:	
Total Trihalomethanes	0.10
002.02B4 Volatile Organic Chemicals (VOC):	
1,1,1-Trichloroethane	0.2
1,1-Dichloroethylene	0.007
1,2-Dichloroethane	0.005
Benzene	0.005
Carbon Tetrachloride	0.005
Trichloroethylene	0.005
Vinyl Chloride	0.002
Para-Dichlorobenzene	0.075

002.02C Turbidity. The maximum contaminant levels for turbidity are applicable to all public water supply systems using surface water, in whole or in part, as a source of supply. Turbidity shall be measured at representative entry points to the distribution system. The maximum permissible levels for turbidity are as follows:

002.02C1 One turbidity unit (TU), as determined by the monthly average of daily examinations, except that up to five turbidity units may be allowed if the owner can demonstrate that the higher turbidity does not interfere with disinfection, will not prevent maintenance of the disinfectant agent throughout the system, or will not interfere with microbiological examinations of the water; and

002.02C2 When the exceptions listed under the preceding paragraph cannot be demonstrated by the owner, the average of two samples taken on consecutive days does not exceed five turbidity units.

002.02C3 The requirements of 002.02C1 and 002.02C2 shall apply until June 29, 1993.

002.02D Microbiological. The maximum contaminant levels for coliform bacteria, applicable to all public water supply systems, are as follows:

002.02D1 The MCL is based on the presence or absence of total coliforms in a sample, rather than coliform density.

002.02D1a For a system which collects at least 40 samples per month, if no more than 5.0 percent of the samples collected during a month are total coliform-positive, the system is in compliance with the MCL for total coliforms.

002.02D1b For a system which collects fewer than 40 samples per month, if no more than one sample collected during a month is total coliform-positive, the system is in compliance with the MCL for total coliforms.

002.02D1c Results of all routine samples and repeat samples (when required by 002.03A7) which are not invalidated must be included in determining compliance with 002.02D1a and 002.02D1b.

002.02D2 Any fecal coliform-positive repeat sample or E. coli-positive repeat sample, or any total coliform-positive repeat sample following a fecal coliform-positive or E. coli-positive routine sample constitutes a violation of the MCL for total coliforms. For purposes of the public notification requirements in 004.02A3 this is a violation that may pose an acute risk to health.

002.02D3 Compliance with the MCL for total coliforms in 002.02D1 and 002.02D2 shall be determined each month for systems which are required to monitor monthly for total coliforms, and each quarter for systems which are required to monitor once per quarter for total coliforms.

002.02E Radium-226, Radium-228 and Gross Alpha Particle Activity. The maximum contaminant levels for Radium-226, Radium-228, and gross alpha particle radioactivity, are:

002.02E1 Combined radium-226 and radium-228 = 5 pCi per liter.

002.02E2 Gross alpha particle activity including radium-226 but excluding radon and uranium = 15 pCi per liter.

002.02F Beta Particle and Photon Radioactivity from Man-Made Radionuclides. The maximum contaminant levels for beta particle and photon radioactivity from man-made radionuclides, are:

002.02F1 The average annual concentration of beta particle and photon radioactivity from man-made radionuclides shall not produce an annual dose equivalent to the total body or any internal organ greater than 4 millirem per year.

002.02F2 Except for the radionuclides listed in Table A, the concentration of man-made radionuclides causing 4 mrem total body or organ dose equivalents shall be calculated on the basis of a 2 liter per day drinking water intake using the 168 hour date listed in "Maximum Permissible Body Burdens and Maximum Permissible Concentration of Radionuclides in Air or Water for Occupational Exposure", NBS Handbook 69, as amended August, 1963, U.S. Department of Commerce, a copy of which is attached and incorporated by reference as if fully set forth with herein. If two or more radionuclides are present, the sum of their annual dose equivalent to the total body or any organ shall not exceed 4 millirem per year.

TABLE A - Average Annual Concentrations Assumed to Produce a Total Body or Organ Dose of 4 millirem per year.

Radionuclide	Critical Organ	pCi per liter
Tritium	Total Body	20,000
Strontium-90	Bone Marrow	8

002.03 Monitoring Requirements. The owners of public water supply systems shall be responsible for accomplishing monitoring requirements as demonstrated by ossession of an official copy of laboratory results. The Director will establish schedules for sampling, and will assist in the collection of chemical and radiological samples. Samples will be examined at timed intervals and on schedules designed to meet monitoring requirements and maintain a uniform laboratory work load. The owner of each public water supply will be informed of this schedule and, if for any reason the schedule is not met, will be responsible for initiating arrangements for an alternate date to effect compliance with established monitoring requirements. The arrangements shall be timed to provide the required number of samples within the designated sample period used to determine compliance with these regulations. All sample analyses needed be meet monitoring requirements of this chapter, unless otherwise stated, shall be examined by a State Health Department Laboratory or a laboratory which has entered into an agreement with the Department of Health pursuant to 002.03H.

<u>002.03A</u> Microbiological.

<u>002.03A1</u> The owners of public water supply systems shall collect total coliform samples at sites which are representative of both population densities and distribution system characteristics according to a written sample site plan. These plans are subject to review and revision by the Director. All biological samples shall be mailed to the assigned laboratory through the U.S. Postal Service, with the owner paying the postage, unless the Director authorizes other means of transportation.

<u>002.03A1a</u> The sample site plan shall consist of sampling points at sites scattered throughout various zones of the distribution system. Each plan shall provide for at least five sampling sites in each zone and there shall be as many zones as the number of routine total coliform samples required each month up to sixteen zones. Systems which are required to collect more than sixteen samples per month may elect to have more than sixteen zones, but it is not required. A map of the area served by the public water supply system, showing the distribution system and the boundaries of the various zones, labeled numerically, shall be included in the plan. A list of all sampling sites, by name and address (or by a readily identifiable location) for each zone shall be included with the map and, except for supplies having only one zone, the location of the sites need not be indicated on the map. All zones shall be sampled monthly. The actual sites used within each zone shall be varied on a scheduled rotation basis. Both the zone number and the site location shall be noted on the laboratory report form by the person taking the sample. The owner of each community water supply system shall submit a copy of the sampling site plan to the Director for review no later than 90 days after the effective date of these regulations, with updated copies submitted annually thereafter. The Director, at any time, may require a plan be modified as a result of population or system changes which may have rendered an existing plan non-representative.

002.03A2 The owner of a community water supply system shall take total coliform samples at regular time intervals established by the Director. The number of samples required shall in no instance be less than as set forth below:

Population Served Minimum Number of Per Month	Minimum Number of Sampling Zones	Minimum Number of Samples Per Month	Population Served	Minimum Number of Sampling Zones	Samples
25 - 1,000	1	1	46,001 - 58,000	16	64
1,001 - 2,500	2	2	58,001 - 70,000	16	76
2,501 - 4,100	4	4	70,001 - 83,000	16	88
4,101 - 7,600	8	8	83,001 - 96,000	16	96
7,601 - 11,000	12	12	96,001 - 130,000	16	112
11,001 - 14,600	16	16	130,001 - 220,000	16	140
14,601 - 18,100	16	20	120,001 - 320,000	16	172
18,101 - 21,500	16	24	320,001 - 450,000	16	200
21,501 - 28,000	16	32	450,001 - 600,000	16	232
28,001 - 37,000	16	40	600,001 - 780,000	16	260
37,001 - 46,000	16	52	780,001 - 970,000	16	292

002.03A3 The owner of a non-community water supply system shall take samples for total coliforms according to a frequency as follows:

002.03A3a A non-community water supply system using only ground water (except ground water under the direct influence of surface water) and serving 1,000 persons or fewer must sample each calendar quarter that the system provides water to the public.

002.03A3b A non-community water supply system using only ground water (except ground water under the direct influence of surface water) and serving more than 1,000 -during any month must sample at the same frequency as a like-sized community water system, as specified in 002.03A2.

002.03A3c The owner of a non-community water supply system using surface water, in total or in part, must sample at the same frequency as a like-sized community water system, as specified in 002.03A2, regardless of the number of persons it serves.

002.03A3d The owner of a non-community water supply system using ground water under the direct influence of surface water, as defined in 001.01W must sample at the same frequency as a like-sized community water supply system, as specified in 002.03A2. The owner must sample at this frequency beginning six months after the Director determines that the ground water is under the direct influence of surface water.

002.03A4 The owner of a public water supply system must collect samples at regular time intervals throughout the month.

002.03A5 The owner of a public water supply system that uses surface water or ground water under the direct influence of surface water, as defined in 001.01W, and does not practice filtration in compliance with 002.04 must collect at least one sample near the first service connection each day the turbidity level of the source water, measured as specified in 002.04H2b exceeds 1 NTU. The owner must collect this coliform sample within 24 hours of the first exceedance. Sample results from this coliform monitoring must be included in determining compliance with the MCL for total coliforms in 002.02D.

002.03A6 Special purpose samples, such as those taken to determine whether disinfection practices are sufficient following pipe placement, replacement, or repair, shall not be used to determine compliance with the MCL for total coliforms in 002.02D. Repeat samples taken pursuant to 002.03A7 are not considered special purpose samples, and must be used to determine compliance with the MCL for total coliforms in 002.02D.

002.03A7 Repeat monitoring.

002.03A7a If a routine sample is total coliform-positive, the owner of the public water supply system must collect a set of repeat samples within 24 hours of being notified of the positive result. A system which is required to collect more than one routine sample per month shall have no fewer than three repeat samples collected for each total coliform-positive sample found. A system which is required to collect one routine sample per month or fewer shall have no fewer than four repeat samples collected for each total coliform-positive sample found. The Director may extend the 24-hour limit on a case-by-case basis if the owner has a logistical problem in collecting the repeat samples within 24 hours that is beyond its control. In the case of an extension, the Director must specify how much time the owner has to collect the repeat samples.

002.03A7b The system owner must collect at least one repeat sample from the sampling tap where the original total coliform-positive sample was taken, and at least one repeat sample at a tap within five service connections upstream and at least one repeat sample at a tap within five service connections downstream of the original sampling site. The fourth repeat sample, if required by 002.03A7, must be collected within five service connections upstream or downstream of the original sampling site. If a total coliform-positive sample is at the end of the distribution system, or one away from the end of the distribution system, the Director may waive the requirement to collect at least one repeat sample upstream or downstream of the original sampling site.

<u>002.03A7c</u> The owner shall collect all repeat samples on the same day, except that an owner of a system with a single service connection shall collect the required set of repeat samples over a four-day period.

<u>002.03A7d</u> If one or more repeat samples in the set is total coliform-positive, the owner of the public water supply system must collect an additional set of repeat samples in the manner specified in 002.03A7a-c. The owner must repeat this process until either total coliforms are not detected in one complete set of repeat samples or the MCL for total coliforms in 002.02D has been exceeded and the Director determines that no additional repeat samples are required.

<u>002.03A7e</u> If a system which is required to collect fewer than five routine samples per month has one or more total coliform-positive samples and the Director does not invalidate the sample(s) under 002.03A8, the owner must collect at least five routine samples during the next month the system provides water to the public, except that the Director may waive this requirement if the conditions of 002.03A7e1-3 are met. The Director shall not waive the requirement for a system to collect repeat samples in 002.03A7a-d.

<u>002.03A7e(1)</u> The Director may waive the requirement to collect five routine samples the next month the system provides water to the public if the Director or an agent approved by the Director performs a site visit before the end of the next month the system provides water to the public. Although a sanitary survey need not be performed, the site visit shall be sufficiently detailed to allow the Director to determine whether additional monitoring and/or any corrective action is needed. The Director shall not approve an employee of the system owner to perform this site visit, even if the employee is an agent approved by the Director to perform sanitary surveys.

<u>002.03A7e(2)</u> The Director may waive the requirement to collect five routine samples the next month the system provides water to the public if the Director has determined why the sample was total coliform-positive

and established that the owner of the system will correct the problem before the end of the next month the system serves water to the public. In this case, the Director must document this decision to waive the following month's additional monitoring requirement in writing, have it approved and signed by the supervisor of the Director's authorized representative who recommends such a decision, and make this document available to the EPA and public. The written documentation must describe the specific cause of the total coliform-positive sample and what action the system has taken and/or will take to correct this problem. The Director shall not waive the requirement to collect five routine samples the next month the system provides water to the public solely on the grounds that all repeat samples are total coliform-negative. If the requirement to collect five routine samples the next month is waived under this section, the system owner must still take at least one additional routine sample before the end of the next month the system serves water to the public and use it to determine compliance with the MCL for total coliforms in 002.02D.

002.03A7e(3) The Director shall consider the waiver of the requirement to collect five routine samples the next month only upon receipt of a request in writing from the system owner. The waiver shall not be considered until after the repeat samples required in 002.03A7a-d have been collected and the results reported to the Director. The waiver shall not be granted if any of the repeat samples are coliform-positive, unless all positive samples have been invalidated under 002.03A8, or if a similar waiver has been granted within the six months previous to the date of the collection of the initial coliform positive sample.

002.03A7f Results of all routine and repeat samples not invalidated by the Director must be included in determining compliance with the MCL for total coliforms in 002.02D.

002.03A8 Invalidation of total coliform samples. A total coliform-positive sample invalidated under this section does not count towards meeting the minimum monitoring requirements of 002.03A. If a total coliform-positive sample is invalidated under this section, the system owner must collect another sample from the same zone as the original sample to meet monitoring requirements.

002.03A8a The Director may invalidate a total coliform-positive sample only if one or more of the conditions of 002.03A8a(1)-002.03A8a(4) are met.

002.03A8a(1) The laboratory establishes that improper sample analysis caused the total coliform-positive result.

002.03A8a(2) The Director, on the basis of the results of repeat samples collected as required by 002.03A7a-d determines that the total coliform-positive sample resulted from a domestic or other non-distribution system plumbing problem. The Director shall not invalidate a sample on the basis of repeat sample results unless all repeat sample(s) collected at the same tap as the original total coliform-positive sample are also total coliform-positive, and all repeat samples collected within five service connections of the original tap are total coliform-negative (e.g., the Director shall not invalidate a total coliform-positive sample on the basis of repeat samples if all the repeat samples are total coliform-negative or if the public water system has only one service connection.)

002.03A8a(3) The Director has substantial grounds to believe that a total coliform-positive result is due to a circumstance or condition which does not reflect water quality in the distribution system. In this case, the system owner must still collect all repeat samples required under 002.03A7 and use them to determine compliance with the MCL for total coliforms in 002.02D. To invalidate a total coliform-positive sample under this paragraph, the decision with the rationale for the decision must be documented in writing and approved and signed by the Director's authorized representative. The Director must make this document available to EPA and the public. The written documentation must state the specific cause of the total coliform-positive sample, and what action the system has taken or will take to correct this problem. The Director may not invalidate a total coliform-positive sample solely on the grounds that all repeat samples are total coliform-negative.

002.03A8a(4) The Director shall consider invalidation of a coliform-positive sample under 002.03A8a(3) only upon receipt of a request in writing from the owner of the public water supply system from which the coliform-positive sample was collected. Such sample shall not be invalidated if any of the repeat samples collected at locations other than that of the coliform-positive sample are coliform-positive. No coliform-positive sample shall be invalidated if any of the most recent six samples collected from the system were coliform-positive.

002.03A8b A laboratory must invalidate a total coliform sample (unless total coliforms are detected) if the sample produces a turbid culture in the absence of gas production using an analytical method where gas formation is examined (e.g., the Multiple-Tube-Fermentation Technique), produces a turbid culture in the absence of an acid reaction in the Presence-Absence (P-A) Coliform Test, or exhibits confluent growth or produces colonies too numerous to count with an analytical methods using a membrane filter (e.g., Membrane Filter Technique). If a laboratory invalidates a sample because of such interference, the system owner must collect another sample from the same location as the original sample within 24 hours of being notified of the interference problem, and have it analyzed for the presence of total coliforms. The system owner must continue to re-sample within 24 hours and have the samples analyzed until a valid result is obtained. The Director may waive the 24-hour time limit on a case-by-case basis.

002.03A9 Sanitary Surveys.

002.03A9a Public water systems which do not collect five or more routine samples per month must undergo an initial sanitary survey by June 29, 1994, for community public water systems and June 29, 1999, for non-community systems. Thereafter, systems must undergo another sanitary survey every five years, except that non-community water systems using only disinfected ground water and wells which have been constructed in accordance with and continue to meet the siting requirements of Section 007, must undergo subsequent sanitary surveys at least every ten years after the initial sanitary survey.

002.03A9b Sanitary surveys must be performed by the Nebraska Department of Health personnel or an agent approved by the Department. The system is responsible for ensuring the survey takes place.

002.03A10 Fecal coliforms/Escherichia coli (E. coli) testing.

002.03A10a If any routine or repeat sample is total coliform-positive, that total coliform-positive culture medium must be analyzed to determine if fecal coliforms are present, except that E. coli may be tested for in lieu of fecal coliforms. If fecal coliforms or E. coli are present in samples analyzed by a laboratory other than the Nebraska Department of Health Laboratory, the system owner must notify the Director by the end of the day when the system owner is notified of the test result, unless the system owner is notified of the result after the Director's office is closed, in which case the system owner must notify the Director before the end of the next business day.

002.03A10b The Director has the discretion to allow the owner of a public water system, on a case-by-case basis, to forgo fecal coliform or E. coli testing on a total coliform-positive sample if the owner assumes that the total coliform-positive sample is fecal-coliform-positive or E. coli-positive. Accordingly, the owner must notify the Director as specified in 002.03A10a and the provisions of 002.02D2 apply.

002.03A11 Response to violation.

002.03A11a A public water system which has exceeded the MCL for total coliforms in 002.02D must report the violation to the State no later than the end of the next business day after it learns of the violation, and notify the public in accordance with 004.02.

002.03A11b A public water system which has failed to comply with a coliform monitoring requirement, including the sanitary survey requirement, must report the monitoring violation to the State within ten days after the system discovers the violation, and notify the public in accordance with 004.03.

002.03B Turbidity. Samples shall be taken by owners of community, non-community and non-transient, non-community surface water supply systems at a representative point of entry to the distribution system at least once a day for the purpose of making turbidity measurements. When the results of a turbidity analysis indicates that the maximum allowable level has been exceeded, the sampling and measurement shall be repeated as soon as practicable and preferably within one hour. The repeat sample shall be the one used in computing the monthly average. This section shall apply until June 29, 1993.

002.03C Inorganic Chemicals, Chlorinated hydrocarbons and Chlorophenoxys.

002.03C1 Chlorinated Hydrocarbons, Chlorophenoxys and Inorganic chemicals other than Nitrate. The owner of a community or non-transient, non-community public water supply system shall take chemical samples at three year intervals when ground water is the source of supply and at one year intervals when surface water is the source of supply except that, regardless of the source of water, the owner shall take samples for sodium analyses at one year intervals. Samples shall be submitted to the State Health Department Laboratory in containers provided by the Laboratory. Owners of systems using approved laboratories pursuant to paragraph 002.03H of this Chapter shall report initial analyses and repeat the analysis and report at one year intervals when surface water is used as a source and shall report initial analysis and repeat the analysis and report at three year intervals when ground water is used as a source.

002.03C1a When any maximum contaminant level is exceeded in a public water supply, the owner of the supply shall be responsible for collecting, preserving, and transporting samples to the State Health Department Laboratory in accordance with the following schedule: three additional samples from the sample sampling point within one month of notification of the result that exceeded the maximum contaminant level. When the average of the original and three confirming samples, rounded to the same number of significant figures as the maximum contaminant level of the substance in question, demonstrates a value equal to or below the maximum contaminant level no further sampling is required until the time interval established in paragraph 002.03C expires.

002.03C1b When the average of the four samples exceeds the maximum contaminant level, samples shall be submitted at quarterly intervals until such time as a variance, exemption, or enforcement action takes effect or until compliance is accomplished.

002.03C1c The Director may reduce the sampling frequency for chlorinated hydrocarbons and chlorophenoxys by systems using only ground water as source of supply after initial sampling demonstrates the absence of the contaminants and when the results of a sanitary survey indicate remote possibility of future contamination.

002.03C2 Nitrate Monitoring. The owner of a public water system shall take samples for nitrate analysis at yearly intervals. Compliance with the maximum contaminant level for nitrate shall be determined on the basis of the mean of two analyses. When a level exceeding the maximum contaminant level is found, a second analysis shall be initiated within 24 hours, and if the mean of the two analyses exceeds the maximum contaminant level, the supplier of water shall notify the public pursuant to section 004 of this Chapter.

002.03D Trihalomethanes. Community and non-transient, non-community water systems which add a disinfectant (oxidant) to the water in any part of the drinking water treatment process shall take samples for total trihalomethane potential analysis at yearly intervals unless provisions of subparts 002.03D1 or 002.03D2 apply and shall submit them to the State Health Department Laboratory in containers provided by the Laboratory. Owners of systems using approved laboratories pursuant to part 002.03H of this Chapter shall report analysis as prescribed in subpart 002.03D1.

002.03D1 Water delivered by systems serving more than 10,000 persons and which utilize surface water, in whole or in part as a source of supply shall be analyzed for Total Trihalomethane concentration at quarterly intervals in at least four water samples for each treatment plant used by the system. All samples shall be collected within a 24-hour period. At least 25 percent of the samples shall be taken at representative locations within the distribution system taking into account the number of persons served, different sources of water, and different treatment methods employed. Results from all samples collected shall be used to determine the quarterly average unless invalidated for technical reasons. Compliance shall be determined by averaging the newest quarterly result with the three previous quarterly results. Systems using approved laboratories pursuant to 002.03H shall report results to the Director within thirty days of the analysis.

002.03D2 Based on the results of one or more maximum total trihalomethane potential examinations the Director may excuse a community water system using only ground water as a source and serving fewer than 10,000 persons from further monitoring when an assessment of local conditions indicate the system is not likely to approach or exceed the maximum contaminant level for total trihalomethanes. Any change in the treatment process or disinfection practices will require reinstatement of monitoring requirements.

002.03E Volatile Organic Chemicals. Analyses of the contaminants listed in 002.02B4 for purposes of determining compliance with the maximum contaminant levels shall be conducted as follows:

002.03E1 Groundwater systems shall sample at points of entry to the distribution system representative of each well after any application of treatment. Sampling must be conducted at the same location or a more representative location each quarter. Groundwater systems must sample every three months for every point to the distribution system except as provided in 002.03E8a.

002.03E2 Surface water systems shall sample at points in the distribution system representative of each source or at entry points to the distribution system after any application of treatment. Surface water systems must sample every three months except as provided in 002.03E8b. Sampling must be conducted at the same location or a more representative location each quarter.

002.03E3 If the system draws water from more than one source and sources are combined before distribution the system must sample at an entry point to the distribution system during periods of normal operating conditions.

002.03E4 All community water supply systems and non-transient, non-community supply systems serving more than 10,000 people shall analyze all distribution or entry-point samples, as appropriate, representing all source waters beginning no later than January 1, 1988. All community water supply systems and non-transient, non-community water supply systems serving from 3,300 to 10,000 people shall analyze all distribution or entry-point samples, as required in this subsection, representing all source waters beginning no later than January 1, 1989. All other community and non-transient, non-community water supply systems shall analyze distribution or entry point samples, as required in the subsection, representing all source waters beginning no later than January 1, 1991.

002.03E5 The Director may require confirmation samples for positive or negative results. If a confirmation sample(s) is required by the Director, than the sample result(s) shall be averaged with the first sampling result and used for compliance determination in accordance with 002.03E9 of this section. The Director shall have discretion to delete results of obvious sampling errors from this calculation.

002.03E6 Analyses for vinyl chloride is required only for groundwater systems that have detected one or more of the following two-carbon organic compounds: Trichloroethylene, tetrachloroethylene, 1,2-dichloroethane, 1,1,1-trichloroethane, cis-1,2-dichloroethylene,trans-1,2-dichloroethylene, or 1,1-dichloroethylene. The analysis for vinyl chloride is required at each distribution or entry point at which one or more of the two-carbon organic compounds were found. If the first analyses does not detect vinyl chloride, the director may reduce the frequency of vinyl chloride monitoring to once every three years for that sample location for other sample locations which are more representative of the same source. Surface water systems may be required to analyze for vinyl chloride at the discretion of the Director.

002.03E7 The Director or individual public water supply systems may choose to composite up to five samples from one or more public water systems. Compositing of samples is to be done in the laboratory by the procedures listed below. Samples must be analyzed within fourteen days of collection. If any organic contaminant listed in 002.02B4 is detected in the original composite sample, a sample from each source that made up the composite sample must be reanalyzed individually within fourteen days of sampling. The sample for reanalyses cannot be the original sample but can be a duplicate sample. If the duplicates of the original samples are not available, new samples must be taken from each source used in the original composite and analyzed for VOCs. Reanalysis must be made within fourteen days of the second sample. To composite samples, the following procedure must be followed:

002.03E7a Compositing samples prior to GC analysis.

002.03E7a(1) Add 5 ml or equal larger amounts of each sample (up to 5 samples are allowed) to a 25 ml glass syringe. Special precautions must be made to maintain zero headspace in the syringe.

002.03E7a(2) The samples must be cooled at 4°C during this step to minimize volatilization losses.

002.03E7a(3) Mix well and draw out a 5-ml aliquot for analysis.

002.03E7a(4) Follow sample introduction, purging and desorption steps described in the method.

002.03E7a(5) If less than five samples are used for compositing, a proportionately smaller syringe may be used.

002.03E7b Compositing samples prior to GC/MS analysis.

002.03E7b(1) Inject 5-ml or equal larger amounts of each aqueous sample (up to 5 samples are allowed) into a 25-ml purging device using the sample introduction technique described in the method.

002.03E7b(2) The total volume of the sample in the purging devise must be 25 ml.

002.03E7b(3) Purge and desorb as described in the method.

002.03E8 The Director may reduce the monitoring frequency specified in 002.03E1 and 002.03E2 as explained in this paragraph:

002.03E8a The monitoring frequency for groundwater systems is as follows:

002.03E8a(1) When VOCs are not detected in the first sample (or any subsequent samples that may be taken) and the system is not vulnerable as defined in 002.03E8d, monitoring may be reduced to one sample and must be repeated every 5 years.

002.03E8a(2) When VOCs are not detected in the first sample (or any subsequent sample that may be taken) and the system is vulnerable as defined in 002.03E8d,

002.03E8a(2)(a) Monitoring (i.e., one sample) must be repeated every 3 years for systems greater than 500 connections.

002.03E8a(2)(b) Monitoring (i.e., one sample) must be repeated every 5 years for systems with less than or equal to 500 connections.

002.03E8a(3) If VOCs are detected in the first sample (or any subsequent sample that may be taken), regardless of vulnerability, monitoring must be repeated every 3 months, as required under 002.03E1.

002.03E8b The repeat monitoring frequency for surface water systems is as follows:

002.03E8b(1) When VOCs are not detected in the first year of quarterly sampling (or any other subsequent sample that may be taken) and the system is not vulnerable as defined in 002.03E8d, monitoring is only required at the discretion of the Director.

002.03E8b(2) When VOCs are not detected in the first year of quarterly sampling (or any other subsequent sample that may be taken) and the system is vulnerable as defined in 002.03Ed.

002.03E8b(2)(a) Monitoring must be repeated every three years (for systems greater than 500 connections).

002.03E8b(2)(b) Monitoring must be repeated every five years (for systems less than or equal to 500 connections).

002.03E8b(3) When VOCs are detected in the first year of quarterly sampling (or any subsequent sample that may be taken) regardless of vulnerability, monitoring must be repeated every 3 months, as required under 002.03E2.

002.03E8c The Director may reduce the frequency of monitoring to once per year for a groundwater system or surface water system detecting VOCs at levels consistently less than the MCL for three consecutive years.

002.03E8d Vulnerability of each public water system shall be determined by the Director based upon an assessment of the following factors:

002.03E8d(1) Previous monitoring results.

002.03E8d(2) Number of persons served by the public water system.

002.03E8d(3) Proximity of a smaller system to a larger system.

002.03E8d(4) Proximity to commercial or industrial use, disposal, or storage of Volatile Synthetic Organic Chemicals.

002.03E8d(5) Protection of the water source.

002.03E8e A system is deemed to be vulnerable for a period of three years after any positive measurement of one or more contaminants listed on 002.02B4 or 002.03K5 except for trihalomethanes or other demonstrated disinfection by-product.

002.03E9 Compliance with 002.02B4 shall be determined based on the results of running annual average of quarterly sampling for each sampling location. If one location's average is greater than the MCL, then the system shall be deemed to be out of compliance. If a public water system has a distribution system separable from other parts of the distribution system with no interconnections, only that part of the system that exceeds any MCL as specified in 002.02B4 will be deemed out of compliance. The Director may reduce the public notice requirement to that portion of the system which is out of compliance. If any one sample result would cause the annual average to be exceeded, than the system shall be deemed to be out of compliance immediately, For systems that only take one sample per location because no VOCs were detected, compliance shall be based on that one sample.

002.03F Radionuclides. The State Health Department will collect and analyze samples from each community water supply for radioactivity by July 1, 1979, when surface water is used as a source, and by July 1, 1980, when ground water is the sole source of supply. Theses analyses will be repeated at four-year intervals, except analysis for beta activity in ground water sources may be less frequent at the discretion of the Director.

002.03G Special Monitoring - Radionuclides. The owner of any community or non-transient, non-community water supply system designated by the Director as utilizing waters receiving effluents from nuclear facilities shall initiate quarterly monitoring for gross beta particle and Iodine-131 radioactivity and annual monitoring for Strontium-90 and Tritium.

002.03G1 Quarterly monitoring for gross beta particle activity shall be based on the analysis of monthly samples or the analysis of a composite of three monthly samples. The former is recommended. If the gross beta particle activity in a sample exceeds 15 pCi per liter, the same or an equivalent sample shall be analyzed for Strontium-89 and Cesium-134. If the gross beta particle activity exceeds 50 pCi per liter, an analysis of the sample must be performed to identify the major radioactive constituents present and the appropriate organ and total body doses shall be calculated to determine compliance with subsection 002.02F of these rules.

002.03G2 For Iodine-131, a composite of five consecutive daily samples shall be analyzed once each quarter. As ordered by the Director, more frequent monitoring shall be conducted when Iodine-131 is identified in the finished water.

002.03G3 Annual monitoring for Strontium-90 and Tritium shall be conducted by means of the analysis of a composite of four consecutive quarterly samples or analysis of four quarterly samples. The latter procedure is recommended.

002.03H Approved Laboratories. The Department of Health shall enter into an agreement with any laboratory demonstrating a continuing capability to perform required analyses, to conduct each specific analysis in an acceptable manner, to participate in quality control activities, and to comply with all requirements of these regulations relative to maintaining records and reports.

002.03I Consecutive Systems. When a public water supply system provides water to one or more other public water supply systems, the Director may modify the monitoring imposed by Section 002 to the extent that the inter-connection of the systems justifies treating them as one system for monitoring purposes.

002.03J Treatment Techniques. Treatment techniques and processes for removing or reducing the levels of any of the contaminants listed in section 002 shall be approved by the Director prior to application and shall conform with tested and proven engineering practices. The Director shall include recommended criteria for acceptance of such techniques and processes in the "Standards for Water System Design" herein incorporated by reference as Attachment 1, required by section 007 of these rules.

002.03K Special Monitoring for Organic Chemicals.

002.03K1 All community and non-transient, non-community water supply systems shall monitor for the contaminants listed in 002.03K5 in this section by the date specified in Table 1.

TABLE 1 Number of Persons Served	Monitoring to begin no later than:
Over 10,000	January 1, 1988
3,300 to 10,000	January 1, 1989
Less than 3,300	January 1, 1991

002.03K2 Surface water systems shall sample at points in the distribution system representative of each water source or at entry points to the distribution system. The minimum number of samples is one year of quarterly samples per water source.

002.03K3 Groundwater systems shall sample at points of entry to the distribution system representative of each well after any application of treatment. The minimum number of samples is one sample per entry point to the distribution system. The sampling frequency shall be specified in 002.03E.

002.03K4 The Director may require confirmation samples for positive or negative results.

002.03K5 Community water supply systems and non-transient, non-community water supply systems shall monitor for the following contaminants except as provided in 002.03K6.

002.03K5a Chloroform

002.03K5b Bromodichloromethane

002.03K5c Chlorodibromomethane

002.03K5d Bromoform

002.03K5e trans-1,2,-Dichloroethylene

002.03K5f Chlorobenzene

002.03K5g m-Dichlorobenzene

002.03K5h Dichloromethane

002.03K5i cis-2,3-Dichloroethylene

002.03K5j o-Dichlorobenzene

002.03K5k Dibromomethane

002.03K5l 1,1-Dichloropropene

002.03K5m Tetrachloroethylene

002.03K5n Toluene

002.03K5o p-Xylene

002.03K5p o-Xylene

002.03K5q m-Xylene

002.03K5r 1,1-Dichloroethane

002.03K5s 1,2-Dichloropropane

002.03K5t 1,1,2,2-Tetrachloroethane

002.03K5u Ethylbenzene

002.03K5v 1,3-Dichloropropane

002.03K5w Styrene

002.03K5x Chloromethane

002.03K5y Bromomethane

002.03K5z 1,2,3-Trichloropropane

002.03K5aa 1,1,1,2-Tetrachloroethane

002.03K5bb Chloroethane

002.03K5cc 1,1,2-Trichloroethane

002.03K5dd 2,2-Dichloropropane

002.03K5ee o-Chlorotoluene

002.03K5ff p-Chlorotoluene

002.03K5gg Bromobenzene

002.03K5hh 1,3-Dichloropropene

002.03K5ii Ethylene dibromide (EDB)

002.03K5jj 1,2-Dibromo-3-chloropropane (DBCP)

002.03K6 Community water supply systems and non-transient, non-community water supply systems must monitor for EDB and DBCP only if the Director determines they are vulnerable to contamination by either or both of these substances. For the purpose of this section, a vulnerable system is defined as a system which is potentially contaminated by EDB and DBCP, including surface water system where these two compounds are applied, manufactured, stored, disposed of, or shipped upstream, and for groundwater systems in areas where the compounds are applied, manufactured, stored, disposed of or shipped in the groundwater recharge basin or groundwater systems that are in proximity to underground storage tanks that contain leaded gasoline.

002.03K7 Public water supply systems may use monitoring data collected any time after January 1, 1983, to meet the requirements for unregulated monitoring, provided that the monitoring program was consistent with the requirements of this section.

002.03K8 Instead of performing the monitoring required by this section, a community water supply system or non-transient, non-community water supply system serving fewer than 150 service connections may send a letter to the Director stating that its system is available for sampling. This letter must be sent to the Director no later than January 1, 1991. The system shall not send such samples to the Director, unless requested to do so by the Director.

002.03K9 All community and non-transient, non-community water supply systems shall repeat the monitoring required in 002.03K no less frequently than every five years from the dates specified in 002.03K1.

002.04 Filtration and Disinfection. This section establishes treatment technique requirements in lieu of maximum contaminant levels for the following contaminants: Giardia lamblia, viruses, heterotrophic plate count bacteria, legionella, and turbidity.

002.04A Each public water supply system with a surface water source or a ground water source under the direct influence of surface water must include treatment of that source water that complies with these treatment technique requirements. The treatment technique requirements consist of installing and properly operating water treatment processes which reliably achieve:

002.04A1 At least 99.9 percent (3-log) removal and/or inactivation of Giardia lamblia cysts between a point where the raw water is not subject to recontamination by surface water runoff and a point downstream before or at the first customer, and

002.04A2 At least 99.99 percent (4-log) removal and/or inactivation of viruses between a point where the raw water is not subject to recontamination by surface water runoff and a point downstream before or at the first customer.

002.04B Each groundwater source providing water to a public system shall be examined to determine whether or not it is under the direct influence of surface water. The examination shall be performed in accordance with the protocol described in Attachment 3 to this rule.

002.04B1 Each groundwater source for a community water supply system shall have been examined no later than June 19, 1994.

002.04B2 Each groundwater source for a non-community system shall have been examined by June 19, 1999.

002.04B3 If the Director determines that a ground water source is under the direct influence of surface water, s/he shall so indicate in writing to the owner or the public water supply system.

002.04C A public water supply system using a surface water as a source must include filtration which meets the requirements of 002.04G.

002.04D A public water supply system using a ground water source under the direct influence of surface water is considered to be in compliance with the requirements of 002.04 if:

002.04D1 It meets the requirements for avoiding filtration in 002.04E and the disinfection requirements in 002.04F1; or

002.04D2 It meets the filtration requirements in 002.04G and the disinfection requirements in 002.04F2.

002.04E Criteria for avoiding filtration. A public water supply system that uses a ground water source under the direct influence of surface water must meet all of the conditions of 002.04E1 and 002.04E2 and is subject to 002.04E3 beginning 18 months after the Director determines that it is under the direct influence of surface water, or December 30, 1991, whichever is later. Within 18 months of the failure of a system using a groundwater source under the direct influence of surface water to meet any one of the requirements of 002.04E1 through 002.04E6 the system owner must have installed filtration and meet the criteria for filtered systems specified in 002.04G.

002.04E1 Source water quality conditions.

002.04E1a The fecal coliform concentration must be equal to or less than 20 per 100 ml or the total coliform concentration must be equal to or less than 100 per 100 ml (measured by the Nebraska Department of Health Laboratory or an approved laboratory pursuant to 002.03H) in representative samples of the source water immediately prior to the first or only point of disinfection application in at least 90 percent of the measurements made for the 6 previous months that the system served water to the public on an ongoing basis. If a system measures both fecal and total coliforms, the fecal coliform criterion, but not the total coliform criterion, in this paragraph must be met.

002.04E1b The turbidity level cannot exceed 5 NTU (measured as specified in 002.04H1a and 002.04H2b) in representative samples of the source water immediately prior to the first or only point of disinfectant application unless:

002.04E1b(1) The Director determines that any such event was caused by circumstances that were unusual and unpredictable; and

002.04E1b(2) As a result of any such event, there have not been more than two events in the past 12 months the system served water to the public, or more than five events in the past 120 months the system served water to the public, in which the turbidity level exceeded 5 NTU. An "event" is a series of consecutive days during which at least one turbidity measurement each day exceeds 5 NTU.

002.04E2 Site-specific conditions.

002.04E2a The public water supply system must meet the requirements of 002.04F1a at least 11 of the 12 previous months that the system served water to the public on an ongoing basis, unless the system fails to meet the requirements during 2 of the 12 previous months that the system served water to the public and the Director determines that at least one of these failures was caused by circumstances that were unusual and unpredictable.

002.04E2b The public water supply system must meet the requirements of 002.04F1b at all times the system serves water to the public.

002.04E2c The public water supply system must meet the requirements of 002.04F1c at all times the system serves water to the public unless the Director determines that any such failure was caused by circumstances that were unusual and unpredictable.

002.04E2d The public water supply system must meet the requirements of 002.04F1d on an ongoing basis unless the Director determines that failure to meet these requirements was not caused by a deficiency in treatment of the source water.

002.04E2e The public water supply system owner must develop and implement a wellhead protection program sufficient to exclude potential sources of microbiological contamination within a one year time-of-travel from the well or well field. A wellhead protection program shall consist of the following:

002.04E2e(1) A wellhead protection area delineation. The area delineated shall be based on a one year time-of-travel and shall be determined by the following formula:

$T = 3.14nbL^2$ divided by Q
where T = Time-of-Travel (days)
L = Distance from the well (feet)
n = Effective porosity (assumed to be 0.2 unless supporting evidence indicates otherwise.
b = Aquifer thickness (feet)
Q = Pumping rate (cubic feet per day)

002.04E2e(2) Identification of all sources of microbiological contamination within the delineated area.

002.04E2e(3) Management of all sources identified in 002.04E2e(2) to prevent contamination from the sources.

002.04E3 The public water supply must be subject to an annual on-site inspection to assess the wellhead protection program specified in 002.04E2e and the disinfection treatment process. Either the Director or a party approved by the Director must conduct the on-site inspection. The inspection must be conducted by competent individuals such as sanitary and civil engineers, sanitarians, or technicians who have experience and knowledge about the operation and maintenance of a public water system, and who have a sound understanding of public health principles and waterborne diseases. A report of the on-site inspection must be prepared every year and indicate to the Director's satisfaction that the disinfection treatment process is adequately designed and maintained. The on-site inspection must include:

002.04E3a A review of the effectiveness of the wellhead protection program required by 002.04E2e.

002.04E3b A review of the physical condition of the well and the extent of protection provided;

002.04E3c A review of the system's equipment maintenance program to ensure there is low probability for failure of the disinfection process;

002.04E3d An inspection of the disinfection equipment for physical deterioration;

002.04E3e A review of operating procedures;

002.04E3f A review of data records to ensure that all required tests are being conducted and recorded and disinfection is effectively practiced; and

002.04E3g Identification of any improvements which are needed in the equipment, system maintenance and operation or data collection.

002.04E4 The public water system must not have been identified as a source of a waterborne disease outbreak, or if it has been so identified, the system must have been modified sufficiently to prevent another such occurrence, as determined by the Director.

002.04E5 The public water supply system must comply with the maximum contaminant level (MCL), for total coliforms in 002.02D at least 11 months of the 12 previous months that the system served water to the public, on an ongoing basis, unless the Director determines that failure to meet this requirement was not caused by a deficiency in treatment of the source water.

002.04E6 The public water supply system must comply with the requirements for trihalomethanes in 002.02B3 and 002.03D.

002.04E7 A system that fails to meet any one of the criteria in 002.04E1-002.04E6 and/or for which filtration is required, and fails to install filtration by the date specified in the introductory paragraph of this subsection is in violation of a treatment technique requirement.

002.04E8 A system that has not installed filtration is in violation of a treatment technique requirement if:

002.04E8a The turbidity level (measured as specified in 002.04H1a) in a representative sample of the source water immediately prior to the first or only point of disinfection application exceeds 5 NTU; or

002.04E8b The system is identified as a source of a waterborne disease outbreak.

002.04F Disinfection. A public water supply system that uses a ground water source under the direct influence of surface water and does not provide filtration treatment must provide disinfection treatment specified in 002.04F1 beginning December 30, 1991, or 18 months after the Director determines that the ground water source is under the direct influence of surface water, whichever is later. If filtration is required in accordance with 002.04G, the system must comply with any interim disinfection requirements the Director deems necessary before filtration is installed. A system that uses a surface water source and provides filtration treatment must provide the disinfection treatment specified in 002.04F2 of this section beginning June 29, 1993, or beginning when filtration is installed, whichever is later. A system that uses a ground water source under the direct influence of surface water and provides filtration treatment must provide disinfection treatment as specified in 002.04F2 of this section by June 29, 1993, or beginning when filtration is installed, whichever is later. Failure to meet any requirement of this section after the applicable date specified in this introductory paragraph is a treatment technique violation.

002.04F1 Disinfection requirements for public water systems that do not provide filtration. Each public water supply system that uses a ground water source under the direct influence of surface water and does not provide filtration treatment must provide disinfection treatment as follows:

002.04F1a The disinfection treatment must be sufficient to ensure at least 99.9 percent (3-log) inactivation of Giardia lamblia cysts and 99.99 percent (4-log) inactivation of viruses, every day the system serves water to the public, except any one day each month. Each day a system serves water to the public, the public water system must calculate the CT value(s) from the system's treatment parameters, using the procedure specified in 002.04H2d and determine whether this value(s) is sufficient to achieve the specified inactivation rates for Giardia lamblia cysts and viruses. If a system uses a disinfectant other than chlorine, the system owner may demonstrate to the Director that CT(99.9) values other than those specified in Tables 2.1 and 3.1 in 002.04H2c or other operational parameters are adequate to demonstrate that the system is achieving minimum inactivation rates required by this section.

002.04F1b The disinfection system must have either redundant components, including an auxiliary power supply with automatic start-up and alarm to ensure that disinfectant application is maintained continuously while water is being delivered to the distribution system, or automatic shut-off of delivery of water to the distribution system whenever there is less than 0.2 mg per liter of residual disinfectant concentration in the water. If the Director determines that automatic shut-off would cause unreasonable risk to health or interference with fire protection, the system must have the above redundant components.

<u>002.04F1c</u> The residual disinfectant concentration in the water entering the distribution system, measured as specified in 002.04H1b and 002.04H2e cannot be less than 0.2 mg per liter for more than 4 hours.

<u>002.04F1d</u> Residual disinfectant concentration

<u>002.04F1d(1)</u> The residual disinfectant concentration in the distribution system, measured as total chlorine, combined chlorine, or chlorine dioxide as specified in 002.04H1b and 002.04H2f shall not be undetectable in more than 5 percent of the samples each month for any two consecutive months that the system serves water to the public. Water in the distribution system with a heterotrophic bacteria concentration less than or equal to 500 per ml, measured as heterotrophic plate count (HPC) by the Nebraska Department of Health Laboratory or an approved laboratory is deemed to have a detectable disinfectant residual for purposes of determining compliance with this requirement. Thus, the value "V" in the following formula cannot exceed 5 percent in one month for any two consecutive months.

$$V = \frac{c + d + e}{a + b} \times 100$$

Where:

a = number of instances where the residual disinfectant concentration is measured.

b = number of instances where the residual disinfectant concentration is not measured, but heterotrophic bacteria plate count (HPC) is measured;

c = number of instances where the residual disinfectant concentration is measured but not detected and no HPC is measured;

d = number of instances where the residual disinfectant concentration is measured but not detected and where the HPC is greater than 500 ml;

e = number of instances where the residual disinfectant concentration is not measured and HPC is greater than 500 per ml; and

V = percent of samples with undetectable residual disinfectant concentration.

002.04F1d(2) If the Director determines, based on site-specific considerations, that a system does not have the means for having a sample transported and analyzed for heterotropic plate count (HPC) by the State Health Department Laboratory or an approved laboratory under the requisite time and temperature conditions required of the laboratory and that the system is providing adequate disinfection in the distribution system, the requirements of 002.04F1d(1) do not apply to that system.

002.04F2 Disinfection requirements for public water systems which provide filtration. Each pubic water system that provides filtration treatment must provide disinfection treatment as follows:

002.04F2a The disinfection treatment must be sufficient to ensure that the total treatment processes of that system achieve at least 99.9 percent (3-log) inactivation and/or removal of Giardia lamblia cysts and at least 99.99 percent (4-log) inactivation and/or removal of viruses, as determined by the Director.

002.04F2b The residual disinfectant concentration in the water entering the distribution system, measured as specified in 002.04H1b and 002.04H3b cannot be less than 0.2 mg per liter for more than 4 hours.

002.04F2c The residual disinfectant concentration in the distribution system, measured as total chlorine, combined chlorine, or chlorine dioxide, as specified in 002.04H1b and 002.04H3b shall not be undetectable in more than 5 percent of the samples each month, for any two consecutive months that the system serves water to the public. Water in the distribution system with a heterotrophic bacteria concentration less than or equal to 500 per ml, measured as heterotrophic plate count

(HPC) as specified in 002.04H1 is deemed to have a detectable disinfectant residual for purposes of determining compliance with this requirement. Thus, the value "V" in the following formula cannot exceed 5 percent in one month, for any two consecutive months.

$$V = \frac{c + d + e}{a + b} \times 100$$

Where:

a = number of instances where the residual disinfectant concentration is measured;

b = number of instances where the residual disinfectant concentration is not measured but heterotrophic bacteria plate count (HPC) is measured;

c = number of instances where the residual disinfectant concentration is measured but not detected and no HPC is measured;

d = number of instances where no residual disinfectant concentration is detected and where the HPC is greater than 500 per ml;

e = number of instances where the residual disinfectant concentration is not measured and HPC is greater than 500 per ml; and

V = percent of samples with undetectable residual disinfectant concentration.

If the Director determines, based on site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by the State Health Department Laboratory or an approved laboratory under the requisite time and temperature conditions required of the laboratory and that the system is providing adequate disinfection in the distribution system, the requirements of 002.04F2c do not apply.

<u>002.04G</u> Filtration. A public water system that uses a surface water source must provide treatment consisting of both disinfection, as specified in 002.04F2 and filtration treatment which complies with the requirements of 002.04G1-002.04G4 by June 29, 1993. A public water system that uses a ground source which is under the direct influence of surface water and does not meet all the criteria in 002.04E for avoiding filtration, must provide treatment consisting of both

disinfection, as specified in 002.04F2 and filtration which complies with the requirements of 002.04G1-002.04G4 within 18 months of the failure to meet any one of the criteria for avoiding filtration in 002.04E. Failure to meet any requirement of this section after the date specified in this introductory paragraph is a treatment technique violation.

002.04G1 Conventional filtration treatment or direct filtration.

002.04G1a For systems using conventional filtration or direct filtration, the turbidity level of representative samples of a system's filtered water must be less than or equal to 0.5 NTU in at least 95 percent of the measurements taken each month, measured as specified in 002.04H1a and 002.04H3a,, except that if the Director determines that the system is capable of achieving at least 99.9 percent removal and/or inactivation of Giardia lamblia cysts at some turbidity level higher than 0.5 NTU in at least 95 percent of the measurements taken each month, the Director may substitute this higher turbidity limit for that system. However, in no case may the Director approve a turbidity limit that allows more than 1 NTU in more than 5 percent of the samples taken each month, measured as specified in 002.04H1a and 002.04H3a.

002.04G1b The turbidity level of representative samples of a system's filtered water must at no time exceed 5 NTU, measured as specified in 002.04H1a and 002.04H3a.

002.04G2 Slow sand filtration

002.04G2a For systems using slow sand filtration, the turbidity level of representative samples of a system's filtered water must be less than or equal to 1 NTU in at least 95 percent of the measurements taken each month, measured as specified in 002.04H1a and 002.04H3a, except that if the Director determines there is no significant interference with disinfection at a higher turbidity level, the Director may substitute this higher turbidity limit for that system.

002.04G2b The turbidity level of representative sample of a system's filtered water must at no time exceed 5 NTU, measured as specified in 002.04H1a and 002.04H3a.

002.04G3 Diatomaceous earth filtration.

002.04G3a For systems using diatomaceous earth filtration, the turbidity level of representative samples of system's filtered water must be less than or equal to 1 NTU in at least 95 percent of the measurements taken each month, measured as specified in 002.04H1a and 002.04H3a.

<u>002.04G3b</u> The turbidity level of representative samples of a system's filtered water must at no time exceed 5 NTU, measured as specified in 002.04H1a 002.04H3a.

<u>002.04G4</u> Other filtration technologies. A public water system may use a filtration technology not listed in 002.04G1 - 002.04G3 if it demonstrates to the Director using pilot plant studies or other means, that the alternative filtration technology, in combination with disinfection treatment that meets the requirements of 002.04F2, consistently achieves 99.9 percent removal and/or inactivation of Giardia lamblia cysts and 99.99 percent removal and/or inactivation of viruses. For a system that makes this demonstration, the requirements of 002.04G2 apply.

<u>002.04H</u> Analytical and monitoring requirements.

<u>002.04H1</u> Analytical requirements. Only the analytical method(s) specified in this subpart shall be used to demonstrate compliance with the requirements of 002.04E, 002.04F and 002.04G. Measurements for pH, temperature, turbidity, and residual disinfectant concentrations shall be conducted by an operator with a certification of competency issued by the Department. Measurements for total coliforms, fecal coliforms, and heterotropic plate count (HPC) shall be conducted by the Nebraska Department of Health Laboratory or an approved laboratory as required in 002.03H.

<u>002.04H1a</u> Turbidity shall be measured using Method 214A (Nephelometric Method -- Nephelometric Turbidity Units), pp. 134-136, as set forth in <u>Standard Methods for the Examination of Water and Wastewater</u>, 1985, American Public Health Association et al, 16th edition a copy of which is attached in Attachment 6 and incorporated by this reference.

<u>002.04H1b</u> Residual disinfectant concentrations for free chlorine and combined chlorine (chloramines) shall be measured by Method 408C (Amperometric Titration Method), pp. 303-306, Method 408D (DPD Ferrus Titrimetric Method), pp. 306-309, Method 408E (DPD Colorimetric Method), pp. 309-310, or Method 408F (Leuco Crystal Violet Method), pp. 310-313, as set forth in <u>Standard Methods for the Examination of Water and Wastewater</u>, 1985, American Public Health Association et al., 16th edition which are incorporated by this reference and are attached in Attachment 6. Residual disinfectant concentrations for free chlorine and combined chlorine may also be measured by using DPD colorimetric test kits. Residual disinfectant

concentrations for ozone shall be measured by the Indigo Method as set forth in Standard Methods for the Examination of Water and Wastewater, 1989, American Pubic Health Association et al., 17th edition, which is incorporated by this reference and is attached in Attachment 6 and incorporated by this reference or by automated methods which are calibrated in reference to the results obtained by the Indigo Method on a regular basis. Residual disinfectant concentrations for chlorine dioxide shall be measured by Method 410B (Amperometric Method) or Method 410C (DPD Method), pp. 322-324, as set forth in Standard Methods for the Examination of Water and Wastewater, 1985, American Public Health Association et al., 16th edition a copy of which is attached in Attachment 6 and incorporated by this reference.

002.04H1c Temperature shall be measured by Method 212 (Temperature), pp. 126-127, as set forth in Standard Methods for the examination of Water and Wastewater, 1985, American Public Health Association et al., 16th edition which is attached in Attachment 6 incorporated by this reference; and

002.04H1d pH shall be measured by Method 423 (pH Value) pp. 429-437, as set forth in Standard Methods for the Examination of Water and Wastewater, 1985, American Public Health Association et al., 16th edition a copy of which is attached in Attachment 6 and incorporated by this reference.

002.04H2 Monitoring requirements for systems that do not provide filtration. A public water supply system that uses a ground water source under the direct influence of surface water and does not provide filtration treatment must begin monitoring as specified in this subpart beginning December 31, 1990, or 6 months after the Director determines that the ground water source is under the direct influence of surface water, whichever is later, unless filtration is required, in which case the Director may specify alternative monitoring requirements, as appropriate, until filtration is in place.

002.04H2a Fecal coliform or total coliform density measurements as required by 002.04E1a must be performed on representative source water samples immediately prior to the first or only point of disinfectant application. The owner of the system must sample for fecal or total coliforms at the following minimum frequency each week the system serves water to the public:

System size (persons served)	Samples per week[1]
500 or less	1
501 to 3,300	2
3,301 to 10,000	3
10,001 to 25,000	4
Over 25,000	5

[1] Must be taken on separate days

Also, one fecal or total coliform density measurement must be made every day the system serves water to the public and the turbidity of the source water exceeds 1 NTU (these samples count toward the weekly coliform sampling requirement) unless the Director determines that the system, for logistical reasons outside the system's control, cannot have the sample analyzed within 30 hours of collection.

<u>002.04H2b</u> Turbidity measurements as required by 002.04E1b must be performed on representative grab samples of source water immediately prior to the first or only point of disinfectant application every four hours (or more frequently) that the system serves water to the public. A public water supply system owner may substitute continous turbidity monitoring for grab sample monitoring if it validates the continuous measurement for accuracy on a regular basis.

<u>002.04H2c</u> The total inactivation ratio for each day that the system is in operation must be determined based on the CT(99.9) values in Tables 1.1-1.6, 2.1 and 3.1 of this section. The parameters necessary to determine the total inactivation ratio must be monitored as follows:

<u>002.04H2c(1)</u> The temperature of the disinfected water must be measured at least once per day at each residual disinfectant concentration sampling point.

<u>002.04H2c(2)</u> If the system uses chlorine, the pH of the disinfected water must be measured at least once per day at each chlorine residual disinfectant concentration sampling point.

<u>002.04H2c(3)</u> The disinfectant contact time(s) ("T") must be determined for each day during peak hourly flow.

<u>002.04H2c(4)</u> The residual disinfectant concentration(s) ("C") of the water before or at the first customer must be measured each day during peak hourly flow.

<u>002.04H2c(5)</u> If a system uses a disinfectant other than chlorine, the system may demonstrate to the Director that CT(99.9) values other than those specified in Tables 2.1 and 3.1 in this section or other operational parameters are adequate to demonstrate that the system is achieving the minimum inactivation rates required by 002.04F1.

TABLE 1.1

CT VALUES [CT(99.9)] FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 0.5 ° C OR LOWER*.

Residual (mg/l)	pH						
	≤6.0	6.5	7.0	7.5	8.0	8.5	≤9.0
≤0.4	137	163	195	237	277	329	390
0.6	141	168	200	239	286	342	407
0.8	145	172	205	246	295	354	422
1.0	148	176	210	253	304	365	437
1.2	152	180	215	259	313	376	451
1.4	155	184	221	266	321	387	464
1.6	157	189	226	273	329	397	477
1.8	162	193	231	279	338	407	489
2.0	165	197	236	286	346	417	500
2.2	169	201	242	297	353	426	511
2.4	172	205	247	298	361	435	522
2.6	175	209	252	304	368	444	533
2.8	178	213	257	310	375	452	543
3.0	181	217	261	316	382	460	552

* These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the CT(99.9) value at the lower temperature and at the higher pH.

TABLE 1.2

CT VALUES [CT(99.9)] FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 5.0 ° C*.

	pH						
Residual (mg/l)	≤6.0	6.5	7.0	7.5	8.0	8.5	≤9.0
≤0.4	97	117	139	166	198	236	279
0.6	100	120	143	171	204	244	291
0.8	103	122	146	175	210	252	301
1.0	105	125	149	179	216	260	312
1.2	107	127	152	183	221	267	320
1.4	109	130	155	187	227	274	329
1.6	111	132	158	192	232	281	337
1.8	114	135	162	196	238	287	345
2.0	116	138	165	200	243	294	353
2.2	118	140	169	204	248	300	361
2.4	120	143	172	209	253	306	368
2.6	122	146	175	213	258	312	375
2.8	124	148	178	217	263	318	382
3.0	126	151	182	221	268	324	389

* These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the CT(99.9) value at the lower temperature and at the higher pH.

TABLE 1.3

CT VALUES [CT(99.9)] FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 10.0° C*.

				pH			
Residual (mg/l)	≤6.0	6.5	7.0	7.5	8.0	8.5	≤9.0
≤0.4	73	88	104	125	149	177	209
0.6	75	90	107	128	153	183	218
0.8	78	92	110	131	158	189	226
1.0	79	94	112	134	162	195	234
1.2	80	95	114	137	166	200	240
1.4	82	98	116	140	170	206	247
1.6	83	99	119	144	174	211	253
1.8	86	101	122	147	179	215	259
2.0	87	104	124	150	182	221	265
2.2	89	105	127	153	186	225	271
2.4	90	107	129	157	190	230	276
2.6	92	110	131	160	194	234	281
2.8	93	111	134	163	197	239	287
3.0	95	113	137	166	201	243	292

* These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the CT(99.9) value at the lower temperature and at the higher pH.

TABLE 1.4

CT VALUES [CT(99.9)] FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 15.0° C*.

Residual (mg/l)	pH ≤6.0	6.5	7.0	7.5	8.0	8.5	≤9.0
≤0.4	49	59	70	83	99	118	140
0.6	50	60	72	86	102	122	146
0.8	52	61	73	88	105	126	151
1.0	53	63	75	90	108	130	156
1.2	54	64	76	92	111	134	160
1.4	55	65	78	94	114	137	165
1.6	56	66	79	96	116	141	169
1.8	57	68	81	98	119	144	173
2.0	58	69	83	100	122	147	177
2.2	59	70	85	102	124	150	181
2.4	60	72	86	105	127	153	184
2.6	61	73	88	107	129	156	188
2.8	62	74	89	109	132	159	191
3.0	63	76	91	111	134	162	195

* These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the CT(99.9) value at the lower temperature and at the higher pH.

TABLE 1.5

CT VALUES [CT(99.9)] FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 20.0° C*.

Residual (mg/l)	pH						
	≤6.0	6.5	7.0	7.5	8.0	8.5	≤9.0
≤0.4	36	44	52	62	74	89	105
0.6	38	45	54	64	77	92	109
0.8	39	46	55	66	79	95	113
1.0	39	47	56	67	81	98	117
1.2	40	48	57	69	83	100	120
1.4	41	49	58	70	85	103	123
1.6	42	50	59	72	87	105	126
1.8	43	51	61	74	89	108	129
2.0	44	52	62	75	91	110	132
2.2	44	53	63	77	93	113	135
2.4	45	54	65	78	95	115	138
2.6	46	55	66	80	97	117	141
2.8	47	56	67	81	99	119	143
3.0	47	57	68	83	101	122	146

* These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the CT(99.9) value at the lower temperature and at the higher pH.

TABLE 1.6

CT VALUES [CT(99.9)] FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 25° C AND HIGHER*.

			pH				
Residual (mg/l)	≤6.0	6.5	7.0	7.5	8.0	8.5	≤9.0
≤0.4	24	29	35	42	50	59	70
0.6	25	30	36	43	51	61	73
0.8	26	31	37	44	53	63	75
1.0	26	31	37	45	54	65	78
1.2	27	32	38	46	55	67	80
1.4	27	33	39	47	57	69	82
1.6	28	33	40	48	58	70	84
1.8	29	34	41	49	60	72	86
2.0	29	35	41	50	61	74	88
2.2	30	35	42	51	62	75	90
2.4	30	36	43	52	63	77	92
2.6	31	37	44	53	65	78	94
2.8	31	37	45	54	66	80	96
3.0	32	38	46	55	67	81	97

* These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the CT(99.9) value at the lower temperature and at the higher pH.

TABLE 2.1

CT VALUES [CT(99.9)] FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY CHLORINE DIOXIDE AND OZONE*.

	Temperature					
	≤1°C	5°C	10°C	15°C	20°C	≥25°C
Chlorine dioxide	63	26	23	19	15	11
Ozone	2.9	1.9	1.4	0.95	0.72	0.48

* These CT values achieve greater than 99.99 percent inactivation of viruses. CT values between the indicated temperatures may be determined by linear interpolation. If no interpolation is used, use the CT(99.9) value at the lower temperature for determining CT(99.9) values between indicated temperatures.

TABLE 3.1

CT VALUES [CT(99.9)] FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY CHLORAMINES*.

	Temperature					
	≤1°C	5°C	10°C	15°C	20°C	25°C
Chloramine	3,800	2,200	1,850	1,500	1,100	750

*These values are for pH values of 6 to 9. These CT values may be assumed to achieve greater than 99.99 percent inactivation of viruses only if chlorine is added and mixed in the water prior to the addition of ammonia. If this condition is not met, the system must demonstrate, in accordance with 002.04F1a, that the system is achieving at least 99.99 percent inactivation of viruses. CT values between the indicated temperatures may be determined by linear interpolation. If no interpolation is used, use the CT (99.9) value at the lower temperature for determining CT(99.9) values between indicated temperatures.

002.04H2d The total inactivation ratio must be calculated as follows:

002.04H2d(1) If the system uses only one point of disinfectant application, the system owner may determine the total inactivation ratio based on either of the following two methods:

002.04H2d(1)(a) One inactivation ratio (CTcalc divided by CT(99.9)) is determined before or at the first customer during peak hourly flow and if CTcalc divided by CT(99.9) is greater than or equal to 1.0, the 99.9 percent Giardia lamblia inactivation requirement has been achieved; or

002.04H2d(1)(b) Successive CTcalc divided by CT(99.9) values, representing sequential inactivation ratios, are determined between the point of disinfectant application and a point before or at the first customer during peak hourly flow. Under this alternative, the following method shall be used to calculate the total inactivation ratio:

(1) Determine CTcalc divided by CT(99.9) for each sequence

(2) Add the CTcalc divided by CT(99.9) values together

(3) If the sum of CTcalc divided by CT(99.9) is greater than or equal to 1.0, the 99.9 percent Giardia lamblia inactivation requirement has been achieved.

002.04H2d(2) If the system used more than one point of disinfectant application before or at the first customer, the system owner shall determine the CT value of each disinfection sequence immediately prior to the next point of disinfectant application during peak hourly flow. The CTcalc divided by CT(99.9) value of each sequence and the sum of CTcalc divided by CT(99.9) shall be calculated using the method in 002.04H2d(1)(b) of this section to determine if the system is in compliance with 002.04F1.

002.04H2d(3) Although not required, the total percent inactivation for a system with one or more points or residual disinfectant concentration monitoring may be calculated by solving the following equation:

Percent inactivation = 100 - 100 divided by 10^Z
where: Z = 3 times the sum of
CTcalc divided by CT(99.9)

002.04H2e The residual disinfectant concentration of the water entering the distribution system shall be monitored continuously, and the lowest value must be recorded each day, except that if there is a failure in the continous monitoring equipment, grab sampling every 4 hours may be conducted in lieu of continuous monitoring, but for no more than 5 working days following the failure of the equipment. Owners of systems serving 3,300 or fewer persons may take grab samples in lieu of providing continuous monitoring on an ongoing basis at the frequencies prescribed below:

System size by population	Samples per day[1]
500 or less	1
501 to 1,000	2
1,001 to 2,500	3
2,501 to 3,300	4

[1] The day's samples cannot be taken at the same time. The sampling intervals are subject to review and approval by the Director.

If at any time the residual disinfectant concentration falls below 0.2 mg per liter in a system using grab sampling in lieu of continuous monitoring, the system must take a grab sample every 4 hours until the residual concentration is equal to or greater than 0.2 mg per liter.

002.04H2f Measurement of Residual Disinfectant.

002.04H2f(1) The residual disinfectant concentration shall be measured at least at the same points in the distribution system and at the same time as total coliforms are sampled, as specified in 002.03A2, except that the Director may allow a public water supply system which uses both a surface water source or a ground water source under direct influence of surface water, and a ground water source, to take disinfectant residual samples at points other than the total coliform sampling points if the Director determines that such points are more representative of treated (disinfected) water quality within the distribution system. Heterotrophic bacteria measured as heterotrophic plate count (HPC) by the Nebraska Department of Health Laboratory or an approved laboratory in accordance with 002.03H may be measured in lieu of residual disinfectant concentration.

002.04H2f(2) If the Director determines, based on site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by an approved laboratory under the requisite time and temperature conditions required of the approved laboratory and that the system is providing adequate disinfection in the distribution system, the requirements of 002.04H2f(1) of this section do not apply to that system.

002.04H3 Monitoring requirements for systems using filtration treatment. A public water supply system that uses a surface water source or a ground water source under the influence of surface water and provides filtration treatment must monitor in accordance with this section beginning June 29, 1993, or when filtration is installed, whichever is later.

002.04H3a Turbidity measurements as required by 002.04G shall be performed on representative samples of the system's filtered water every four hours (or more frequently) that the system serves water to the public. A public water supply system owner may substitute continuous turbidity monitoring for grab sample monitoring if the owner validates the continuous measurement for accuracy on a regular basis. For any systems using slow sand filtration or filtration treatment other than conventional treatment, direct filtration, or diatomaceous earth filtration, the Director may reduce the sampling frequency to once per day if the Director determined that less frequent monitoring is sufficient to indicate effective filtration performance. For systems serving 500 or fewer persons, the Director may reduce the turbidity sampling frequency to once per day, regardless of the type of filtration treatment used, if the Director determines that less frequent monitoring is sufficient to indicate effective filtration performance.

002.04H3b The residual disinfectant concentration of the water entering the distribution system shall be monitored continuously, and the lowest value must be recorded each day, except that if there is a failure in the continuous monitoring equipment, grab sampling every 4 hours may be conducted in lieu of continuous monitoring, but for no more than 5 working days following the failure of the equipment. Owners of systems serving 3,300 or fewer persons may take grab samples in lieu of providing continuous monitoring on an ongoing basis at the frequencies each day prescribed below:

System size by population	Samples per day[1]
500 or less	1
501 to 1,000	2
1,001 to 2,500	3
2,501 to 3,300	4

[1] The day's samples cannot be taken at the same time. The sampling intervals are subject to review and approval by the Director.

If at any time the residual disinfectant concentration falls below 0.2 mg per liter in a system using grab sampling in lieu of continuous monitoring, the systems must take a grab sample every 4 hours until the residual concentration is equal to or greater than 0.2 mg per liter.

<u>002.04H3c</u> Measurement of Residual Disinfectant. The residual disinfectant concentration must be measured at least at the same point in the distribution system and at the same time as total coliforms are sampled, as specified in 002.03A2 except that the director may allow a public water supply system which uses both a surface water source or a ground water source under direct influence of surface water, and a ground water source to take disinfectant residual samples at points other than the total coliform sampling points if the Director determines that such points are more representative of treated (disinfected) water quality within the distribution system. Heterotrophic bacteria, measured as heterotrophic plate count (HPC) as specified in 002.04H1 may be measured in lieu of residual disinfectant concentration.

003 LEAD BAN (INCLUDES AMENDMENTS MADE THROUGH SEPTEMBER 15, 1991)

003.01 Solder and Pipes. After July 1, 1988, any pipe, pipe fitting, solder, or flux which is used in the installation or repair of any public water supply system shall be lead free.

003.02 Adoption of Code(s); Ordinances. By July 1, 1988, the owner of any public water supply system shall, by the adoption of plumbing codes or ordinances, contract, or other enforceable means, require that any pipe, pipe filling, solder, or flux used in the installation or repair of any residential or nonresidential facility which is connected to the public water supply system be lead free.

003.02A The owner of any public water supply system shall inspect the installation or repair of facilities as described in 003.02 constructed or repaired after July 1, 1988. to determine compliance with this subsection.

003.02B The owner of any public water supply system shall cause any joint or pipe in facilities as described in 003.02 constructed or repaired after July 1, 1988, to be replaced if the owner or the Director finds that such joint or pipe in not lead free.

003.03 Application. This section shall not apply to the repair of leaded joints in cast iron pipes in any public water supply systems that are in existence and use on July 1, 1988.

003.04 Evidence Required. The owner of each public water supply system shall, by January 1, 1989, submit to the Director a copy of the plumbing code, ordinances, contracts other enforceable means required in 003.02.

004 PUBLIC NOTIFICATION AS CONDITION OF OPERATING A PUBLIC WATER SUPPLY SYSTEM (INCLUDES AMENDMENTS MADE THROUGH SEPTEMBER 15, 1991)

004.01 When Required. If a community water supply system fails to comply with an applicable maximum contaminant level or fails to submit any sample required by section 002 of these rules; is granted a variance or exemption from an applicable maximum contaminant level as provided in section 006 of these rules; fails to comply with the requirements of any schedule prescribed pursuant to a variance or exemption; or otherwise violates any provisions of the Nebraska Safe Drinking Water Act (NEB REV STAT §71-5301, et seq), the supplier of water shall notify persons served by the system of the failure, grant or violation.

004.02 Maximum Contaminant Level (MCL), Treatment Technique, and Variance and Exemption Schedule Violations. The owner or operator of a public water supply system which fails to comply with an applicable MCL or treatment technique established by these regulations or which fails to comply with the requirements of any schedule prescribed pursuant to a variance or exemption, shall notify persons served by the system as follows:

004.02A Except as provided in 004.02C, the owner or operator of a public water supply system must give notice:

004.02A1 By publication in a daily newspaper of general circulation in the area served by the system as soon as possible, but in no case later then 14 days after the violation or failure. If the area served by a public water supply system is not served by a daily newspaper of general circulation, notice shall instead be given by publication in a weekly newspaper of general circulation serving the area; and

004.02A2 By mail delivery (by direct mail or with the water bill) or by hand delivery, not later than 45 days after the violation or failure. The Director may waive mail or hand delivery if he or she determined that the owner or operator of the public water supply system in violation has corrected the violation or failure within the 45-day period. The Director must make the waiver in writing and within the 45-day period; and

004.02A3 For violations of the MCLs of contaminants that may pose an acute risk to human health, by furnishing a copy of the notice to the radio and television stations serving the area served by the public water system as soon as possible but in no case later than 72 hours after the violation. The following violations are acute violations:

004.02A3a Any violations specified by the Director as posing an acute risk to human health.

004.02A3b Violation of the MCL for nitrate as defined in 002.02A and determined according to 002.03C3.

004.02A3c Violation of the MCL for total coliforms, when fecal coliforms or E. coli are present in the water distribution system as specified in 002.02D2.

004.02A3d Occurrence of a waterborne disease outbreak in an unfiltered system subject to the requirements of section 002.04 after December 30, 1991, or the effective date of 002.04 and 004.02A3c and 004.02A3d of these regulations, if later.

004.02B Except as provided in 004.02C, following the initial notice given under 004.02A, the owner or operator of the pubic water supply system must give notice at least once every three months by mail delivery (by direct mail or with the water bill) or by hand delivery, for as long as the violation or failure exists.

004.02C Exceptions.

004.02C1 In lieu of the requirements of 004.02A and 004.02B, the owner or operator of a community water supply system in an area that is not served by a daily or weekly newspaper or general circulation must give notice by hand delivery or by continuous posting in conspicuous places within the area served by the system. Notice by hand delivery or posting must begin as soon as possible, but no later than 72 hours after the violation or failure for acute violations or 14 days after the violation or failure of any other violation or failure specified in 004.02. Posting must continue for as long as the violation or failure exists. Notice by hand delivery must be repeated at least every three months for as long as the violation or failure exists.

004.02C2 In lieu of the requirements of 004.02A and 004.02B, the owner or operator of a non-community water supply system may give notice by hand delivery or by the continuous posting in conspicuous places within the area served by the system. Notice by hand delivery or posting must begin as soon as possible, but in no case later than 72 hours after the violation or failure for acute violations or 14 days after the violation or failure for any other violation or failure specified in 004.02.

004.03 Other Violations, Variances, Exemptions. The owner or operator of a public water supply system which fails to perform monitoring required by section 002.03 of these rules, fails to comply with a testing procedure established by this part, is subject to a variance or is subject to an exemption shall notify persons served by the system as follows:

004.03A Except as provided in 004.03C or 004.03D, the owner or operator of a public water supply system must give notice within three months of the violation or granting of a variance or exemption by publication in a daily newspaper of general circulation in the area served by the system. If the area served by a public water supply system is not served by a daily newspaper of general circulation, notice shall instead be given by publication in a weekly newspaper of general circulation serving the area.

<u>004.03B</u> Except as provided in 004.03C or 004.03D, following the initial notice given under 004.03A, the owner or operator of the pubic water supply system must give notice at least once every three months by mail delivery (by direct mail or with the water bill) or by hand delivery, for as long as the violation exists. Repeat notice of the existence of a variance or exemption must be given every three months for as long as the variance or exemption remains in effect.

<u>004.03C</u> In lieu of the requirements of 004.03A and 004.03B of this section, the owner or operator of a community water supply system in the area that is not served by a daily or weekly newspaper of general circulation must give notice within three months of the violation or granting of the variance or exemption by hand delivery or by continuous posting in conspicuous places within the area served by the system. Posting must continue for as long as the violation exists or a variance or exemption remains in effect. Notice by hand delivery must be repeated at least every three months for as long as the violation exists or a variance or exemption remains in effect.

<u>004.03C1</u> In lieu of the requirements of 004.03A and 004.03B of this section, the owner or operator of a non-community water supply system may give notice, within three months of the violation or the granting of the variance or exemption, by hand delivery or by continuous posting in conspicuous places within the area served by the system. Posting must continue for as long as the violation exists, or a variance or exemption remains in effect. Notice by hand delivery must be repeated at least every three months for as long as the violation exists or a variance or exemption remains in effect.

<u>004.03D</u> In lieu of the requirements of 004.03A, 004.03B and 004.03C of this section, the owner or operator of a public water supply system, at the discretion of the Director may provide less frequent notice for minor monitoring violations as defined by the Director, if EPA has approved the Director's application for a program revision. Notice of such violation must be given no less frequently than annually.

<u>004.04 Notice to new billing units.</u> The owner or operator of a community water supply system must give a copy of the most recent pubic notice for any outstanding violation of any maximum contaminant level, or any treatment technique requirement, or any variance or exemption schedule to all new billing units or new hookups prior to or at the time service begins.

<u>004.05 General content of public notice.</u> Each notice required by this section must provide a clear and readily understandable explanation of the violation, any potential adverse health effects, the population at risk, the steps that the public water system is taking to correct such violation, the necessity for seeking alternative water supplies, if any, and any preventative measures the consumer should take until the violation is corrected. Each notice shall be conspicuous and shall not contain unduly technical language, unduly small print, or similar problem that frustrate the purpose of the notice. Each notice shall

include the telephone number of the owner, operator, or designee of the public water supply system as a source of additional information concerning the notice. Where appropriate, the notice shall be multilingual.

004.06 Mandatory health effects language. When providing the information on potential adverse health effects required by 004.05 of this section in notices of violation of maximum contaminant levels or treatment technique requirements, or notices of the granting or the continued existence of exemptions or variances, or notices of failure to comply with a variance or exemption schedule, the owner or operator of a public water supply system shall include the language specified below for each contaminant. (If language for a particular contaminant is not specified below at the time notice is required, this subsection does not apply.)

004.06A Trichloroethylene. "The Director of the Nebraska Department of Health (NDOH) sets drinking water standards for the State of Nebraska and has determined, based upon information provided by the U.S. Environmental Protection Agency (EPA) and other scientific and medical information that trichloroethylene is a health concern at certain levels of exposure. This chemical is a common metal cleaning and dry cleaning fluid. It generally gets into drinking water by improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. NDOH has set forth the enforceable drinking water standard for trichloroethylene at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animal. Drinking Water which meets this standard is associated with little to none of this risk and should be considered safe."

004.06B Carbon tetrachloride. "The Nebraska Department of Health (NDOH) sets drinking water standards and has determined, based on information provided by the U.S. Environmental Protection Agency and other scientific and medical information that carbon tetrachloride is a health concern at certain levels of exposure. This chemical was once a popular household cleaning fluid. If generally gets into drinking water by improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. NDOH has set the enforceable drinking water standard for carbon tetrachloride at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe."

004.06C 1,2-Dichloroethane. "The Director of the Nebraska Department of Health (NDOH) sets drinking water standards and has determined, based on information provided by the U.S. Environmental Protection Agency and other scientific and medical information that 1,2-Dichloroethane is a health concern at certain levels of exposure. This chemical is used as a cleaning fluid for fats, oils, waxes, and resins. It generally gets into drinking water from improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. NDOH has set the enforceable drinking water standard for 1,2-Dichloroethane at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe."

004.06D Vinyl chloride. "The Nebraska Department of Health (NDOH) sets drinking water standards and has determined, based on information provided by the U.S. Environmental Protection Agency and other scientific and medical information that vinyl chloride is a health concern at certain levels of exposure. This chemical is used in industry and is found in drinking water as a result of the breakdown of related solvents. The solvents are used as cleaners and degreasers of metals and generally get into by improper waste disposal. This chemical has been associated with significantly increased risks of cancer among certain industrial workers who were exposed to relatively large amounts of this chemical during their working careers. This chemical has also been shown to cause cancer in laboratory animals when the animals are exposed at high levels over their lifetimes. Chemicals that cause increased risk of cancer among exposed industrial workers and in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. NDOH has set the enforceable drinking water standard for vinyl chloride and 0.002 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in humans and laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe."

004.06E Benzene. "The Nebraska Department of Health (NDOH) sets drinking water standards and has determined, based on information provided by the U.S. Environmental Protection Agency and other scientific and medical information that benzene is a health concern at certain levels of exposure. This chemical is used as a solvent and degreaser of metals. It is also a major component of gasoline. Drinking water contamination generally results from leaking underground gasoline and petroleum tanks or improper waste disposal. This chemical has been associated with significantly increased risks of leukemia among ceratin industrial workers who were exposed to relatively large amounts of this chemical during their working careers. This chemical has also been shown to cause cancer in laboratory animals when these animals are exposed at high levels over their lifetimes. Chemicals that cause increased risk of cancer among exposed industrial workers and in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. NDOH has set the enforceable drinking water standard for benzene at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in humans and laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe."

004.06F 1,1-Dichloroethylene. "The Nebraska Department of Health (NDOH) sets drinking water standard and has determined, based on information provided by the U.S. Environmental Protection Agency and other scientific and medical information that 1,1-Dichloroethylene is a health concern at ceratin levels of exposure. This chemical is used in industry and is found in drinking water as a result of the breakdown or related solvents. The solvents are used as cleaners and degreasers of metals and generally get into drinking water by improper waste disposal. This chemical has been shown to cause liver and kidney damager in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals which cause adverse effects in humans who are exposed at lower levels over long periods of time. NDOH has set the enforceable drinking water standard for 1,1-Dichloroethylene at 0.007 parts per million (ppm) to reduce the risk of these adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe."

004.06G Para-dichlorobenzene. "The Nebraska Department of Health (NDOH) sets drinking water standard and has determined, based on information provided by the U.S. Environmental Protection Agency and other scientific and medical information that para-dichlorobenzene in a health concern at ceratin levels of exposure. This chemical is a component of deodorizers, moth balls, and pesticides. It generally gets into drinking water by improper waste disposal. This chemical has been shown to cause liver and kidney damage in laboratory animals such as rats and mice when the animals are exposed to high levels over their lifetimes. Chemicals which cause adverse effects in laboratory animals also may cause adverse health effects in humans who are exposed at lower levels over long periods of time. NDOH has set the enforceable drinking water standard for para-dichlorobenzene at0.075 parts per million (ppm) to reduce the risk of these adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe."

004.06H 1,1,1-Trichloroethane. "The Nebraska Department of Health (NDOH) sets drinking water standards and has determined, based on information provided by the U.S. Environmental Protection Agency and other scientific and medical information that 1,1,1-Trichloroethane is a health concern at ceratin levels of exposure. This chemical is used as a cleaner and degreaser of metals. It generally gets into drinking water by improper waste disposal. This chemical has been shown to cause liver, nervous system and circulatory system of laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Some industrial workers who were exposed to relatively large amounts of this chemical during their working careers also suffered damage to the liver, nervous system and circulatory system. Chemicals which cause adverse effects among exposed in humans who are exposed at lower levels over long periods of time. NDOH has set the enforceable drinking water standard for 1,1,1-Trichloroethane at 0.2 parts per million (ppm) to reduce the risk of these adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe."

004.06I Coliforms.

004.06I1 Total Coliforms (to be used when there is a violation of 002.02D1 and not a violation of 002.02D2). "The Nebraska Department of Health (NDOH) sets drinking water standards and has determined, based on information provided by the Environmental Protection Agency and other scientific and medical information, that the presence of total coliforms is a possible health concern. Total coliforms are common in the environment and are generally not harmful in themselves. The presence of these bacteria in drinking water, however, generally is a result of a problem with water treatment or the pipes which distribute the water, and indicates the water may be contaminated with organisms that can cause disease. Disease symptoms may include diarrhea, cramps, nausea, and possible jaundice, and any associated headaches and fatigue. These symptoms, however, are not just associated with disease-causing organisms in drinking water, but also may be caused by a number of factors other than your drinking water. NDOH has set an enforceable drinking water standard for total coliforms to reduce the risk of these adverse health effects. Under this standard, not more than 5.0 percent of the samples collected during a month can contain these bacteria, except that systems collecting fewer than 40 samples per month that have one total coliform-positive sample per month are not violating the standard. Drinking water which meets this standard is usually not associated with a health risk from disease-causing bacteria and should be considered safe."

004.06I2 Fecal coliforms/E. coli (to be used when there is a violation of 002.02D2 or both 002.02D1 and 002.02D2). The Nebraska Department of Health (NDOH) sets drinking water standards and has determined, based on information provided by the U.S. Environmental Protection Agency and other scientific and medical information, that the presence of fecal coliforms or E. coli is a serious health concern. Fecal coliforms and E. coli are generally not harmful in themselves, but their presence in drinking water is serious because they usually are associated with sewage or animal wastes. The presence of these bacteria in drinking water is generally a result of a problem with water treatment or the pipes which distribute the water, and indicates that the water may be contaminated with organisms that can cause disease. Disease symptoms may include diarrhea, cramps, nausea, and possibly jaundice, and associated headaches and fatigue. These symptoms, however, are not just associated with disease-causing organisms in drinking water, but also may be caused by a number of factors other than your drinking water. NDOH has set an enforceable drinking water standard for fecal coliforms and E. coli to reduce the risk of these adverse health effects. Under this standard, all drinking water samples must be free of these bacteria. Drinking water which meets this standard is associated with little or none of this risk and should be considered safe. State and local health authorities recommend that consumers take the following precautions: [To be inserted by the public water system according to instructions from the Director or local authorities]."

004.06J Microbiological contaminants (for use when there is a violation of the treatment technique requirements for filtration and disinfection in 002.04). "The Nebraska Department of Health (NDOH) sets drinking water standards and has determined that the presence of microbiological contaminants are a health concern at certain levels of exposure. If water is inadequately treated, microbiological contaminants in that water may cause disease. Disease symptoms may include diarrhea, cramps, nausea, and possibly jaundice, and any associated headaches and fatigue. These symptoms, however, are not just associated with disease-causing organisms in drinking water, but also may be caused by a number of factors other than your drinking water. NDOH has set enforceable requirements for treating drinking water to reduce the risk of these adverse health effects. Treatment such as filtering and disinfecting the water removes or destroys microbiological contaminants. Drinking water which is treated to meet NDOH requirements is associated with little to none of this risk and should be considered safe."

004.07 Public notices for fluoride. Notice of violations of the maximum contaminant level for fluoride, notices of variances and exemptions from the maximum contaminant level for fluoride, and notices of failure to comply with variance and exemption schedules for the maximum contaminant level for fluoride shall consist of the public notice prescribed in 004.02A and 004.02B of this section, plus a description of any steps which the system is taking to come into compliance.

004.07A Compliance with the secondary maximum contaminant level (2.0 mg per liter) for fluoride shall be calculated base on the last single sample taken in accordance with the requirements of 002.03C of this rule.

004.07B Using a notice as described in 004.07C of this section, public water systems which exceed the secondary MCL for fluoride shall notify:

004.07B1 all billing units annually;

004.07B2 all dentists and doctors listed as such in the telephone directories covering the areas served by the public water supply system, local officials, and local newspapers annually; and

004.07B3 all new billing units at the time service begins.

004.07C The notice to be used by systems which exceed the secondary MCL shall read as follows:

"Dear User:

Many public water supply systems are required by federal regulation to routinely analyze for fluoride levels in the drinking water they provide to consumers. Analyses of the drinking water in your community has found a level of * mg per liter of fluoride. The Director of the Nebraska Department of Health (NDOH), based upon information provided by the U.S. Environmental Protection Agency and other scientific and medical information has set a standard at 2.0 mg per liter to minimize the occurrence of objectionable dental fluorosis. That guideline was established to protect public welfare, but it is not federally enforceable. NDOH requires that this notice of monitoring results be provided to you.

Fluoride, at the appropriate levels in the drinking water of children up to the age of nine, reduces cavities. However, children exposed to levels of fluoride greater than 1.0 to 2.0 mg per liter may develop dental fluorosis in their permanent teeth. Dental fluorosis, in its moderate and severe forms, is a discoloration (brown staining) and pitting of teeth.

Because fluoride affects only developing teeth, households without children would not be expected to be affected by this level of fluoride. Individuals with children under the age of nine are encouraged to seek other sources of drinking water for their children.

The NDOH Maximum Contaminant Level (MCL) for fluoride is 4.0 mg per liter. That standard is based upon protection from crippling skeletal fluorosis, which may result from levels of 4.0 mg per liter or more. The MCL is an enforceable standard and has been set to protect pubic health. Your water supplier can lower the concentration of fluoride to the level where beneficial effects still occur (cavity prevention), and where the occurrence of dental fluorosis in minimal. Technology for the removal of fluoride is currently available, but the use of this technology by your utility may increase your monthly water bill. Treatment systems for the removal of fluoride from drinking water are also available for home use. Information of such systems is available at the address given below. Low fluoride bottled drinking water that would meet all standards is also available.

For further information, contact ** at your water utility."

* PWS shall insert the compliance result which triggered notification under this part.

** PWS shall insert the name, address and telephone number of a contact person at the PWS.

004.08 Public notification by the Director. The Director may give notice to the public required by this section on behalf of the owner or operator of the public water supply system. However, the owner or operator of the public water supply system remains legally responsible for ensuring that the requirements of this section are met.

004.09 Public notice requirements pertaining to lead.

004.09A Applicability of public notice requirement.

004.09A1 Except as provided in 004.09A2 of this section, within 30 days after the effective date of these regulations, the owner or operator of each community water supply system and each non-transient, non-community water supply system shall issue notice to persons served by the system that may be affected by lead contamination of their drinking water. The Director may require subsequent notices. The owner of operator shall provide notice under this section even if there is not violation of the national primary drinking water regulation for lead.

004.09A2 Notice under 004.09A1 of this section is not required if the system demonstrates to the Director that the water supply system, including the residential and non-residential portions connected to the water supply system, are lead free, as defined in 001.01Z.

004.09B Manner of notice. Notice shall be given to persons served by the system either by (1) three newspaper notices (one for each of three consecutive months and the first no later than 30 days after the effective date of there regulations); (2) once by mail notice with the water bill or in a separate mailing 30 days after the effective date of these regulations; or (3) once by hand delivery 30 days after the effective dater of these regulations. For non-transient, non-community water supply systems, notice may be given by continuous posting. If posting is used, the notice shall be posted in a conspicuous place in the area served by the system and start no later than 30 days after the effective date of these regulations, and continue for three months.

004.09C General content of notice.

004.09C1 Notices issued under this section shall provide a clear and readily understandable explanation of the potential sources of lead in drinking water, potential adverse health effects, reasonably available methods of mitigating known or potential lead content in drinking water, any steps the water system is taking to mitigate lead content in drinking water, and the necessity for seeking alternative water supplies, if any. Use of the mandatory language on 004.09D of this section in the notice will be sufficient to explain potential adverse health effects.

004.09C2 Each notice shall also include specific advise on how to determine if materials containing lead have been used in homes or the water distribution system and how to minimize exposure to water likely to contain high levels of lead. Each notice shall be conspicuous and shall not contain unduly technical language, unduly small printing, or similar problems that frustrate the purpose of the notice. Each notice shall contain the telephone number of the owner. operator, or designee of the pubic water supply system as a source of additional information regarding the notice. Where appropriate, the notice shall be multilingual.

(NOTE: Optional Information): Each notice should advise persons served by the system to use only the cold water faucet from drinking and for use in cooking or preparing baby formula, and to run the water until it gets as cold as it is going to get before each use. If there has recently been major water use in the household, such as showering or bathing, flushing toilets, or doing laundry with cold water, flushing the pipes should take 5 to 30 seconds; if not, flushing the pipes could take as long as several minutes. Each notice should also advise persons served by the system to check to see if lead pipes, solder, or flux have been used in plumbing that provides tap water and to ensure that new plumbing and plumbing repairs use lead-free materials.

The only way to be sure of the amount of lead in the household water is to have the water tested by a competent laboratory. Testing is especially important to apartment dwellers because flushing may not be effective in high-rise buildings that have lead-soldered central piping. As appropriate, the notice should provide information on testing).

004.09D Mandatory health effects information. When providing the information in public notices required under 004.09C of this section on the potential adverse health effects of lead in drinking water, the owner or operator of the water supply system shall include the following specific language in the notice:

"The Nebraska Department of Health (NDOH) sets drinking water standards and has determined that lead is a health concern at certain levels of exposure. There is currently a standard of 0.050 parts per million (ppm).

Based on new health information, NDOH is likely to lower this standard significantly.

Part of the purpose of this notice is to inform you of the potential adverse health effects of lead. This is being done even though your water may not be in violation of the current standard.

NDOH and others are concerned about lead in drinking water. Too much lead in the human body can cause serious damage to the brain, kidneys, nervous system, and red blood cells. The greatest risk, even with short-term exposure, is to young children and pregnant women.

Lead levels in your drinking water are likely to be highest:

- If your home or water system has lead pipes, or
- If your home has copper pipes with lead solder, and
- If the home is less than five years old, or
- If you have soft or acidic water, or
- If water sits in the pipes for several hours.

004.09E Notice by the Director. The Director may give notice to the public required by this section on behalf of the owner or operator of the water supply system if the Director meets the requirements of 004.09B and the notice contains all the information specified in 004.07C and 004.07D of this section. However, the owner or operator or the water supply system remains legally responsible for ensuring that the requirements of this section are met.

004.10 Reporting to the Director. The owner of any public water supply system required to give notice under section 004 shall submit a copy of such notice and evidence of publication of the notice to the Director in accordance with 005.01E.

005 REPORTING AND RECORDKEEPING (INCLUDES AMENDMENTS MADE THROUGH SEPTEMBER 15, 1991)

005.01 Required reports.

005.01A The owner of a public water supply shall report to the Director within the time prescribed on each of the following occurrences which influence or alter the reliability of the system of the safety of drinking water delivered to the consumer:

005.01A1 Any disruption in service that results in a likelihood for the entrance of contaminants into the system, including reduced pressure due to fire fighting operations or breakdown should be recorded by the owner of the system. Any loss of pressure of disruption of service for reasons beyond normal repair or replacement shall be reported to the Director when more than 10% of the consumers are affected.

005.01A2 Any proposed change in source of supply or treatment processes, or any expansion of storage or to the distribution service area shall be reported to the Director before entering into any financial commitment for implementing a change.

005.01A3 All reports, studies, and investigations pertinent to the operation and management of a public water supply system shall be made available to the Director on request.

005.01A4 Any change in personnel, elected or appointed having primary responsibility for the operation of a public water supply system, shall be promptly reported to the Director by the owner or a representative of the owner.

005.01A5 Any change in ownership of a public water supply system shall be reported to the Director by the prior owner, and the prior owner shall be responsible for informing the new owner of the general provisions of the Nebraska Safe Drinking Water Act and the regulations implementing it.

005.01B Except where a shorter period is specified in this chapter, the owner of a public water system shall report to the Director the results of any test measurement or analysis required by this chapter within (1) the first ten days following the month in which the result is received, or (2) the first ten days following the end of the required monitoring period as stipulated by the Director, which ever of these is shortest.

005.01C Except where a different reporting period is specified in this chapter, the owner of a public water supply system must report to the Director within 48 hours the failure to comply with any regulation (including failure to comply with monitoring requirements) set forth in this chapter.

005.01D The owner of a public water supply system is not required to report analytical results to the Director in cases where the Department of Health Laboratory performs all of the analysis and reports all of the results.

005.01E Within ten days of completion of each pubic notification required pursuant to 004, the owner of a public water supply system shall submit to the Director a representative copy of each type of notice distributed, published, posted and/or made available to the persons served by the system and/or to the media.

005.01F The owner of a public water supply system shall submit to the Director within the time stated in the request copies of any record required to be maintained under this chapter.

005.02 Reporting and Recordkeeping Requirements, Filtration and Disinfection.

005.02A A public water system that uses a ground water source under the direct influence of surface water and does not provide filtration treatment must report monthly to the Director the information specified in this part beginning 6 months after the Director determines that the ground water source is under the direct influence of surface water, unless filtration is required, in which case the Director may specify alternative reporting requirements, as appropriate, until filtration is in place. The reports shall be submitted on Forms PWS 403, PWS 404 and PWS 405 in Attachment 4 to this Rule.

005.02A1 Source water quality information must be reported to the Director within 10 days after the end of each month the system serves water to the public. Information that must be reported includes:

005.02A1a The cumulative number of months for which result are reported.

005.02A1b The number of fecal and/or total coliform samples, whichever are analyzed during the month (if a system monitors for both, only fecal coliforms must be reported), the dates of sample collection, and the dates when the turbidity level exceeded 1 NTU.

005.02A1c The number of samples during the month that had equal to or less than 20 per 100 ml fecal coliforms and/or equal to or less than 100 per 100 ml total coliforms.

005.02A1d The cumulative number of fecal or total coliform samples, whichever are analyzed, during the previous six months the system served water to the public.

005.02A1e The cumulative number of samples that had equal to or less than 20 per 100 ml of fecal coliforms or equal to or less than 100 per 100 ml total coliforms, whichever are analyzed, during the previous six months the system served water to the public.

005.02A1f The percentage of samples that had equal to or less than 20 per 100 ml fecal coliforms or equal to or less than 100 per 100 ml total coliforms, whichever are analyzed, during the previous six months the system served water to the public.

005.02A1g The maximum turbidity level measured during the month, the date(s) of occurrence for any measurement(s) which exceeded 5 NTU, and the date(s) the occurrences was reported to the Director.

005.02A1h For the first 12 months of recordkeeping, the dates and cumulative number of events during which turbidity exceeded 5 NTU, and after one year of recordkeeping for turbidity measurement, the dates and cumulative number of events during which the turbidity exceeded 5 NTU in the previous 12 months the system served water to the public.

005.02A1i For the first 120 months of recordkeeping, the dates and cumulative number of events during which the turbidity exceeded 5 NTU, and after 10 years of recordkeeping for turbidity measurements, the dates and cumulative number of events during which the turbidity exceeded 5 NTU in the previous 120 months the system served water to the public.

005.02A2 Disinfection information specified on 002.04H2 shall be reported to the Director within 10 days after the end of each month the system serves water to the public. Information that must be reported includes:

005.02A2a For each day, the lowest measurement of residual disinfectant concentration in mg per liter in water entering the distribution system.

005.02A2b The date and duration of each period when the residual disinfectant concentration in water entering the distribution system fell below 0.2 mg per liter and when the Director was notified of the occurrence.

005.02A2c The daily residual disinfectant concentration(s) in mg per liter and disinfectant contact time(s) in minutes used for calculating the CT value(s).

005.02A2d If chlorine is used, the daily measurement(s) of pH of disinfected water following each point of chlorine disinfection.

005.02A2e The daily measurement(s) of water temperature in °C following each point of chlorine disinfection.

005.02A2f The daily CTcalc and CTcalc divided by CT(99.9) values for each disinfectant measurement or sequence and the sum of all CTcalc divided by CT(99.9) values before or at the first customer.

005.02A2g The daily determination of whether disinfection achieves adequate Giardia cyst and virus inactivation, i.e., whether CTcalc divided by CT(99.9) is at least 1.0 or, where disinfectants other than chlorine are used, other indicator conditions that the Director determines are appropriate, are met.

005.02A2h The following information on the samples taken in the distribution system in conjunction with total coliform monitoring pursuant to 002.04F.

005.02A2h(1) Number of instances where the residual disinfectant concentration is measured;

005.02A2h(2) Number of instances where the residual disinfectant concentration is not measured but heterotrophic bacteria plate count (HPC) is measured;

005.02A2h(3) Number of instances where the residual disinfectant concentration is measured but not detected and no HPC is measured;

005.02A2h(4) Number of instances where the residual disinfectant concentration is detected and where HPC is greater than 500 per ml;

005.02A2h(5) Number of instances where the residual disinfectant concentration is not measured and HPC is greater then 500 per ml;

005.02A2h(6) For the current and previous month the system served water to the pubic, the value of "V" in the following formula:

$$V = \frac{c + d + e}{a + b} \times 100$$

Where:

a = the value in 005.02A2h(1)

b = the value in 005.02A2h(2)

c = the value in 005.02A2h(3)

d = the value in 005.02A2h(4)

e = the value in 005.02A2h(5)

V = percent of samples with undetectable residual disinfectant concentration

005.02A2h(7) If the Director determines, based in site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by the State Health Department Laboratory or an approved laboratory under the requisite time and temperature conditions required of the laboratory and that the system is providing adequate disinfection in the distribution system, the requirements of 005.02F2h do not apply to that system.

005.02A2i A system need not report the data listed in 005.02A2a and 005.02A2c-f, if all data listed in 005.02A2a-h remain on file at the system, and the Director determines that:

005.02A2i(1) The system owner has submitted to the Director all the information required by 005.02A2a-h for at least 12 months, and

005.02A2i(2) The Director has determined that the system is not required to provide filtration treatment.

005.02A3 No later than ten days after the end of each federal fiscal year (September 30), each system owner must provide to the Director a report which summarizes its compliance with all wellhead protection requirements specified in 002.04E2e.

005.02A4 No later than ten days after the end of each federal fiscal year (September 30) each system must provide to the Director a report on the on-site inspection conducted during that year pursuant to 002.04E3, unless the on-site inspection was conducted by the Director.

005.02A5 Special reports.

005.02A5a Each system, upon discovering that a water- borne disease outbreak potentially attributable to the water system has occurred, must report that occurrence to the Director as soon as possible, but not later than by the end of the next business day.

005.02A5b If at any time the turbidity exceeds 5 NTU, the system must inform the Director as soon as possible, but no later than the end of the next business day.

005.02A5c If at any time the residual falls below 0.2 mg per liter in the water entering the distribution system, the system must notify the Director as soon as possible, but no later than by the end of the next business day. The system also must notify the Director by the end of the next business day whether or not the residual was restored to at least 0.2 mg per liter within 4 hours.

005.02B A public water supply system that uses a surface water source or a ground water source under the direct influence of surface water and provides filtration treatment must report monthly to the Director the information specified in this section beginning June 29, 1993, or when filtration is installed, whichever is later. The reports shall be submitted on Forms PWS 405 and PWS 408 in Attachment 4 to this rule.

005.02B1 Turbidity measurements as required by 002.04H3a must be reported within 10 days after the end of each month the system serves water to the public. Information that must be reported includes:

005.02B1a The total number of filtered water turbidity measurements taken during the month.

005.02B1b The number and percentage of filtered water turbidity measurements taken during the month which are less than or equal to the turbidity limits specified in 002.04G.

005.02B1c The date and value of any turbidity measurements taken during the month which exceed 5 NTU.

005.02B2 Disinfection information specified in 002.04H3 shall be reported to the Director within 10 days after the end of each month the system serves water to the pubic. Information that must be reported includes:

005.02B2a For each day, the lowest measurement of residual disinfectant concentration in mg per liter in water entering the distribution system.

005.02B2b The date and duration of each period when the residual disinfectant concentration in water entering the distribution system fell below 0.2 mg per liter and when the Director was notified of the occurrence.

005.02B2c The following information on the samples taken in the distribution system in conjunction with total coliform monitoring pursuant to 002.04F:

005.02B2c(1) Number of instances where the residual disinfectant concentration is measured;

005.02B2c(2) Number of instances where the residual disinfectant concentration is not measured but heterotrophic bacteria plate count (HPC) is measured;

005.02B2c(3) Number of instances where the residual disinfectant concentration is measured but not detected and no HPC is measured;

005.02B2c(4) Number of instances where no residual disinfectant concentration is detected and the HPC is greater than 500 per ml;

005.02B2c(5) Number of instances where the residual disinfectant concentration is not measured and HPC is greater than 500 per ml;

005.02B2c(6) For the current and previous month the system serves water to the public, the value of "V" in the following formula:

$$V = \frac{c + d + e}{a + b} \times 100$$

Where:

a = the value in 005.02B2c(1)

b = the value in 005.02B2c(2)

c = the value in 005.02B2c(3)

d = the value in 005.02B2c(4)

e = the value in 005.02B2c(5)

V = percent of samples with undetectable residual disinfectant concentration

005.02B2c(7) If the Director determines, based on site-specific considerations, that a system has no means of having a sample transported and analyzed for HPC by an the State Health Department Laboratory or an approved laboratory under the requisite time and temperature conditions required of the laboratory and that the system is providing adequate disinfection in the distribution system, the requirements of 005.02B2c(1) - 005.02B2c(6) do not apply.

005.02B2d A system need not report the data listed in 005.02B2a if all the data listed in 005.02B2a-c of this section remain on file at the system and the Director determined that the system has submitted all the information required by 005.02B2a-02B2c of this section for at least 12 months.

005.02B3 Special Reports

005.02B3a Each system owner, upon discovering that a waterborne disease outbreak potentially attributable to that water system has occurred, must report that occurrence to the Director as soon as possible, but no later than by the end of the next business day.

005.02B3b If at any time the turbidity exceeds 5 NTU, the system owner must inform the Director as soon as possible, but no later than the end of the next business day.

005.02B3c If at any time the residual falls below 0.2 mg per liter in the water entering the distribution system, the system owner must notify the Director as soon as possible, but no later than by the end of the next business day. The system owner must also notify the Director by the end of the next business day whether or not the residual was restored to at least 0.2 mg per liter within 4 hours.

005.03 Required Records. Any owner of a public water supply system subject to the provisions of this Chapter shall retain on the premises of the public water supply system or a convenient location near such premises, the following records:

005.03A Records of bacteriological, turbidity, temperature, pH, and disinfectant residual analyses made pursuant to this rule shall be kept for not less than five years. Records of chemical analyses made pursuant to this rule shall be kept for not less than ten years. Actual laboratory reports may be kept of data may be transferred to tabular summaries, provided that the following information be included:

005.03A1 The date, place, time of sampling, and the name of the person who collected the sample;

005.03A2 Identification of the sample as to whether it was a routine distribution system sample, a check sample, a raw or processed sample, or any other special purpose sample;

005.03A3 Dates of analyses;

005.03A4 Laboratory and person responsible for performing analysis;

005.03A5 The analytical technique or method used; and

005.03A6 The results of the analysis.

005.03A7 Owners of public water systems that use a ground water source under the direct influence of surface water which does not provide filtration shall keep records of source water bacteriological an turbidity measurements on Department Forms PWS 401 and PWS 402.

005.03A8 Owners of public water systems that use a surface water source or a ground water source under the direct influence of surface water which provides filtration shall keep records of the disinfection residual and turbidity measurements on Department Form PWS 407.

005.03B Records of action taken by the owner or the system to correct violations of primary drinking water regulations shall be kept for a period of not less than three years after the last action taken with respect to the particular violation involved.

005.03C Copies of any written reports, summaries or communications relating to sanitary surveys of the system conducted by the owner of the system, by a private consultant, or by any local, state or federal agency including the Department of Health shall be kept for a period of not less than ten years after completion of the sanitary survey involved.

005.03D Records concerning a variance or exemption granted to the system and records relating to receipt and compliance with Administrative Orders to correct construction or operational deficiencies shall be kept for a period ending not less than five years following the expiration of such variance, exemption or order.

005.04 Availability of Records. All records required by this section shall be available for public inspection at any reasonable hour and will be reviewed periodically for completeness by the Director. In addition, the Director shall publish annually, a listing of all public water supply systems together with violation of maximum contaminant levels, monitoring requirements, and record keeping requirements as prescribed in sections 002 and 005 of these regulations.

006 VARIANCES AND EXEMPTIONS (INCLUDES AMENDMENTS MADE THROUGH SEPTEMBER 15, 1991)

006.01 Variance. The Director, with the concurrence of the Advisory Council on Public Water Supply may authorize a variance from a maximum contaminant level adopted in section 002 of this rule, except that variances from the MCL for total coliforms and variances from any of the treatment techniques requirements of 002.04 may not be authorized. Such variance may be granted when:

006.01A The raw water sources which are reasonably available to the system cannot meet the maximum contaminant levels specified in these regulations despite application of the best technology, treatment techniques, or other means, which the Director finds are generally available (taking costs into consideration);

006.01B The concentration of the contaminant, or contaminant, for which the maximum contaminant level is exceeded by granting such variance, will not result in unreasonable risk to health; and

006.01C At the same time the variance is granted, a schedule for compliance, or increments of compliance, is issued and the owner of the supply agrees to implement such schedule.

006.02 Exemption. The Director, with the concurrence of the Advisory Council on Public Water Supply may exempt any public water supply system from any requirement respecting a maximum contaminant level or treatment technique, or from both, as adopted in section 002 of this rule, except that exemptions from the MCL for total coliforms may not be authorized. Such exemption may be granted upon finding that:

006.02A Due to compelling factors, which may include economic factors, the public water supply system is unable to comply with such contaminant level or treatment technique;

006.02B The public water supply system was in operation on the effective date of such contaminant level or treatment technique regulation;

006.02C The granting of the exemption will not result in an unreasonable risk to health; and

006.02D At the same time the exemption is granted, a schedule for compliance, or increments of compliance, is issued and the owner of the supply agrees to implement such schedule. The schedule shall require compliance not later than 12 months after the date of issuance of the exemption.

006.02E The final date for the compliance provided in any schedule in the case of any exemption may be extended by the Director for a period not to exceed 3 years after the date of the issuance of the exemption if the public water supply establishes that:

006.02E1 The system cannot meet the standard without capitol improvements which cannot be completed within the period of such exemption;

006.02E2 In the case of a system which needs financial assistance for the necessary improvements, the system has entered into an agreement to obtain such financial assistance; or

006.02E3 The system has entered into an enforceable agreement to become part of a regional public water system; and the system is taking all practicable steps to meet the standard.

006.02F In the case of a system which does not serve more than 500 service connections and which needs financial assistance for the necessary improvements, an exemption granted under 006.02E1 or 006.02E2 of this section may be renewed for one or more additional 2-year periods if the system establishes that it is taking all practicable steps to meet the requirements of 006.02E.

006.03 Procedure. Action to consider a variance or exemption from the requirements contained in section 002 of this rule may be initiated by the Director or by the owner of the supply through a formal request submitted to the Director. Before a variance or exemption proposed to be granted by the Director may take effect, the Director shall provide notice and opportunity for public hearing on the proposal; inform the Advisory Council on Public Water Supply of all facts and findings relative to the proposed action; and obtain Council approval of the proposed action, provided:

006.03A Prior to granting a variance or an exemption, the Director shall provide notice, in a newspaper of general circulation serving the area served by the public water supply system of the proposes exemption or variance, that interested persons may request a public hearing on the proposed exemption or variance. The Director may require the system to provide other appropriate notice as he or she deems necessary to provide adequate notice to persons served by the system;

006.03B If a public hearing is requested, the Director shall set a time and place for the hearing and such hearing shall be held before the Department prior to the variance to the variance or exemption being issued. Frivolous and insubstantial requests for a hearing may be denied by the Director;

006.03C An exemption or variance shall be conditioned on monitoring, testing, analyzing, or other requirements to insure the protection of the public health;

006.03D The conditions for issuing the variance or exemptions are not less stringent than conditions under which variances and exemptions may be granted under the provisions of the Federal Safe Drinking Water Act, P.L. 93-523, 93rd Congress; and further provided that,

006.03E The procedures for notification and public hearings are in conformance with the requirements of Chapter 84, Article 9, Reissue Revised Statutes of Nebraska, 1943. The same procedures shall be carried out prior to prescribing a compliance schedule for conforming to the requirements of section 002 of these rules.

007 SITING, DESIGN AND CONSTRUCTION OF PUBLIC WATER SUPPLY SYSTEMS (INCLUDES AMENDMENTS MADE THROUGH SEPTEMBER 15, 1991)

007.01 Siting. All wells, treatment and storage facilities, and other appurtenances necessary for the continued operation of a community water supply system shall be located as to:

007.01A Assure against damage or breakdown as a result of floods, fire, earthquakes, or other natural disasters;

007.01B Prevent contamination of the drinking water by existing sources of pollution;

007.01C Permit control, by the owner, over the location of future potential sources of contamination within the proximity of the system in order to prevent or minimize any hazard to the safety of the drinking water; and

007.01D Provide a sufficient property interest for the owner of the public water supply system in order to operate, maintain and replace the system components.

007.02 Design. Plans and specifications for all major construction, extension or alteration to community water supply systems shall be prepared by a registered professional engineer and shall be submitted to the Director for review and written approval prior to entering into a financial commitment for construction, the awarding of a contract, or the beginning of construction.

007.02A Plans and specifications shall be submitted for the following types of projects:

007.02A1 All components of new water supply systems;

007.02A2 New wells and intake structures;

007.02A3 Alterations that influence the capacity of existing wells or intake structures;

007.02A4 New treatment plants and modifications to existing treatment plants;

007.02A5 Installation of chemical feed equipment beyond replacement of existing equipment;

007.02A6 Storage facilities and repair to existing storage facilities, including interior lining, painting and/or coatings;

007.02A7 Pump stations;

007.02A8 Transmission mains from the source of supply to the service area; and

007.02A9 Replacement of mains within a service area for the purpose of balancing pressure or improving the efficiency of the distribution system.

007.02B Submission of plans and specifications is not required for extension of water mains within an established service area.

007.02C Plans and specifications shall be reviewed for general conformance with "Guidelines for Water System Design", herein incorporated by reference as Attachment 1, published and distributed by the Director to reflect current technology in the design of public water supply systems and their components, and shall be:

007.02C1 Presented in legible form and of sufficient scale to establish construction requirements and facilitate effective review;

007.02C2 Submitted by the engineer in triplicate and in sufficient time to permit thirty working days for review and comment or approval and with time for the incorporation of changes if required;

007.02C3 Amended in a manner that will alter the function or efficiency of components which may affect the chemical quality of the water only after change orders directing such amendments are approved by the Director;

007.02C4 Replaced by "as built" plans when change orders reflect relocations or affect the operation or replacement of the improvement, and supplemented by information relative to the location of other utilities, basis of design performance of proprietary materials or products and similar information, as may be requested by the Director.

007.02D An initial fee for the review of plans and specifications for the types of projects described in subsection 007.02A shall be submitted with the plans and specifications to be reviewed and in the amount of one hundred dollars plus five tents of one percent of the engineers estimate of the cost of the project, alteration or improvement described in the documents to be reviewed but such total fee shall not exceed $7,600 and:

007.02D1 Documentation of the contract or actual cost of the project shall be provided to the Director by the engineer or owner of the system for the purpose of determining the final fee amount;

007.02D2 Payment of the final fee amount based on one hundred dollars plus five tenths on one percent of the engineers estimate of the contract or actual cost of the project, whichever is larger, shall be made before the project is placed in service;

007.02D3 Failure to pay the final fee amount shall constitute cause to deny or revoke the permit to operate the system as required by Section 008 of this rule.

007.03 Construction. All major construction, extensions or alterations shall be completed in accordance with approved plans and specifications or approved change orders and shall comply with the following requirements:

007.03A No part of a community water supply system falling within the definition of major construction shall be placed in service prior to: certification by the owner or designated representative of the owner for conformance to the approved plans and specifications or approved change orders; a final inspection; and issuance of approval by the Director;

007.03B Any part of community water supply system falling within the definition of major construction found not to be constructed in accordance with approved plans and specifications or change orders, or for which plans and specifications were not approved, shall not be placed in service until such time as the Director determines the construction to be in conformance with current published guidelines;

007.03C Construction of water distribution mains within an established service area and not requiring prior approval, shall be accomplished in the manner specified in the "Guidelines for Water System Design".

007.04 Distinctions Applied to Non-Community Water Supply Systems. The owner of a non-community water system may:

007.04A Accomplish construction, extension or alteration to the system in the same manner applicable to the siting, design and construction of community water supply systems; or

007.04B Report to the Director any intention to locate, construct, extend, or alter a public water supply system and complete the work in accordance with TITLE 178, NEBRASKA ADMINISTRATIVE CODE, CHAPTER 12, REGULATIONS GOVERNING WATER WELL CONSTRUCTION, PUMP INSTALLATION AND WATER WELL ABANDONMENT STANDARDS.

007.05 Declaratory Ruling About Substantially Equivalent Siting, Design and Construction.

007.05A No siting, design or construction for a Public Water Supply System shall be carried out by a procedure which is inconsistent with these regulations unless the Director makes a declaratory ruling that such siting, design or construction is substantially equivalent to the standards prescribed in these regulations.

007.05B Any owner of a Public Water Supply System may request a declaratory ruling by the Director.

<u>007.05B1</u> Such a request must be submitted in writing at least thirty (30) days prior to the initiation of construction or alteration of the Public Water Supply System involved, unless good cause is shown for a shorter review period.

<u>007.05B2</u> The request shall include a description of the design; material(s), and/or construction procedure(s) proposed (including appropriate plans and specifications); identify the procedure or material required by the prescribed standards and include proof of the alleged equivalency and such written arguments as are deemed appropriate by the requesting party and/or the Director. Such request shall be submitted by a Registered Professional Engineer.

<u>007.05B3</u> Such request shall be made generally in accordance with 184 NAC 1.008, <u>Rules of Practice and Procedure Regarding Declaratory Rulings,</u> but unless the requesting party at the time of the request demands a hearing thereon, the matter will be deemed submitted on the written request, attachments thereto and facts of which the Director takes judicial notice.

<u>007.05B4</u> Any ruling issued by the Director hereunder shall be binding between the Director and the requesting party on the facts alleged unless it is altered or set aside by a court. The Director may, in situations when the submission of the request thirty (30) days in advance would result in an immediate environmental threat, significant economic hardship on or pose a health threat to the owner or other persons, waive the thirty (30) day review period.

TITLE 179 - NEBRASKA DEPARTMENT OF HEALTH
CHAPTER 2 - REGULATIONS GOVERNING PUBLIC WATER SUPPLY SYSTEMS (INCLUDING AMMENDMENTS MADE THROUGH MARCH 30, 1992)

008 OPERATION AND MAINTENANCE OF PUBLIC WATER SUPPLY SYSTEMS

008.01 Operation. All public water supply systems shall be operated and supervised by competent personnel and such personnel must possess a certificate of competency issued by the Director. In addition, all community and non-transient, non-community water supply systems shall:

008.01A Provide service on a twenty-four hour a day basis with a certified operator on call at all times unless the Director allows the water supply system to establish an electronic or otherwise dependable means for mitigating disruption of service or which assures an adequate supply of safe water on a continuous basis.

008.01B Notify the Director of any situation with the water system which presents or may present an imminent and substantial hazard to health;

008.01C Maintain an emergency plan of operations for safe-guarding the water supply, protecting the drinking water, and, if necessary, providing for an alternate drinking water supply in the event of natural or man-made disasters. The plan must include a list of individuals who may be called for help in times of disaster, their titles and their phone numbers. Such plans shall state the basic domestic water needs and usage under normal conditions. Any special institutional, commercial or industrial users shall be shown. Any special back up or standby equipment or power supply available shall be included as well as alternate sources of supply or bottled water sources. Any toxic substances within 1000 feet of the public water supply system wells or water sources shall be noted. All available chemicals and equipment for the purpose of disinfection shall be listed. The emergency plan must outline all emergency operations and must be updated at least every 4 years with copies provided to the Department of Health for inclusion in the state Drinking Water Emergency plan located in the Division of Drinking Water and Environmental Sanitation of the Department of Health. The emergency plan shall be placed at key locations, clearly marked and readily accessible to utility personnel and the public.

008.01D Flush and disinfect all newly constructed or repaired water mains and storage facilities in accordance with methods acceptable to the Director before placing the new or repaired portion of the system into service;

008.01E Conduct an on-going program for the effective detection and elimination of cross-connections and the prevention of backflow or backsiphonage. Such program is subject to review by the Director and shall include and require:

008.01E1 That there be no physical connection between the public water supply system and any pipes, pumps, hydrants, tanks, steam condensate returns, engine jackets, heat exchangers, or other water supplies whereby potentially unsafe water or contaminating materials may be discharged or drawn into the public water supply system.

008.01E2 That there be no interconnection with the public water supply system and another potable water system unless first approved by the public water supply system and reported immediately to the Director.

008.01E3 That the public water supply system shall install or require installation of properly located backflow prevention devices appropriate to the potential hazards enumerated in Tables 2 and 3 when such hazards exist and where, in the opinion of the public water supply system, effective measures consistent with a potential risk have not been taken.

TABLE 2

Cross-Connections Rated by Degree of Hazard for Commonly Encountered Equipment, Fixtures, Facilities, and Their Use

(For a more complete list, refer to the Manual of Cross-Connection Control referenced in section 008.01E9a.)

Direct or Indirect Potable Water Connections	Hazard	
	High	Low
I. Subject to Back Pressure		
A. Pumps, tanks & lines handling:		
1. Sewage	X	
2. Toxic substances	X	
3. Nontoxic substances		X
B. Water connection to steam and steam boiler:		
1. Boiler or steam connection to toxic substances	X	
2. Boiler or steam connection to nontoxic substances (boiler blowoff through air gap)		X
II. Not Subject to Back Pressure		
A. Sewer-connected water line (not subject to waste stoppages)	X	
B. Low inlets to receptacles containing:		
1. Toxic substances	X	
2. Nontoxic substances		X
C. Coils or jackets used as heat exchangers in compressors in lines carrying:		
1. Sewage	X	
2. Toxic substances	X	
3. Nontoxic substances		X
D. Flush valve toilets or urinals	X	
E. Toilet, urinal tanks and approved bathtubs		X
F. Bidets, sitz tanks, or spa, therapy and roman pools not otherwise isolated by design or backflow protectors	X	
G. Trough urinals		X
H. Valved outlets or fixtures with hose attachments that may constitute a cross-connection to:		
1. Toxic substances	X	
2. Nontoxic substances		X
I. Aspirators that may constitute a cross-connection to:		
1. Toxic substances	X	
2. Nontoxic substances		X
III. Other Equipment and Facilities Subject to a Variety of Backflow Conditions		
A. Lawn sprinkling systems that may constitute a cross-connection to:		
1. Toxic substances including lawn chemicals	X	
2. Nontoxic substances		X
B. Fire suppression systems employing toxic chemicals.	X	
C. Soft drink dispenser or bar carbonators	X	
D. Radiological, photographic, dental, medical, biological or chemical laboratories or facilities	X	
E. Swimming pools	X	
F. Tank truck loading station	X	

TABLE 3

Permitted Backflow Assemblies, Devices and Methods

Assembly, Device or Method[1]	Degree of Hazard				Installation[2,3,4,6]
	Low		High		
	Back siphonage	Back pressure	Back siphonage	Back pressure	
Air Gap	X	X	X	X	Shall be a minimum of 1 inch but not less than 2 times the diameter of the effective spout opening when not affected by side walls, and 3 times the diameter of the effective opening when affected by side walls. Side walls will be assumed to not affect air gaps when they are spaced horizontally a distance greater than 4 times the effective opening from the spout opening.
Atmospheric Vacuum Breaker	X		X		Upright position. No valves downstream. Minimum of 6 inches or listed distance above all downstream piping & flood level rim of receptor[5]
Double Check Valve Assembly	X	X			Horizontal unless otherwise listed. Requires 1 foot below & sufficient side & head room for testing & maintenance with a maximum of 5 feet above the ground, work floor, or a permanently installed working platform with stairs or ladder affixed. Does not discharge water.
Pressure Vacuum Breaker Assembly	X		X		Upright position. May have valves downstream. Minimum of 12 inches above all downstream piping & flood level rim of receptor. May discharge water.
Reduced Pressure Principle Backflow Prevention Assembly	X	X	X	X	Same as Double Check Valve Assembly above except may discharge water & wherever installed, provision for draining away at least 2 times the rated gallons per minute of the device shall be made.
Dual Check Valves	X	X			Residential services only, & where high hazards are not known to exist on the premises. Properly protected lawn sprinkling systems are assumed to be low hazard for this purpose. Dual checks are not subject to annual inspection unless so stipulated by the manufacturer. Otherwise, reasonable inspection periods will be assumed to be every 5 years.

Footnotes:

[1] For description of assemblies and devices, refer to the Cross-Connection Control Standards. Backflow preventers described herein and in the guidelines as "assemblies" must be installed as assemblies keeping the shutoff valves intact.

[2] Previous approval by the public water supply system is required for use of a pit or vault (normally prohibited due to possible flooding) or for parallel and bypass installations (normally prohibited without special design considerations and proper cross connection controls).

[3] Backflow preventers shall not be located in any area containing fumes that are toxic, poisonous or corrosive; nor in any area in which they could be damaged by freezing, or by excessively high temperatures or pressures, vibration, physical impact or structural stress; nor knowingly be allowed to conduct highly corrosive or sandy waters without a special testing and maintenance program to assure proper & safe operation.

[4] Refer to general and specific installation requirements as stated in the Cross-Connection Control Standards provided for in section 008.01E9 for conditions or situations not otherwise covered in these regulations.

[5] Not to be subjected to operating pressure for more than 12 hours in any 24-hour period. Hose bibb vacuum breakers are permitted for some uses described in the Cross-Connection Control Standards listed in section 008.01E9. Garden hose bibbs shall be protected with approved, non-removable or integral, frost-proof, self-draining, anti-siphoning vacuum breakers.

[6] Fire protection systems as a minimum shall be equipped with backflow prevention devices as described in AWWA Manual M-14, second edition. Backflow preventers under this regulation and connected to fire protection systems shall be considered part of those systems. As such, they shall not be installed, moved, removed, replaced, shut off or in any way altered unless in strict compliance with the rules and regulations promulgated by the State Fire Marshal.

> 008.01E4 That all backflow or backsiphonage protection devices equipped with test ports be tested as often as required by the public water supply system but at least once each year by a backflow preventer test and repair technician certified by the Director, with test results certified immediately to the public water supply system.
>
> 008.01E5 That the public water supply system consumers be required to assess and report potential backflow hazards on their premises and take any steps necessary for protection of public health and safety as often as reasonably requested by the public water supply system and which shall be no less often than every five (5) years.

<u>008.01E6</u> That the public water supply system shall maintain, or cause to be maintained, records of locations, types, tests and repairs of backflow preventers for a period of five (5) years of said tests and repairs.

<u>008.01E7</u> That backflow preventers required by this regulation shall have been tested and approved or listed for the intended use by one of the following organizations:

<u>008.01E7a</u> Foundation for Cross Connection Control and Hydrologic Research, University of Southern California, University Park, Los Angeles, California 90089.

<u>008.01E7b</u> American National Standards Institute, 1430 Broadway, New York, New York 10018.

<u>008.01E7c</u> Underwriters Laboratories Inc., 333 Pfingsten Road, Northbrook, Illinois 60062.

<u>008.01E7d</u> National Sanitation Foundation, 2355 West Stadium Boulevard, P.O. Box 1468, Ann Arbor, Michigan 48106.

<u>008.01E7e</u> International Association of Plumbing and Mechanical Officials, 5032 Alhambra Avenue, Los Angeles, California 90032.

<u>008.01E8</u> That an on-going public information program shall be conducted by the public water supply system to further the public water supply system customers' understanding and awareness of cross-connection hazards, the types of remedies available and the need to protect the public water supply system against backflow.

<u>008.01E9</u> That approval of Cross-Connection Control Programs (including as a minimum, backflow preventers, pressure regulators, vacuum breakers, their installation, operation, testing, maintenance and repair) shall be based on the following standards, and where conflicts may exist, the Director will be the sole judge as to which standard shall prevail:

<u>008.01E9a</u> <u>Manual of Cross-Connection Control</u>, published by the Foundation for Cross-Connection Control and Hydraulic Research, University of Southern California, eighth edition, a copy of which is attached and incorporated by reference as if fully set forth with herein.

<u>008.01E9b</u> <u>American Water Works Manual</u>, M-14, second edition, a copy of which is attached and incorporated by reference as if fully set forth with herein.

008.01F Provide sufficient personnel, tools, spare parts, work areas, chemicals, and other essentials necessary to accomplish continuous operation of the system without undue or unnecessary interruption of service.

008.02 Maintenance. All public water supply systems shall adopt and carry out a preventative maintenance program incorporating the following elements:

008.02A Neat and orderly premises used solely for the purpose of producing, treating, storing, or distributing safe drinking water with emphasis on easy access to those system components requiring periodic attention:

008.02B Routine inspection and servicing and record keeping of all mechanical equipment in accordance with manufacturer's recommendations for such maintenance;

008.02C Timely replacement of worn or deteriorated system components and equipment parts as identified by routine inspection and record keeping;

008.02D Elimination of rust and corrosion by application of paint, protective coatings, or cathodic protection or other treatment capable of prolonging the useful life of the system; and determination of the corrosive nature of delivered water for the purpose of protecting the delivered water from harmful contamination occurring from soluble metals and other materials present in household and institutional conveyance pipes;

008.02E Maintaining a system of records for annual review and reporting of the capability of the source of supply, treatment, storage, and distribution facilities to provide for future service demands both short-term and long-term (2 and 10 year plans);

008.02F Action as necessary to protect the system and its components from encroachments which are likely hazards to the safety of the drinking water quality, or which could have a substantial impact on the system pressure or economies delivered by the system. Such action shall include the adoption of ordinances, regulations, contracts, or other enforceable instruments necessary to insure adequate protection from such encroachments. This may include issues such as zoning, water rights, condemnation, land purchases, easements, abandonment of old wells, and establishing lakes, lagoons, drainage ways, special use areas, and sanitary and water districts.

009 PERMIT FOR OPERATING A PUBLIC WATER SUPPLY SYSTEM (INCLUDES AMENDMENTS MADE THROUGH SEPTEMBER 15, 1991)

009.01 Permit Required. Each person operating or maintaining a public water supply system within the state shall apply to the Director for a permit to continue the operation of such system beyond the date of January 1, 1978. The application shall be submitted on a form provided by the Director (Attachment 5).

009.02 Inspection of the System. The Director shall, as promptly as is possible, inspect and survey the system, reporting findings to the owner of the system, and issue or deny a permit based on compliance with the requirements of the Nebraska Safe Drinking Water Act and of these rules and regulations.

009.03 Information Required. Any person constructing, or otherwise acquiring a public water supply system subsequent to July 1, 1977, shall provide the information required in subsection 009.01 of this section with the submission of plans and specifications as required in section 007 of these rules. An operating permit will be issued upon confirmation that the completed works are constructed in accordance with approved plans and specifications and upon demonstration the system will be maintained and operated as prescribed by these rules and regulations.

009.04 Enforcement. Any person operating a public water supply system prior to the issuance of a permit in the manner prescribed in subsection 009.02 or 009.03 of this section, and found to be in violation of any provisions of these rules or the provisions of the Nebraska Safe Drinking Water Act, shall be subject to enforcement action as provided by law.

009.05 Permit Time Frame. Permits shall be issued for an indefinite period of time, subject only to continued compliance with the Nebraska Safe Drinking Water Act and rules and regulations promulgated thereunder.

009.06 Opportunity for Hearing. Any person shall be granted, upon request, an opportunity for a hearing before the Department under the provisions of Chapter 84, Article 9, Reissue Revised Statues of Nebraska, 1943, and amendments thereto, prior to the denial or revocation of a permit. Judicial review of such denial or revocation may be obtained as provided by such Chapter and Article.

TITLE 179 - NEBRASKA DEPARTMENT OF HEALTH
CHAPTER 2 - REGULATIONS GOVERNING PUBLIC WATER SUPPLY SYSTEMS
(INCLUDES AMMENDMENTS MADE THROUGH MARCH 30, 1992)

010 OPERATOR CERTIFICATION

010.01 Certified Operator Necessary. No public water supply system shall be issued or otherwise hold a permit to operate a public water supply system, granted by the Department, unless its operator possesses a certificate of competency issued by the Department.

010.01A Two or more public water supply systems may share or utilize the services of a single certified operator provided that the conditions in 010.01A1-010.01A4 are met. The Director shall approve or disapprove each application for a shared operator based on the following conditions:

010.01A1 The shared operator shall hold that grade required for the operator of the highest classification of the systems involved;

010.01A2 The systems are located so as to permit reasonable travel time between work areas with sufficient time remaining to perform necessary routine supervisory maintenance and operational activity for each system. As a guide, forty (40) miles is considered to be an acceptable distance;

010.01A3 Each system involved must provide a local person or persons who shall perform routine activities, including the collection of samples, under the supervision of the shared operator;

010.01A4 Companies or corporations established for the purpose of providing operational service to owners of public water supply systems shall employ one or more persons holding a certificate appropriate to the highest classification of the systems served. These persons shall have demonstrated experience in the reasonable operation or regulation of such system for at least one year within the last five-year period.

010.01B Application for a certificate of competency to act as a certified operator of a public water supply system shall be made upon a form prepared by the Director (Attachment 2). Application for operator sharing shall be made upon a form prepared by the Director (Attachment 2A).

010.01C In determining the degree of competency required for proper operation of a system, the following classification will be applied to public water supply systems;

010.01C1 Class I - All community water supply systems and non-transient, non-community supply systems using any treatment technology involving filtration to remove harmful materials from the raw water source or to improve the aesthetic quality of delivered water, and which are designed to serve a population in excess of 15,000 persons; and all other community water supply systems designed to serve a population in excess of 50,000 persons;

010.01C2 Class II - All community water supply systems and non-transient, non-community supply systems using any treatment technology involving filtration to remove harmful materials from the raw water source or to improve the aesthetic quality of the delivered water, and which are designed to serve between 2,000 and 15,000 persons; and all other community water supply systems serving a population between 15,000 and 50,000;

010.01C3 Class III - All community water supply systems and non-transient, non-community supply systems using any treatment technology involving filtration to remove harmful materials from the raw water source or to improve the aesthetic quality of the delivered water, and which are designed to serve fewer than 2,000 persons; and all other community water supply systems which serve between 2,000 and 15,000 persons; and all community supply systems which purchase water from one or more systems for the purpose of supplementing or providing service to more than 15,000 persons;

010.01C4 Class IV - All community water supply systems and non-transient, non-community supply systems which do not use a treatment technology involving filtration and which serve fewer than 2,000 persons; and all other community water supply systems which purchase water from one or more systems for the purpose of supplementing or providing service to fewer than 15,000 persons;

010.01D Class V - All other public water supply systems.

The individual or individuals placed in responsible charge of each system, or system operation, shall hold a valid Certificate of Competency comparable in Grade to the Class within which the public water supply system is described.

010.02 Water Operator Certification Requirements. Certificates of Competency will be issued by the Director, in behalf of the Department, to those operators applying for such certificates and meeting the following requirements:

010.02A In adequate physical condition;

010.02B Able to read and write the English language;

010.02C Produces evidence of satisfactory attendance at operators training meetings or short courses, or completion of education courses available that are consistent with the Grade applied for;

010.02D Able to maintain logs and records of operation and perform maintenance consistent with the Grade applied for;

010.02E Produces evidence of good moral character, integrity, ability to cooperate with others, industry, reliability, initiative, and judgement to the degree necessary to secure satisfactory operating results; and

010.02F Compliance with the requirements of subsection 010.04 of this section in regard to the appropriate Grade with the exception that the Director may issue a Provisional Certificate of Competency to any applicant during that period in which experience is being acquired.

010.03 Grades of Certification. Six grades of certification shall be made available: five grades to public water system operators, and one grade of certification to public water system authorized backflow preventer testing and repair technicians, in accordance with the criteria described in subsection 010.04 of this section. Operators possessing Grade I Certificates of Competency are qualified to operate a Class I community water supply system; those possessing Grade II certification are qualified to operate a Class II public water supply system, with the grades following having similar relationships with the other public water supply systems respectively. Operators working under the supervision of a certified operator, but in responsible charge while on duty, shall be required to hold a certificate of at least Grade IV. However, technicians who for the purpose of this regulation shall fit the definition of operators responsible for continued performance of ...any part of (the) system during assigned duty hours (001.01QQ) and who inspect, test and repair backflow preventers with test ports which are used to protect potable drinking water from contamination and/or pollution or from potential contamination and/or pollution, and including both isolation and containment types of installation, shall be required to hold a Grade VI certificate for all classes of public water supply systems. Inspection of backflow preventers without test ports does not require Grade VI certification.

010.04 Education and Experience Required. All applicants of a Certificate of Competency shall meet the following education and experience requirements for the appropriate Grade requested:

010.04A Grade I Certification -

010.04A1 Prior successful completion of a Grade II certification examination and successful completion of an examination on the subject of operation of a public water supply system recommended by the Advisory Council on Public Water Supply and approved by the Director; and

010.04A2 A degree of Bachelor of Science (with special courses in sanitary sciences) and three years in responsible charge or operation of a public water supply system or as a regulatory agent of public water supply systems (e.g., State or Federal Safe Drinking Water Act enforcement experience); or

010.04A3 Four years of college and four years of responsible charge of a public water system or as a regulatory agent of public water supply systems (e.g., State or Federal Safe Drinking Water Act enforcement experience); or

010.04A4 High school education or equivalent and six years responsible charge of a Class II or III public water supply system or eight years operation of a system under the supervision of a person possessing a Certificate of Competency as a Grade I Operator.

010.04B Grade II Certification -

010.04B1 Prior successful completion of a Grade III certification examination and successful completion of an examination recommended by the Advisory Council on Public Water Supply and approved by the Director; and

010.04B2 Two years of college, plus three years responsible charge of a public water supply system or three years as a regulatory agent of public water supply systems (e.g., State or Federal Safe Drinking Water Act enforcement experience); or

010.04B3 High school education or equivalent and six years responsible charge of a Class III, II or I public water supply system or six years operation of a system under the supervision of a person possessing a Certificate of Competency as a Grade I or Grade II operator or six years as a regulatory agent of public water supply systems (e.g., State or Federal Safe Drinking Water Act enforcement experience).

010.04C Grade III Certification -

010.04C1 Successful completion of an examination recommended by the Advisory Council on Public Water Supply and approved by the Director; and

010.04C2 High school education or equivalent and two years responsible charge of a community water supply system; or

010.04C3 Two years of high school or equivalent, and four years responsible charge of a community water supply system or six years operation of a system under the supervision of a person possessing a Certificate of Competency as a Grade I, Grade II, or Grade III operator.

010.04D Grade IV Certification -

010.04D1 Successful completion of an examination following a basic training course for water system operators recommended by the Council and approved by the Director; or

010.04D2 Successful completion of a correspondence course recommended by the Council and approved by the Director; and

010.04D3 Two years of high school or equivalent and one year in responsible charge of a community water supply system.

010.04E Grade V Certification -

010.04E1 Demonstrated ability in the collection of water samples, interpretation of results of biological examination and the maintenance of required records.

010.04F The following substitution or equivalents for required experience or training will be accepted by the Department:

010.04F1 Two years employment by a community water system under the supervision of a certified operator may be substituted for one year of grammar school or high school.

010.04F2 The Director, with the advice of the Council, may accept a substitution for required experience or training when deemed equivalent to the requirements of this section and when requested and documented by the applicant for certification.

010.04G Grade VI Certification -

010.04G1 Successful completion of an examination recommended by the Council and approved by the Director on the subject of cross connections, backflow preventer operation, maintenance, testing, and repair. Such examination shall include a written test as well as a hands-on portion to test actual testing and repair proficiency.

010.05 Issuance and Renewal of Certificates of Competency.

010.05A Certificates of Competency to act as operators of public water supply systems or any part of such systems shall be issued by the Department for the calendar years applied for and shall expire at midnight on December 31, of the third year. The Department shall notify each certificate holder at least ninety days before the expiration of the certificate by a letter addressed to him at his last place of residence as noted upon its records.

010.05B Any such Certificate of Competency may be denied, suspended, revoked or refused renewal by the Director for due cause. Due cause shall include, but not be limited to:

010.05B1 Fraud in processing the certificate;

010.05B2 Habitual intoxication or addiction to the use of drugs;

010.05B3 Conviction of a felony;

010.05B4 Physical or mental incapacity to perform professional duties;

010.05B5 Violation of any of the provisions of the Nebraska Safe Drinking Water Act or any rules or regulations adopted under such act; and

010.05B6 Failure to pay the required fee.

010.05C Except in cases of failure to pay the required fees, no license shall be denied, suspended, or revoked except after due notice and opportunity for a hearing. Any denial, suspension, or revocation of such license shall be subject to review pursuant to the provisions of Chapter 84, Article 9.

010.05D Certificates of Competency may be renewed triennially upon submission of information updating that contained in the original application and upon the submission of evidence that the applicant for renewed certification has received at least fifteen hours of acceptable continuing education during the past three year period.

010.06 Application Fees. A fee of ten dollars shall be submitted with each application of a Certificate of Competency and with each application for renewal of a Certificate of Competency.

010.07 Correspondence Course Fee. A fee of eighty dollars shall be submitted with each application for entrance into a correspondence course conducted by the Department.

010.08 Training Course Fee. A fee of forty dollars for each day of a scheduled basic training course delivered by the Department shall be submitted with each application for enrollment in a course that terminates with a written examination qualifying the attendee to apply for a Certificate of Competency.

010.09 Other Fees. A fee of fifty dollars shall be submitted with each request for individual examination without attendance at a scheduled basic training course delivered by or approved by the Department.

ATTACHMENT 1

STATE OF NEBRASKA DEPARTMENT OF HEALTH
Division of Drinking Water and Environmental Sanitation

GUIDELINES FOR WATER SYSTEM DESIGN

Introduction

Chapter 71, Article 53, Supp., 1976, the NEBRASKA SAFE DRINKING WATER ACT, required that plans and specifications for major construction, extension, or alteration of public water supply systems in Nebraska be prepared by a Professional Engineer and reviewed and approved by the Nebraska State Department of Health. These guidelines are established under this authority in conformance with NEBRASKA STATE DEPARTMENT OF HEALTH, RULE 5, REGULATIONS GOVERNING PUBLIC WATER SUPPLY SYSTEMS. The definitions contained in RULE 5 apply to these guidelines.

The Guidelines are intended as a guide to the engineer in the preparation of plans and specifications and will be used as the basis for review by the Department of Health.

Guidelines

The MANUAL OF WATER WELL CONSTRUCTION PRACTICES, published by the United States Environmental Protection Agency, Office of Water Supply; and RECOMMENDED STANDARDS FOR WATER WORKS, 1987 Edition, published by the Great Lakes-Upper Mississippi River Board of State Sanitary Engineers, are hereby adopted by the Nebraska State Department of Health as guidelines for water system design. In the event of discrepancy, the MANUAL OF WATER WELL CONSTRUCTION PRACTICES shall govern.

In addition, the following guidelines concerning the groundwater sources are established:

1. Location. Every well, infiltration line or spring serving or intended to provide water for a public water supply system, insofar as possible, should be located, constructed, or modified in such a manner that neither underground or surface contamination by any biological, chemical or radioactive substance or by the physical property of any substance from any cesspool, privy, septic tank, sub-surface tile system, sewer, drain, pit below ground surface, abandoned well, animal or avian wastes, or any other possible source of pollution can adversely affect such water supply. The minimum recommended horizontal distance in feet separating the well or spring from potential sources of contamination should be as described below. The Department will consider location of wells and springs at closer proximity than the minimum distances below. Approval for such location will be given when circumstances require such location and when, in the opinion of the Director, the engineer demonstrates that such location will not constitutes a pollution hazard to the supply.

(1. Continued)

CATEGORY	DISTANCE FEET	DISTANCE METERS
Non-Potable Water Well	1,000	300
Sewage Lagoon	1,000	300
Absorption or Disposal Field for Waste	500	150
Cesspool	500	150
Dump	500	150
Feedlot or Feedlot Runoff	500	150
Corral	500	150
Pit Toilet	500	150
Sanitary Landfill	500	150
Chemical or Petroleum Product Storage	500	150
Septic Tank	500	150
Sewage Treatment Plant	500	150
Sewage Wet Well	500	150
Sanitary Sewer Connection	100	30
Sanitary Sewer Manhole	100	30
Sanitary Sewer Line	50	15
Sanitary Sewer Line (Permanently Water Tight)	10	3

When surface runoff or underground movement from potential sources of contamination may adversely affect the quality of water from such supplies, the distance separating these potential sources of contamination and the well or spring should be greater than that listed in the above schedule.

2. A test hole will be required for all proposed well sites.

3. The annular space shall be grouted to a minimum depth of 10 feet below the ground surface. Grouting to greater depths will be required when warranted by the method of

construction or is necessary, in the judgement of the Director, to prevent possible contamination of the well.

4. The following information must be submitted with the plans and specifications or as addendum thereto:

   a. Test hole driller's logs and reports;

   b. All sieve analysis and calculations used in gravel pack and screen design. This information shall be submitted prior to the placement of the screen and gravel pack.

ATTACHMENT 2

Water Operator Application Form -
Nebraska Department of Health
Division of Drinking Water and
Environmental Sanitation
P.O. Box 95007
Lincoln, NE 68509-5007

OFFICE USE ONLY
Cert #________
In Comp.________
Cert. Mailed______

Date ______________ Training Course Completed __________

Last Name ____________ First Name and M.I. ____________

Street Address ________________________________

City and State ________________ Zip Code __________

EMPLOYER INFORMATION:

Name of Water System ______________________________

Street Address ________________________________

City and State ________________ Zip Code __________

County System is located in ___________ Work phone ________

Population Served ______________ No. of Service Conn. ____

Type(s) of Water Treatment used _______________________

PERSONAL INFORMATION:

Home Phone ____________ Social Security No. ____________

(Disclosure of your social security number is voluntary. Its usage by the Department of Health, in regard to this application, is to allow the Department to distinguish between persons who have the same or similar names. You will not be denied a license or certificate if you do not disclose your Social Security number.)

High School Graduate? YES NO (Circle Response)

Post High School education __________________________

Certification Grade Requested? 5 4 3 2 1 (Circle one)

Will you be in responsible charge of the water system? YES NO

Months of experience as a water operator ______ Where? ______

Name, address and phone of the person who will be your immediate supervisor.

____

Signature ___________________________________

ATTACHMENT 2A

APPLICATION TO SERVE AS A CERTIFIED OPERATOR FOR MORE THAN ONE NEBRASKA PUBLIC WATER SUPPLY SYSTEM

I. This form constitutes a request that ____________________ (Name of Operator) holding a Class ___ water operator's certificate and operating the ____________________________ (Name & Address of Water Supply)

____

____

a system classified as Class ____ be allowed to act in the additional capacity as the operator in responsible charge of the ______________ (Name & Address of Water Supply)

____

____

a system classified as Class ____.

II. Additional Information to be Supplied:

Distance between the two systems _________ miles.

Name of person responsible for shared system in absence of certified operator ____

III. We the undersigned agree to allow ____________________ (Name of Operator) to serve as the operator for the above named systems and in carrying out this responsibility to spend the time indicated at each water supply sytem.

System I	System II
______________________________	______________________________
Signature (Mayor, Chairman or Owner)	Signature (Mayor, Chairman or Owner)
______________________________	______________________________
Title	Title
______________________________	______________________________
Printed or Typed Name	Printed or Typed Name
______________________________	______________________________
Date	Date

System III

______________________________	______________________________
Signature (Mayor, Chairman or Owner)	Title
______________________________	______________________________
Printed or Typed Name	Date

ATTACHMENT 3

Protocol for the Determination of Influence of Surface Water on Ground Water Sources

Nebraska Department of Health
Division of Drinking Water and Environmental Sanitation
P.O. Box 95007
Lincoln, NE 68509

The Surface Water Treatment Rule (SWTR) promulgated by EPA requires that treatment sufficient to produce a three-log (99.9%) reduction in Giardia lamblia cysts and a four-log (99.99%) reduction in viruses be provided for all surface water sources and all ground water sources under the direct influence of surface water. In most cases, treatment which will effect a three-log reduction of Giardia will be more than sufficient to effect a four-log reduction in viruses. The basic criteria which determines direct influence of surface on ground water sources, within the context of the SWTR, is the possibility of Giardia cysts being carried into the ground water by infiltrating surface water. The state must make a determination for all ground water sources as to whether or not they are under the direct influence of surface water. This determination must be made for all ground water sources supplying community systems by June 19, 1994, and by June 19, 1999, for those serving non-community systems.

The definitive determination of the influence of surface water is to perform particulate analyses on water samples collected from ground water sources. Such analyses will be time consuming, possibly to the degree that the analyses on each ground water source could not be accomplished within the deadlines established in the SWTR. A screening protocol is necessary to limit the number of ground water sources for which particulate analysis will be necessary. The screening protocol is as follows:

1. Department of Health personnel will examine information on file to determine if a source of water is potentially at risk to the direct influence of surface water or not. If any one of the following conditions is true, then the source of water is potentially at risk to the direct influence of surface water:

    A. The source of water is a spring or an infiltration gallery;

    B. The top of the uppermost intake structure of a well is less than or equal to 50 feet from the ground surface;

    C. There is evidence of fecal contamination of the discharge within the preceding three years, excluding distribution system samples;

    D. There are defects in the condition of the upper terminus (e.g., defective pump base seals, missing or unscreened casing vents, cracked pump bases);

    E. There is inadequate grouting of the upper annulus;

F. There is inadequate information on file to assess whether the source of water meets one or more of the conditions in 1A through 1E. In this case, owners of the Public Water Supply System will be given the opportunity to provide the required information.

2. A. If a source of water is determined not to be potentially at risk to the direct influence of surface water in accordance with protocol number one, then it is not considered to be at risk to the direct influence of surface water and Section 002.04 does not apply (no further analyses are required).

B. If a well is determined to be potentially at risk to the direct influence of surface water solely because of either condition 1D or 1E and it is subsequently repaired and no longer meets any of the conditions in protocol number 1, then it is not considered to be at risk to the direct influence of surface water and Section 002.04 does not apply (no further analyses are required).

3. If a source of water is determined to be potentially at risk to the direct influence of surface water in accordance with protocol number one, then it shall be investigated further for the direct influence of surface water as follows:

A. Monthly examination of temperature, pH, or turbidity shall be conducted over twelve consecutive months from the water source discharge. A minimum of two of these parameters shall be examined and recorded.

(1) Sample collection and certification.

(a) Prior to the collection of each sample or measurement, the water source pump shall be operated long enough to pump at least ten times the volume of the pump column.

(b) The certified operator in responsible charge of the public water supply system shall certify the location, analysis method and results of each test.

(2) Analytical Methods. Samples shall be analyzed by the following methods which are incorporated by the following references and are attached in Attachment 6.

(a) Turbidity analyses shall be performed in accordance with 002.04H1a (Method 214A - Nephelometric Method).

(b) pH shall be measured by one of the following:

(i) Method 150.1, Electrometric, "Methods of Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, (EPA-600/4-79-020), March 1983. Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268;

(ii) Method D1293-84B, Electrometric, Annual Book of ASTM Standards, Vol. 11.01, American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103; or

(iii) Method 4500-H, Electrometric, Standard Methods for the Examination of Water and Wastewater, 17th edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1989.

(c) Temperature shall be measured as follows:

(i) Method 2550, Thermometric, Standard Methods for the Examination of Water and Wastewater, 17th edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1989.

(3) Evaluation of measurements.

(a) A variation of less than 15 percent between high and low results of all of the parameters measured will be considered evidence that the source of water is not under the direct influence of surface water. Section 002.04 does not apply (no further analyses are required).

(b) A variation of 15 percent or greater between high and low results of any of the parameters measured will be considered evidence that the source of water is under the direct influence of surface water. The owner of the water system may accept the designation of surface water and provide treatment as required by 002.04 or may cause particulate analyses to be performed as outlined in 3B.

(4) In lieu of performing analyses outlined in 3A, the owner may elect to perform particulate analyses in accordance with 3B.

B. Perform particulate analyses of samples taken from the well.

(1) A minimum of two samples shall be collected and analyses for the presence of diatoms, rotifers, coccidia, insect parts, and Giardia. Samples shall be collected in accordance with the 17th Edition of Standard Methods, and collection shall be at such times as the ground water source is most vulnerable to surface water infiltration (e.g., during periods of high surface water stages, after heavy rainfall or runoff events). Sample volumes shall be at least 500 gallons. Analysis shall be made by a certified laboratory in accordance with EPA consensus protocol given in 3B(2).

(2) EPA Consensus Method:

Filter unwound into quarters
↓
Rinsed in distilled water with polysorbate 20
↓
Settled overnight, or centrifuged
↓
Collect sediment and add 2% Formaldehyde in PBS
↓
Settled overnight, or centrifuged
↓
Collect sediment

↓ > 1 g.
↓ < 1 g.

Sucrose or Percoll-sucrose gradient → ← $ZnSO_4$ Flotation
↓
Microscopic observation of the entire concentrate (Brightfield/Phase-contrast)

(3) The presence of any of the indicators given in 3B1) in all samples shall be considered conclusive evidence of surface water influence and the well is subject to section 002.04.

(4) If sample results are inconsistent or inconclusive, additional sampling shall be taken as directed by the Department of Health.

ATTACHMENT 4

SURFACE WATER TREATMENT FORMS

PWS 401

SOURCE WATER QUALITY CONDITIONS FOR UNFILTERED SYSTEMS (1)

(For System Use Only)

Month_______________

Year_______________

System/Treatment Plant_______________________________________

PWSID_______________________

	Coliform Measurements (2)				Turbidity Measurements	
	No. of Samples		No. of Samples Meeting Specified Limits			
Date	Fecal	Total	Fecal (< = 20/100 mL)	Total (< = 100/100 mL)	Maximum (3) Turbidity (NTU)	Turbidity (4) "Event" (Yes or No)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
Totals					Maximum Daily Turbidity = _____ NTU Total No. of Turbidity Events = ___	

Continued on Back

Notes:

1. Samples are taken form the source water immediately prior to the first disinfection point included in the CT determination.

2. As specified in 40 CFR 141.74(b)(1), a fecal or total coliform sample must be taken on each day that the system operates and a source water turbidity measurement exceeds 1 NTU.

3. For each day that the maximum turbidity exceeds 5 NTU, the date should also be entered for the day that the Sate was notified of this exceedance, e.g., "7.3-22 Apr."

4. A "yes" response is required each day the maximum turbidity exceeds 5 NTU and the previous day did not. This is indicative of the beginning of a turbidity "event". The total number of "yes" responses equals the number of turbidity "events" in the month.

PWS 402

LONG-TERM SOURCE WATER QUALITY CONDITIONS FOR UNFILTERED SYSTEMS
(For System Use Only)

Year____________________

System/Treatment Plant____

PWSID____________________

	Coliform Measurements				Turbidity Measurements	
	No. of Samples		No. of Samples Meeting Specified Limits		Days with Turbidity >5 NTU	Number of Turbidity Events
Month	Fecal	Total	Fecal (<= 20/100 mL)	Total (<= 100/100 mL)		
January						
February						
March						
April						
May						
June						
July						
August						
September						
October						
November						
December						
					Total:	

PWS 403

CT DETERMINATION FOR UNFILTERED SYSTEMS -- MONTHLY REPORT TO PRIMACY AGENCY (3)(2)

Month____________________
Year____________________
System/Treatment Plant____
PWSID____________________
Disinfectant/Sequence of Application____

Date	Disinfectant (3) Concentration, C (mg/L)	Disinfectant (3) Contact Time, T (min.)	CT calc (4) (=C x T)	pH (3,5)	Water (3) Temp. (deg. C)	CT99.9 (6)	(CTcalc/CT99.9)
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							
31							

Prepared By____________________
Date____________________

Notes:
(1) To be included in the monthly report for at least 12 months after the initiation of reporting. After that time, the Primacy Agency may no longer require this form.
(2) Use a separate form for each disinfectant/sampling site. Enter disinfection and sequence position, e.g., "ozone/1st" or "C102/3rd".
(3) Measurement taken at peak hourly flow.
(4) CTcalc - c (mg/L) x T (Min.)
(5) Only required if the disinfectant is free chlorine.
(6) From Tables 1.1 - 1.6, 2.1, and 3.1, 40 CFR 141.74(b)(3)

PWS 404

DISINFECTION INFORMATION FOR UNFILTERED SYSTEMS -- MONTHLY REPORT TO PRIMACY AGENCY

Month________________________

Year________________________

System/Treatment Plant____

PWSID____________________

Date	Minimum Disinfectant Residual at Point-of Entry to Distribution System (mg/L) (1)	(CTcalc/CT99.9) (from Table 6-3)						(2) Sum (CTcalc/CT99.9)	(3) SUM (CTcalc/CT99.9)<1 (Yes or No)
		Disinfectant Sequence							
		1st	2nd	3rd	4th	5th	6th		
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									
31									

Prepared By_______________________________________

Date____

Notes:

(1) If less than 0.2 mg/L, the lowest level and duration of the period must be reported, e.g., "0.1-3 hrs.".

(2) To determine SUM (CTcalc/CT99.9), add (CTcalc/CT99.9) values from the first disinfection sequence to the last.

(3) If SUM (CTcalc/CT99.9) <1, a treatment technique violation has occurred, and a "yes" response must be entered.

PWS 405

DISTRIBUTION SYSTEM DISINFECTANT RESIDUAL DATA FOR UNFILTERED AND FILTERED SYSTEMS
MONTHLY REPORT FOR PRIMACY AGENCY

Month____________________

Year____________________

System/Treatment Plant____

PWSID_______________

Date	No. of sites where disinfectant residual was measured (=a)	No. of sites where no disinfectant residual was measured, but HPC measured (=b)	No. of sites where disinfectant residual not detected, no HPC measured (=c)	No. of sites where disinfectant residual not detected, HPC > 500/ml (=d)	No. of sites where disinfectant residual not measured, HPC > 500 ml (=c)
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
26					
27					
28					
29					
30					
31					
Total	a=	b=	c=	d=	e=

V = (c+d+e)/(a=b) x 100 = (_____ + _____ + _____)/(_____ + _____) x 100 = _____%

Prepared By________________________

Date_____________________________

PWS 406

MONTHLY REPORT TO PRIMACY AGENCY FOR COMPLIANCE DETERMINATION -- UNFILTERED SYSTEMS

Month________________________

Year________________________

System/Treatment Plant____

PWSID____________

Source Water Quality Conditions

A. Cumulative number of months for which results are reported

For source water coliform monitoring ______ (No. of months)

For turbidity monitoring ______ (No. of months) (1)

B. Coliform Criteria

	No. of Samples		No. of Samples Meeting Specified Limits	
	Fecal	Total	Fecal (<=20/100mL)	Total (<=100/100mL)
Previous 6 months	w= ______	x=______	y=______________	z=______________

Percentage of samples <= 20/100 mL fecal coliforms, F = y/w x 100 = ______ %

Percentage of samples <= 100/100 mL total coliforms, T = z/x x 100 = ______ %

is F < 90%?: Yes______ No______ N/A______; is T < 90%?: Yes_____ No______ N/A______

C. Turbidity Criteria

Maximum turbidity level for reporting (current) month = ______ NTU

Enter the month (120 months prior to the reporting month or January 1991 (whichever is later) ______________

Dates of 5 NTU Exceedances Since Latest Month Recorded Above		
Beginning Date	Duration (days)	Date Reported

Disinfection Criteria

A. Point-of-Entry Minimum Disinfectant Residual Criteria

Days the Residual was <0.2 mg/L		
Day	Duration of Low Level (hrs.)	Date Reported to Primacy Agency

B. Distribution System Disinfectant Residual Criteria

The value of a, b, c, d, and e, from Table 6-5, as specified in 40 CFR 141.75(b)(2)(iii)(A)-(E)

a = _____, b = _____, c = _____, d = _____, e = _____

$V = \frac{c+d+e}{a+b} \times 100 =$ ______%

For previous month, V = ______%

C. Disinfection Requirement Criteria

Record the date and value of SUM (CTcalc/CT99.9) for any SUM (CTcalc/CT99.9) <1 (from table 6-4):
If none, enter "none".

Date	SUM (CTcalc/99.9)

Prepared By ______________________

Date______________________

Notes:

(1) The current 6-month cumulatives are required to determine whether compliance with the coliform criteria has been achieved. These totals are calculated from: the previous 6-month cumulatives, the current month's, and totals from the earliest of 6 previous months.

PWS 407

DAILY DATA SHEET FOR FILTERED SYSTEMS
(For system use only)

Month________________

Year________________

System/Treatment Plant____

Filtration Technology____

PWSID________________

Date	(1) Minimum Disinfection Residual at Point-of-Entry to Distribution System (mg/L)	(2) Maximum Filtered Water Turbidity				(3) No. of Turbidity Measurements	(4) No. of Turbidity Measurements <= Specified Limit	(5) No. of Turbidity Measurements > 5 NTU
		Filter #	Combined Filter Effluent	Clear-well Effluent	Plant Effluent			
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								
26								
27								
28								
29								
30								
31								
					Totals:			

CONTINUED ON BACK

PWS 408

MONTHLY REPORT TO PRIMACY AGENCY FOR COMPLIANCE DETERMINATION - FILTERED SYSTEMS

Month______________ System/Treatment Plant____
Year_______________ Type of Filtration____
Turbidity Limit______________________ PWSID________________

Turbidity Performance Criteria

A. Total number of filtered water turbidity measurements = ____________

B. Total number of filtered water turbidity measurements that are less than or equal to the specified limits for the filtration technology employed = __________

C. The percentage of turbidity measurements meeting the specified limits = B/A x 100 = _____/_____ x 100 = _____%

D. Record the date and turbidity value for any measurements exceeding 5 NTU: if none, enter "none".

Date	Turbidity, NTU

Disinfection Performance Criteria

A. Point-of-Entry Minimum Disinfectant Residual Criteria

Date	Minimum Disinfectant Residual at Point-of-Entry to Distribution System (mg/L)	Date	Minimum Disinfectant Residual at Point-of-Entry to Distribution System (mg/L)	Date	Minimum Disinfectant Residual at Point-of-Entry to Distribution System (mg/L)
1		11		21	
2		12		22	
3		13		23	
4		14		24	
5		15		25	
6		16		26	
7		17		27	
8		18		28	
9		19		29	
10		20		30	
				31	

Days the Residual was <0.2 mg/L		
Day	Duration of Low Level (hrs.)	Date Reported to Primacy Agency

B. Distribution System Disinfectant Residual Criteria

The value of a, b, c, d, and e, from Table 6-5, as specified in 40 CFR 141.75 (b)(2)(iii)(a)-(e):

a = _____, b = _____, c = _____, d = _____, e = _____

$V = \frac{c + d + e}{a + b} \times 100 =$ ______%

For previous month, V = _____%

Prepared By___________________________
Date__________________________________

Notes:

1. For multiple disinfectants, this column must only be completed for the last disinfectant added prior to entering the distribution system. If less than 0.2 mg/L, the duration of the period must be reported, d.g., "1.2-3 hrs.".

2. For systems using conventional treatment, direct filtration, or technologies other than slow sand or diatomaceous earth filtration, turbidity measurements may be taken at the combined filter effluent, clearwell effluent, or plant effluent prior to entry into the distribution system. The turbidity may also be measured for each individual filter with a separate sheet maintained for each distribution system. The turbidity may also be measured for each individual filter with a separate sheet maintained for each.

3. For continuous monitors, count each 4 hour period as 1 sample.

4. Depending on the filtration technology employed, the number of turbidity samples meeting the following levels must be recorded: conventional treatment or direct filtration - 0.5 NTU, slow sand filtration - 1 NTU, diatomaceous earth filtration - 1 NTU. The State may specify alternate performance levels for conventional treatment or direct filtration, not exceeding 1 NTU, and slow sand filtration not exceeding 5 NTU, in which case the number of turbidity measurements meeting these levels must be recorded.

5. In recording the number of turbidity measurements exceeding 5 NTU, the turbidity values should also be recorded, e.g., "3: 5.6; 6.2; 6.0".

ATTACHMENT 5

NEBRASKA DEPARTMENT OF HEALTH
Division of Drinking Water and Environmental Sanitation

APPLICATION FOR PERMIT TO OPERATE A PUBLIC WATER SUPPLY SYSTEM

1. Name of system ______________________________
2. Location of system ____________________________
3. Area served (attach map) ________________________
4. Is the system open and serving water to the public year-round?
   _______ If the answer is no, what part of the year are you open?
   ____
5. Owner's name ________________________________
6. Owner's address
   Street or RFD ______________________________
   City, State, ZIP ____________________________
   Telephone number ___________________________
7. Name of individual responsible for operation and maintenance:
   Name ______________________________________
   Street or RFD ______________________________
   City, State, ZIP ____________________________
   Telephone number ___________________________
8. Name of individual responsible for retaining records (if different from # 6): ______________________________
9. Certified water operator(s) (include those person(s) currently applying for certification):

Name	Grade

10. Type and population of service areas:
    a. Year-round residential population: ________________
    b. Seasonal residential population: ________________
    c. Non-residential, non-transient population:
       - school ___________
       - medical facility _____
       - day care center _____
       - institution ___________
       - industrial/agricultural ___
       - other _____________

d. Non-residential, transient population (daily average - during the peak season if applicable):

* rec area ________ * service station ________
* summer camp ________ * restaurant ________
* highway rest area ____ * hotel/motel ________

11. Number of service connections (generally equals the number of services which are billed for water usage):

* residential ________ * commercial ________
* institutional ______ * other ________

12. Source water utilized:

a. Non-purchased source water:
   1) % from ground water source(s) = ____
   2) % from surface water source(s) = ____

b. Purchased water (from another public water system):
   1) % purchased from a public water using ground water = ____
   2) % purchased from a public water system using surface water = ____

(Total should equal 100%)

13. Description of (use additional pages if necessary):

a. Source(s) of supply:

b. Treatment techniques (i.e., fluoridation, disinfection, sequestration, etc.):

c. Storage facilities:

d. Distribution system:

14. Attach Emergency Plan. If an emergency plan is currently not in effect, then complete the attached "Local Emergency Plan" and submit it with the completed application.

15. Other information:

16. Submitted by Name ________________

Title ________________

Signature ________________

Date ________________

ATTACHMENT 6

ANALYSIS METHODS

2550 TEMPERATURE*

2550 A. Introduction

Temperature readings are used in the calculation of various forms of alkalinity, in studies of saturation and stability with respect to calcium carbonate, in the calculation of salinity, and in general laboratory operations. In limnological studies, water temperatures as a function of depth often are required. Elevated temperatures resulting from discharges of heated water may have significant ecological impact. Identification of source of water supply, such as deep wells, often is possible by temperature measurements alone. Industrial plants often require data on water temperature for process use or heat-transmission calculations.

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* Approved by Standard Methods Committee, 1988.

2550 B. Laboratory and Field Methods

1. Laboratory and Other Non-Depth Temperature Measurements

Normally, temperature measurements may be made with any good mercury-filled Celsius thermometer. As a minimum, the thermometer should have a scale marked for every 0.1°C, with markings etched on the capillary glass. The thermometer should have a minimal thermal capacity to permit rapid equilibration. Periodically check the thermometer against a precision thermometer certified by the National Institute of Standards and Technology (NIST, formerly National Bureau of Standards)* that is used with its certificate and correction chart. For field operations use a thermometer having a metal case to prevent breakage.

2. Depth Temperature Measurements

Depth temperature required for limnological studies may be measured with a reversing thermometer, thermophone, or thermistor. The thermistor is most convenient and accurate; however, higher cost may preclude its use. Calibrate any temperature measurement devices with a NIST-certified thermometer before field use. Make readings with the thermometer or device immersed in water long enough to permit complete equilibration. Report results to the nearest 0.1 or 1.0°C, depending on need.

The thermometer commonly used for depth measurements is of the reversing type. It often is mounted on the sample collection apparatus so that a water sample may be obtained simultaneously. Correct readings of reversing thermometers for changes due to differences between temperature at reversal and temperature at time of reading. Calculate as follows:

$$\Delta T = \left[\frac{(T' - t)(T' - V_0)}{K}\right] \times \left[1 + \frac{(T' - t)(T' - V_0)}{K}\right] + L$$

where:

ΔT = correction to be added algebraically to uncorrected reading.

T' = uncorrected reading at reversal.

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* Some commercial thermometers may be as much as 3°C in error.

t = temperature at which thermometer is read.
V_0 = volume of small bulb end of capillary up to 0°C graduation.
K = constant depending on relative thermal expansion of mercury and glass (usual value of K = 6100), and
L = calibration correction of thermometer depending on T'

If series observations are made it is convenient to prepare graphs for a thermometer to obtain ΔT from any values of T' and t.

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2710 TESTS ON SLUDGES*

2710 A. Introduction

This section presents a series of tests uniquely applicable to sludges or slurries. The test data are useful in designing facilities for solids separation and concentration and for assessing operational behavior, especially of the activated sludge process.

2710 B. Oxygen-Consumption Rate

1. General Discussion

This test is used to determine the oxygen consumption rate of a sample of a biological suspension such as activated sludge. It is useful in laboratory and pilot-plant studies as well as in the operation of full-scale treatment plants. When used as a routine plant operation test, it often will indicate changes in operating conditions at an early stage. However, because test conditions are not necessarily identical to conditions at the sampling site, the observed measurement may not be identical with actual oxygen consumption rate.

2. Apparatus

a. Oxygen-consumption rate device: Either:

1) *Probe with an oxygen-sensitive electrode* (polarographic or galvanic), or

2) *Manometric or respirometric device* with appropriate readout and sample capacity of at least 300 mL. The device should have an oxygen supply capacity greater than the oxygen consumption rate of the biological suspension, or at least 150 mg/L·h.

b. Stopwatch or other suitable timing device.

c. Thermometer to read to ± 0.5°C.

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*Approved by Standard Methods Committee, 1985.

4500-H$^+$ B. Electrometric Method

1. General Discussion

a. Principle: The basic principle of electrometric pH measurement is determination of the activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode. The hydrogen electrode consists of a platinum electrode across which hydrogen gas is bubbled at a pressure of 101 kPa. Because of difficulty in its use and the potential for poisoning the hydrogen electrode, the glass electrode commonly is used. The electromotive force (emf) produced in the glass electrode system varies linearly with pH. This linear relationship is described by plotting the measured emf against the pH of different buffers. Sample pH is determined by extrapolation.

Because single ion activities such as a_{H^+} cannot be measured, pH is defined operationally on a potentiometric scale. The pH measuring instrument is calibrated potentiometrically with an indicating (glass) electrode and a reference electrode using National Institute of Standards and Technology (NIST, formerly National Bureau of Standards) buffers having assigned values so that:

$$pH_s = -\log_{10} a_{H^+}$$

where:

pH_s = assigned pH of NIST buffer.

The operational pH scale is used to measure sample pH and is defined as:

$$pH_x = pH_s \pm \frac{F(E_s - E_x)}{2.303\ RT}$$

where:

pH_x = potentiometrically measured sample pH,
F = Faraday: 9.649×10^4 coulomb/mole,
E_x = sample emf, V,
E_s = buffer emf, V,
R = gas constant; 8.314 joule/(mole °K), and
T = absolute temperature, °K.

NOTE: Although the equation for pH_x appears in the literature with a plus sign, the sign of emf readings in millivolts for most pH meters manufactured in the U.S. is negative. The choice of negative sign is consistent with the IUPAC Stockholm convention concerning the sign of electrode potential.[1,2]

The activity scale gives values that are higher than those on Sorenson's scale by 0.04 units:

$$pH\ (activity) = pH\ (Sorenson) + 0.04$$

The equation for pH_x assumes that the emf of the cells containing the sample and buffer is due solely to hydrogen ion activity unaffected by sample composition. In practice, samples will have varying ionic species and ionic strengths, both affecting H^+ activity. This imposes an experimental limitation on pH measurement; thus, to obtain meaningful results, the differences between E_x and E_s should be minimal. Samples must be dilute aqueous solutions of simple solutes ($<0.2M$). (Choose buffers to bracket the sample.) Determination of pH cannot be made accurately in nonaqueous media, suspensions, colloids, or high-ionic-strength solutions.

b. Interferences: The glass electrode is relatively free from interference from color, turbidity, colloidal matter, oxidants, reductants, or high salinity, except for a sodium error at pH > 10. Reduce this error by using special "low sodium error" electrodes.

pH measurements are affected by temperature in two ways: mechanical effects that are caused by changes in the properties of the electrodes and chemical effects caused by equilibrium changes. In the first instance, the Nernstian slope increases with increasing temperature and electrodes take time to achieve thermal equilibrium. This can cause long-term drift in pH. Because chemical equilibrium affects pH, standard pH buffers have a specified pH at indicated temperatures.

Always report temperature at which pH is measured.

2. Apparatus

a. pH meter consisting of potentiometer, a glass electrode, a reference electrode, and a temperature-compensating device. A circuit is completed through the potentiometer when the electrodes are immersed in the test solution. Many pH meters are capable of reading pH or millivolts and some have scale expansion that permits reading to 0.001 pH unit, but most instruments are not that precise.

For routine work use a pH meter accurate and reproducible to 0.1 pH unit with a range of 0 to 14 and equipped with a temperature-compensation adjustment.

Although manufacturers provide operating instructions, the use of different descriptive terms may be confusing. For most instruments, there are two controls: intercept (set buffer, asymmetry, standardize) and slope (temperature, offset); their functions are shown diagramatically in Figures 4500-H$^+$:1 and 2. The intercept control shifts the response curve laterally to pass through the isopotential point with no change in slope. This permits bringing the instrument on scale (0 mV) with a pH 7 buffer that has no change in potential with temperature.

The slope control rotates the emf/pH slope about the isopotential point (0 mV/pH 7). To adjust slope for temperature without disturbing the intercept, select a buffer that brackets the sample with pH 7 buffer and adjust slope control to pH of this buffer. The instrument will indicate correct millivolt change per unit pH at the test temperature.

b. Reference electrode consisting of a half cell that provides a constant electrode potential. Commonly used are calomel and silver: silver-chloride electrodes. Either is available with several types of liquid junctions.

The liquid junction of the reference electrode is critical because at this point the

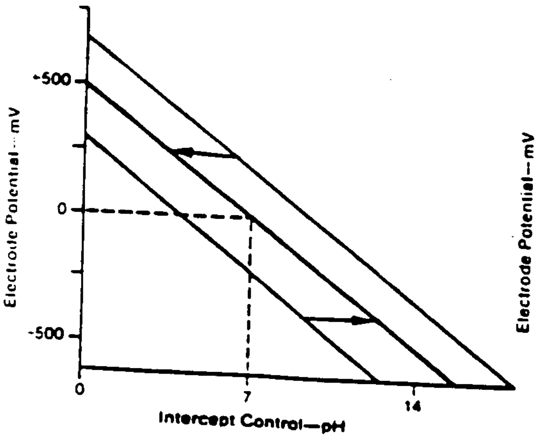

Figure 4500-H$^+$:1. Electrode potential vs. pH. Intercept control shifts response curve laterally.

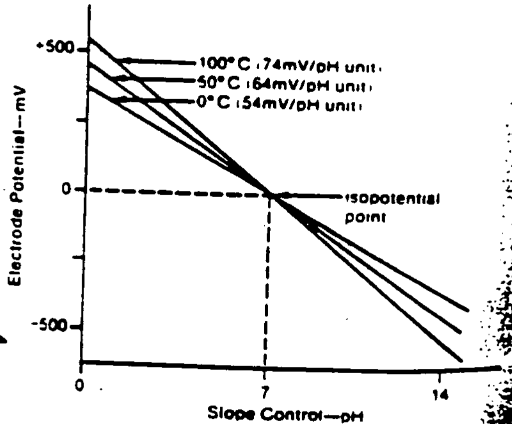

Figure 4500-H$^+$:2. Typical pH electrode response as a function of temperature.

electrode forms a salt bridge with the sample or buffer and a liquid junction potential is generated that in turn affects the potential produced by the reference electrode. Reference electrode junctions may be annular ceramic, quartz, or asbestos fiber, or the sleeve type. The quartz type is most widely used. The asbestos fiber type is not recommended for strongly basic solutions. Follow the manufacturer's recommendation on use and care of the reference electrode.

Refill nonsealed electrodes with the correct electrolyte to proper level and make sure junction is properly wetted.

c. Glass electrode: The sensor electrode is a bulb of special glass containing a fixed concentration of HCl or a buffered chloride solution in contact with an internal reference electrode. Upon immersion of a new electrode in a solution the outer bulb surface becomes hydrated and exchanges sodium ions for hydrogen ions to build up a surface layer of hydrogen ions. This, together with the repulsion of anions by fixed, negatively charged silicate sites, produces at the glass-solution interface a potential that is a function of hydrogen ion activity in solution.

Several types of glass electrodes are available. Combination electrodes incorporate the glass and reference electrodes into a single probe. Use a "low sodium error" electrode that can operate at high temperatures for measuring pH over 10 because standard glass electrodes yield erroneously low values. For measuring pH below 1 standard glass electrodes yield erroneously high values; use liquid membrane electrodes instead.

d. Beakers: Preferably use polyethylene or TFE* beakers.

e. Stirrer: Use either a magnetic, TFE-coated stirring bar or a mechanical stirrer with inert plastic-coated impeller.

f. Flow chamber: Use for continuous flow measurements or for poorly buffered solutions.

3. Reagents

a. General preparation: Calibrate the electrode system against standard buffer solutions of known pH. Because buffer solutions may deteriorate as a result of mold growth or contamination, prepare fresh as needed for accurate work by weighing the amounts of chemicals specified in Table 4500-H$^+$:I, dissolving in distilled water at 25°C, and diluting to 1000 mL. This is particularly important for borate and carbonate buffers.

Boil and cool distilled water having a conductivity of less than 2 μmhos/cm. To 50 mL add 1 drop of saturated KCl solution suitable for reference electrode use. If the pH of this test solution is between 6.0 and 7.0, use it to prepare all standard solutions.

Dry KH_2PO_4 at 110 to 130°C for 2 h before weighing but do not heat unstable hydrated potassium tetroxalate above 60°C nor dry the other specified buffer salts.

Although ACS-grade chemicals generally are satisfactory for preparing buffer solutions, use certified materials available from the National Institute of Standards and Technology when the greatest accuracy is required. For routine analysis, use commercially available buffer tablets, powders, or solutions of tested quality. In preparing buffer solutions from solid salts, insure complete solution.

As a rule, select and prepare buffer solutions classed as primary standards in Table 4500-H$^+$:I; reserve secondary standards for extreme situations encountered in wastewater measurements. Consult Table 4500-H$^+$:II for accepted pH of standard buffer solutions at temperatures other than 25°C. In routine use, store buffer solutions and samples in polyethylene bottles. Replace buffer solutions every 4 weeks.

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*Teflon or equivalent.

TABLE 4500-H$^+$:I. PREPARATION OF pH STANDARD SOLUTIONS[1]

Standard Solution (molality)	pH at 25°C	Weight of Chemicals Needed/1000 mL Aqueous Solution at 25°C
Primary standards:		
Potassium hydrogen tartrate (saturated at 25°C)	3.557	> 7 g $KHC_4H_4O_6$*
0.05 potassium dihydrogen citrate	3.776	11.41 g $KH_2C_6H_5O_7$
0.05 potassium hydrogen phthalate	4.004	10.12 g $KHC_8H_4O_4$
0.025 potassium dihydrogen phosphate + 0.025 disodium hydrogen phosphate	6.863	3.387 g KH_2PO_4 + 3.533 g Na_2HPO_4†
0.008 695 potassium dihydrogen phosphate + 0.030 43 disodium hydrogen phosphate	7.415	1.179 g KH_2PO_4 + 4.303 g Na_2HPO_4†
0.01 sodium borate decahydrate (borax)	9.183	3.80 g $Na_2B_4O_7 \cdot 10H_2O$†
0.025 sodium bicarbonate + 0.025 sodium carbonate	10.014	2.092 g $NaHCO_3$ + 2.640 g Na_2CO_3
Secondary standards:		
0.05 potassium tetroxalate dihydrate	1.679	12.61 g $KH_3C_4O_8 \cdot 2H_2O$
Calcium hydroxide (saturated at 25°C)	12.454	> 2 g $Ca(OH)_2$*

* Approximate solubility.

† Prepare with freshly boiled and cooled distilled water (carbon-dioxide free).

2 g Ca(OH)$_2$*

Approximate solubility.

pare with freshly boiled and cooled distilled water (carbon dioxide-free)

TABLE 4500-H$^+$:II. STANDARD pH VALUES'

Temperature °C	Primary Standards							Secondary Standards	
	Tartrate (Saturated)	Citrate (0.05 *M*)	Phthalate (0.05 *M*)	Phosphate (1:1)	Phosphate (1:3.5)	Borax (0.01 *M*)	Bicarbonate-Carbonate (0.025 *M*)	Tetroxalate (0.05 *M*)	Calcium Hydroxide (Saturated)
0			4.003	6.982	7.534	9.460	10.321	1.666	
5			3.998	6.949	7.501	9.392	10.248	1.668	
10			3.996	6.921	7.472	9.331	10.181	1.670	
15			3.996	6.898	7.449	9.276	10.120	1.672	
20			3.999	6.878	7.430	9.227	10.064	1.675	
25	3.557	3.776	4.004	6.863	7.415	9.183	10.014	1.679	12.454
30	3.552		4.011	6.851	7.403	9.143	9.968	1.683	
35	3.549		4.020	6.842	7.394	9.107	9.928	1.688	
37			4.024	6.839	7.392	9.093			
40	3.547		4.030	6.836	7.388	9.074	9.891	1.694	
45	3.547		4.042	6.832	7.385	9.044	9.859	1.700	
50	3.549		4.055	6.831	7.384	9.017	9.831	1.707	
55	3.554		4.070					1.715	
60	3.560		4.085					1.723	
70	3.580		4.12					1.743	
80	3.609		4.16					1.766	
90	3.650		4.19					1.792	
95	3.674		4.21					1.806	

b. Saturated potassium hydrogen tartrate solution: Shake vigorously an excess (5 to 10 g) of finely crystalline $KHC_4H_4O_6$ with 100 to 300 mL distilled water at 25°C in a glass-stoppered bottle. Separate clear solution from undissolved material by decantation or filtration. Preserve for 2 months or more by adding one thymol crystal (8 mm diam) per 200 mL solution.

c. Saturated calcium hydroxide solution: Calcine a well-washed, low-alkali grade $CaCO_3$ in a platinum dish by igniting for 1 h at 1000°C. Cool, hydrate by slowly adding distilled water with stirring, and heat to boiling. Cool, filter, and collect solid $Ca(OH)_2$ on a fritted glass filter of medium porosity. Dry at 110°C, cool, and pulverize to uniformly fine granules. Vigorously shake an excess of fine granules with distilled water in a stoppered polyethylene bottle. Let temperature come to 25°C after mixing. Filter supernatant under suction through a sintered glass filter of medium porosity and use filtrate as the buffer solution. Discard buffer solution when atmospheric CO_2 causes turbidity to appear.

d. Auxiliary solutions: 0.1*N* NaOH, 0.1*N* HCl, 5*N* HCl (dilute five volumes 6*N* HCl with one volume distilled water), and acid potassium fluoride solution (dissolve 2 g KF in 2 mL conc H_2SO_4 and dilute to 100 mL with distilled water).

4. Procedure

a. Instrument calibration: In each case follow manufacturer's instructions for pH meter and for storage and preparation of electrodes for use. Recommended solutions for short-term storage of electrodes vary with type of electrode and manufacturer, but generally have a conductivity greater than 4000 μmhos/cm. Tap water is a better substitute than distilled water, but pH 4 buffer is best for the single glass electrode and saturated KCl is preferred for a calomel and Ag/AgCl reference electrode. Saturated KCl is the preferred solution for a combination electrode. Keep electrodes wet by returning them to storage solution whenever pH meter is not in use.

Before use, remove electrodes from storage solution, rinse, blot dry with a soft tissue, place in initial buffer solution, and set the isopotential point (¶ 2*a* above). Select a second buffer within 2 pH units of sample pH and bring sample and buffer to same temperature, which may be the room temperature, a fixed temperature such as 25°C, or the temperature of a fresh sample. Remove electrodes from first buffer, rinse thoroughly with distilled water, blot dry, and immerse in second buffer. Record temperature of measurement and adjust temperature dial on meter so that meter indicates pH value of buffer at test temperature (this is a slope adjustment).

Use the pH value listed in the tables for the buffer used at the test temperature. Remove electrodes from second buffer, rinse thoroughly with distilled water and dry electrodes as indicated above. Immerse in a third buffer below pH 10, approximately 3 pH units different from the second; the reading should be within 0.1 unit for the pH of the third buffer. If the meter response shows a difference greater than 0.1 pH unit from expected value, look for trouble with the electrodes or potentiometer (see ¶s 5*a* and *b* below).

The purpose of standardization is to adjust the response of the glass electrode to the instrument. When only occasional pH measurements are made standardize instrument before each measurement. When frequent measurements are made and the instrument is stable standardize less frequently. If sample pH values vary widely, standardize for each sample with a buffer having a pH within 1 to 2 pH units of the sample.

b. Sample analysis: Establish equilibrium between electrodes and sample by stirring sample to insure homogeneity; stir gently to minimize carbon dioxide entrainment. For buffered samples or those of high ionic strength, condition electrodes after clean-

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ing by dipping them into sample for 1 min. Blot dry, immerse in a fresh portion of the same sample, and read pH.

With dilute, poorly buffered solutions, equilibrate electrodes by immersing in three or four successive portions of sample. Take a fresh sample to measure pH.

5. Trouble Shooting

a. Potentiometer: To locate trouble source disconnect electrodes and, using a short-circuit strap, connect reference electrode terminal to glass electrode terminal. Observe change in pH when instrument calibration knob is adjusted. If potentiometer is operating properly, it will respond rapidly and evenly to changes in calibration over a wide scale range. A faulty potentiometer will fail to respond, will react erratically, or will show a drift upon adjustment. Switch to the millivolt scale on which the meter should read zero. If inexperienced, do not attempt potentiometer repair other than maintenance as described in instrument manual.

b. Electrodes: If potentiometer is functioning properly, look for the instrument fault in the electrode pair. Substitute one electrode at a time and cross-check with two buffers that are about 4 pH units apart. A deviation greater than 0.1 pH unit indicates a faulty electrode. Glass electrodes fail because of scratches, deterioration, or accumulation of debris on the glass surface. Rejuvenate electrode by alternately immersing it three times each in 0.1*N* HCl and 0.1*N* NaOH. If this fails, immerse tip in KF solution for 30 s. After rejuvenation, soak in pH 7.0 buffer overnight. Rinse and store in pH 7.0 buffer. Rinse again with distilled water before use. Protein coatings can be removed by soaking glass electrodes in a 10% pepsin solution adjusted to pH 1 to 2.

To check reference electrode, oppose the emf of a questionable reference electrode against another one of the same type that is known to be good. Using an adapter, plug good reference electrode into glass electrode jack of potentiometer; then plug questioned electrode into reference electrode jack. Set meter to read millivolts and take readings with both electrodes immersed in the same electrolyte (KCl) solution and then in the same buffer solution. The millivolt readings should be 0 ± 5 mV for both solutions. If different electrodes are used, i.e., silver: silver-chloride against calomel or vice versa, the reading will be 44 ± 5 mV for a good reference electrode.

Reference electrode troubles generally are traceable to a clogged junction. Interruption of the continuous trickle of electrolyte through the junction causes increase in response time and drift in reading. Clear a clogged junction by applying suction to the tip or by boiling tip in distilled water until the electrolyte flows freely when suction is applied to tip or pressure is applied to the fill hole. Replaceable junctions are available commercially.

6. Precision and Bias

By careful use of a laboratory pH meter with good electrodes, a precision of ±0.02 pH unit and an accuracy of ±0.05 pH unit can be achieved. However, ±0.1 pH unit represents the limit of accuracy under normal conditions, especially for measurement of water and poorly buffered solutions. For this reason, report pH values to the nearest 0.1 pH unit. A synthetic sample of a Clark and Lubs buffer solution of pH 7.3 was analyzed electrometrically by 30 laboratories with a standard deviation of ±0.13 pH unit.

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4500-I IODINE*

4500-I A. Introduction

1. Uses and Forms

Iodine may be used to disinfect potable and swimming pool waters. For wastewaters, iodine has had limited application. Use of iodine generally is restricted to personal or remote water supplies where ease of application, storage stability, and an inertness toward organic matter are important considerations. Some swimming pool waters are treated with iodine to lessen eye burn among swimmers and to provide a stable disinfectant residual less affected by adverse environmental conditions.

Iodine is applied in the elemental form or produced in situ by the simultaneous addition of an iodide salt and a suitable oxidant. In the latter case, an excess of iodide may be maintained to serve as a reservoir for iodine production; the determination of iodide is desirable for disinfectant control (see Iodide, Section 4500-I⁻).

Because of hydrolysis, active iodine exists in the forms of elemental I_2, hypoiodous acid (HOI) or a form thereof, hypoiodite ion (OI^-), and, in the presence of excess iodide, the triiodide ion (I_3^-). Most analytical methods use the oxidizing power of all forms of active iodine for its determination and the results usually are expressed as an equivalent concentration of elemental iodine.

2. Selection of Method

For potable and swimming pool waters treated with elemental iodine, both the am-

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*Approved by Standard Methods Committee, 1988.

pH

Method 150.1 (Electrometric)

STORET NO.
Determined on site 00400
Laboratory 00403

1. Scope and Application
   1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.
2. Summary of Method
   2.1 The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.
3. Sample Handling and Preservation
   3.1 Samples should be analyzed as soon as possible preferably in the field at the time of sampling.
   3.2 High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.
4. Interferences
   4.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.
   4.2 Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.
   4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with hydrochloric acid (1 + 9) may be necessary to remove any remaining film.
   4.4 Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample dependent and cannot be controlled, it should therefore be noted by reporting both the pH and temperature at the time of analysis.
5. Apparatus
   5.1 pH Meter-laboratory or field model. A wide variety of instruments are commercially available with various specifications and optional equipment.

Approved for NPDES
Issued 1971
Editorial revision 1978

5.2 Glass electrode.

5.3 Reference electrode–a calomel, silver-silver chloride or other reference electrode of constant potential may be used.

NOTE 1: Combination electrodes incorporating both measuring and reference functions are convenient to use and are available with solid, gel type filling materials that require minimal maintenance.

5.4 Magnetic stirrer and Teflon-coated stirring bar.

5.5 Thermometer or temperature sensor for automatic compensation.

6. Reagents

6.1 Primary standard buffer salts are available from the National Bureau of Standards and should be used in situations where extreme accuracy is necessary.

6.1.1 Preparation of reference solutions from these salts require some special precautions and handling[1] such as low conductivity dilution water, drying ovens, and carbon dioxide free purge gas. These solutions should be replaced at least once each month.

6.2 Secondary standard buffers may be prepared from NBS salts or purchased as a solution from commercial vendors. Use of these commercially available solutions, that have been validated by comparison to NBS standards, are recommended for routine use.

7. Calibration

7.1 Because of the wide variety of pH meters and accessories, detailed operating procedures cannot be incorporated into this method. Each analyst must be acquainted with the operation of each system and familiar with all instrument functions. Special attention to care of the electrodes is recommended.

7.2 Each instrument/electrode system must be calibrated at a minimum of two points that bracket the expected pH of the samples and are approximately three pH units or more apart.

7.2.1 Various instrument designs may involve use of a "balance" or "standardize" dial and/or a slope adjustment as outlined in the manufacturer's instructions. Repeat adjustments on successive portions of the two buffer solutions as outlined in procedure 8.2 until readings are within 0.05 pH units of the buffer solution value.

8. Procedure

8.1 Standardize the meter and electrode system as outlined in Section 7.

8.2 Place the sample or buffer solution in a clean glass beaker using a sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar.

8.2.1 If field measurements are being made the electrodes may be immersed directly in the sample stream to an adequate depth and moved in a manner to insure sufficient sample movement across the electrode sensing element as indicated by drift free (< 0.1 pH) readings.

8.3 If the sample temperature differs by more than 2°C from the buffer solution the measured pH values must be corrected. Instruments are equipped with automatic or manual

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(1)National Bureau of Standards Special Publication 260.

compensators that electronically adjust for temperature differences. Refer to manufacturer's instructions.

8.4 After rinsing and gently wiping the electrodes, if necessary, immerse them into the sample beaker or sample stream and stir at a constant rate to provide homogeneity and suspension of solids. Rate of stirring should minimize the air transfer rate at the air water interface of the sample. Note and record sample pH and temperature. Repeat measurement on successive volumes of sample until values differ by less than 0.1 pH units. Two or three volume changes are usually sufficient.

9. Calculation

9.1 pH meters read directly in pH units. Report pH to the nearest 0.1 unit and temperature to the nearest °C.

10. Precision and Accuracy

10.1 Forty-four analysts in twenty laboratories analyzed six synthetic water samples containing exact increments of hydrogen-hydroxyl ions, with the following results:

pH Units	Standard Deviation pH Units	Accuracy as	
		Bias, %	Bias, pH Units
3.5	0.10	−0.29	−0.01
3.5	0.11	−0.00	
7.1	0.20	+1.01	+0.07
7.2	0.18	−0.03	−0.002
8.0	0.13	−0.12	−0.01
8.0	0.12	+0.16	+0.01

(FWPCA Method Study 1, Mineral and Physical Analyses)

10.2 In a single laboratory (EMSL), using surface water samples at an average pH of 7.7, the standard deviation was ±0.1.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, 14th Edition, p 460, (1975).
2. Annual Book of ASTM Standards, Part 31, "Water", Standard D1293–65, p 178 (1976).

Designation: D 1293 – 84

Standard Test Methods for pH of Water[1]

This standard is issued under the fixed designation D 1293; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 These test methods cover the determination of pH by electrometric measurement using the glass electrode as the sensor. Two test methods are given as follows:

	Sections
Test Method A—Precise Laboratory Measurement	8 to 15
Test Method B—Routine or Continuous Measurement	16 to 24

1.2 Test Method A covers the precise measurement of pH in water utilizing at least two of seven standard reference buffer solutions for instrument standardization.

1.3 Test Method B covers the routine measurement of pH in water and is especially useful for continuous monitoring. Two buffers are used to standardize the instrument under controlled parameters, but the conditions are somewhat less restrictive than those in Test Method A.

1.4 Neither test method is considered to be adequate for measurement of pH in water whose conductivity is less than about 5 μS/cm. (See Appendixes X1.5.3 and X1.5.4.)

1.5 Precision and bias data were obtained using buffer solutions only. It is the user's responsibility to assure the validity of these test methods for untested types of water.

1.6 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1066 Practice for Sampling Steam[2]

D 1067 Test Methods for Acidity or Alkalinity of Water[2]

D 1129 Definitions of Terms Relating to Water[2]

D 1192 Specification for Equipment for Sampling Water and Steam[2]

D 1193 Specification for Reagent Water[2]

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[2]

D 3370 Practices for Sampling Water[2]

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode[3]

3. Terminology

3.1 For definitions of terms used in these test methods, refer to Definitions D 1129.

3.2 *Description of Term Specific to This Standard:*

3.2.1 *pH*—the pH of an aqueous solution is derived from E, the electromotive force (emf) of the cell

glass electrode | solution ‖ reference electrode

(where the double vertical line represents a liquid junction) when the electrodes are immersed in the solution in the diagrammed position, and E_s is the electromotive force obtained when the electrodes are immersed in a reference buffer solution. For use in the operational definition, the sign of the measured potential difference indicated by many pH meters must be reversed because the electrode configuration used with these meters is the following:

reference electrode ‖ solution | glass electrode

With the assigned pH of the reference buffer desig pH$_s$, and E and E_s expressed in volts is the following:

$$\mathrm{pH} = \mathrm{pH}_s - \frac{(E - E_s)F}{2.3026\,RT}$$

where:

F = Faraday,

R = gas constant, and

T = absolute temperature, t(°C) + 273.15.

Values of the factor $F/(2.3026\,RT)$ at different temperatures are given in Table 1. (See Appendix X1.1 for comment on the meaning of pH.)

4. Summary of Test Method

4.1 The pH meter and associated electrodes are standardized against two reference buffer solutions that closely bracket the anticipated sample pH. The sample measurement is made under strictly controlled conditions and prescribed techniques.

5. Significance and Use

5.1 The determination of pH in water is an indication of its acidic or alkaline tendency. It is not a measure of the acidity or alkalinity of a water sample. (Refer to Test Methods D 1067 and Appendix X1.) A pH value less than 7.0 shows a tendency toward acidity while a value greater than 7.0 shows a tendency toward alkalinity. Most natural waters range between 6.0 and 9.0, but there are r exceptions, such as mine drainage water and unbuffered rain water. The pH measurement is an important consideration in determining the corrosive action of water and assessing water treatment practices for industrial processes. It is also

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[1] These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.11 on Water for Power Generation and Process Use.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 15.05.

TABLE 1 $F/(2.3026\ RT)^A$

Temperature, °C	$F/(2.3026\ RT)$, V−1
0	18.4512
5	18.1195
10	17.7996
15	17.4907
20	17.1924
25	16.9041
30	16.6253
35	16.3555
40	16.0944
45	15.8414
50	15.5963
55	15.3587
60	15.1282
65	14.9045
70	14.6873
75	14.4764
80	14.2714
85	14.0722
90	13.8784
95	13.6899

[A] The above data were calculated using a precise value of the logarithmic conversion factor (2.302585) and values of the fundamental constants.

$F = 96\ 487.0$ C/eq
$R = 8.31433$ J/K·mol
$T = 273.15 +$°C

used to assess the extent of pollution in precipitation.

6. Purity of Reagents

6.1 Reagent grade chemicals shall be used in all tests, except as specifically noted for preparation of reference buffer solutions. Unless otherwise indicated, it is intended reagents shall conform to the specifications of the ttee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type I.

7. Sampling

7.1 Collect samples in accordance with Practice D 1066 or Specifications D 1192 or D 3370, whichever is applicable.

TEST METHOD A—PRECISE LABORATORY MEASUREMENT OF pH

8. Scope

8.1 This test method covers the precise measurement of pH in water under strictly controlled laboratory conditions.

9. Interferences

9.1 The glass electrode reliably measures pH in nearly all aqueous solutions and in general is not subject to solution interference from color, turbidity, colloidal matter, oxidants, or reductants.

9.2 The reference electrode may be subject to interferences and should be chosen to conform to all requirements of Sections 10 and 12. Refer also to Appendix X1.3.

9.3 The true pH of an aqueous solution or extract is affected by the temperature. The electromotive force between the glass and the reference electrode is a function of temperature as well as pH. The temperature effect can be compensated for automatically in many instruments or can be manually compensated for in most other instruments. The temperature compensation corrects for the effect of the water temperature on the instrument, including the electrodes, but does not correct for temperature effects on the chemical system being monitored. It does not adjust the measured pH to a common temperature; therefore, the temperature should be reported for each pH measurement. Temperature effects are discussed further in Appendix X1.2.

9.4 The pH response of most glass electrodes is imperfect at both ends of the pH scale. The indicated pH value of highly alkaline solutions, as measured with the glass electrode, will be too low. The indicated pH value of strong aqueous solutions of salts and strong acids having a pH less than 1, will often be higher than the true pH value. Interferences can be minimized by the selection of the proper glass and reference electrodes for measurements in highly alkaline or acidic solutions.

9.5 A few substances sometimes dispersed in water appear to poison the glass electrode. A discussion of this subject is given in Appendix X1.4.

10. Apparatus

10.1 *Laboratory pH Meter*—Table 2 gives the most important characteristics of four typical pH meters commercially available. Each type of meter (together with its associated glass and reference electrodes) is permissible. The choice will depend on the desired precision of measurement.

10.2 *Glass Electrode*—The pH response of the glass electrode shall conform to the requirements set forth in 12.1 through 12.5. The glass electrode lead wire shall be shielded. New glass electrodes and those that have been stored dry shall be conditioned and maintained as recommended by the manufacturer.

10.3 *Reference Electrode*—A calomel, silver-silver chloride or other reference electrode of constant potential shall be used. There are two basic types of reference electrodes: the flowing and the nonflowing liquid junction-type electrodes. The flowing liquid junction-type unit ensures that a fresh liquid junction is formed for each measurement and shall be used for Test Method A determinations. The temperature response of the internal elements of the reference and glass electrode should be closely matched to avoid thermal hysteresis. If a saturated calomel electrode is used, some potassium chloride crystals shall be contained in the saturated potassium chloride solution. If the reference electrode is of the flowing junction type, the design of the electrode shall permit a fresh liquid junction to be formed between the reference electrode solution and the buffer standard or tested water at each measurement and shall allow traces of solution to be washed from the outer surfaces of the electrodes. To ensure the desired slow outward flow of reference electrode solution, the solution pressure inside the liquid junction should be kept somewhat in excess of that outside the junction.

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[4] gent Chemicals, American Chemical Society Specifications," Am. Chem- ... Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

TABLE 2 Laboratory pH Meter

[illegible]	Type I	Type II	Type III	Type IV
[illegible] normal	0 to 14	0 to 14	0 to 14	0 to 14 000
[illegible] expanded		2 pH units	1.4 pH units	
[illegible] division	0.1	0.01	0.01	0.001
Accuracy	±0.05	±0.01	±0.007	±0.002
Repeatability	±0.02	±0.005	±0.002	±0.002
Temperator compensation:				
Manual or automatic	yes	yes	yes	yes
Range, °C	0 to 100	0 to 100	0 to 100	0 to 100
Smallest graduation, °C	2	2	2	2
[illegible] compensator	–	yes	yes	yes

nonpressurized applications, this requirement can be met by maintaining the inside solution level higher than the outside water level. If the reference electrode is of the nonflowing junction type, these outward flow and pressurization considerations do not apply. The reference electrode and junction shall perform satisfactorily as required in the standardizing procedure described in 12.1 through 12.5. A discussion of reference electrodes is given in Appendix X1.3.

10.4 *Temperature Compensator*—The thermocompensator is a temperature-sensitive resistance element immersed in the water sample with the electrodes. The thermocompensator automatically corrects for the change in slope of the glass electrode (with change of temperature) but does not correct for actual changes in sample pH with temperature. The automatic thermocompensator is not required if the water temperature is essentially constant and the analyst chooses to use the manual temperature compensation feature of the pH meter.

11. Reagents

11.1 *Reference Buffer Solutions*—The pH values of the reference buffer solutions measured at several temperatures are listed in Table 3. Table 4 identifies each buffer salt by its National Institute of Standards and Technology (NIST) number and provides a recommended drying procedure prior to use. Keep the five reference buffer solutions with pH less than 9.5 in bottles of chemically resistant glass. Keep the calcium hydroxide solutions in a plastic bottle that is nonporous to air (that is, polypropylene or high density polyethylene). Keep all the reference buffer solutions well-stoppered and replace at a shelf age of 3 months, or sooner if a visible change is observed.

11.1.1 *Borax Reference Buffer Solution* (pH$_s$ = 9.18 at 25°C)—Dissolve 3.80 g of sodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) in water and dilute to 1 L.

11.1.2 *Calcium Hydroxide Reference Buffer Solution* (pH$_s$ = 12.45 at 25°C)—Prepare pure calcium hydroxide ($Ca(OH)_2$) from well-washed calcium carbonate ($CaCO_3$) of low-alkali grade by slowly heating the carbonate in a platinum dish at 1000°C and calcining for at least 45 min at that temperature. After cooling, add the calcined product slowly to water with stirring, heat the resultant suspension to boiling, cool, and filter through a funnel having a fritted-glass disk of medium porosity. Collect the solid from the filt [illegible] it in an oven at 110°C, and crush it to a uniform an [illegible] granular state. Prepare a saturated calcium hydroxide solution by vigorously shaking a considerable excess (about 3 g/L) of the fine granular product in water at 25°C in a stoppered plastic bottle (that is, polypropylene or high density polyethylene) that is essentially nonporous to gases. Allow the gross excess of solid to settle and filter the solution with suction through a fritted-glass funnel of medium porosity. The filtrate is the reference buffer solution. Contamination of the solution with atmospheric carbon dioxide

TABLE 3 pH$_s$ of Reference Buffer Solutions[A]

Temperature, °C	Tetroxalate Solution	Tartrate Solution	Phthalate Solution	Phosphate Solution	Borax Solution	Sodium Bicarbonate Sodium Carbonate	Calcium Hydroxide Solution
0	1.67	...	4.00	6.98	9.46	10.32	13.42
5	1.67	...	4.00	6.95	9.39	10.25	13.21
10	1.67	...	4.00	6.92	9.33	10.18	13.00
15	1.67	...	4.00	6.90	9.28	10.12	12.81
20	1.68	...	4.00	6.88	9.23	10.06	12.63
25	1.68	3.56	4.00	6.86	9.18	10.01	12.45
30	1.68	3.55	4.01	6.85	9.14	9.97	12.29
35	1.69	3.55	4.02	6.84	9.11	9.93	12.13
40	1.69	3.55	4.03	6.84	9.07	9.89	11.98
45	1.70	3.55	4.04	6.83	9.04	9.86	11.84
50	1.71	3.55	4.06	6.83	9.02	9.83	11.71
55	1.72	3.55	4.07	6.83	8.99	...	11.57
60	1.72	3.56	4.09	6.84	8.96	...	11.45
70	1.74	3.58	4.12	6.85	8.92	...	...
80	1.77	3.61	4.16	6.86	8.89	...	...
90	1.79	3.65	4.19	6.88	8.85	...	...
95	1.81	3.67	4.21	6.89	8.83	...	...

[A] For a discussion of the manner in which these pH values were assigned, see Bates, R. G., "Revised Standard Values for pH Measurements from 0 to 95°C," *Journal of Research*, NIST, Vol 66A, 1962, p. 179.

TABLE 4 National Institute of Standards and Technology (NIST) Materials for Reference Buffer Solutions

NIST Standard Sample Designation	Buffer Salt[A]	Drying Procedure
187-b	Borax (sodium tetraborate decahydrate)	Drying not necessary (this salt should not be oven-dr
186-II-c	disodium hydrogen phosphate	2 h in oven at 130°
186-I-c	potassium dihydrogen phosphate	2 h in oven at 130°C
185-e	potassium hydrogen phthalate	2 h in oven at 110°C
188	potassium hydrogen tartrate	drying not necessary
189	potassium tetroxalate dihydrate	should not be dried
191	sodium bicarbonate	should not be dried
192	sodium carbonate	2 h in oven at 275°C

[A] The buffer salts listed can be purchased from the Office of Standard Reference Materials, National Institute of Standards and Technology, Washington, DC 202

enders it turbid and indicates need for replacement.

11.1.3 *Phosphate Reference Buffer Solution* (pH$_s$ = 6.86 at 25°C)—Dissolve 3.39 g of potassium dihydrogen phosphate (KH$_2$PO$_4$) and 3.53 g of anhydrous disodium hydrogen phosphate (Na$_2$HPO$_4$) in water and dilute to 1 L.

11.1.4 *Phthalate Reference Buffer Solution* (pH$_s$ = 4.00 at 25°C)—Dissolve 10.12 g of potassium hydrogen phthalate (KHC$_8$H$_4$O$_4$) in water and dilute to 1 L.

11.1.5 *Tartrate Reference Buffer Solution* (pH$_s$ = 3.56 at 25°C)—Shake vigorously an excess (about 75 g/L) of potas-ium hydrogen tartrate (KHC$_4$H$_4$O$_6$) with 100 to 300 mL of ater at 25°C in a glass-stoppered bottle. Filter, if necessary, o remove suspended salt. Add a crystal of thymol (about 0.1) as a preservative.

11.1.6 *Tetroxalate Reference Buffer Solution* (pH$_s$ = 1.68 25°C)—Dissolve 12.61 g of potassium tetroxalate ihydrate (KHC$_2$O$_4$ · H$_2$C$_2$O$_4$ · 2H$_2$O) in water and dilute to 1

Sodium Bicarbonate—Sodium Carbonate Refer-fer Solution (pH$_s$ = 10.01 at 25°C)—Dissolve 2.092 of sodium bicarbonate (NaHCO$_3$) and 2.640 g of sodium arbonate (Na$_2$CO$_3$) in water and dilute to 1 L.

11.2 *Other Buffer Solutions*—A buffer solution other than at specified may be used as a working standard in the ethod providing that in each case such a solution is first ecked against the corresponding reference buffer solution, sing the procedures of the method, and is found to differ by ot more than 0.02 pH unit.

11.3 *Commercial Buffer Solutions*—Commercially avail-ble prepared buffer solutions are not acceptable for the andardization in Test Method A.

2. Standardization of Assembly

12.1 Turn on the instrument, allow it to warm up oroughly, and bring it to electrical balance in accordance ith the manufacturer's instructions. Wash the glass and ference electrodes and the sample container with three anges of water or by means of flowing stream from a wash ttle. Form a fresh liquid junction if a sleeve-type reference nction is used. Note the temperature of the water to be sted. If temperature compensation is to be manual, adjust e temperature dial of the meter to correspond to the mperature of the water to be tested and allow time for all ffer utions, and electrodes to equilibrate thermally.

elect at least two reference buffer solutions, the pH$_s$ of which bracket the anticipated pH (refer to Table 3). arm or cool the reference solutions as necessary to match thin 2°C the temperature of the solution to be tested. Fill the sample container with the first reference buffer solutio and immerse the electrodes.

12.3 Set the pH$_s$ value of the reference buffer solution a the temperature of the buffer, as read from Table 3 interpolated from the data therein, according to the ma facturer's instructions.

12.4 Empty the sample container and repeat, using su cessive portions of the reference buffer solution, until tw successive readings are obtained without adjustment of t system. These readings should differ from the pH$_s$ value o the buffer solution by not more than 0.02 pH unit.

NOTE 1—If the temperature of the electrode differs appreciably fr that of the solution to be tested, use several portions of solution an immerse the electrodes deeply to assure that both the electrodes and th solution are at the desired temperature. To reduce the effects of therm lag, keep the temperature of electrodes, reference buffer solutions, an the wash as close to that of the water sample as possible.

12.5 Wash the electrodes and the sample container thre times with water. Place the second reference buffer solutio in the sample container, and measure the pH. Adjust the slope control only until the reading corresponds to the temperature corrected value of the second reference buffe solution. Use additional portions of the second referenc buffer solution, as before, until two successive readings diffe by not more than 0.02 pH unit. The assembly shall be judge to be operating satisfactorily if the pH reading obtained fo the second reference buffer solution agrees with its assigne pH$_s$ value within 0.05 pH unit for Type I meters or withi 0.03 pH unit for Type II, III, or IV meters (see Table 2).

12.6 If only an occasional pH determination is made standardize the assembly each time it is used. In a long serie of measurements, supplemental interim checks at regular intervals are recommended. Inasmuch as commerciall available pH assemblies exhibit different degrees of measure ment stability, conduct these checks at intervals of 30 min unless it is ascertained that less frequent checking is satisfac tory to ensure the performance described in 12.2 to 12.5.

13. Procedure

13.1 Standardize the assembly with two reference buffe solutions as described in 12.2 to 12.5 and then wash the electrodes with three changes of water or by means of a flowing stream from a wash bottle.

13.2 Place the water sample in a clean glass beake provided with a thermometer and a stirring bar.

13.3 Stir during the period of pH measurement at a rate that will prevent splashing and that will avoid loss or gain of acidic or basic gases by interchange with the atmosphere. When necessary, stir briskly enough to intermix the phases of

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a nonhomogeneous water sample. Stop the stirrer during the period of measurement if streaming potentials are noted.

13.4 Insert the electrodes and determine a preliminary pH value (since this value may drift somewhat, it should be considered an estimated value). Measure successive portions of the water sample until readings on two successive portions differ by no more than 0.03 pH unit, and show drifts of less than 0.02 pH unit in 1 min. Two or three portions will usually be sufficient if the water is well buffered.

13.5 Record the pH and temperature of the sample.

13.6 Measure the pH of slightly buffered waters (that are in equilibrium with air) essentially as described in 13.1 to 13.5, but measure the pH of successive portions until the readings for two successive portions differ by no more than 0.1 pH unit. Six or more portions may be necessary.

NOTE 2—Take special precautions if the sample is not in equilibrium with the carbon dioxide of the atmosphere protecting the sample from exposure to the air during measurement. *Measurement of unbuffered or slightly buffered samples is more reliably made in flow-type cells as described in NOTES 4 and 5.* Appendix X1.5.3 describes additional precautions that should be taken if the electrical conductivity of the sample is less than about 5 µS/cm.

14. Report

14.1 Report the temperature of the measurement of the nearest 1°C.

14.2 Report the pH of the test solution to the nearest 0.01 pH unit when the pH measurement lies between 1.0 and 12.0.

14.3 Report the pH of the test solution to the nearest 0.1 pH unit when the pH measurement is less than 1.0 and greater than 12.0.

15. Precision and Bias[5]

15.1 The information summarized in this section was derived from an interlaboratory study performed in 1973 on four buffer solutions having pH values of approximately 3.7, 6.5, 8.2, and 8.4. Eleven laboratories (fourteen operators, with one laboratory providing four operators) analyzed each solution in duplicate and replicated the analysis on another day for a total of 224 determinations. A variety of commercial meters was used in this study. It is assumed that all measurements were made at room temperature.

15.2 Statistical treatment of the data conforms to the recommendations of Practice D 2777. Further information, based on a different statistical interpretation, can be found in Test Method E 70.

15.3 *Precision*—The overall and single-operator precision of this test method varies with pH as shown in Fig. 1.

15.4 *Bias*—The pH values of the buffer solutions, as determined using a gaseous hydrogen electrode, are compared with values obtained using this test method in Table 5.

15.5 Precision and bias data were obtained using buffer solutions only. It is the user's responsibility to assure the validity of the standards for untested types of water.

TEST METHOD B—ROUTINE OR CONTINUOUS MEASUREMENT OF pH

16. Scope

16.1 This test method is used for the routine measurement of pH in the laboratory and the measurement of pH under various process conditions.

17. Summary of Test Method

17.1 A direct standardization technique is employed in this test method for routine batch samples. Two buffers are used to standardize the instrument under controlled parameters, but the conditions are somewhat less restrictive than those in Test Method A. An indirect standardization procedure is used on flowing systems in which grab samples are removed periodically in order to compare a monitored pH value (of the system) with the reading of a laboratory pH meter.

18. Interferences

18.1 For information on interferences, see Section 9 and Appendix X1.4.

19. Apparatus

19.1 *Laboratory pH Meter*—See 10.1.

19.2 *Glass Electrode*—See 10.2.

19.3 *Reference Electrode*—See 10.3.

19.4 *Temperature Compensator*—See 10.4.

19.5 *Process pH Measurement Instrumentation*—Instruments that are used for process pH measurement generally much more rugged than those which are used very accurate measurements in the laboratory. The characteristics of three types of process pH analyzers are presented in Table 6. Each of these analyzers is satisfactory for process pH measurements. The choice of analyzer is generally based on how closely the characteristics of the analyzer match the requirements of the application.

19.5.1 *Electrode Chamber*—For process pH measurements; the electrodes and thermocompensator are mounted in an electrode chamber or cell.

19.5.1.1 *Flow-Through Chamber* completely encloses the electrodes and the sample is piped to and from the chamber in a flow-through configuration. Commercially available chambers generally can tolerate temperatures as high as 100°C over a pH range from 0 to 14, and pressures up to 1034 kPa (approximately 150 psi).

19.5.1.2 *Immersion Type Chamber*, suitable for measurement in open streams or tanks, shields but does not completely enclose the electrodes. Immersion-style chambers are available for use at depths to 30 m (100 ft).

19.5.2 *Signal Transmission*—The glass electrode is usually a high-impedance device from which only an extremely small current can be drawn. Shielded cable must be used to connect the electrode to the pH analyzer. The signal can frequently be transmitted up to 300 m (approximately 1000 ft) with no loss in accuracy if the manufacturer's recommendations are followed carefully. The signal can usual amplified for transmission over even greater distances.

19.5.3 *pH Meter Associated with Remote Measurement*—Usually a different style instrument is used to indicate or display remote pH measurements. Table 6 shows the characteristics of three types of meters that are suitable.

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[5] Supporting data for these test methods have been filed at ASTM Headquarters. Request Research Report RR: D19-1111.

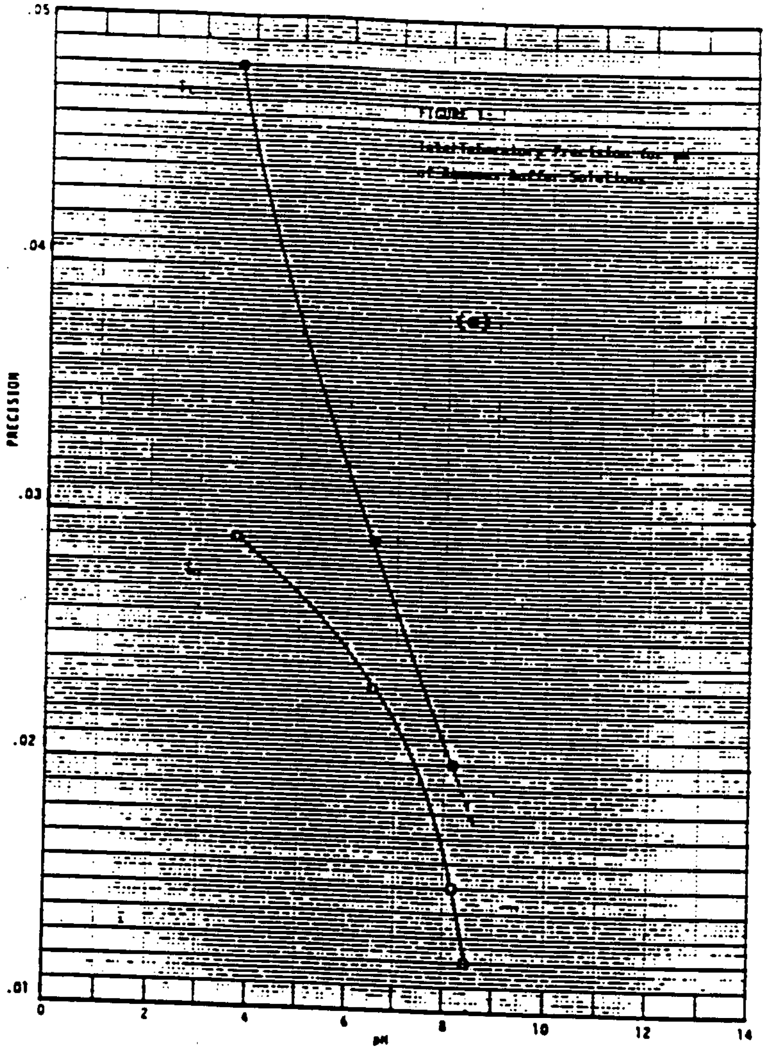

FIG. 1 Interlaboratory Precision for pH of Aqueous Buffer Solutions

. Reagents

20.1 *Commercial Buffer Solutions*—Commercially available prepared buffer should be adequate for the standardizaon in Test Method B. These commercial buffer solutions ually have pH values near 4, 7, and 10, the exact pH and e temperature being provided by the purveyor of the ecific buffer. The pH buffer is susceptible to contaminaon from atmospheric carbon dioxide, and frequently used partially filled bottles are particularly vulnerable to this ror.

20.2 For more information on reagents, see Section 11.

. Standardization of Assembly

21.1 Turn on the analyzer, allow it to warm up thorghly and bring it to electrical balance in accordance with e manufacturer's instructions. Wash the electrodes, the ermocompensator, and the sample container with three ang[illegible] water or by means of flowing stream for a wash [illegible]rm a fresh liquid junction if a sleeve reference junction is used. If manual temperature compensaon is to be used, note the temperature of the water sample d adjust the temperature dial of the meter to correspond.

21.2 *Direct Standardization:*

21.2.1 Select two reference buffer solutions that have pH_s values that bracket the anticipated pH of the water sample. Warm or cool the reference solution to within 2°C of the temperature of the water sample.

21.2.2 Fill the sample container with the first reference buffer solution and immerse the electrodes. Set the known pH_s of the reference buffer solution according to the instrument manufacturer's instructions. Repeat with successive portions of the reference buffer solution until two successive instrument readings are obtained which differ from the pH_s value of the buffer solution by no more than 0.02 pH unit.

21.2.3 Wash the electrodes and sample container three times with water. Place the second reference buffer solution in the sample container, and measure the pH. Adjust the slope control only until the reading corresponds to the temperature corrected value of the second reference buffer

TABLE 5 Determination of Bias

pH Expected	pH Found	Bias, ± %	Statistically Significant (95 % Confidence Level)
3.714	3.73	+0.48	No
6.517	6.53	+0.20	Yes
8.147	8.19	+0.53	Yes
8.470	8.45	−0.24	Yes

TABLE 6 Remote Measurement of pH

	Type I	Type II	Type III
Range—normal	0 to 14	0 to 14	0 to 14
expanded	any 2 pH or wider units	any 2, 4, 5, 7, 10, or 14 pH units	any 2, 5, or 10 units
Signal to recorder	to 1000 mV	to 50 mA	to 5000 mV, 4 to 20 or 10 to 50 mA
Accuracy	±0.02	± 0.005 to 0.04 dependent upon span	±0.02
Temperature compensation:			
Manual or automatic	yes	yes	yes
Range	0 to 100°C	0 to 100°C	0 to 100°C
Stability	0.005 pH/24 h	0.005 pH/24 h	±0.02 pH units/24 h

solution. Use additional portions of the second reference buffer solution, as before, until two successive readings differ by not more than 0.02 pH unit.

21.2.4 If only an occasional pH determination is made, standardize the assembly each time it is used. In a long series of measurements, supplement initial and final standardizations by interim checks at regular intervals. As commercially available, pH assemblies exhibit different degrees of measurement stability, conduct these checks at intervals of 30 min, unless it is ascertained that less frequent checking is satisfactory to ensure performance.

21.3 *Indirect Standardization:*

21.3.1 This procedure is to be employed when it is not convenient or practical to remove the electrodes from the flowing stream or container on which the pH is being determined. Use of a laboratory pH meter or an additional analyzer is required.

21.3.2 Standardize the laboratory pH meter or additional process analyzer as outlined in 21.2.

21.3.3 Collect a grab sample of the water from the immediate vicinity of the electrodes or from the discharge of a flow-through chamber. Measure the pH of this grab sample immediately, using the standardized laboratory pH meter.

21.3.4 Adjust the standardization control on the process analyzer until the reading corresponds to the pH of the grab sample. Repeat the grab sampling, analyzing, and adjusting procedure until two successive readings are obtained that differ by no more than 0.05 pH unit or within an acceptable accuracy.

NOTE 3—Indirect standardization as described above cannot be employed when the pH of the water being tested fluctuates by more than 0.05 pH unit. The standardization must be accomplished in the shortest possible time if the pH is fluctuating. It is absolutely essential that the grab sample be representative of the water in contact with the electrodes of the analyzer being standardized. The integrity of the grab sample must be maintained until its pH has been measured by the standardized meter, and its temperature must remain constant.

21.3.5 Indirect standardization is a one-point calibration and does not establish the proper response of the electrodes over a pH range.

22. Procedure, Batch Samples

22.1 Standardize the assembly as described in 21.2 and wash the electrodes with three changes of water or by means of a flowing stream from a wash bottle.

22.2 Place the water sample in a clean glass beaker provided with a thermometer and a stirring bar. Stir during the period of pH measurement at a rate that will prevent splashing and that will avoid loss or gain of acidic or basic gases by interchange with the atmosphere. When necessary, stir briskly enough to intermix the phases of a nonhomogeneous water sample.

22.3 Insert the electrodes and determine a preliminary pH value (the reading may drift). Measure successive portions of the water sample until readings on two successive portions differ by no more than 0.05 pH unit. Two portions will usually be sufficient if the water is well-buffered.

22.4 Record the pH and temperature of the sample.

NOTE 4—*Continuous Determination of pH*—Make the selection of the electrodes and the electrode chamber to suit the physical and chemical characteristics of the process water. Locate a submersion style electrode chamber so that fresh representative sampling is provided continuously across the electrodes. Agitation may be required to improve homogeneity. Process pH measurements generally employ automatic temperature compensation. The pH value is usually displayed continuously and can be noted at any specific time. Also, record the successive pH values frequently to provide a permanent record. If the temperature of the sample fluctuates significantly with time, the temperature should also be recorded to interpret the pH values correctly.

NOTE 5—*Continuous Determination of the pH of High-Purity Water*—The analyzer used to determine the pH of high purity water must have a high-impedance differential input in order to minimize environmental effects. Use a metallic electrode chamber in order to shield the electrodes, and electrically isolate it from all stray a-c fields. Pay particular attention to grounding and the use of metallic connectors, as recommended by the equipment manufacturer. Place the reference electrode upstream of the glass electrode to impart the small amount of electrolyte essential to the sensing of the pH of the water sample. One successful flow pattern uses a bottom inlet, a full chamber, and a top overflow; the flow rate and the pressure are held constant and the discharge is to atmospheric pressure. Follow the manufacturer's instructions very closely.

23. Report

23.1 Report the temperature of measurement to the nearest 1°C.

23.2 Report the pH to the nearest 0.1 pH unit.

24. Precision and Bias[5]

24.1 Because of the wide variability in measurement conditions and the changeable character of the pH of many process waters, the precision of this test method is probably less than that of Test Method A; however, a precision of pH unit should be attainable under controlled conditions.

24.2 Precision and bias data were obtained using buffer solutions only. It is the user's responsibility to assure the validity of this test method for untested types of water.

APPENDIX

(Nonmandatory Information)

X1. MISCELLANEOUS NOTES ON GLASS ELECTRODE MEASUREMENTS AND EQUIPMENT

X1.1 Meaning of the Term pH

X1.1.1 The term pH historically has referred to the hydrogen ion activity of a solution and has been expressed as the logarithm to the base 10 of the reciprocal (negative logarithm) of the activity of hydrogen ions at a given temperature, as follows:

$$pH = \log 1/(H^+) = -\log(H^+)$$

where:

(H^+) = activity of hydrogen ions.

X1.1.2 Although this expression is helpful in giving theoretical meaning to the term pH and can be used as an approximate definition, it may not be rigorously related to empirical pH measurements. The definition given in 3.2.1 has gained wide acceptance.

X1.2 Temperature Effects

X1.2.1 The effects of temperature on electrometric pH measurements arise chiefly from two sources: (*1*) temperature effects that are common to all electrometric measurements and (*2*) variations of sample pH with temperature. The first category includes the effect of temperature on the [illegible] $F/2.3026\ RT$ that occurs in the definition of pH (see [illegible] values of this factor for various temperatures are given in Table 1. The first category also includes variations of glass electrode asymmetry potential and reference electrode liquid junction potential and temperature. If the pH measurement is made in accordance with Sections 13 and 21, the effects of these potentials will be negligible.

X1.2.2 Secondly, because of changes in activity coefficients and equilibrium constants with temperature, the pH of a sample will change with temperature. These changes are independent of the method of measurement. In general, the rate of change of pH with temperature is not constant, and it may be positive or negative. The data in Table 3, showing changes in pH_s of buffer solutions with temperature, are typical examples.

X1.3 Reference Electrodes

X1.3.1 In making pH measurements with the glass electrode, the reference electrode used to complete the cell assembly functions simply as a source of reproducible potential. The absolute value of the reference electrode potential is of no consequence owing to the way the measurements are made. Both the saturated calomel and silver-silver-chloride electrodes have proven themselves over the years to be satisfactory reference electrodes. For measurements at normal room temperatures, either of these elec[illegible] is satisfactory and thus are recommended. The [illegible]lver-chloride is recommended for measurements at [illegible]ed temperatures, where its potential is more stable than that of the saturated calomel electrode. Depending on the environmental conditions, other electrodes may serve satisfactorily as reference electrodes.

X1.3.2 If a saturated calomel electrode is used under significantly changeable temperature conditions, care must be taken to see that sufficient solid potassium chloride is present at all the temperatures to ensure solution saturation throughout, both in the free solution in the electrode tube and in the solution permeating the electrode element. The electrode must be given 5 or 10 min to accommodate itself to a new temperature condition before a pH measurement is made. If the temperature falls appreciably, crystallization of potassium chloride may cause plugging of the liquid junction; one result may be high resistance and false or erratic potential at the junction. Any such accumulation of potassium chloride should therefore be removed by aqueous washing.

X1.3.3 Reference electrodes of the unsaturated type have been used preferentially in continuous mechanized pH monitoring where the temperature is likely to fluctuate. The selected potassium chloride concentration is frequently saturation at the lowest temperature of use (for example, approximately 3.3 N for 0°C). Such a reference electrode has the advantage of being free from the annoying effects caused by variable solubility, but take considerable care to prepare the required concentration and to maintain the prescribed value under plant operating conditions. Follow the instrument manufacturer's recommendations on choosing and maintaining reference electrodes. Changing an electrode from the saturated type to the unsaturated type (or vice versa) by changing the concentration of potassium chloride may produce drifting potentials for weeks or even months.

X1.3.4 Reference electrodes are available with any number of means to establish the liquid junction. These include, but are not limited to, dependence on the porosity of wood, fibrous materials, glass-encased noble metal, ground-glass sleeves, ceramic frits, and nonflowing polymeric bodies.

X1.4 Faulty Glass Electrode Response and Restorative Techniques

X1.4.1 *Detecting Faulty Electrodes*—The pH measuring assembly is standardized with two reference buffer solutions (see 12.2) to verify the linearity of response of the electrode combination at different pH values. Standardization also detects a faulty glass or reference electrode or an incorrect temperature compensator. The faulty electrode is indicated by a failure to obtain a reasonably correct value for the pH of the second reference buffer solution after the meter has been standardized with the first. A cracked glass electrode will often yield pH readings that are essentially the same for both standards and should be discarded. Even though a normal glass electrode responds remarkably well to moderate pH changes, it is not necessarily a perfect pH-measuring device, and may miss the rigid requirements of 12.2, if, for example the pH span is made as great as 5 pH units (phthalate to borax).

X1.4.2 *Imperfect pH Response*—The pH response of the glass electrode may be impaired by a few coating substances (certain oily materials or even some particulates). When the faulty condition is disclosed by the check with the two reference buffer solutions, the electrode can frequently be restored to normal by an appropriate cleaning procedure.

X1.4.3 *Glass Electrode Cleaning Techniques*—Where emulsions of free oil and water are to be measured for pH, it is absolutely necessary that the electrodes be cleaned thoroughly after each measurement. This may be done by washing with soap or detergent and water, followed by several rinses with water, after which the lower third of the electrodes should be immersed in HCl (1+9) to remove any film that may have been formed. Rinse the electrode thoroughly by washing it in several changes of water before returning it to service. Process pH analyzers used for continuous measurement may be provided with an ultrasonic cleaner to lessen or even eliminate the need for manual cleaning of electrodes.

X1.4.4 Thorough cleaning with a suitable solvent may be necessary after each measurement if the sample contains stickly soaps or suspended particles. If this fails, a chemical treatment designed to dissolve the particular deposited coating may prove successful. After the final rinsing of the electrode in the cleaning solvent, immerse the lower third of the electrodes in HCl (1+9) to remove a possible residual film. Wash the electrode thoroughly in several changes of water before subjecting it to the standardization procedure.

X1.4.5 If an electrode has failed to respond to the treatment suggested in X1.4.3, try a more drastic measure as a last resort. This drastic treatment, which will limit the life of the electrode and should be used only as an alternative to discarding it, is immersing it in chromic acid cleaning solution for a period of several minutes (or longer if necessary). Chromic acid is particularly effective in cleaning foreign substances from the surface of the glass, but it also has a dehydrating effect on the glass. Consequently allow an electrode so treated, after thoroughly rinsing, to stand in water overnight before using it for measurements. Finally, if the electrode fails to respond to the chromic acid solution, it may be subjected to mild etching in ammonium bifluoride solution. Immerse the electrode for about 1 min in a 20 % solution of ammonium bifluoride (NH_4HF_2) in water, in a polyethylene cup. The bifluoride actually removes a portion of the bulb glass, and should be used only as a last resort (and then only infrequently). Follow the fluoride etch by thorough rinsing and conditioning as is recommended for a new electrode. The electrode manufacturer may have a suggestions. specific to his own product.

X1.4.6 Techniques for cleaning flow cell electrodes clude the use of ultrasonics, brushes, and high-veloc submerged jets.

X1.5 Special Measurements Techniques

X1.5.1 *Measurements on Alkaline Waters*—Although t high-alkalinity electrode exhibits a lower sodium ion err than an ordinary glass electrode, the high-alkalinity electroc can nevertheless introduce an appreciable error when tr sodium ion content and the pH or temperature of the wate are unusually high. Corrections may in some instances b applied to the measurement if suitable data are availab from the electrode manufacturer.

X1.5.2 Carbon dioxide from the air tends to react with a alkaline water and to change its pH. Make all measuremen with alkaline waters or buffer solutions as quickly as possibl with the water exposed to the air no longer than is absolute necessary.

X1.5.3 *pH Measurement of Static Solutions of Low Con ductivity*—When the sample electrical conductivity is lowe than about 5 μS/cm, the electrical resistance of the wate path between the glass and reference electrodes may becom high enough to cause measuring difficulties. If placing th junction of the reference electrode as close as possible to th pH responsive bulb of the glass electrode does not c [illegible] difficulties, the electrode pair should be protect [illegible] electrostatic charges by surrounding the nonconducting cel with a grounded metallic shield. Also helpful is the use of a metallic connector at the inlet and outlet, electrically connected external to the chamber.

X1.5.4 *pH Measurement of Flowing Solutions of Low Conductivity*—In making measurements on flowing samples having an electrical conductivity lower than about 5 μS/cm, take precautions to avoid difficulties from streaming potentials. If available, observe special precautions recommende by the instrument manufacturer. Preferably, make the flow chamber of a conductive material such as stainless steel an ground it. Because streaming potential increases with flow velocity, it is advisable to adjust the sample flow rate at a value not exceeding that needed to keep the flow cell properly flushed. This can be done by conveying the sample water to the flow cell through a short line leading from a constant-head overflow chamber. Provide the line to the cell with suitable flow-controlling means.

X1.5.5 *Magnetic Stirring*—Do not use magnetic stirring in those few instances where it may affect pH measurement.

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4500-O_3 OZONE (RESIDUAL)* (PROPOSED)

4500-O_3 A. Introduction

Ozone, a potent germicide, is used also as an oxidizing agent for the destruction of organic compounds producing taste and odor in drinking water, for the destruction of organic coloring matter, and for the oxidation of reduced iron or manganese salts to insoluble oxides.

4500-O_3 B. Indigo Colorimetric Method

1. General Discussion

The indigo colorimetric method is quantitative, selective, and simple; it replaces methods based on the measurement of total oxidant. The method is applicable to lake water, river infiltrate, manganese-containing groundwaters, extremely hard groundwaters, and even biologically treated domestic wastewaters.

a. Principle: In acidic solution, ozone rapidly decolorizes indigo. The decrease in absorbance is linear with increasing concentration. The proportionality constant at

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*Approved by Standard Methods Committee, 1988.

600 nm is 0.42 ± 0.01/cm/mg/L ($\Delta\epsilon$ = 20 000/*M*·cm) compared to the ultraviolet absorption of pure ozone of ϵ = 2950/*M*·cm at 258 nm).[1]

b. Interferences: Hydrogen peroxide (H_2O_2) and organic peroxides decolorize the indigo reagent very slowly. H_2O_2 does not interfere if ozone is measured in less than 6 h after adding reagents. Organic peroxides may react more rapidly. Fe(III) does not interfere. Mn(II) does not interfere but it is oxidized by ozone to forms that decolorize the reagent. Correct for this interference by making the measurement relative to a blank in which the ozone has been destroyed selectively. Without the corrective procedure, 0.1 mg/L ozonated manganese gives a response of about 0.08 mg/L apparent ozone. Chlorine also interferes but it can be masked by malonic acid. Bromine, which can be formed by oxidation of Br^-, interferes (1 mole HOBr corresponds to 0.4 mole ozone).

c. Minimum detectable concentration: For the spectrophotometric procedure using thermostated cells and a high-quality photometer, the low-range procedure will measure down to 2 µg O_3/L. For the visual method the detection limit is 10 µg/L.

2. Apparatus

a. Photometer: Spectrophotometer or filter colorimeter for use at 600 ± 5 nm.

b. Glass cylinders (for visual procedure): 100-mL graduated glass cylinders, preferably with flat bottoms.

3. Reagents

a. Indigo stock solution: Add about 500 mL distilled water and 1 mL conc phosphoric acid to a 1-L volumetric flask. With stirring, add 770 mg potassium indigo trisulfonate, $C_{16}H_7N_2O_{11}S_3K_3$ (commercially available at about 80 to 85% purity). Fill to mark with distilled water. A 1:100 dilution exhibits an absorbance of 0.20 ± 0.010 cm at 600 nm. The stock solution is stable for about 4 months when stored in the dark. Discard when absorbance of a 1:100 dilution falls below 0.16/cm.

b. Indigo reagent I: To a 1-L volumetric flask add 20 mL indigo stock solution, 10 g sodium dihydrogen phosphate (NaH_2PO_4), and 7 mL conc phosphoric acid. Dilute to mark. Prepare solution fresh when its absorbance decreases to less than 80% of its initial value, typically within a week.

c. Indigo reagent II: Proceed as with indigo reagent I, but add 100 mL indigo stock solution instead of 20 mL.

d. Malonic acid reagent: Dissolve 5 g malonic acid in water and dilute to 100 mL.

e. Glycine reagent: Dissolve 7 g glycine in water and dilute to 100 mL.

4. Procedure

a. Spectrophotometric procedure:

1) Concentration range 0.01 to 0.1 mg O_3/L—Add 10.0 mL indigo reagent I to two 100-mL volumetric flasks. Fill one flask (blank) to mark with distilled water. Fill other flask to mark with sample. Add sample so that completely decolorized zones are eliminated quickly by stirring but no ozone degassing occurs. Measure absorbance of both solutions at 600 ± 5 nm as soon as possible but at least within 4 h. Preferably use 10-cm cells. Calculate the ozone concentration from the difference between the absorbances found in sample and blank (¶ 5*a* below). (NOTE: A maximum delay of 4 h before spectrophotometric reading can be tolerated only for drinking water samples. For other sample types test the time drift.)

2) Range 0.05 to 0.5 mg O_3/L—Proceed as above using 10.0 mL indigo reagent II instead of reagent I. Preferably measure absorbance in 4- or 5-cm cells.

3) Concentrations greater than 0.3 mg O_3/L—Proceed using indigo reagent II, but for these higher ozone concentrations

use a correspondingly smaller sample volume. Dilute resulting mixture to 100 mL with distilled water. Use a glass pipet for dosing sample; let sample flow through an erlenmeyer flask for at least 1 min without generating bubbles. Rinse pipet with sample and add measured amount to flask while keeping the pipet tip below the surface.

4) Control of interferences—In presence of chlorine, place 1 mL malonic acid reagent in both flasks before adding sample and/or filling to mark. Measure absorbance as soon as possible, within 60 min (Br^-, Br_2, and HOBr are only partially masked by malonic acid).

In presence of manganese prepare a blank solution using sample, in which ozone is selectively destroyed by addition of glycine. Place 0.1 mL glycine reagent in 100-mL volumetric flask (blank) and 10.0 mL indigo reagent II in second flask (sample). Pipet exactly the same volume of sample into each flask. Adjust dose so that decolorization in second flask is easily visible but complete bleaching does not result (maximum 80 mL).

Insure that pH of glycine/sample mixture in blank flask (before adding indigo) is not below 6 because reaction between ozone and glycine becomes very slow at low pH. Stopper flasks and mix by carefully inverting. Add 10.0 mL indigo reagent II to blank flask only 30 to 60 s after sample addition. Fill both flasks to the mark with ozone-free water and mix thoroughly. Measure absorbance of both solutions at comparable contact times of approximately 30 to 60 min (after this time, residual manganese oxides further discolor indigo only slowly and the drift of absorbance in blank and sample become comparable). Reduced absorbance in blank flask results from manganese oxides while that in sample flask is due to ozone plus manganese oxide.

5) Calibration—Because ozone is unstable, base measurements on known and constant loss of absorbance of the indigo reagent ($f = 0.42 \pm 0.01$/cm/mg O_3/L). For maximum accuracy analyze the lot of potassium indigo trisulfonate (no commercial lot has been found to deviate from $f = 0.42$) using the iodometric procedure.

When using a filter photometer, readjust the conversion factor, f, by comparing photometer sensitivity with absorbance at 600 nm by an accurate spectrophotometer.

b. Visual procedure:

1) Concentration range 0.01 to 0.1 mg O_3/L—Add 10.0 mL indigo reagent I to each of two identical 100-mL graduated glass cylinders. Fill reference cylinder (blank) to the mark with distilled water and other cylinder with sample. Add sample to cylinder so that completely decolorized zones are eliminated quickly by mixing but no degassing occurs. Pour off blank by portions until liquid height gives the same apparent color intensity as the sample when viewed from top. Record volume in blank cylinder. Color comparisons may be made up to 4 h after sample addition.

2) Concentrations greater than 0.1 mg O_3/L—Proceed as above, adding either 30 or 45 mL of sample and dilute to the mark.

3) Manganese-containing waters—The visual method is not suitable for these waters when the manganese concentration is comparable to that of the ozone because the difference measurement becomes too inaccurate.

5. Calculations

a. Spectrophotometric procedure:

$$\text{mg } O_3/L = \frac{100 \times \Delta A}{f \times b \times V}$$

where:

ΔA = difference in absorbance between sample and blank,
b = path length of cell, cm,
V = volume of sample, mL (normally 90 mL), and
f = 0.42.

The factor f is based on a sensitivity factor of 20 000/cm for the change of absorbance (600 nm) per mole of added ozone per liter. It was calibrated by iodometric titration. The UV absorbance of ozone in pure water may serve as a secondary standard: the factor $f = 0.42$ corresponds to an absorption coefficient for aqueous ozone, $\epsilon = 2950/M \cdot \text{cm}$ at 258 nm.

b. Visual procedure:

$$\text{mg } O_3/L = \frac{(100 - V) \times k}{100}$$

where:

V = volume of reference solution in blank cylinder, mL, and

k = conversion factor for indigo stock solution, calibrated by a spectrophotometric analysis of ozone. The value is about 0.10 mg O_3/L if the 1:100 dilution gives an absorbance of 0.19/cm.

When adding only 45 or 30 mL of sample, the conversion factor becomes $2k$ or $3k$, respectively.

6. Precision and Bias

a. Spectrophotometric procedure: In the absence of interferences, the relative error is less than 5% without special sampling setups. In laboratory testing this may be reduced to 1%.

Because this method is based on the differences in absorbance between the sample and blank (ΔA) the method is not applicable in the presence of chlorine. If the manganese content exceeds the ozone, precision is reduced. If the ratio of manganese to ozone is less than 10:1, ozone concentrations above 0.02 mg/L may be determined with a relative error of less than 20%.

b. Visual procedure: Duplicate determinations gave an average deviation of 1 to 1.5% within the pair. If the manganese concentration is comparable to that of ozone, this method is not applicable.

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212 TEMPERATURE*

1. General Discussion

Temperature readings are used in the calculation of various forms of alkalinity, in studies of saturation and stability with respect to calcium carbonate, in the calculation of salinity, and in general laboratory operations. In limnological studies, water temperatures as a function of depth often are required. Elevated temperatures resulting from discharges of heated water may have significant ecological impact. Identification of source of water supply, such as deep wells, often is possible by temperature measurements alone. Industrial plants often require data on water temperature for process use or heat-transmission calculations.

Normally, temperature measurements may be made with any good mercury-filled Celsius thermometer. As a minimum, the thermometer should have a scale marked for every 0.1°C, with markings etched on the capillary glass. The thermometer should have a minimal thermal capacity to permit rapid equilibration. Periodically check the thermometer against a precision thermometer certified by the National Bureau of Standards† that is used with its certificate and correction chart. For field operations use a thermometer having a metal case to prevent breakage.

Depth temperature required for limnological studies may be measured with a reversing thermometer, thermophone, or thermistor. The thermistor is most convenient and accurate; however, higher cost may preclude its use. Calibrate any temperature measurement devices with a National Bureau of Standards certified thermometer before field use. Make readings with the thermometer or device immersed in water long enough to permit complete equilibration. Report results to the nearest 0.1 or 1.0°C, depending on need.

2. Reversing Thermometer

The thermometer commonly used for depth measurements is of the reversing type. It often is mounted on the sample collection apparatus so that a water sample may be obtained simultaneously. Correct readings of reversing thermometers for changes due to differences between temperature at reversal and temperature at time of reading. Calculate as follows:

$$\Delta T = \left[\frac{(T' - t)(T' + V_0)}{K}\right] \times \left[1 + \frac{(T' - t)(T' + V_0)}{K}\right] + L$$

where:

ΔT = correction to be added algebraically to uncorrected reading,
T' = uncorrected reading at reversal,
t = temperature at which thermometer is read,
V_0 = volume of small bulb end of capillary up to 0°C graduation,
K = constant depending on relative thermal expansion of mercury and glass (usual value of K = 6100), and
L = calibration correction of thermometer depending on T'.

If series observations are made it is convenient to prepare graphs for a thermometer to obtain ΔT from any values of T' and t.

3. Bibliography

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213 TESTS ON SLUDGES*

This section presents a series of tests uniquely applicable to sludges or slurries. The test data are useful in designing facilities for solids separation and concentration and for assessing operational behavior, especially of the activated sludge process.

213 A. Oxygen-Consumption Rate

1. General Discussion

This test is used to determine the oxygen consumption rate of a sample of a biological suspension such as activated sludge. It is useful in laboratory and pilot-plant studies as well as in the operation of full-scale treatment plants. When used as a routine plant operation test, it often will indicate changes in operating conditions at an early stage. However, because test conditions are not necessarily identical to conditions at the sampling site, the observed measurement may not be identical with actual oxygen consumption rate.

2. Apparatus

a. Oxygen-consumption rate device: Either:

1) A probe with an oxygen-sensitive electrode (polarographic or galvanic), or

2) A manometric or respirometric device with appropriate readout and sample capacity of at least 300 mL. The device should have an oxygen supply capacity greater than the oxygen consumption rate of the biological suspension, or at least 150 mg/L · h.

b. Stopwatch or other suitable timing device.

c. Thermometer to read to ± 0.5°C.

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* Approved by Standard Methods Committee, 1981.

† Some commercial thermometers may be as much as 3°C in error.

*Approved by Standard Methods Committee, 1985.

direct sunlight at all times and keep from all contact with rubber.

4. Procedure

a. Sample collection: Collect an 800-mL sample in a 1-L gas washing bottle.

b. Ozone absorption: Pass a stream of pure air or N_2 through sample and then through an absorber containing 400 mL KI solution. Continue for 5 to 10 min at a rate of 0.2 to 1.0 L/min to insure that all ozone is swept from sample and absorbed in KI solution.

c. Titration: Transfer KI solution to a 1-L beaker, rinse absorber, and add 20 mL 1*N* H_2SO_4 to reduce pH below 2.0. Titrate with 0.005*M* $Na_2S_2O_3$ titrant until yellow color of liberated iodine almost is discharged. Add 4 mL starch indicator solution and continue titrating carefully but rapidly to the end point, at which the blue color just disappears. Long contact of iodine and starch develops a blue compound that is difficult to decolorize. The end point may be determined amperometrically as described in Section 408C.4*b* except that $Na_2S_2O_3$ can be used as the titrant. Other procedures given in Section 408 for measuring iodine may be used.

d. Blank test: Correct sample titration result by determining blank contributed by such reagent impurities as free iodine or iodate in KI, or traces of reducing agents that might reduce liberated iodine.

Take 400 mL KI solution, 20 mL 1*N* H_2SO_4, and 4 mL starch indicator solution. Perform whichever blank titration below applies:

1) If a blue color appears, titrate with 0.005*M* $Na_2S_2O_3$ to disappearance of blue and record result.

2) If no blue color appears, titrate with 0.005*M* iodine solution until a blue color appears. Back-titrate with 0.005*M* $Na_2S_2O_3$ to disappearance and record difference.

Before calculating ozone concentration subtract blank titration in ¶ 4*d*1) from sample titration, or add result of ¶ 4*d*2) above.

5. Calculation

$$\text{mg } O_3/\text{L} = \frac{(A \pm B) \times M \times 24\,000}{\text{mL sample}}$$

where:

A = mL titrant for sample,
B = mL titrant for blank (positive or negative), and
M = molarity of $Na_2S_2O_3$.

6. Interpretation of Results

The precision of the test is within ±1% for concentrations of 3 mg O_3/L or greater. However, rapid decrease of the residual occurs in the time elapsing between sampling and testing. Temperature also is an important factor in the decrease.

7. Bibliography

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423 pH VALUE*

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment, e.g., acid-base neutralization, water softening, precipitation, coagulation, disinfection, and corrosion control, is pH-dependent. pH is used in alkalinity and carbon dioxide measurements and many other acid-base equilibria. At a given temperature the *intensity* of the acidic or basic character of a solution is indicated by pH or hydrogen ion activity. Alkalinity and acidity are the acid- and base-neutralizing capacities of a water and usually are expressed as milligrams $CaCO_3$ per liter. Buffer capacity is the amount of strong acid or base, usually expressed in moles per liter, needed to change the pH value of a 1-L sample by 1 unit. pH as defined by Sorenson[1] is $-\log [H^+]$; it is the "intensity" factor of acidity. Pure water is very slightly ionized and at equilibrium the ion product is

$$[H^+][OH^-] = K_w$$
$$= 1.01 \times 10^{-14} \text{ at } 25°\text{C} \quad (1)$$

and

$$[H^+] = [OH^-]$$
$$= 1.005 \times 10^{-7}$$

where:

$[H^+]$ = activity of hydrogen ions, moles/L,
$[OH^-]$ = activity of hydroxyl ions, moles/L, and
K_w = ion product of water.

Because of ionic interactions in all but very dilute solutions, it is necessary to use the "activity" of an ion and not its molar concentration. Use of the term pH assumes that the activity of the hydrogen ion, a_{H^+}, is being considered. The *approximate* equivalence to molarity, $[H^+]$ can be presumed only in very dilute solutions (ionic strength < 0.1).

A logarithmic scale is convenient for expressing a wide range of ionic activities. Equation 1 in logarithmic form and corrected to reflect activity is:

$$(-\log_{10} a_{H^+}) + (-\log_{10} a_{OH^-}) = 14 \quad (2)$$

or

$$\text{pH} + \text{pOH} = \text{p}K_w$$

where:

$\text{pH} = -\log_{10} a_{H^+}$ and
$\text{pOH} = -\log_{10} a_{OH^-}$.

Equation 2 states that as pH increases pOH decreases correspondingly and vice versa because pK_w is constant for a given temperature. At 25°C, pH 7.0 is neutral, the activities of the hydrogen and hydroxyl ions are equal, and each corresponds to an approximate activity of 10^{-7} moles/L. The neutral point is temperature-dependent and is pH 7.5 at 0°C and pH 6.5 at 60°C.

The pH value of a highly dilute solution is approximately the same as the negative common logarithm of the hydrogen ion concentration. Natural waters usually have pH values [illegible] of 4 to 9, and most

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*Approved by Standard Methods Committee, [illegible]

[1] p designates $-\log_{10}$ of a number.

ment yet devised will duplicate the results obtained on the Jackson candle turbidimeter for all samples. Because of fundamental differences in optical systems, the results obtained with different types of secondary instruments frequently will not check closely with one another, even though the instruments are precalibrated against the candle turbidimeter.

Most commercial turbidimeters available for measuring low turbidities give comparatively good indications of the intensity of light scattered in one particular direction, predominantly at right angles to the incident light. These nephelometers are unaffected relatively by small changes in design parameters and therefore are specified as the standard instrument for measurement of low turbidities. Nonstandard turbidimeters, such as forward-scattering devices, are more sensitive than nephelometers to the presence of larger particles and are useful for process monitoring.

A further cause of discrepancies in turbidity analysis is the use of suspensions of different types of particulate matter for the preparation of instrumental calibration curves. Like water samples, prepared suspensions have different optical properties depending on the particle size distributions, shapes, and refractive indices. A standard reference suspension having reproducible light-scattering properties is specified for nephelometer calibration.

Because there is no direct relationship between the intensity of light scattered at a 90° angle and Jackson candle turbidity, there is no valid basis for the practice of calibrating a nephelometer in terms of candle units. To distinguish between turbidities derived from nephelometric and visual methods, report the results from the former as nephelometric turbidity units (NTU) and from the latter as Jackson turbidity units (JTU).

1. Selection of Method

Its greater precision, sensitivity, and applicability over a wide turbidity range make the nephelometric method preferable to visual methods. The candle turbidimeter, with a lower limit of 25 turbidity units, has its principal usefulness in examining highly turbid waters. The bottle standards offer a practical means for checking raw and conditioned water at various stages of the treatment process.

2. Storage of Sample

Determine turbidity on the day the sample is taken. If longer storage is unavoidable, store samples in the dark for up to 24 h. Do not store for long periods because irreversible changes in turbidity may occur. Vigorously shake all samples before examination.

214 A. Nephelometric Method—Nephelometric Turbidity Units

1. General Discussion

a. Principle: This method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the intensity of scattered light, the higher the turbidity. Formazin polymer is used as the reference turbidity standard suspension. It is easy to prepare and is more reproducible in its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter; therefore, nephelometric turbidity units based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

b Interference: Turbidity can be determined for any water sample that is free of debris and rapidly settling coarse sediments. Dirty glassware, the presence of air bubbles, and the effects of vibrations that disturb the surface visibility of the sample will give false results. "True color," that is, water color due to dissolved substances that absorb light, causes measured turbidities to be low. This effect usually is not significant in the case of treated water.

2. Apparatus

a. Turbidimeter consisting of a nephelometer with a light source for illuminating the sample and one or more photoelectric detectors with a readout device to indicate intensity of light scattered at 90° to the path of incident light. Use a turbidimeter designed so that little stray light reaches the detector in the absence of turbidity and free from significant drift after a short warmup period. The sensitivity of the instrument should permit detecting turbidity differences of 0.02 NTU or less in waters having turbidity of less than 1 NTU with a range from 0 to 40 NTU. Several ranges are necessary to obtain both adequate coverage and sufficient sensitivity for low turbidities.

Differences in turbidimeter design will cause differences in measured values for turbidity even though the same suspension is used for calibration. To minimize such differences, observe the following design criteria:

1) Light source—Tungsten-filament lamp operated at a color temperature between 2200 and 3000°K.

2) Distance traversed by incident light and scattered light within the sample tube—Total not to exceed 10 cm.

3) Angle of light acceptance by detector—Centered at 90° to the incident light path and not to exceed ± 30° from 90°. The detector, and filter system if used, shall have a spectral peak response between 400 and 600 nm.

b. Sample tubes, clear colorless glass. Keep tubes scrupulously clean, both inside and out, and discard when they become scratched or etched. Never handle them where the light strikes them. Use tubes with sufficient extra length, or with a protective case, so that they may be handled properly. Fill tubes with samples and standards that have been agitated thoroughly and allow sufficient time for bubbles to escape.

3. Reagents

a. Turbidity-free water: Turbidity-free water is difficult to obtain. The following method is satisfactory for measuring turbidity as low as 0.02 NTU.

Pass distilled water through a membrane filter having precision-sized holes of 0.2 µm;* the usual membrane filter used for bacteriological examinations is not satisfactory. Rinse collecting flask at least twice with filtered water and discard the next 200 mL.

Some commercial bottled demineralized waters are nearly particle-free. These may be used when their turbidity is lower than can be achieved in the laboratory. Dilute samples to a turbidity not less than 1 with distilled water.

b. Stock turbidity suspension:

1) Solution I—Dissolve 1.000 g hydrazine sulfate, $(NH_2)_2 \cdot H_2SO_4$, in distilled water and dilute to 100 mL in a volumetric flask.

2) Solution II—Dissolve 10.00 g hexamethylenetetramine, $(CH_2)_6N_4$, in distilled water and dilute to 100 mL in a volumetric flask.

3) In a 100-mL volumetric flask, mix 5.0 mL Solution I and 5.0 mL Solution II. Let stand 24 h at 25 ± 3°C, dilute to mark, and mix. The turbidity of this suspension is 400 NTU.

4) Prepare solutions and suspensions monthly.

c. Standard turbidity suspension: Dilute 10.00 mL stock turbidity suspension to 100 mL with turbidity-free water. Prepare

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*Nuclepore Corporation, 7035 Commerce Circle, Pleasanton, Calif., or equivalent.

daily. The turbidity of this suspension is defined as 40 NTU.

d. Alternate standards: As an alternative to preparing and diluting formazin, use commercially available standards such as styrene divinylbenzene beads† if they are demonstrated to be equivalent to freshly prepared formazin.

e. Dilute turbidity standards: Dilute portions of standard turbidity suspension with turbidity-free water as required. Prepare daily.

4. Procedure

a. Turbidimeter calibration: Follow the manufacturer's operating instructions. In the absence of a precalibrated scale, prepare calibration curves for each range of the instrument. Check accuracy of any supplied calibration scales on a precalibrated instrument by using appropriate standards. Run at least one standard in each instrument range to be used. Make certain that turbidimeter gives stable readings in all sensitivity ranges used. High turbidities determined by direct measurement are likely to differ appreciably from those determined by the dilution technique, ¶ 4*c*.

b. Measurement of turbidities less than 40 NTU: Thoroughly shake sample. Wait until air bubbles disappear and pour sample into turbidimeter tube. When possible, pour shaken sample into turbidimeter tube and immerse it in an ultrasonic bath for 1 to 2 s, causing complete bubble release. Read turbidity directly from instrument scale or from appropriate calibration curve.

c. Measurement of turbidities above 40 NTU: Dilute sample with one or more volumes of turbidity-free water until turbidity falls between 30 and 40 NTU. Compute turbidity of original sample from turbidity of diluted sample and the dilution factor. For example, if five volumes of turbidity-free water were added to one volume of sample and the diluted sample showed a turbidity of 30 NTU, then the turbidity of the original sample was 180 NTU.

d. Calibrate continuous turbidity monitors for low turbidities by determining turbidity of the water entering or leaving them, using a laboratory-model turbidimeter. When this is not possible, use an appropriate dilute turbidity standard, ¶ 3*e*. For turbidities above 40 NTU use undiluted stock solution.

5. Calculation

$$\text{Nephelometric turbidity units (NTU)} = \frac{A \times (B + C)}{C}$$

where:

A = NTU found in diluted sample,
B = volume of dilution water, mL, and
C = sample volume taken for dilution, mL.

6. Interpretation of Results

a. Report turbidity readings as follows:

Turbidity Range *NTU*	Report to the Nearest *NTU*
0–1.0	0.05
1–10	0.1
10–40	1
40–100	5
100–400	10
400–1000	50
> 1000	100

b. For comparison of water treatment efficiencies estimate turbidity more closely than is specified above. Uncertainties and discrepancies in turbidity measurements make it unlikely that two or more laboratories will duplicate results on the same sample more closely than specified.

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† AMCO-AEPA-1 Standard, Advanced Polymer Systems, 3696 C Haven Ave., Redwood City, Calif.

214 B. Visual Methods—Jackson Turbidity Units

1. General Discussion

a. Principle: Turbidity measurements by the candle turbidimeter are based on the light path through a suspension that just causes the image of the flame of a standard candle to disappear—that is, to become indistinguishable against the general background illumination—when the flame is viewed through the suspension. The longer the light path, the lower the turbidity.

b. Interference: Turbidity can be determined for any water sample that is free of rapidly settling debris and coarse sediments. Dirty glassware, the presence of air bubbles, and the effects of vibrations that disturb the surface visibility of the sample give false results.

2. Apparatus

a. Candle turbidimeter consisting of a glass tube calibrated according to Table 214:I, a standard candle, and a support that aligns candle and tube. The glass tube and candle are supported in a vertical position so that the center line of the tube passes through the center line of the candle. The candle is supported by a spring-loaded cylinder designed to keep the top of the candle pressed against the top of the support as the candle gradually burns away. The top of the support for the candle is 7.6 cm below the bottom of the glass tube. The glass tube has a flat, polished optical-glass bottom and conforms to specifications for nessler tubes given in Section 102.6. It is graduated to read directly in JTU. Keep tube clean and free from scratches. Keep most of glass tube enclosed within a metal tube when observations are being made, both to protect against breakage and to exclude extraneous light.

Use a candle made of beeswax and spermaceti, designed to burn within the limits of 114 to 126 grains/h. To insure uniform results, keep flame as near constant size and constant distance from bottom of glass tube as possible by frequently trimming charred portion of the wick and making sure that the candle is pushed to the top of its support. Eliminate all drafts during measurements to prevent flame from flickering. Do not burn candle for more than a few minutes at a time because the flame tends to increase in size. Before lighting candle each time, remove any portions of charred wick that can be broken off easily when manipulated with the fingers.

b. Bottles for visual comparison: A matched set of 1-L-capacity, glass-stoppered bottles made of borosilicate or other resistant glass.

3. Preparation of Standard Suspensions

a. Turbidity-free water: See Section 214A.3*a*.

b. Visual comparison standards: Prepare from natural turbid water or kaolin.

1) Natural water—For best results, prepare from natural turbid water from the same source as that to be tested. Determine turbidity with candle turbidimeter, then dilute portions of suspension to turbidity values desired.

Weekly prepare suspensions of turbidities below 25 units by diluting a freshly checked, more concentrated suspension.

2) Kaolin—Add approximately 5 g kaolin to 1 L distilled water, thoroughly agitate, and let stand for 24 h. Withdraw supernatant without disturbing sediment. Determine turbidity with candle turbidimeter. Dilute to turbidity values desired. Preserve standard suspensions by adding 1 g $HgCl_2$/L suspension. Shake suspensions vigorously before each reading and check monthly with candle turbidimeter

eral hours take sample, add KI, and measure total chlorine with a colorimetric method using a nessler tube to increase sensitivity. Do not use before last trace of free and combined chlorine has been removed.

Distilled water commonly contains ammonia and also may contain reducing agents. Collect good-quality distilled or deionized water in a sealed container from which water can be drawn by gravity. To the air inlet of the container add an H_2SO_4 trap consisting of a large test tube half filled with 1 + 1 H_2SO_4 connected in series with a similar but empty test tube. Fit both test tubes with stoppers and inlet tubes terminating near the bottom of the tubes and outlet tubes terminating near the top of the tubes. Connect outlet tube of trap containing H_2SO_4 to the distilled water container, connect inlet tube to outlet of empty test tube. The empty test tube will prevent discharge to the atmosphere of H_2SO_4 due to temperature-induced pressure changes. Stored in such a container, chlorine-demand-free water is stable for several weeks unless bacterial growth occurs.

4. Procedure

a. Preparation for titration:

1) Volume of sample—For chlorine concentrations of 10 mg/L or less, titrate 200 mL. For greater chlorine concentrations, use proportionately less sample and dilute to 200 mL with chlorine-demand-free water. Use a sample of such size that not more than 10 mL phenylarsine oxide solution is required.

2) Preparation for titration—Measure 5 mL 0.005 64*N* reductant for chlorine concentrations from 2 to 5 mg/L, and 10 mL for concentrations of 5 to 10 mg/L, into a flask or casserole for titration with standard iodine or iodate. Start stirring. For titration by amperometry or standard iodine, also add excess KI (approximately 1 g) and 4 mL acetate buffer solution or enough to reduce the pH to between 3.5 and 4.2.

b. Titration: Use one of the following:

1) Amperometric titration—Add 0.0282*N* iodine titrant in small increments from a 1-mL buret or pipet. Observe meter needle response as iodine is added: the pointer remains practically stationary until the end point is approached, whereupon each iodine increment causes a temporary deflection of the microammeter, with the pointer dropping back to its original position. Stop titration at end point when a small increment of iodine titrant gives a definite pointer deflection upscale and the pointer does not return promptly to its original position. Record volume of iodine titrant used to reach end point.

2) Colorimetric (iodine) titration—Add 1 mL starch solution and titrate with 0.0282*N* iodine to the first appearance of blue color that persists after complete mixing.

3) Colorimetric (iodate) titration—To suitable flask or casserole add 200 mL chlorine-demand-free water and add, with agitation, the required volume of reductant, an excess of KI (approximately 0.5 g), 2 mL 10% H_3PO_4 solution, and 1 mL starch solution in the order given, and titrate immediately* with 0.005 64*N* iodate solution to the first appearance of a blue color that persists after complete mixing. Designate volume of iodate solution used as *A*. Repeat procedure, substituting 200 mL sample for the 200 mL chlorine-demand-free water. If sample is colored or turbid, titrate to the first change in color, using for comparison another portion of sample with H_3PO_4 added. Designate this volume of iodate solution as *B*.

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*Titration may be delayed up to 10 min without appreciable error if H_3PO_4 is not added until immediately before titration.

5. Calculation

a. Titration with standard iodine:

$$\text{mg Cl as Cl}_2\text{/L} = \frac{(A - 5B) \times 200}{C}$$

where:

A = mL 0.005 64*N* reductant,
B = mL 0.0282*N* I_2, and
C = mL sample.

b. Titration with standard iodate:

$$\text{mg Cl as Cl}_2\text{/L} = \frac{(A - B) \times 200}{C}$$

where:

A = mL $Na_2S_2O_3$,
B = mL iodate required to titrate $Na_2S_2O_3$, and
C = mL sample.

408 C. Amperometric Titration Method

1. General Discussion

Amperometric titration requires a higher degree of skill and care than the colorimetric methods. Chlorine residuals over 2 mg/L are measured best by means of smaller samples or by dilution with water that has neither residual chlorine nor a chlorine demand. The method can be used to determine total chlorine and can differentiate between free and combined chlorine. A further differentiation into monochloramine and dichloramine fractions is possible by control of KI concentration and pH.

a. Principle: The amperometric method is a special adaptation of the polarographic principle. Free chlorine is titrated at a pH between 6.5 and 7.5, a range in which the combined chlorine reacts slowly. The combined chlorine, in turn, is titrated in the presence of the proper amount of KI in the pH range 3.5 to 4.5. When free chlorine is determined, the pH must not be greater than 7.5 because the reaction becomes sluggish at higher pH values, nor less than 6.5 because at lower pH values some combined chlorine may react even in the absence of iodide. When combined chlorine is determined, the pH must not be less than 3.5 because substances such as oxidized manganese interfere at lower pH values, nor greater than 4.5 because the iodide reaction is not quantitative at higher pH values. The tendency of monochloramine to react more readily with iodide than does dichloramine provides a means for further differentiation. The addition of a small amount of KI in the neutral pH range enables estimation of monochloramine content. Lowering the pH into the acid range and increasing the KI concentration allows the separate determination of dichloramine.

Organic chloramines can be measured as free chlorine, monochloramine, or dichloramine, depending on the activity of the chlorine in the organic compound.

Phenylarsine oxide is stable even in dilute solution and each mole reacts with two equivalents of halogen. A special amperometric cell is used to detect the end point of the residual chlorine-phenylarsine oxide titration. The cell consists of a nonpolarizable reference electrode that is immersed in a salt solution and a readily polarizable noble-metal electrode that is in contact both with the salt solution and the sample being titrated. In some applications, end-point selectivity is improved by adding +200 mV to the platinum electrode versus silver, silver chloride. Another approach to end-point detection uses dual platinum electrodes, a mercury cell with voltage divider to impress a potential across the electrodes, and a microammeter. If there is no chlorine residual in the sample, the microammeter reading will be comparatively low because of cell polarization. The greater the residual, the greater the microammeter reading. The meter acts merely as a null-point indicator—that is, the actual meter reading is not important, but rather the relative readings as the titration proceeds. The gradual addition of phenylarsine oxide causes the cell to become more and more polarized because of the decrease in chlorine. The end point is recognized when no further decrease in meter reading can be obtained by adding more phenylarsine oxide.

b. Interference: Accurate determinations of free chlorine cannot be made in the presence of nitrogen trichloride, NCl_3, or chlorine dioxide, which titrate partly as free chlorine. When present, NCl_3 can titrate partly as free chlorine and partly as dichloramine, contributing a positive error in both fractions. Some organic chloramines also can be titrated in each step. Monochloramine can intrude into the free chlorine fraction and dichloramine can interfere in the monochloramine fraction, especially at high temperatures and prolonged titration times. Free halogens other than chlorine also will titrate as free chlorine. Combined chlorine reacts with iodide ions to produce iodine. When titration for free chlorine follows a combined chlorine titration, which requires addition of KI, erroneous results may occur unless the measuring cell is rinsed thoroughly with distilled water between titrations.

Interference from copper has been noted in samples taken from copper pipe or after heavy copper sulfate treatment of reservoirs, with metallic copper plating out on the electrode. Silver ions also poison the electrode. Interference occurs in some highly colored waters and in waters containing surface-active agents. Very low temperatures slow response of measuring cell and longer time is required for the titration, but precision is not affected. A reduction in reaction rate is caused by pH values above 7.5; overcome this by buffering all samples to pH 7.0 or less. On the other hand, some substances, such as manganese, nitrite, and iron, do not interfere. The violent stirring of some commercial titrators can lower chlorine values by volatilization. When dilution is used for samples containing high chlorine content, take care that the dilution water is free of chlorine and ammonia and possesses no chlorine demand.

See 408.1 for a discussion of other interferences.

2. Apparatus

a. End-point detection apparatus, consisting of a cell unit connected to a microammeter, with necessary electrical accessories. The cell unit includes a noble-metal electrode of sufficient surface area, a salt bridge to provide an electrical connection without diffusion of electrolyte, and a reference electrode of silver-silver chloride in a saturated sodium chloride solution connected into the circuit by means of the salt bridge.

Keep noble-metal electrode free of deposits and foreign matter. Vigorous chemical cleaning generally is unnecessary. Occasional mechanical cleaning with a suitable abrasive usually is sufficient. Keep salt bridge in good operating condition; do not allow it to become plugged nor permit appreciable flow of electrolyte through it. Keep solution surrounding reference electrode free of contamination and maintain it at constant composition by insuring an adequate supply of undissolved salt at all times. A cell with two metal electrodes polarized by a small DC potential also may be used. (See Bibliography.)

b. Agitator, designed to give adequate agitation at the noble-metal electrode surface to insure proper sensitivity. Thoroughly clean agitator and exposed electrode system to remove all chlorine-consuming contaminants by immersing them in water containing 1 to 2 mg/L free chlorine for a few minutes. Add KI to the same water and let agitator and electrodes remain immersed for 5 min. After thorough rinsing with chlorine-demand-free water or the sample to be tested, sensitized electrodes and agitator are ready for use. Remove iodide reagent completely from cell.

c. Buret: Commercial titrators usually are equipped with suitable burets (1 mL). Manual burets are available.*

d. Glassware, exposed to water containing at least 10 mg/L chlorine for 3 h or more before use and rinsed with chlorine-demand-free water.

3. Reagents

a. Standard phenylarsine oxide titrant: See Method 408B.3a.

b. Phosphate buffer solution, pH 7: Dissolve 25.4 g anhydrous KH_2PO_4 and 34.1 g anhydrous Na_2HPO_4 in 800 mL distilled water. Add 2 mL sodium hypochlorite solution containing 1% chlorine and mix thoroughly. Protect from sunlight for 2 d. Determine that free chlorine still remains in the solution. Then expose to sunlight until no chlorine remains. If necessary, carry out the final dechlorination with an ultraviolet lamp. Determine that no total chlorine remains by adding KI and measuring with one of the colorimetric tests. Dilute to 1 L with distilled water and filter if any precipitate is present.

c. Potassium iodide solution: Dissolve 50 g KI and dilute to 1 L with freshly boiled and cooled distilled water. Store in the dark in a brown glass stoppered bottle, preferably in the refrigerator. Discard when solution becomes yellow.

d. Acetate buffer solution, pH 4: See Method 408B.3e.

4. Procedure

a. Sample volume: Select a sample volume requiring no more than 2 mL phenylarsine oxide titrant. Thus, for chlorine concentrations of 2 mg/L or less, take a 200-mL sample; for chlorine levels in excess of 2 mg/L, use 100 mL or proportionately less.

b. Free chlorine: Unless sample pH is known to be between 6.5 and 7.5, add 1 mL pH 7 phosphate buffer solution to produce a pH of 6.5 to 7.5. Titrate with standard phenylarsine oxide titrant, observing current changes on microammeter. Add titrant in progressively smaller increments until all needle movement ceases. Make successive buret readings when needle action becomes sluggish, signaling approach of end point. Subtract last very small increment that causes no needle response because of overtitration.

Continue titrating for combined chlorine as described in ¶ 4c below or for the separate monochloramine and dichloramine fractions as detailed in ¶s 4e and 4f.

c. Combined chlorine: To sample remaining from free-chlorine titration add 1.00 mL KI solution and 1 mL acetate buffer solution, in that order. Titrate with phenylarsine oxide titrant to the end point, as above. Do not refill buret but simply continue titration after recording figure for free chlorine. Again subtract last increment to give amount of titrant actually used in reaction with chlorine. (If titration was continued without refilling buret, this figure represents total chlorine. Subtracting free chlorine from total gives combined chlorine.) Wash apparatus and sample cell thoroughly to remove iodide ion to avoid inaccuracies when the titrator is used subsequently for a free chlorine determination.

d. Separate samples: If desired, determine total chlorine and free chlorine on separate samples. If sample pH is between

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*Kimax 17110-F, 5 ml, Kimble Products, Box 1035, Toledo, Ohio, or equivalent.

3.5 and 9 [illegible] total chlorine alone is required, treat sample immediately with 1 mL KI solution followed by 1 mL acetate buffer solution, and titrate with phenylarsine oxide titrant as described in ¶ 4*c* preceding.

e. Monochloramine: After titrating for free chlorine, add 0.2 mL KI solution to same sample and, without refilling buret, continue titration with phenylarsine oxide titrant to end point. Subtract last increment to obtain net volume of titrant consumed by monochloramine.

f. Dichloramine: Add 1 mL acetate buffer solution and 1 mL KI solution to same sample and titrate final dichloramine fraction as described above.

5. Calculation

Convert individual titrations for free chlorine, combined chlorine, total chlorine, monochloramine, and dichloramine by the following equation:

$$\text{mg Cl as Cl}_2\text{/L} = \frac{A \times 200}{\text{mL sample}}$$

where:

A = mL phenylarsine oxide titration.

6. Precision and Accuracy

See 408A.5.

408 D. DPD Ferrous Titrimetric Method

1. General Discussion

a. Principle: N,N-diethyl-p-phenylenediamine (DPD) is used as an indicator in the titrimetric procedure with ferrous ammonium sulfate (FAS). Where complete differentiation of chlorine species is not required, the procedure may be simplified to give only free and combined chlorine or total chlorine.

In the absence of iodide ion, free chlorine reacts instantly with DPD indicator to produce a red color. Subsequent addition of a small amount of iodide ion acts catalytically to cause monochloramine to produce color. Addition of iodide ion to excess evokes a rapid response from dichloramine. In the presence of iodide ion, part of the nitrogen trichloride (NCl_3) is included with dichloramine and part with free chlorine. A supplementary procedure based on adding iodide ion before DPD permits estimating proportion of NCl_3 appearing with free chlorine.

Chlorine dioxide (ClO_2) appears, to the extent of one-fifth of its total chlorine content, with free chlorine. A full response from ClO_2, corresponding to its total chlorine content, may be obtained if the sample first is acidified in the presence of iodide ion and subsequently is brought back to an approximately neutral pH by adding bicarbonate ion. Bromine, bromamine, and iodine react with DPD indicator and appear with free chlorine.

b. pH control: For accurate results careful pH control is essential. At the proper pH of 6.2 to 6.5, the red colors produced may be titrated to sharp colorless end points. *Titrate as soon as the red color is formed in each step.* Too low a pH in the first step tends to make the monochloramine show in the free-chlorine step and the dichloramine in the monochloramine step. Too high a pH causes dissolved oxygen to give a color.

c. Temperature control: In all methods for differentiating free chlorine from chloramines, higher temperatures increase the tendency for chloramines to react and lead to increased apparent free-chlorine results. Higher temperatures also increase color fading. Complete measurements rapidly, especially at higher temperature.

d. Interference: The most significant interfering substance likely to be encountered in water is oxidized manganese. To correct for this, place 5 mL buffer solution and 0.5 mL sodium arsenite solution in the titration flask. Add 100 mL sample and mix. Add 5 mL DPD indicator solution, mix, and titrate with standard FAS titrant until red color is discharged. Subtract reading from Reading *A* obtained by the normal procedure as described in ¶ 3*a*1) of this method or from the total chlorine reading obtained in the simplified procedure given in ¶ 3*a*4). If the combined reagent in powder form (see below) is used, first add KI and arsenite to the sample and mix, then add combined buffer-indicator reagent.

As an alternative to sodium arsenite use a 0.25% solution of thioacetamide, adding 0.5 mL to 100 mL sample.

Interference by copper up to approximately 10 mg Cu/L is overcome by the EDTA incorporated in the reagents. EDTA enhances stability of DPD indicator solution by retarding deterioration due to oxidation, and in the test itself, provides suppression of dissolved oxygen errors by preventing trace metal catalysis.

High concentrations of combined chlorine can break through into the free chlorine fraction. At 10°C this amounts to 2% and at 25°C to 4% of the monochloramine present that reacts after standing 1 min. Adding thioacetamide (0.5 mL 0.25% solution to 100 mL) immediately after mixing DPD reagent with sample completely stops further reaction with combined chlorine in the free chlorine measurement. Continue immediately with FAS titration to obtain free chlorine. Obtain total chlorine from the normal procedure, i.e., without using thioacetamide.

Because high concentrations of iodide are used to measure combined chlorine and only traces of iodide greatly increase chloramine interference in free chlorine measurements, take care to avoid iodide contamination by rinsing between samples or using separate glassware.

See 408.I for a discussion of other interferences.

e. Minimum detectable concentration: Approximately 18 µg Cl as Cl_2/L.

2. Reagents

a. Phosphate buffer solution: Dissolve 24 g anhydrous Na_2HPO_4 and 46 g anhydrous KH_2PO_4 in distilled water. Combine with 100 mL distilled water in which 800 mg disodium ethylenediamine tetraacetate dihydrate (EDTA) have been dissolved. Dilute to 1 L with distilled water and add 20 mg $HgCl_2$ to prevent mold growth and interference in the free chlorine test caused by any trace amounts of iodide in the reagents. (CAUTION: *$HgCl_2$ is toxic—take care to avoid ingestion*).

b. N,N-Diethyl-p-phenylenediamine (DPD) indicator solution: Dissolve 1 g DPD oxalate,* or 1.5 g DPD sulfate pentahydrate,† or 1.1 g anhydrous DPD sulfate in chlorine-free distilled water containing 8 mL 1 + 3 H_2SO_4 and 200 mg disodium EDTA. Make up to 1 L, store in a brown glass-stoppered bottle in the dark, and discard when discolored. Periodically check solution blank for absorbance and discard when absorbance at 515 nm exceeds 0.002/cm. (The buffer and indicator sulfate are available commercially as a combined reagent in stable powder form.) CAUTION: *The oxalate is toxic—take care to avoid ingestion.*

c. Standard ferrous ammonium sulfate (FAS) titrant: Dissolve 1.106 g $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in distilled water containing 1 mL 1 + 3 H_2SO_4 and make

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*Eastman chemical No. 7102 or equivalent

†Available from Gallard-Schlesinger Chemical Mfg Corp., 584 Mineola Avenue, Carle Place, N.Y. 11514, or equivalent

up to 1 L with freshly boiled and cooled distilled water. This standard may be used for 1 month, and the titer checked by potassium dichromate. For this purpose add 10 mL 1 + 5 H_2SO_4, 5 mL conc H_3PO_4, and 2 mL 0.1% barium diphenylamine sulfonate indicator to a 100-mL sample of FAS and titrate with 0.100*N* primary standard potassium dichromate to a violet end point that persists for 30 s. The FAS titrant is equivalent to 100 µg Cl as Cl_2/1.00 mL.

d. Potassium iodide, KI, crystals.

e. Potassium iodide solution: Dissolve 500 mg KI and dilute to 100 mL, using freshly boiled and cooled distilled water. Store in a brown glass-stoppered bottle, preferably in a refrigerator. Discard when solution becomes yellow.

f. Potassium dichromate solution: See 408A.2*c*2).

g. Barium diphenylaminesulfonate, 0.1%: Dissolve 0.1 g $(C_6H_5NHC_6H_4\text{-}4\text{-}SO_3)_2Ba$ in 100 mL distilled water.

h. Sodium arsenite solution: Dissolve 5.0 g $NaAsO_2$ in distilled water and dilute to 1 L. (CAUTION: *Toxic—take care to avoid ingestion.*)

i. Thioacetamide solution: Dissolve 250 mg CH_3CSNH_2 in 100 mL distilled water. (CAUTION: *Cancer suspect agent. Take care to avoid skin contact or ingestion.*)

j. Chlorine-demand-free water: See 408B.3*m*.

3. Procedure

The quantities given below are suitable for concentrations of total chlorine up to 5 mg/L. If total chlorine exceeds 5 mg/L, use a smaller sample and dilute to a total volume of 100 mL. Mix usual volumes of buffer reagent and DPD indicator solution, or usual amount of DPD powder, with distilled water *before* adding sufficient sample to bring total volume to 100 mL. (If sample is added before buffer, test does not work.)

a. Free chlorine or chloramine: Place 5 mL each of buffer reagent and DPD indicator solution in titration flask and mix (or use about 500 mg DPD powder). Add 100 mL sample, or diluted sample, and mix.

1) Free chlorine—Titrate rapidly with standard FAS titrant until red color is discharged (Reading *A*).

2) Monochloramine—Add one very small crystal of KI (about 0.5 mg) or 0.1 mL (2 drops) KI solution and mix. Continue titrating until red color is discharged again (Reading *B*).

3) Dichloramine—Add several crystals KI (about 1 g) and mix to dissolve. Let stand for 2 min and continue titrating until red color is discharged (Reading *C*). For dichloramine concentrations greater than 1 mg/L, let stand 2 min more if color driftback indicates slightly incomplete reaction. When dichloramine concentrations are not expected to be high, use half the specified amount of KI.

4) Simplified procedure for free and combined chlorine or total chlorine—Omit 2) above to obtain monochloramine and dichloramine together as combined chlorine. To obtain total chlorine in one reading, add full amount of KI at the start, with the specified amounts of buffer reagent and DPD indicator, and titrate after 2 min standing.

b. Nitrogen trichloride: Place one very small crystal of KI (about 0.5 mg) or 0.1 mL KI solution in a titration flask. Add 100 mL sample and mix. Add contents to a second flask containing 5 mL each of buffer reagent and DPD indicator solution (or add about 500 mg DPD powder direct to the first flask). Titrate rapidly with standard FAS titrant until red color is discharged (Reading *N*).

4. Calculation

For a 100-mL sample, 1.00 mL standard FAS titrant = 1.00 mg Cl as Cl_2/L.

Reading	NCl_3 Absent	NCl_3 Present
A	Free Cl	Free Cl
B-*A*	NH_2Cl	NH_2Cl
C-*B*	$NHCl_2$	$NHCl_2$ + ½NCl_3
N	—	Free Cl + ½NCl_3
2(*N*-*A*)	—	NCl_3
C-*N*	—	$NHCl_2$

In the event that monochloramine is present with NCl_3, it will be included in *N*, in which case obtain NCl_3 from 2(*N* − *B*).

Chlorine dioxide, if present, is included in *A* to the extent of one-fifth of its total chlorine content.

In the simplified procedure for free and combined chlorine, only *A* (free Cl) and *C* (total Cl) are required. Obtain combined chlorine from *C*−*A*.

The result obtained in the simplified total chlorine procedure corresponds to *C*.

5. Precision and Accuracy

See 408A.5.

408 E. DPD Colorimetric Method

1. General Discussion

a. Principle: This is a colorimetric version of the DPD method and is based on the same principles. Instead of titration with standard ferrous ammonium sulfate (FAS) solution as in the titrimetric method, a colorimetric procedure is used.

b. Interference: See 408.1 and 408D.1*d*. Compensate for color and turbidity by using sample to zero photometer.

c. Minimum detectable concentration: Approximately 10 µg Cl as Cl_2/L.

2. Apparatus

Colorimetric equipment: One of the following is required:

a. Spectrophotometer, for use at a wavelength of 515 nm and providing a light path of 1 cm or longer.

b. Filter photometer, equipped with a filter having maximum transmission in the wavelength range of 490 to 530 nm and providing a light path of 1 cm or longer.

c. Glassware: Use separate glassware, including separate spectrophotometer cells, for free and combined (dichloramine) measurements, to avoid iodide contamination in free chlorine measurement.

3. Reagents

See Section 408D.2*a*, *b*, *d*, *e*, *h*, *i*, and *j*.

4. Procedure

a. Calibration of photometer or colorimeter: Calibrate instrument with chlorine (1) or potassium permanganate (2) solutions.

1) Chlorine solutions—Prepare chlorine standards in the range of 0.05 to 4 mg/L from about 100 mg/L chlorine water standardized as directed in Section 409A.3*g*. Use chlorine-demand-free water and glassware to prepare these standards. Develop color by first placing 5 mL phosphate buffer solution and 5 mL DPD indicator reagent in flask and then adding 100 mL chlorine standard with thorough mixing as described in *b* and *c* below. Fill photometer or colorimeter cell from flask and read color at 515 nm. Return cell contents to flask and titrate with standard FAS titrant as a check on chlorine concentration.

2) Potassium permanganate solutions—Prepare a stock solution containing 891 mg $KMnO_4$/1000 mL. Dilute 10.00 mL stock solution to 100 mL with distilled water in a volumetric flask. When 1 mL of this solution is diluted to 100 mL with

mercial product,† a stable powdered mixture containing 42.8% $KHSO_5$ by weight and a mixture of $KHSO_4$ and K_2SO_4. Dissolve 1.0 g powder in water and dilute to 1 L.

4. Procedure

a. Calibration of photometer: Prepare chlorine standards in the range of 0.05 to 2 mg/L by diluting the chlorine water standardized as directed in Section 409A.3*g*. Prepare separate calibration curves for free available chlorine and total chlorine as described in ¶s 4*b* and *c* below.

b. Free chlorine: Measure 100 mL dilute chlorine solution into a 250-mL beaker. Add 2.0 mL pH 4.0 buffer solution and mix. Fill pipet to mark with LCV indicator and place pipet tip just under beaker spout. Let 2.0 mL of indicator flow down inside glass surface into sample with minimum agitation. Stir sample to develop color. For best reproducibility add LCV indicator and stir sample to develop color in a standardized manner. Compare absorbance measured at 592 nm with calibration curve and report result as milligrams free chlorine per liter.

c. Total chlorine:

1) Concentration below 2.0 mg/L—Measure 100 mL sample into suitable flask and add 2.0 mL buffer solution, pH 4.0. Add 1.0 mL KI solution. Mix and wait at least 60 s. Add 2.0 mL LCV indicator, mix, and dilute to 200 mL. Read absorbance photometrically at 592 nm and compare with calibration curve.

2) Concentrations above 2.0 mg/L—Dilute sample with water so that when sample is treated with reagents in 1) above, the absorbance may be read from the calibration curve. Apply the appropriate dilution factor and report result as milligrams total chlorine per liter.

d. Elimination of interference from high concentrations of combined chlorine: To determine free chlorine in the presence of high concentrations of combined chlorine, immediately add 10.0 mL $NaAsO_2$ solution after adding and mixing 2.0 mL LCV indicator. Compare photometrically with standards prepared by adding 10.0 mL distilled water after adding indicator to compensate for dilution by $NaAsO_2$ solution.

e. Compensation for manganic [Mn (IV)] manganese: For free chlorine determination, follow the procedure given in ¶ 4*b* and record absorbance as A_1. To a second 100-mL sample, add 0.8 mL 1*N* NaOH. Add 2.0 mL dilute $NaAsO_2$ solution and let react for 2 min. Add 4.0 mL potassium peroxymonosulfate solution, and wait 1.0 min. Add 4.0 mL pH 4.0 buffer and 2.0 mL LCV indicator, mix, and record absorbance as A_2. Calculate absorbance, A_3, due to free chlorine as follows:

$$A_3 = A_1 - 1.084\,A_2$$

Refer absorbance A_3 to the free chlorine standard curve to obtain free chlorine concentration.

For total chlorine determination, make the total chlorine test as in ¶ 4*c* and record absorbance as B_1. To a second 100-mL sample, add 1*N* NaOH (approximately 0.8 mL) to adjust to pH 11.0. Add dilute $NaAsO_2$, potassium peroxymonosulfate, pH 4.0 buffer, and LCV indicator solutions as in ¶ 4*e*. Dilute to 200 mL and record absorbance as B_2. Calculate absorbance, B_3, due to total chlorine as follows:

$$B_3 = B_1 - B_2$$

Refer absorbance B_3 to total chlorine standard curve to obtain total chlorine concentration.

f. Compensation for turbidity and color: Compensate for interference by natural color or turbidity by measuring sample absorbance at 592 nm and subtracting this reading from absorbance of treated free or total chlorine sample.

5. Calculation

$$\text{mg/L total Cl as Cl}_2 = \frac{A \times 100}{\text{mL sample}}$$

$$\text{mg/L combined Cl as Cl}_2 = B - C$$

where:

A = total chlorine in mg/L measured in diluted sample,
B = total chlorine in mg/L in sample, and
C = free chlorine in mg/L.

6. Precision and Accuracy

See 408A.5.

[†] Oxone, a product of E I du Pont de Nemours and Co., Inc. Wilmington, Del., or equivalent.

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408 G. Syringaldazine (FACTS) Method (TENTATIVE)

1. General Discussion

a. Principle: The free (available) chlorine test, syringaldazine (FACTS) measures free chlorine over the range of 0.1 to 10 mg/L. A saturated solution of syringaldazine (3,5-dimethoxy-4-hydroxybenzaldazine) in 2-propanol is used. Syringaldazine is stable when stored as a solid or as a solution in 2-propanol. It is oxidized by free chlorine on a 1:1 molar basis to produce a colored product with an absorption maximum of 530 nm. The color product is only slightly soluble in water; therefore, at chlorine concentrations greater than 1 mg/L, the final reaction mixture must contain 2-propanol to prevent product precipitation and color fading.

The optimum color and solubility (minimum fading) are obtained in a solution having a pH between 6.5 and 6.8. At a pH less than 6, color development is slow and reproducibility is poor. At a pH greater than 7, the color develops rapidly but fades quickly. A buffer is required to maintain the reaction mixture pH at approximately 6.7. Care should be taken with waters of high acidity or alkalinity to assure that the added buffer maintains the proper pH.

Temperature has a minimal effect on the color reaction. The maximum error observed at temperature extremes of 5 and 35°C is ± 10%.

b. Interferences: Interferences common to other methods for determining free chlorine do not affect the FACTS procedure. Monochloramine concentrations up to 18 mg/L, dichloramine concentrations up to 10 mg/L, and manganese concentrations (oxidized forms) up to 1 mg/L do not interfere. Very high concentrations of monochloramine (≥ 35 mg/L) and oxidized manganese (≥ 2.6 mg/L) produce a color with syringaldazine slowly. Ferric iron can react with syringaldazine; however, concentrations up to 10 mg/L do not interfere. Nitrite (≤ 250 mg/L), nitrate (≤ 100 mg/L), sulfate (≤ 1000 mg/L), and chloride (≤ 1000 mg/L) do not interfere. Waters with high hardness (≥ 500 mg/L) will produce a cloudy solution that can be compensated for by using a blank or recalibrating using the alternative buffer of Section 408E.3*c*. Oxygen does not interfere.

Other strong oxidizing agents, such as iodine, bromine, and ozone, will produce a color.

c. Minimum detectable concentration: The FACTS procedure is sensitive to free chlorine concentrations of 0.1 mg/L or less.

2. Apparatus

Colorimetric equipment: One of the following is required:

a. Filter photometer, providing a light distilled water, [illegible]orine equivalent of 1.00 mg/L will be produced in the DPD reaction. Prepare a series of $KMnO_4$ standards covering the chlorine equivalent range of 0.05 to 4 mg/L. Develop color by first placing 5 mL phosphate buffer and 5 mL DPD indicator reagent in flask and adding 100 mL standard with thorough mixing as described in *b* and *c* below. Fill photometer or colorimeter cell from flask and read color at 515 nm. Return cell contents to flask and titrate with FAS titrant as a check on any absorption of permanganate by distilled water.

b. Volume of sample: Use a sample volume appropriate to the photometer or colorimeter. The following procedure is based on using 10-mL volumes; adjust reagent quantities proportionately for other sample volumes. Dilute sample with chlorine-demand-free water when total chlorine exceeds 4 mg/L.

c. Free chlorine: Place 0.5 mL each of buffer reagent and DPD indicator reagent in a test tube or photometer cell. Add 10 mL sample and mix. Read color immediately (Reading *A*).

d. Monochloramine: Continue by adding one very small crystal of KI (about 0.1 mg) and mix. If dichloramine concentration is expected to be high, instead of small crystal add 0.1 mL (2 drops) freshly prepared KI solution (0.1 g/100 mL). Read color immediately (Reading *B*).

e. Dichloramine: Continue by adding several crystals of KI (about 0.1 g) and mix to dissolve. Let stand about 2 min and read color (Reading *C*).

f. Nitrogen trichloride: Place a very small crystal of KI (about 0.1 mg) in a clean test tube or photometer cell. Add 10 mL sample and mix. To a second tube or cell add 0.5 mL each of buffer and indicator reagents; mix. Add contents to first tube or cell and mix. Read color immediately (Reading *N*).

5. Calculation

Reading	NCl_3 Absent	NCl_3 Present
A	Free Cl	Free Cl
B-A	NH_2Cl	NH_2Cl
C-B	$NHCl_2$	$NHCl_2$ + $\frac{1}{2}NCl_3$
N	—	Free Cl + $\frac{1}{2}NCl_3$
2(*N-A*)	—	NCl_3
C-N	—	$NHCl_2$

In the event that monochloramine is present with NCl_3, it will be included in Reading *N*, in which case obtain NCl_3 from 2(*N-B*).

408 F. Leuco Crystal Violet Method

1. General Discussion

The leuco crystal violet method measures separately the free and the total chlorine. The combined chlorine may be determined by difference.

a. Principle: The compound 4,4′,4″-methylidynetris (N,N-dimethylaniline), also known as leuco crystal violet, reacts instantaneously with free chlorine to form a bluish color. Interference from combined chlorine can be avoided by completing the test within a 5-min interval.

The total chlorine determination involves the reaction of free and combined chlorine with iodide ion to produce hypoiodous acid, which in turn reacts instantaneously with leuco crystal violet to form the dye crystal violet. The color is stable for days and follows Beer's law over a wide range of total chlorine.

b. Interference: No significant interference from combined chlorine occurs when free chlorine is determined within 5 min after indicator addition. Fifteen minutes after adding indicator the apparent error in the free chlorine determination is about 0.04 mg/L at 25°C in a sample containing 5.0 mg/L combined chlorine.

For combined chlorine concentrations above 5.0 mg/L use the arsenite addition procedure to minimize interference. The major interference in the determination of free chlorine is manganic ion, which increases the apparent chlorine reading. When manganic ion is known to be present, use the photometric procedure in which absorbance due to manganic ion is determined separately and subtracted from total absorbance to yield that produced by free chlorine alone.

Ferric and nitrate compounds do not interfere and nitrite ion does not interfere in the absence of monochloramine. Where nitrite ion and monochloramine are present together, as in certain wastewaters, serious interference will occur in the determination of free chlorine. Adding arsenite will minimize but not entirely eliminate this interference.

If suspended matter or organic color is present, compensate by incorporating appropriate turbidity or color blanks into the visual or photometric procedures.

c. Minimum detectable concentration: 10 μg free chlorine/L; 5 μg total chlorine/L.

2. Apparatus

a. Colorimetric equipment: One of the following is required:

1) *Filter photometer,* providing a light path of 1 cm or longer and equipped with an orange filter having maximum transmittance near 592 nm.

2) *Spectrophotometer,* for use at 592 nm, providing a light path of 1 cm or longer. If cells become stained, rinse with acetone.

b. Glassware: See 408C.2*d*.

3. Reagents

a. Chlorine-demand-free water: See 408B.3*m*. Use to prepare reagent solutions and sample dilutions.

b. Stock chlorine solution: Prepare 1000 mg/L stock chlorine solution as described in Section 409A.3*g*.

c. Buffer solution, pH 4.0: Dissolve 480 g glacial acetic acid and 146 g sodium acetate, $NaC_2H_3O_2$, or 243 g $NaC_2H_3O_2 \cdot 3H_2O$, in 400 mL water and dilute to 1 L.

d. Leuco crystal violet (LCV) indicator: Measure 500 mL water and 14.0 mL 85% orthophosphoric acid into a brown glass container of at least 1-L capacity. Introduce a magnetic stirring bar and mix at moderate speed. Add 3.0 g 4,4′,4″-methylidynetris-(N,N-dimethylaniline)* and with a small amount of water wash down any reagent adhering to neck or sides of container. Add 2.7 g mercuric chloride, $HgCl_2$, and stir until dissolved. (CAUTION: *Label container with warning that $HgCl_2$ is poisonous and corrosive.*)

Continue agitation until solution is complete. Finally, add 500 mL water. Store in brown bottle at room temperature away from direct sunlight. Discard after 6 months. If a rubber stopper must be used, wrap with plastic wrapping material to protect from contact with reagent.

e. Potassium iodide solution: Dissolve 3 g KI in water and dilute to 1 L. Store in a brown glass bottle and protect from sunlight.

f. Sodium hydroxide, 1*N*.

g. Dilute sodium arsenite solution: Dissolve 26 mg $NaAsO_2$ in water and dilute to 100 mL.

h. Sodium arsenite solution: Dissolve 5.0 g $NaAsO_2$ in water and dilute to 1 L. (CAUTION: *Toxic—take care to avoid ingestion.*)

i. Potassium peroxymonosulfate solution: Obtain this reagent, $KHSO_5$, as the com

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*Eastman chemical No. 3651 or equivalent.

410 B. Amperometric Method

1. General Discussion

a. Principle: The amperometric titration of ClO_2 is an extension of the amperometric method for chlorine. By performing four titrations with phenylarsine oxide, free chlorine (including hypochlorite and hypochlorous acid), chloramines, chlorite, and ClO_2 may be determined separately. The first titration step consists of conversion of ClO_2 to chlorite and chlorate through addition of sufficient NaOH to produce a pH of 12, followed by neutralization to a pH of 7 and titration of free chlorine. In the second titration KI is added to a sample that has been treated similarly with alkali and had the pH readjusted to 7; titration yields free chlorine and monochloramine. The third titration involves addition of KI and pH adjustment to 7, followed by titration of free chlorine, monochloramine, and one-fifth of the available ClO_2. In the fourth titration, addition of sufficient H_2SO_4 to lower the pH to 2 enables all available ClO_2 and chlorite, as well as the total free chlorine, to liberate an equivalent amount of iodine from the added KI and thus be titrated.

b. Interference: The interferences described in Section 408C.*1b* apply also to determination of ClO_2.

2. Apparatus

The apparatus required is given in Sections 408C.*2a* through *d*.

3. Reagents

All reagents listed for the determination of chlorine in Section 408C.3 are required. Also needed are the following:

a. Sodium hydroxide, NaOH, 6*N*.

b. Sulfuric acid, H_2SO_4, 6*N*, 1 + 5.

4. Procedure

Minimize effects of pH, time, and temperature of reaction by standardizing all conditions.

a. Titration of free available chlorine (hypochlorite and hypochlorous acid): Add sufficient 6*N* NaOH to raise sample pH to 12. After 10 min, add 6*N* H_2SO_4 to lower pH to 7. Titrate with standard phenylarsine oxide titrant to the amperometric end point as given in Section 408C. Record result as *A*.

b. Titration of free available chlorine and chloramine: Add 6*N* NaOH to raise sample pH to 12. After 10 min, add 6*N* H_2SO_4 to reduce pH to 7. Add 1 mL KI solution. Titrate with standard phenylarsine oxide titrant to the amperometric end point. Record result as *B*.

c. Titration of free available chlorine, chloramine, and one-fifth of available ClO_2: Adjust sample pH to 7 with pH 7 phosphate buffer solution. Add 1 mL KI solution. Titrate with standard phenylarsine oxide titrant to the amperometric end point. Record result as *C*.

d. Titration of free available chlorine, chloramines, ClO_2, and chlorite: Add 1 mL KI solution to sample. Add sufficient 6*N* H_2SO_4 to lower pH to 2. After 10 min, add sufficient 6*N* NaOH to raise pH to 7. Titrate with standard phenylarsine oxide titrant to the amperometric end point. Record result as *D*.

5. Calculation

Convert individual titrations (*A*, *B*, *C*, and *D*) into chlorine concentration by the following equation:

$$\text{mg Cl as Cl}_2\text{/L} = \frac{E \times 200}{\text{mL sample}}$$

where

E = mL phenylarsine oxide titration for each individual sample *A*, *B*, *C*, or *D*

Calculate ClO_2 and individual chlorine fractions as follows:

$$\text{mg ClO}_2 \text{ as ClO}_2\text{/L} = 1.9\,(C - B)$$

$$\text{mg ClO}_2 \text{ as Cl}_2\text{/L} = 5\,(C - B)$$

$$\text{mg free available chlorine/L} = A$$

$$\text{mg chloramine/L as chlorine} = B - A$$

$$\text{mg chlorite/L as chlorine} = 4B - 5C + D$$

410 C. DPD Method (TENTATIVE)

1. General Discussion

a. Principle: This method is an extension of the N,N-diethyl-p-phenylenediamine (DPD) method for determining free chlorine and chloramines in water. ClO_2 appears in the first step of this procedure but only to the extent of one-fifth of its total available chlorine content corresponding to reduction of ClO_2 to chlorite ion. If the sample is then acidified in the presence of iodide the chlorite also reacts. When neutralized by subsequent addition of bicarbonate, the color thus produced corresponds to the total available chlorine content of the ClO_2. If chlorite is present in the sample, this will be included in the step involving acidification and neutralization. Chlorite that did not result from ClO_2 reduction by the procedure will cause a positive error equal to twice this chlorite concentration. In evaluating mixtures of these various chloro-compounds, it is necessary to suppress free chlorine by adding glycine before reacting the sample with DPD reagent. Differentiation is based on the fact that glycine converts free chlorine instantaneously into chloroaminoacetic acid but has no effect on ClO_2.

b. Interference: The interference by oxidized manganese described in Section 408D.*1d* applies also to ClO_2 determination. Manganese interference appears as an increase in the first titrations after addition of DPD, with or without KI, and irrespective of whether there has been prior addition of glycine. Titration readings must be corrected suitably. Interference by chromate in wastewaters may be corrected similarly.

Iron contributed to the sample by adding ferrous ammonium sulfate (FAS) titrant may activate chlorite so as to interfere with the first end point of the titration. Suppress this effect with additional EDTA, disodium salt.

2. Reagents

Reagents required in addition to those for the DPD free-combined chlorine method as listed in Section 408D.2 are as follows:

a. Glycine solution: Dissolve 10 g NH_2CH_2COOH in 100 mL distilled water.

b. Sulfuric acid solution: Dilute 5 mL conc H_2SO_4 to 100 mL with distilled water.

c. Sodium bicarbonate solution: Dissolve 27.5 g $NaHCO_3$ in 500 mL distilled water.

d. EDTA: Disodium salt of ethylenediamine tetraacetic acid, solid.

3. Procedure

For samples containing more than 5 mg/L total available chlorine follow the dilution procedure given in Section 408D.3.

a. Chlorine dioxide: Add 2 mL glycine solution to 100 mL sample and mix. Place 5 mL each of buffer reagent and DPD indicator solution in a separate titration flask and mix (or use about 500 mg DPD powder). Add about 200 mg EDTA, disodium salt. Then add glycine-treated sample and mix. Titrate rapidly with standard FAS titrant until red color is discharged (Reading *G*).

b. Free available chlorine and chloramine: Using a second 100-mL sample follow the procedures of Section 408D.3*a* adding about 200 mg EDTA, disodium salt, initially with the DPD reagents (Readings *A*, *B*, and *C*).

c. Total available chlorine including chlorite: After obtaining Reading *C* add 1 mL H_2SO_4 solution to the same sample in titration flask, mix, and let stand about 2 min. Add 5 mL $NaHCO_3$ solution, mix, and titrate (Reading *D*).

d. Colorimetric procedure: Instead of titration with standard FAS solution, colorimetric procedures may be used to obtain the readings at each stage. Calibrate colorimeters with standard permanganate solution as directed in Section 408E.4*a*. Use of additional EDTA, disodium salt, with the DPD reagents is not required in colorimetric procedures.

4. Calculations

For 100 mL sample, 1 mL FAS solution = 1 mg available chlorine/L.

In the absence of chlorite:

Chlorine dioxide = $5G$ (or $1.9G$ expressed as ClO_2)

Free available chlorine = $A - G$

Monochloramine = $B - A$

Dichloramine = $C - B$

Total available chlorine = $C + 4G$

If the step leading to Reading *B* is omitted, monochloramine and dichloramine are obtained together when:

Combined available chlorine = $C - A$

If it is desired to check for presence of chlorite in sample, obtain Reading *D*. Chlorite is indicated if *D* is greater than $C + 4G$.

In the presence of chlorite:

Chlorine dioxide = $5G$ (or $1.9G$ expressed as ClO_2)

Chlorite = $D - (C + 4G)$

Free available chlorine = $A - G$

Monochloramine = $B - A$

Dichloramine = $C - B$

Total available chlorine = D

If *B* is omitted,

Combined available chlorine = $C - A$

410 D. Bibliography

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411 CHLORINE REQUIREMENT*

Chlorine demand is the quantity of chlorine that must be added to a unit volume of water to react with all the chlorine-reactable materials. Because some chlorination processes do not require complete satisfaction of the chlorine demand, chlorine requirement is the more applicable term.

Chlorine requirement is the quantity of chlorine that must be added to a unit volume of water under specified conditions (pH, contact time, temperature) to achieve a specified result. Examples include the quantity of chlorine required to limit the maximum bacterial count in wastewater or to oxidize iron and manganese in a potable water for subsequent removal to specified levels.

Where applicable, chlorine residuals may be determined by any of the methods of Section 408; the same method must be used for both laboratory testing and operational control.

In reporting results, include all conditions of testing such as pH, contact time, temperature, and the analytical procedures used.

Chlorine requirement is not an absolute test and the results of laboratory studies must be applied with caution to plant operations. The primary purpose of the test is to provide guidance in the control of chlorination for disinfection or other purposes.

411 A. Method for Control of Disinfection

1. General Discussion

Chlorine requirement can be determined on a plant or laboratory scale but, in most cases, it is better determined in the plant, under plant conditions, and with plant equipment.

In the plant test, the flow of wastewater

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*Approved by Standard Methods Committee, 1981 are sligh[illegible]ic because of the presence of bicarbon[illegible]nd carbonates of the alkali and alkaline earth metals.

1. General Discussion

a. Principle: The basic principle of electrometric pH measurement is determination of the activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode. The hydrogen electrode consists of a platinum electrode across which hydrogen gas is bubbled at a pressure of 101 kPa. Because of difficulty in its use and the potential for poisoning the hydrogen electrode, the glass electrode commonly is used. The electromotive force (emf) produced in the glass electrode system varies linearly with pH. This linear relationship is described by plotting the measured emf against the pH of different buffers. Sample pH is determined by extrapolation.

Because single ion activities such as a_{H^+} cannot be measured, pH is defined operationally on a potentiometric scale. The pH measuring instrument is calibrated potentiometrically with an indicating (glass) electrode and a reference electrode using National Bureau of Standards buffers having assigned values so that:

$$pH_s = -\log_{10} a_{H^+}$$

where:

pH_s = assigned pH of NBS buffer.

The operational pH scale is used to measure sample pH and is defined as:

$$pH_x = pH_s \pm \frac{F(E_x - E_s)}{2.303\,RT}$$

where:

pH_x = potentiometrically measured sample pH,
F = Faraday: 9.649×10^4 coulomb/mole,
E_x = sample emf, V,
E_s = buffer emf, V,
R = gas constant; 8.314 joule/(mole °K), and
T = absolute temperature, °K

NOTE: Although the equation for pH_x appears in the literature with a plus sign, the sign of emf readings in millivolts for most pH meters manufactured in the U.S. is negative. The choice of negative sign is consistent with the IUPAC Stockholm convention concerning the sign of electrode potential.[2,3]

The activity scale gives values that are higher than those on Sorenson's scale by 0.04 units:

$$pH\ (\text{activity}) = pH\ (\text{Sorenson}) + 0.04$$

The equation for pH_x assumes that the emf of the cells containing the sample and buffer is due solely to hydrogen ion activity unaffected by sample composition. In practice, samples will have varying ionic species and ionic strengths, both affecting H^+ activity. This imposes an experimental limitation on pH measurement; thus, to obtain meaningful results, the differences between E_x and E_s should be minimal. Samples must be dilute aqueous solutions of simple solutes ($< 0.2M$). (Choose buffers to bracket the sample.) Determination of pH cannot be made accurately in nonaqueous media, suspensions, colloids, or high-ionic-strength solutions.

b. Interferences: The glass electrode is relatively free from interference from color, turbidity, colloidal matter, oxidants, reductants, or high salinity, except for a sodium error at $pH > 10$. Reduce this error by using special "low sodium error" electrodes.

pH measurements are affected by temperature in two ways: mechanical effects that are caused by changes in the properties of the electrodes and chemical effects caused by equilibrium changes. In the first instance, the Nernstian slope increases with increasing temperature and electrodes take time to achieve thermal equilibrium. This can cause long-term drift in pH. Because chemical equilibrium affects pH, standard pH buffers have a specified pH at indicated temperatures.

Always report temperature at which pH is measured.

2. Apparatus

a. pH meter consisting of potentiometer, a glass electrode, a reference electrode, and a temperature compensating device. A circuit is completed through the potentiometer when the electrodes are immersed in the test solution. Many pH meters are capable of reading pH or millivolts and some have scale expansion that permits reading to 0.001 pH unit, but most instruments are not that precise.

For routine work use a pH meter accurate and reproducible to 0.1 pH unit with a range of 0 to 14 and equipped with a temperature compensation adjustment.

Although manufacturers provide operating instructions, the use of different descriptive terms may be confusing. For most instruments, there are two controls: intercept (set buffer, asymmetry, standardize) and slope (temperature, offset); their functions are shown diagramatically in Figures 423:1 and 2. The intercept control shifts the response curve laterally to pass through the isopotential point with no change in slope. This permits bringing the instrument on scale (0 mV) with a pH 7 buffer that has no change in potential with temperature.

The slope control rotates the emf/pH slope about the isopotential point (0 mV/pH 7). To adjust slope for temperature without disturbing the intercept, select a buffer that brackets the sample with pH 7 buffer and adjust slope control to pH of this buffer. The instrument will indicate correct millivolt change per unit pH at the test temperature.

b. Reference electrode consisting of a half cell that provides a constant electrode potential. Commonly used are calomel and silver: silver-chloride electrodes. Either is available with several types of liquid junctions.

The liquid junction of the reference electrode is critical because at this point the electrode forms a salt bridge with the sample or buffer and a liquid junction potential is generated that in turn affects the potential produced by the reference electrode. Reference electrode junctions may be annular ceramic, quartz, or asbestos fiber, or the sleeve type. The quartz type is most widely used. The asbestos fiber type is not recommended for strongly basic solutions. Follow the manufacturer's recommenda-

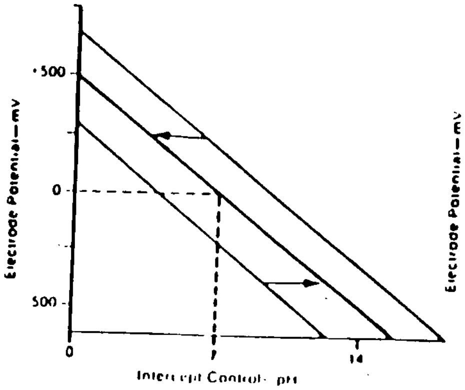

Figure 423:1. Electrode potential vs. pH. Intercept control shifts response curve laterally.

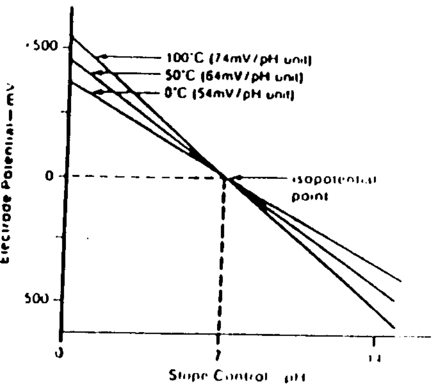

Figure 423:2. Typical pH electrode response as a function of temperature.

tion on use an[illegible]e of the reference electrode.

Refill nonsealed electrodes with the correct electrolyte to proper level and make sure junction is properly wetted.

c. Glass electrode: The sensor electrode is a bulb of special glass containing a fixed concentration of HCl or a buffered chloride solution in contact with an internal reference electrode. Upon immersion of a new electrode in a solution the outer bulb surface becomes hydrated and exchanges sodium ions for hydrogen ions to build up a surface layer of hydrogen ions. This, together with the repulsion of anions by fixed, negatively charged silicate sites, produces at the glass-solution interface a potential that is a function of hydrogen ion activity in solution.

Several types of glass electrodes are available. Combination electrodes incorporate the glass and reference electrodes into a single probe. Use a "low sodium error" electrode that can operate at high temperatures for measuring pH over 10 because standard glass electrodes yield erroneously low values. For measuring pH below 1 standard glass electrodes yield erroneously high values; use liquid membrane electrodes instead.

d. Beakers: Preferably use polyethylene or TFE‡ beakers.

e. Stirrer: Use either a magnetic, TFE-coated stirring bar or a mechanical stirrer with inert plastic-coated impeller.

f. Flow chamber: Use for continuous flow measurements or for poorly buffered solutions.

3. Reagents

a. General preparation: Calibrate the electrode system against standard buffer solutions of known pH. Because buffer solutions may deteriorate as a result of mold growth or contamination, prepare fresh as needed for accurate work by weighing the amounts of [illegible]icals specified in Table 423:I, dissol[illegible] distilled water at 25°C, and diluting [illegible] mL. This is particularly importan[illegible] borate and carbonate buffers.

Boil and cool distilled water having a conductivity of less than 2 µmhos/cm. To 50 mL add 1 drop of saturated KCl solution suitable for reference electrode use. If the pH of this test solution is between 6.0 and 7.0, use it to prepare all standard solutions.

Dry KH_2PO_4 at 110 to 130°C for 2 h before weighing but do not heat unstable hydrated potassium tetroxalate above 60°C nor dry the other specified buffer salts.

Although ACS-grade chemicals generally are satisfactory for preparing buffer solutions, use certified materials available from the National Bureau of Standards when the greatest accuracy is required. For routine analysis, use commercially available buffer tablets, powders, or solutions of tested quality. In preparing buffer solutions from solid salts, insure complete solution.

As a rule, select and prepare buffer solutions classed as primary standards in Table 423:I; reserve secondary standards for extreme situations encountered in wastewater measurements. Consult Table 423:II for accepted pH of standard buffer solutions at temperatures other than 25°C. In routine use, store buffer solutions and samples in polyethylene bottles. Replace buffer solutions every 4 weeks.

b. Saturated potassium hydrogen tartrate solution: Shake vigorously an excess (5 to 10 g) of finely crystalline $KHC_4H_4O_6$ with 100 to 300 mL distilled water at 25°C in a glass-stoppered bottle. Separate clear solution from undissolved material by decantation or filtration. Preserve for 2 months or more by adding one thymol crystal (8 mm diam) per 200 mL solution.

c. Saturated calcium hydroxide solution: Calcine a well-washed, low-alkali grade $CaCO_3$ in a platinum dish by igniting for

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‡Teflon or equivalent.

TABLE 423:I. PREPARATION OF pH STANDARD SOLUTIONS*

Standard Solution (molality)	pH at 25°C	Weight of Chemicals Needed/1000 mL Aqueous Solution at 25°C
Primary standards:		
Potassium hydrogen tartrate (saturated at 25°C)	3.557	> 7 g $KHC_4H_4O_6$†
0.05 potassium dihydrogen citrate	3.776	11.41 g $KH_2C_6H_5O_7$
0.05 potassium hydrogen phthalate	4.004	10.12 g $KHC_8H_4O_4$
0.025 potassium dihydrogen phosphate + 0.025 disodium hydrogen phosphate	6.863	3.387 g KH_2PO_4 + 3.533 g Na_2HPO_4‡
0.008 695 potassium dihydrogen phosphate + 0.030 43 disodium hydrogen phosphate	7.415	1.179 g KH_2PO_4 + 4.303 g Na_2HPO_4‡
0.01 sodium borate decahydrate (borax)	9.183	3.80 g $Na_2B_4O_7 \cdot 10H_2O$‡
0.025 sodium bicarbonate + 0.025 sodium carbonate	10.014	2.092 g $NaHCO_3$ + 2.640 g Na_2CO_3
Secondary standards:		
0.05 potassium tetroxalate dihydrate	1.679	12.61 g $KH_3C_4O_8 \cdot 2H_2O$
Calcium hydroxide (saturated at 25°C)	12.454	> 2 g $Ca(OH)_2$†

* DURST, R. A. 1975. Standard Reference Materials: Standardization of pH Measurements. NBS Spec. Publ. 260-53, National Bur. Standards, Washington D.C.

† Approximate solubility

‡ Prepare with freshly boiled and cooled distilled water (carbon-dioxide-free)

TABLE 423:II. STANDARD pH VALUES ASSIGNED BY THE NATIONAL BUREAU OF STANDARDS*

Temperature °C	Primary Standards							Secondary Standards	
	Tartrate (Saturated)	Citrate (0.05 *M*)	Phthalate (0.05 *M*)	Phosphate (1:1)	Phosphate (1:3.5)	Borax (0.01 *M*)	Bicarbonate-Carbonate (0.025 *M*)	Tetroxalate (0.05 *M*)	Ca[illegible] (S[illegible]ated)
0			4.003	6.982	7.534	9.460	10.321	1.666	
5			3.998	6.949	7.501	9.392	10.248	1.668	
10			3.996	6.921	7.472	9.331	10.181	1.670	
15			3.996	6.898	7.449	9.276	10.120	1.672	
20			3.999	6.878	7.430	9.227	10.064	1.675	
25	3.557	3.776	4.004	6.863	7.415	9.183	10.014	1.679	12.454
30	3.552		4.011	6.851	7.403	9.143	9.968	1.683	
35	3.549		4.020	6.842	7.394	9.107	9.928	1.688	
37			4.024	6.839	7.392	9.093			
40	3.547		4.030	6.836	7.388	9.074	9.891	1.694	
45	3.547		4.042	6.832	7.385	9.044	9.859	1.700	
50	3.549		4.055	6.831	7.384	9.017	9.831	1.707	
55	3.554		4.070					1.715	
60	3.560		4.085					1.723	
70	3.580		4.12					1.743	
80	3.609		4.16					1.766	
90	3.650		4.19					1.792	
95	3.674		4.21					1.806	

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* DURST, R. A. 1975. Standard Reference Materials: Standardization of pH Measurements. NBS Spec. Publ. 260-53. National Bur. Standards, Washington, D.C.

1 h at 1000°C. Cool, hydrate by slowly adding distilled water with stirring, and heat to boiling. Cool, filter, and collect solid $Ca(OH)_2$ on a fritted glass filter of medium porosity. Dry at 110°C, cool, and pulverize to uniformly fine granules. Vigorously shake an excess of fine granules with distilled water in a stoppered polyethylene bottle. Let temperature come to 25°C after mixing. Filter supernatant under suction through a sintered glass filter of medium porosity and use filtrate as the buffer solution. Discard buffer solution when atmospheric CO_2 causes turbidity to appear.

d. Auxiliary solutions: 0.1*N* NaOH, 0.1*N* HCl, 5*N* HCl (dilute five volumes 6*N* HCl with one volume distilled water), and acid potassium fluoride solution (dissolve 2 g KF in 2 mL conc H_2SO_4 and dilute to 100 mL with distilled water).

4. Procedure

a. Instrument calibration: In each case follow manufacturer's instructions for pH meter and for storage and preparation of electrodes for use. Recommended solutions for short-term storage of electrodes vary with type of electrode and manufacturer, but generally have a conductivity greater than 4000 µmhos/cm. Tap water is a better substitute than distilled water, but pH 4 buffer is best for the single glass electrode and saturated KCl is preferred for a calomel and Ag/AgCl reference electrode. Saturated KCl is the preferred solution for a combination electrode. Keep electrodes wet by returning them to storage solution whenever pH meter is not in use.

Before use, remove electrodes from storage solution, rinse, blot dry with a soft tissue, place in initial buffer solution, and set the isopotential point (¶ 2*a* above). Select a second buffer within 2 pH units of sample pH and bring sample and buffer to same temperature, which may be the room temperature, a fixed temperature such as 25°C, or the temperature of a fresh sample. Remove electrodes from first buffer, rinse thoroughly with distilled w[illegible] blot dry, and immerse in second buffer[illegible]cord temperature of measurement and adjust temperature dial on meter so that meter indicates pH value of buffer at test temperature (this is a slope adjustment).

Use the pH value listed in the tables for the buffer used at the test temperature. Remove electrodes from second buffer, rinse thoroughly with distilled water and dry electrodes as indicated above. Immerse in a third buffer below pH 10, approximately 3 pH units different from the second; the reading should be within 0.1 unit for the pH of the third buffer. If the meter response shows a difference greater than 0.1 pH unit from expected value, look for trouble with the electrodes or potentiometer (see ¶s 5*a* and *b* below).

The purpose of standardization is to adjust the response of the glass electrode to the instrument. When only occasional pH measurements are made standardize instrument before each measurement. When frequent measurements are made and the instrument is stable, standardize less frequently. If sample pH values vary widely, standardize for each sample with a buffer having a pH within 1 to 2 pH units of the sample.

b. Sample analysis: Establish equilibrium between electrodes and sample by stirring sample to insure homogeneity; stir gently to minimize carbon dioxide entrainment. For buffered samples or those of high ionic strength, condition electrodes after cleaning by dipping them into sample for 1 min. Blot dry, immerse in a fresh portion of the same sample, and read pH.

With dilute, poorly buffered solutions, equilibrate electrodes by immersing in three or four successive portions of sample. Take a fresh sample to measure pH.

5. Trouble Shooting

a. Potentiometer: To locate trouble source disconnect electrodes and, using a short-circuit strap, connect reference electrode termin[illegible] glass electrode terminal. Observe change in pH when instrument calibration knob is adjusted. If potentiometer is operating properly, it will respond rapidly and evenly to changes in calibration over a wide scale range. A faulty potentiometer will fail to respond, will react erratically, or will show a drift upon adjustment. Switch to the millivolt scale on which the meter should read zero. If inexperienced, do not attempt potentiometer repair other than maintenance as described in instrument manual.

b Electrodes: If potentiometer is functioning properly, look for the instrument fault in the electrode pair. Substitute one electrode at a time and cross-check with two buffers that are about 4 pH units apart. A deviation greater than 0.1 pH unit indicates a faulty electrode. Glass electrodes fail because of scratches, deterioration, or accumulation of debris on the glass surface. Rejuvenate electrode by alternately immersing it three times each in 0.1*N* HCl and 0.1*N* NaOH. If this fails, immerse tip in KF solution for 30 s. After rejuvenation, soak in pH 7.0 buffer overnight. Rinse and store in pH 7.0 buffer. Rinse again with distilled water before use. Protein coatings can be removed by soaking glass electrodes in a 10% pepsin solution adjusted to pH 1 to 2.

To check reference electrode, oppose the emf of a questionable reference electrode against another one of the same type that is known to be good. Using an adapter, plug good reference electrode into glass electrode jack of potentiometer; then plug questioned electrode into reference electrode jack. Set meter to read millivolts and take readings with both electrodes immersed in the same electrolyte (KCl) solution and then in the same buffer solution. The millivolt readings should be 0 ± 5 mV for both solutions. If different electrodes are used, i.e., silver: silver-chloride against calomel or vice versa, the reading will be 44 ± 5 mV for a good reference electrode.

Reference electrode troubles generally are traceable to a clogged junction. Interruption of the continuous trickle of electrolyte through the junction causes increase in response time and drift in reading. Clear a clogged junction by applying suction to the tip or by boiling tip in distilled water until the electrolyte flows freely when suction is applied to tip or pressure is applied to the fill hole. Replaceable junctions are available commercially.

6. Precision and Accuracy

By careful use of a laboratory pH meter with good electrodes, a precision of ±0.02 pH unit and an accuracy of ±0.05 pH unit can be achieved. However, ±0.1 pH unit represents the limit of accuracy under normal conditions, especially for measurement of water and poorly buffered solutions. For this reason, report pH values to the nearest 0.1 pH unit. A synthetic sample of a Clark and Lubs buffer solution of pH 7.3 was analyzed electrometrically by 30 laboratories with a standard deviation of ±0.13 pH unit.

7. References

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424 PHOSPHORUS*

Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates, condensed phosphates (pyro-, meta-, and other polyphosphates), and organically bound phosphates. They occur in solution, in particles or detritus, or in the bodies of aquatic organisms.

These forms of phosphate arise from a variety of sources. Small amounts of certain condensed phosphates are added to some water supplies during treatment. Larger quantities of the same compounds may be added when the water is used for laundering or other cleaning, because these materials are major constituents of many commercial cleaning preparations. Phosphates are used extensively in the treatment of boiler waters. Orthophosphates applied to agricultural or residential cultivated land as fertilizers are carried into surface waters with storm runoff and to a lesser extent with melting snow. Organic phosphates are formed primarily by biological processes. They are contributed to sewage by body wastes and food residues, and also may be formed from orthophosphates in biological treatment processes or by receiving water biota.

Phosphorus is essential to the growth of organisms and can be the nutrient that limits the primary productivity of a body of water. In instances where phosphate is a growth-limiting nutrient, the discharge of raw or treated wastewater, agricultural drainage, or certain industrial wastes to that water may stimulate the growth of photosynthetic aquatic micro- and macroorganisms in nuisance quantities.

Phosphates also occur in bottom sediments and in biological sludges, both as precipitated inorganic forms and incorporated into organic compounds.

1. Definition of Terms

Phosphorus analyses embody two general procedural steps: (*a*) conversion of the phosphorus form of interest to dissolved orthophosphate, and (*b*) colorimetric determination of dissolved orthophosphate. The separation of phosphorus into its various forms is defined analytically but the analytical differentiations have been selected so that they may be used for interpretive purposes.

Filtration through a 0.45 μm pore diam

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*Approved by Standard Methods Committee, 1984.