commentletters

From: Jerry Tamura <jerryitamura@yahoo.com>
Sent: Friday, September 16, 2016 6:00 AM
To: commentletters
Subject: Comment Letter – ELAP Regulations Development/Laboratory Standard
Attachments: SOP_for_pH_2010.pdf

To the Members of the State Water Resources Control Board,

I have worked in the Environmental Laboratories in California since 1983 and I would like to share an example of a laboratory procedure to perform one of the simplest analysis, reading pH's.

I have attached a pdf file titled SOP_for_pH_2010 which was written six years. In this file, you will find a Power Point presentation presented to the Wastewater Operators titled "Preparing for the Regional Water Board Audit on pH Measurements" and the training manual handout contents of the following: I. Table of Contents; II. Phone Log; III. Methods - Initial Demonstration of Capability; IV. 40 CFR 136; V. Section 4500-H+ pH Value of Standard Methods for the Examination of Water and Wastewater 20th Edition 1998; Safety Data Sheets (SDS); Standard Operating Procedure Methods - Written almost in TNI standards; pH Form for Logging in Data (Worksheet); and Certification of Completion.

I respectfully ask the Board Members,
How much more 2016 TNI Standards do you need apply to read pH's?

If you have any questions then I can be reached at (559) 259-3083

Sincerely,

Jerry I. Tamura

Sierra Foothill Laboratory, Inc.

Three Locations in California
Jackson
Colusa
El Dorado Hills

Preparing for the Regional Water Board Audit on pH Measurements

Presented by
Jerry I. Tamura
Sierra Foothill Laboratory, Inc.
Jackson, CA

California Water Boards

What is the purpose of this talk?

On several California Regional Water Quality Control Board Waste Discharge Requirements

NPDES

The following was written:

Chemical, bacteriological, and bioassay analyses shall be conducted at a laboratory certified for such analyses by the State Department of Health Services. In the event a certified laboratory is not available to the Discharger, analyses performed by a non-certified laboratory will be accepted provided a Quality Assurance-Quality Control Program is instituted by the laboratory. A manual containing the steps followed in this program must be kept in the laboratory and shall be available for inspection by Regional Water Board staff. The Quality Assurance-Quality Control Program must conform to USEPA guidelines or to procedures approved by the Regional Water Board.

According to
40 Code of Federal Regulation
(CFR) Monday; March 26, 2007
Part 136 & 503
Table II.-Required Container, Preservation Techniques, and Holding Times
Table 1B-Inorganic Tests:

Hydrogen ion (pH)

Preservation: None Required

Maximum Holding Time:
Analyze within 15 minutes

TABLE II:
REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

D:\March23_2011_Colusa_CWEA_Seminar

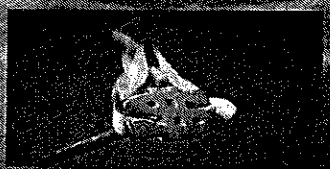
State Certified Laboratory Environmental Laboratory Accreditation Program (ELAP)

Field of Testing 108: Inorganic Chemistry of Wastewater

Subgroup	Analyte Code	Method	Analyte
108.490	001	SM4500-H+ B	pH

CDPH

ELAP Cost Analyses



The Basic Fees are $1003.00 and the Field of Testing Fee is $452.00.

Basic Fee + Number of Fields of Testing Requested times the Field of Testing Fee = Total Fee

Wastewater + Water

$1003 + $452 = $ $1455

Base Fee + (Number of FOTs X $452) = Total Fee Amount

ELAP Definition of a Laboratory Director

The person who is in charge of all analytical and laboratory processes; supervision of laboratory persons, including those designated as Principal Analysts; and is the final person responsible for the quality of data.

TITLE 22. Social Security

Division 4. Environmental Health

Chapter 19. Certification of Environmental Laboratories

Article 9. Laboratory Personnel

Minimum Requirements will be CWEA or CA-NV/AWWA Certificate

Laboratory Analyst II

Minimum Elements

GUIDANCE ON PREPARATION OF LABORATORY QUALITY ASSURANCE PLANS

DOCUMENT CONTROL NUMBER

EPA 910/9-92-032

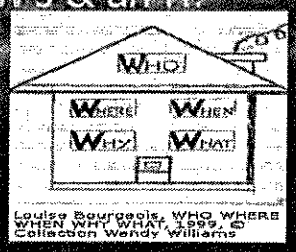
U.S. ENVIRONMENTAL PROTECTION AGENCY
1200 SIXTH AVENUE
SEATTLE, WASHINGTON 98101

- Title Page
- Table of Contents
- Quality Assurance Policy Statement
- Corporate Ethics Policy on Waste, Fraud, and Abuse
- Quality Assurance Management
- Administrative Organization
- Personnel Qualifications
- Facility Description and Capital Equipment
- Preventive Maintenance

- Corrective Action
- Laboratory Evaluation and Audits
- Quality Assurance Reports to Management
- Lab Documentation and Forms
- Sub Contracting of Services
- Standard Operating Procedures
- Laboratory Personnel Training Record
- This will consist of revision, date and page number on each page of the document.

The Five W's & an H!

- Who
- What
- When
- Where
- Why
- How



According to the

CDPH ELAP's

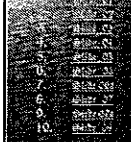
CHECKLIST FOR LABORATORY ON-SITE INSPECTION

Sample Collection Information

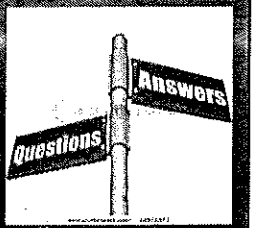
- Identification (number)
- Source (location collected)
- Name of system
- Date collected
- Time collected
- Name of collector

Sample type –
DW, WW, surface, process, routine, repeat, etc.

Manual Prepared by Sierra Foothill Laboratory, Inc.



Questions



Contact Information
Jerry I. Tamura
Jerry@sierralab.com
Phone: 209 223 2800

I. Table of Contents

II

Phone Log to Sierra Foothill Laboratory, Inc. (209) 223 2800

Date: ___/___/___ **Time:**_____ **Contact Person:**_____

Initials: ____

Date: ___/___/___ **Time:**_____ **Contact Person:**_____

Initials: ____

Date: ___/___/___ **Time:**_____ **Contact Person:**_____

Initials: ____

Date: ___/___/___ **Time:**_____ **Contact Person:**_____

Initials: ____

Prepared by Sierra Foothill Laboratory, Inc.
For _____
Standard Operating Procedure

METHODS
Initial Demonstration of Capability and Authorization

SCOPE AND APPLICATION
Describes sequence of events which are to take place prior to employee of El Dorado Irrigation District using the method.

NAME (TYPE OR PRINT)	INITIALS

SOP pH
METHOD NAME

4500-H+ B. Electrometric Method, Standard Methods For The Examination of Water and Wastewater 20th Edition 1998
METHOD REFERENCES

STEP 1=
I HAVE READ, UNDERSTAND, AND WILL COMPLY WITH THE FOLLOWING:

METHOD REFERENCE(S) LISTED ABOVE, READ BY ME_____DATE/INITIAL

SOPs LISTED ABOVE, READ BY ME _____DATE/INITIAL

MSDS FOR EVERY CHEMICAL OR REAGENT USED IN THE ANALYSIS, READ BY ME _____DATE/INITIAL

STEP 2=
Perform five analyses of ***real world samples*** under the auspices of the Supervisor.
Supervisor indicates on line below the date/time of each analytical run, and then initials it.

Date/time/initial Supervisor _____ _____ _____
#1 #2 #3

_____ _____
#4 #5

STEP 3=
Operator signs below, sheet goes to Supervisor to be reviewed by the Assessor.

_____ Date
Signature of Operator

STEP 4=
Supervisor will be reviewing the data on the log book of the four lab analyses performed by the Operator.
The Assessor will finalize Initial Document of Competence and authorize analyst to proceed.

METHODS
Initial Demonstration of Capability and Authorization

Date:_____

Demonstration of Capability
Certification Statement

Sierra Foothill Laboratory
255 Scottsville Blvd
PO Box 1268
Jackson, CA 95642

Operator(s) Name(S):_____

Matrix:_____
(Examples: laboratory pure water, water, and wastewater)

Method:_____

We, the undersigned, CERTIFY that:

1. The Operator(s) above, using the cited test method(s), which is in use at this facility for the analysis for the analyses under the assessment of the Sierra Foothill Laboratory have met the Demonstration of Capability.
2. The test method(s) was performed by the Operator (s) identified on this certification.
3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.
4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Jerry I. Tamura
Laboratory Technical Director _____ _____
Signature Date

Karen Lantz
Quality Assurance Director _____ _____
Signature Date

METHODS
Initial Demonstration of Capability and Authorization

Effective Date	Revisions Made	Date/Initials	

IV

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS FOR WASTEWATER AND SEWAGE SLUDGE

Parameter and units	Method [1]	EPA	Standard methods 18th, 19th, 20th ed.	Standard methods online	AOAC, ASTM, USGS	Other
Bacteria:						
1. Coliform (fecal), number per 100 mL or number per gram dry weight.	Most Probable Number (MPN),[5] tube 3 dilution, or	p. 132[3] ..... 1680[12,14] ..... 1681[12,19]	9221 C E	9221 C E–99.		
	Membrane filter (MF)[2], single step.	p. 124[3]	9222 D	9222 D–97	B–0050–85[6].	
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 132[3]	9221 C E	9221 C E–99.		
	MF[2], single step	p. 124[3]	9222 D	9222 D–97.		
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B	9221 B–99.		
	MF[2], single step or two step.	p. 108[3]	9222 B	9222 B–97	B–0025–85[5].	
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114[3]	9221 B	9221 B–99.		
	MF[2] with enrichment ...	p. 111[3]	9222 (B+B.5c)	9222 (B+B.5c)–97.		
5. *E. coli*, number per 100 mL[20].	MPN[7,9,15] multiple tube/multiple well.	.....	9223 B[13]	9223 B–97[13]	991.15[11]	Colilert®[13,17] Colilert-18®[13,16,17]
	MF[2,6,7,8,9] single step ..	1603[21]	.....	.....	.....	mColiBlue 24®[18]
6. Fecal streptococci, number per 100 mL.	MPN, 5 tube 3 dilution,	p. 139[3]	9230 B	9230 B–93.		
	MF[2], or	p. 136[3]	9230 C	9230 C–93	B–0055–85[5].	
	Plate count	p. 143[3].				
7. Enterococci, number per 100 mL[20].	MPN[7,9], multiple tube/multiple well.	.....	.....	.....	D6503–99[10]	Enterolert®[13,23]
	MF[2,6,7,8,9] single step ..	1600[24].				
8. Salmonella, number per gram dry weight[12].	MPN multiple tube	1682[22].				
Aquatic Toxicity:						
9. Toxicity, acute, fresh water organisms, LC_{50}, percent effluent.	*Ceriodaphnia dubia* acute.	2002.0[25].				
	Daphnia puplex and *Daphnia magna* acute.	2021.0[25].				
	Fathead Minnow, *Pimephales promelas*, and Bannerfin *shiner*, *Cyprinella leedsi*, acute.	2000.0[25].				

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS FOR WASTEWATER AND SEWAGE SLUDGE—Continued

Parameter and units	Method[1]	EPA	Standard methods 18th, 19th, 20th ed.	Standard methods online	AOAC, ASTM, USGS	Other
	Rainbow Trout, *Oncorhynchus mykiss*, and brook trout, *Salvelinus fontinalis*, acute.	2019.0 [25].				
10. Toxicity, acute, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, LC_{50}, percent effluent.	Mysid, *Mysidopsis bahia*, acute.	2007.0 [25].				
	Sheepshead Minnow, *Cyprinodon variegatus*, acute.	2004.0 [25].				
	Silverside, *Menidia beryllina*, *Menidia menidia*, and *Menidia peninsulae*, acute.	2006.0 [25].				
11. Toxicity, chronic, fresh water organisms, NOEC or IC_{25}, percent effluent.	Fathead minnow, *Pimephales promelas*, larval survival and growth.	1000.0 [26].				
	Fathead minnow, *Pimephales promelas*, embryo-larval survival and teratogenicity.	1001.0 [26].				
	Daphnia, *Ceriodaphnia dubia*, survival and reproduction.	1002.0 [26].				
	Green alga, *Selenastrum capricornutum*, growth.	1003.0 [26].				
12. Toxicity, chronic, estuarine and marine organisms of the Atlantic Ocean and Gulf of Mexico, NOEC or IC_{25}, percent effluent.	Sheepshead minnow, *Cyprinodon variegatus*, larval survival and growth.	1004.0 [27].				
	Sheepshed minnow, *Cyprinodon variegatus*, embryo-larval survival and teratogenicity.	1005.0 [27].				
	Inland silverside, *Menidia beryllina*, larval survival and growth.	1006.0 [27].				
	Mysid, *Mysidopsis bahia*, survival, growth, and fecundity.	1007.0 [27].				
	Sea urchin, *Arbacia punctulata*, fertilization.	1008.0 [27].				

[1] The method must be specified when results are reported.
[2] A 0.45 µm membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.
[3] USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH, EPA/600/8–78/017.
[4] [Reserved].
[5] USGS. 1989. U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of the Interior, Reston, VA.
[6] Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.
[7] Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

[8] When the MF method has been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

[9] To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

[10] ASTM. 2000, 1999, 1996. Annual Book of ASTM Standards—Water and Environmental Technology. Section 11.02. ASTM International. 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[11] AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. Association of Official Analytical Chemists International. 481 North Frederick Avenue, Suite 500, Gaithersburg, MD 20877–2417.

[12] Recommended for enumeration of target organism in sewage sludge.

[13] These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme β-glucuronidase produced by *E. coli*.

[14] USEPA. July 2006. Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium. US Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–012.

[15] Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert® may be enumerated with the multiple-well procedures, Quanti-Tray® Quanti-Tray® 2000, and the MPN calculated from the table provided by the manufacturer.

[16] Colilert-18® is an optimized formulation of the Colilert® for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 °C rather than the 24 h required for the Colilert® test and is recommended for marine water samples.

[17] Descriptions of the Colilert®, Colilert-18®, Quanti-Tray®, and Quanti-Tray®/2000 may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

[18] A description of the mColiBlue24® test, Total Coliforms and *E. coli*, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.

[19] USEPA. July 2006. Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A–1 Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–013.

[20] Recommended for enumeration of target organism in wastewater effluent.

[21] USEPA. July 2006. Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–011.

[22] USEPA. July 2006. Method 1682: *Salmonella* in Sewage Sludge (Biosolids) by Modified Semisolid Rappaport-Vassiliadis (MSRV) Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–014.

[23] A description of the Enterolert® test may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

[24] USEPA. July 2006. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-β-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA–821–R–06–009.

[25] USEPA. October 2002. Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms. Fifth Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R–02/012.

[26] USEPA. October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Fourth Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R–02/013.

[27] USEPA. October 2002. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Third Edition. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA/821/R–02/014.

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
Table IA—Bacterial Tests:			
1–5. Coliform, total, fecal, and *E. coli*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours.[22,23]
6. Fecal streptococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours.[22]
7. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours.[22]
8. Salmonella	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours.[22]
Table IA—Aquatic Toxicity Tests:			
9–11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6 °C [16]	36 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, FP, G	Cool, ≤6 °C [18]	14 days.
2. Alkalinity	P, FP, G	Cool, ≤6 °C [18]	14 days.
4. Ammonia	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6 °C [18]	48 hours.
10. Boron	P, FP, or Quartz	HNO_3 to pH<2	6 months.
11. Bromide	P, FP, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous.	P, FP G	Cool, ≤6 °C [18]	48 hours.
15. Chemical oxygen demand	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
16. Chloride	P, FP, G	None required	28 days.
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes.
21. Color	P, FP, G	Cool, ≤6 °C [18]	48 hours.
23–24. Cyanide, total or available (or CATC)	P, FP, G	Cool, ≤6 °C [18], NaOH to pH>12 [6], reducing agent [5].	14 days.
25. Fluoride	P	None required	28 days.
27. Hardness	P, FP, G	HNO_3 or H_2SO_4 to pH<2	6 months.
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes.
31, 43. Kjeldahl and organic N	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
Table IB—Metals: [7]			
18. Chromium VI	P, FP, G	Cool, ≤6 °C [18], pH = 9.3–9.7 [20]	28 days.
35. Mercury (CVAA)	P, FP, G	HNO_3 to pH<2	28 days.
35. Mercury (CVAFS)	FP, G; and FP-lined cap [17].	5 mL/L 12N HCl or 5 mL/L BrCl [17]	90 days.[17]
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO_3 to pH<2, or at least 24 hours prior to analysis [19].	6 months.
38. Nitrate	P, FP, G	Cool, ≤6 °C [18]	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
40. Nitrite	P, FP, G	Cool, ≤6 °C [18]	48 hours.
41. Oil and grease	G	Cool to ≤6 °C [18], HCl or H_2SO_4 to pH<2.	28 days.
42. Organic Carbon	P, FP, G	Cool to ≤6 °C [18], HCl, H_2SO_4, or H_3PO_4 to pH<2.	28 days.
44. Orthophosphate	P, FP, G	Cool, ≤6 °C [18]	Filter within 15 minutes; Analyze within 48 hours.
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes.
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours.
48. Phenols	G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
49. Phosphorous (elemental)	G	Cool, ≤6 °C [18]	48 hours.
50. Phosphorous, total	P, FP, G	Cool, ≤6 °C [18], H_2SO_4 to pH<2	28 days.
53. Residue, total	P, FP, G	Cool, ≤6 °C [18]	7 days.
54. Residue, Filterable	P, FP, G	Cool, ≤6 °C [18]	7 days.
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, ≤6 °C [18]	7 days.
56. Residue, Settleable	P, FP, G	Cool, ≤6 °C [18]	48 hours.
57. Residue, Volatile	P, FP, G	Cool, ≤6 °C [18]	7 days.
61. Silica	P or Quartz	Cool, ≤6 °C [18]	28 days.
64. Specific conductance	P, FP, G	Cool, ≤6 °C [18]	28 days.
65. Sulfate	P, FP, G	Cool, ≤6 °C [18]	28 days.
66. Sulfide	P, FP, G	Cool, ≤6 °C [18], add zinc acetate plus sodium hydroxide to pH>9.	7 days.
67. Sulfite	P, FP, G	None required	Analyze within 15 minutes.
68. Surfactants	P, FP, G	Cool, ≤6 °C [18]	48 hours.
69. Temperature	P, FP, G	None required	Analyze.
73. Turbidity	P, FP, G	Cool, ≤6 °C [18]	48 hours.
Table IC—Organic Tests [8]			

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 76, 104, 105, 108–111, 113. Purgeable Halocarbons.	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5]	14 days.
6, 57, 106. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5], HCl to pH 2 [9],	14 days.[9]
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5], pH to 4–5 [10],	14 days.[10]
23, 30, 44, 49, 53, 77, 80, 81, 98, 100, 112. Phenols [11],	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction.
7, 38. Benzidines [11, 12]	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction.[13]
14, 17, 48, 50–52. Phthalate esters [11]	G, FP-lined cap	Cool, ≤6 °C [18]	7 days until extraction, 40 days after extraction.
82–84. Nitrosamines [11, 14]	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
88–94. PCBs [11]	G, FP-lined cap	Cool, ≤6 °C [18]	1 year until extraction, 1 year after extraction.
54, 55, 75, 79. Nitroaromatics and isophorone [11],	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
1, 2, 5, 8–12, 32, 33, 58, 59, 74, 78, 99, 101. Polynuclear aromatic hydrocarbons [11],	G, FP-lined cap	Cool, ≤6 °C [18], store in dark, 0.008% $Na_2S_2O_3$ [5].	7 days until extraction, 40 days after extraction.
15, 16, 21, 31, 87. Haloethers [11]	G, FP-lined cap	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5]	7 days until extraction, 40 days after extraction.
29, 35–37, 63–65, 107. Chlorinated hydrocarbons [11],	G, FP-lined cap	Cool, ≤6 °C [18]	7 days until extraction, 40 days after extraction.
60–62, 66–72, 85, 86, 95–97, ,102, 103. CDDs/CDFs [11],			
Aqueous Samples: Field and Lab Preservation	G	Cool, ≤6 °C [18], 0.008% $Na_2S_2O_3$ [5], pH<9.	1 year.
Solids and Mixed-Phase Samples: Field Preservation.	G	Cool, ≤6 °C [18]	7 days.
Tissue Samples: Field Preservation	G	Cool, ≤6 °C [18]	24 hours.
Solids, Mixed-Phase, and Tissue Samples: Lab Preservation.	G	Freeze, ≤−10 °C	1 year.
Table ID—Pesticides Tests:			
1–70. Pesticides [11]	G, FP-lined cap	Cool, ≤6 °C [18], pH 5–9 [15]	7 days until extraction, 40 days after extraction.
Table IE—Radiological Tests:			
1–5. Alpha, beta, and radium	P, FP, G	HNO_3 to pH<2	6 months.
Table IH—Bacterial Tests:			
1. *E. coli*	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours.[22]
2. Enterococci	PA, G	Cool, <10 °C, 0.0008% $Na_2S_2O_3$ [5]	6 hours.[22]
Table IH—Protozoan Tests:			
8. Cryptosporidium	LDPE; field filtration	0–8 °C	96 hours.[21]
9. Giardia	LDPE; field filtration	0–8 °C	96 hours.[21]

[1] "P" is polyethylene; "FP" is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon®), or other fluoropolymer, unless stated otherwise in this Table II; "G" is glass; "PA" is any plastic that is made of a sterlizable material (polypropylene or other autoclavable plastic); "LDPE" is low density polyethylene.

[2] Except where noted in this Table II and the method for the parameter, preserve each grab sample within 15 minutes of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), refrigerate the sample at ≤6 °C during collection unless specified otherwise in this Table II or in the method(s). For a composite sample to be split into separate aliquots for preservation and/or analysis, maintain the sample at ≤6 °C, unless specified otherwise in this Table II or in the method(s), until collection, splitting, and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of a grab sample, a composite sample, or an aliquot split from a composite sample; otherwise, preserve the grab sample, composite sample, or aliquot split from a composite sample within 15 minutes of collection. If a composite measurement is required but a composite sample would compromise sample integrity, individual grab samples must be collected at prescribed time intervals (e.g., 4 samples over the course of a day, at 6-hour intervals). Grab samples must be analyzed separately and the concentrations averaged. Alternatively, grab samples may be collected in the field and composited in the laboratory if the compositing procedure produces results equivalent to results produced by arithmetic averaging of the results of analysis of individual grab samples. For examples of laboratory compositing procedures, see EPA Method 1664A (oil and grease) and the procedures at 40 CFR 141.34(f)(14)(iv) and (v) (volatile organics).

[3] When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that, for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). For a grab sample, the holding time begins at the time of collection. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), the holding time begins at the time of the end of collection of the composite sample. For a set of grab samples composited in the field or laboratory, the holding time begins at the time of collection of the last grab sample in the set. Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability. See § 136.3(e) for details. The date and time of collection of an individual grab sample is the date and time at which the sample is collected. For a set of grab samples to be composited, and that are all collected on the same calendar date, the date of collection is the date on which the samples are collected. For a set of grab samples to be composited, and that are collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15. For a composite sample collected automatically on a given date, the date of collection is the date on which the sample is collected. For a composite sample collected automatically, and that is collected across two calendar dates, the date of collection is the dates of the two days; e.g., November 14–15.

[5] Add a reducing agent only if an oxidant (e.g., chlorine) is present. Reducing agents shown to be effective are sodium thiosulfate ($Na_2S_2O_3$), ascorbic acid, sodium arsenite ($NaAsO_2$), or sodium borohydride ($NaBH_4$). However, some of these agents have been shown to produce a positive or negative cyanide bias, depending on other substances in the sample and the analytical method used. Therefore, do not add an excess of reducing agent. Methods recommending ascorbic acid (e.g., EPA Method 335.4) specify adding ascorbic acid crystals, 0.1–0.6 g, until a drop of sample produces no color on potassium iodide (KI) starch paper, then adding 0.06 g (60 mg) for each liter of sample volume. If $NaBH_4$ or $NaAsO_2$ is used, 25 mg/L $NaBH_4$ or 100 mg/L $NaAsO_2$ will reduce more than 50 mg/L of chlorine (see method "Kelada-01" and/or Standard Method 4500–CN^- for more information). After adding reducing agent, test the sample using KI paper, a test strip (e.g. for chlorine, SenSafe™ Total Chlorine Water Check 480010) moistened with acetate buffer solution (see Standard Method 4500–Cl.C.3e), or a chlorine/oxidant test method (e.g., EPA Method 330.4 or 330.5), to make sure all oxidant is removed. If oxidant remains, add more reducing agent. Whatever agent is used, it should be tested to assure that cyanide results are not affected adversely.

[6] Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to > 12 with sodium hydroxide solution (e.g., 5% w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to > 12 and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH > 12 for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary.

(1) Sulfur: To remove elemental sulfur (S_8), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to > 12 with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration.

(2) Sulfide: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4.4 L collapsible container (e.g., Cubitainer™). Acidify with concentrated hydrochloric acid to pH < 2. Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to > 12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Dynamic stripping: In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH < 2. Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to > 12, refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide (> 10%). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. For removal of sulfide by precipitation, raise the pH of the sample to > 12 with NaOH solution, then add approximately 1 mg of powdered cadmium chloride for each mL of sample. For example, add approximately 500 mg to a 500-mL sample. Cap and shake the container to mix. Allow the precipitate to settle and test the sample with lead acetate paper. If necessary, add cadmium chloride but avoid adding an excess. Finally, filter through 0.45 micron filter. Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in µg or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand-exchange reagent to offset any excess of cadmium chloride.

(3) Sulfite, thiosulfate, or thiocyanate: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA–1677) to preclude cyanide loss or positive interference.

(4) Aldehyde: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(5) Carbonate: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to ≥12 using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500–CN.B.3.d).

(6) Chlorine, hypochlorite, or other oxidant: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.

[7] For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] If the sample is not adjusted to pH 2, then the sample must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to ≤6 °C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 30 days at < 0 °C.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[16] Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the preservation temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

[17] Samples collected for the determination of trace level mercury (<100 ng/L) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

[18] Aqueous samples must be preserved at ≤6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of "≤°C" is used in place of the "4 °C" and "< 4 °C" sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤6 °C requirement. The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).

[19] An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

[20] To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

[21] Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

[22] Samples analysis should begin immediately, preferably within 2 hours of collection. The maximum transport time to the laboratory is 6 hours, and samples should be processed within 2 hours of receipt at the laboratory.

[23] For fecal coliform samples for sewage sludge (biosolids) only, the holding time is extended to 24 hours for the following sample types using either EPA Method 1680 (LTB–EC) or 1681 (A–1): Class A composted, Class B aerobically digested, and Class B anaerobically digested.

PART 503—STANDARDS FOR THE USE OR DISPOSAL OF SEWAGE SLUDGE

■ 3. The authority citation for Part 503 continues to read as follows:

Authority: Secs. 405(d) and (e) of the Clean Water Act, as amended by Pub. L. 95–217, sec. 54(d), 91 Stat. 1591 (33 U.S.C. 1345(d) and (e)); and Pub. L. 100–4, title IV, sec. 406(a), (b), 101 Stat., 71, 72 (33 U.S.C. 1251 et seq.).

■ 4. Section 503.8 is amended by revising paragraph (b) introductory text to read as follows:

§ 503.8 Sampling and analysis.

* * * * *

(b) *Methods.* The materials listed below are incorporated by reference in this part. These incorporations by reference were approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. The materials are incorporated as they exist on the date of approval, and notice of any change in these materials will be published in the **Federal Register**. They are available for inspection at the HQ Water Docket Center, EPA/DC, EPA West, Room B102, 1301 Constitution Ave., NW., Washington, DC, and at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202–741–6030, or go to: *http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.*

Copies may be obtained from the standard producer or publisher listed in the regulation. The methods in the materials listed below (or in 40 CFR Part 136) shall be used to analyze samples of sewage sludge.

* * * * *

[FR Doc. 07–1455 Filed 3–23–07; 8:45 am]

BILLING CODE 6560–50–P

d. Combination ion-selective electrode.
e. Injection valve control and data acquisition system.

3. Reagents

Use reagent water (>10 megohm) for all solutions. To prevent bubble formation, degas carrier and buffer with helium. Pass He at 140 kPa (20 psi) through a helium degassing tube. Bubble He through 1 L solution for 1 min.

a. Carrier, 1.0 mg F^-/L: Add 10 mL or 10 g stock fluoride standard (¶ 3*d*) to 990 mL water and mix well.

b. Buffer: To a tared 1-L polyethylene container add 929.5 g water, 59.8 g glacial acetic acid, 30.0 g sodium hydroxide, NaOH, 58.0 g sodium chloride, NaCl, 0.5 g stock fluoride standard (¶ 3*d*), and 4.0 g 1,2-cyclohexyldiaminetetraacetic acid (CDTA) (also called trans-1,2-diaminocyclohexane). Stir on a magnetic stir plate until all material has dissolved.

c. Electrode conditioning solution: To a tared 1-L container, add 534 g buffer (¶ 3*b*) and 500 g carrier (¶ 3*a*). Shake or stir to mix thoroughly. Store fluoride electrode in this solution when it is not in use.

d. Stock fluoride standard, 100.0 mg F^-/L: In a 1-L volumetric flask, dissolve 0.2210 g sodium fluoride, NaF, in approximately 950 mL water. Dilute to mark with water and mix well. Store in a polyethylene bottle.

e. Standard fluoride solutions: Prepare fluoride standards in the desired concentration range, using the stock standard (¶ 3*d*), and diluting with water. A blank or zero concentration standard cannot be prepared for this method because it will give an undefined response from the fluoride electrode.

4. Procedure

Set up a manifold equivalent to that in Figure 4500-F^-:3 and follow method supplied by manufacturer or laboratory standard operating procedure for this method. Follow quality control procedures outlined in Section 4020.

5. Calculations

Prepare standard curves by plotting the electrode response to standards processed through the manifold vs. fluoride concentration. Standards greater than 1.0 mg F^-/L will give positive peaks, standards less than 1.0 mg F^-/L will give negative peaks, and the 1.0 mg F^-/L standard having the same concentration as the carrier will give no peak. The calibration curve gives a good fit to a second-order polynomial.

It is not necessary to plot the response versus log[F^-]; if this is done the calibration curve will still be a second-order polynomial because there is a concentration-dependent kinetic effect in the flowing stream electrode system.

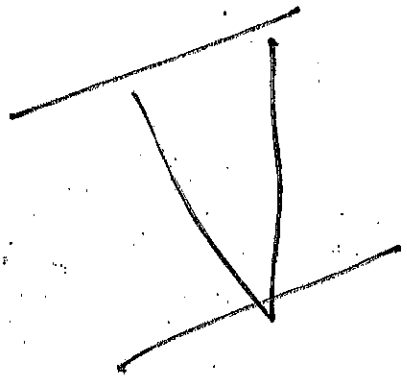
6. Precision and Bias

The samples used in the studies described below were not distilled.

a. Recovery and relative standard deviation: The results of single-laboratory studies with various matrices are given in Table 4500-F^-:II.

b. MDL: A 390-µL sample loop was used in the method described above. Ten replicates of a 1.0-mg F^-/L standard were run to obtain an MDL of 0.02 mg F^-/L.

c. Precision: Ten replicate standards of 2.0 mg F^-/L gave a % RSD of 0.5%.



4500-H^+ pH VALUE*

4500-H^+ A. Introduction

1. Principles

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

$$[H^+][OH^-] = K_w = 1.01 \times 10^{-14} \text{ at } 25°C \quad (1)$$

and

$$[H^+] = [OH^-] = 1.005 \times 10^{-7}$$

* Approved by Standard Methods Committee, 1996.

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

$$pH + pOH = pK_w$$

where:

pH† = $\log_{10} a_{H^+}$ and
pOH = $\log_{10} a_{OH^-}$.

† p designates $-\log_{10}$ of a number.

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 in the range of 4 to 9, and most are slightly basic because of the presence of bicarbonates and carbonates of the alkali and alkaline earth metals.

2. Reference

1. Sorenson, S. 1909. Über die Messung und die Bedeutung der Wasserstoff ionen Konzentration bei Enzymatischen Prozessen. *Biochem. Z.* 21:131.

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) buffers having assigned values so that:

$$pH_B = -\log_{10} a_{H^+}$$

where:

pH_B = assigned pH of NIST buffer.

The operational pH scale is used to measure sample pH and is defined as:

$$pH_x = pH_B \pm \frac{F(E_x - E_s)}{2.303\ RT}$$

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.[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_o should be minimal. Samples must be dilute aqueous solutions of simple solutes (<0.2*M*). (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 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.

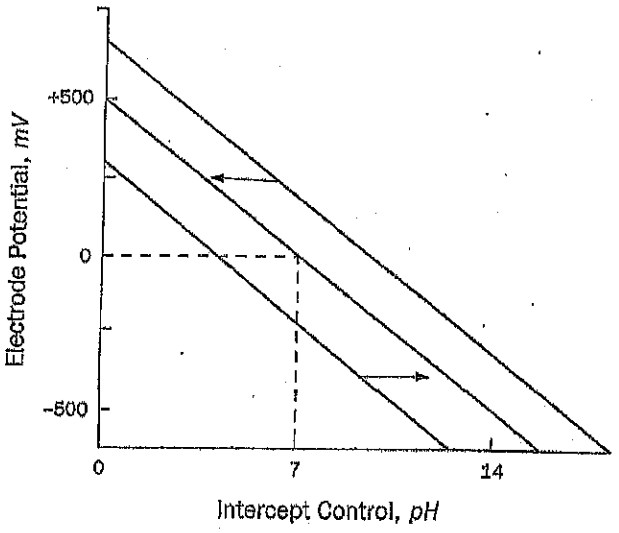


Figure 4500-H⁺:1. Electrode potential vs. pH. Intercept control shifts response curve laterally.

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.

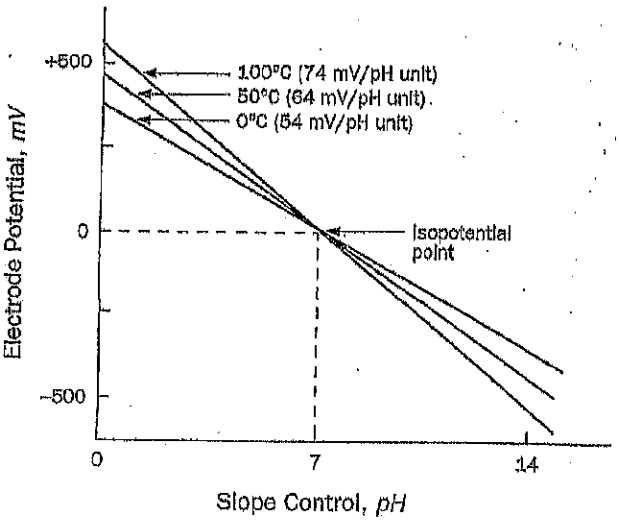


Figure 4500-H⁺:2. Typical pH electrode response as a function of temperature.

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.

* Teflon, or equivalent.

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.

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

TABLE 4500-H⁺:I. PREPARATION OF pH STANDARD SOLUTIONS[3]

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).

TABLE 4500-H$^+$:II. STANDARD pH VALUES[3]

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					1.765	
90	3.650		4.19					1.792	
85	3.674		4.21					1.806	

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 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 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 elec-

trode 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.

7. References

1. Bates, R.G. 1978. Concept and determination of pH. *In* I.M. Kolthoff & P.J. Elving, eds. Treatise on Analytical Chemistry. Part 1, Vol. 1, p. 821. Wiley-Interscience, New York, N. Y.
2. Licht, T.S. & A.J. de Bethune. 1957. Recent developments concerning the signs of electrode potentials. *J. Chem. Educ.* 34:433.
3. Durst, R.A. 1975. Standard Reference Materials: Standardization of pH Measurements. NBS Spec. Publ. 260-53, National Bur. Standards, Washington, D.C.

8. Bibliography

Clark, W.M. 1928. The Determination of Hydrogen Ions, 3rd ed. Williams & Wilkins Co., Baltimore, Md.

Dole, M. 1941. The Glass Electrode. John Wiley & Sons, New York, N.Y.

Bates, R.G. & S.F. Acree. 1945. pH of aqueous mixtures of potassium dihydrogen phosphate and disodium hydrogen phosphate at 0 to 60°C. *J. Res. Nat. Bur. Standards* 34:373.

Langelier, W.F. 1946. Effect of temperature on the pH of natural water. *J. Amer. Water Works Assoc.* 38:179.

Feldman, I. 1956. Use and abuse of pH measurements. *Anal. Chem.* 28: 1859.

Britton, H.T.S. 1956. Hydrogen Ions, 4th ed. D. Van Nostrand Co., Princeton, N.J.

Kolthoff, I.M. & H.A. Laitinen. 1958. pH and Electrotitrations. John Wiley & Sons, New York, N.Y.

Kolthoff, I.M. & P.J. Elving. 1959. Treatise on Analytical Chemistry. Part I, Vol. 1, Chapter 10. Wiley-Interscience, New York, N.Y.

Bates, R.G. 1962. Revised standard values for pH measurements from 0 to 95°C. *J. Res. Nat. Bur. Standards* 66A:179.

American Water Works Association. 1964. Simplified Procedures for Water Examination. Manual M12, American Water Works Assoc., New York, N.Y.

Winstead, M. 1967. Reagent Grade Water: How, When and Why? American Soc. Medical Technologists, The Steck Company, Austin, Tex.

Staples, B.R. & R.G. Bates. 1969. Two new standards for the pH scale. *J. Res. Nat. Bur. Standards* 73A:37.

Bates, R.G. 1973. Determination of pH, Theory and Practice, 2nd ed. John Wiley & Sons, New York, N.Y.

4500-I IODINE*

4500-I A. Introduction

1. Uses and Forms

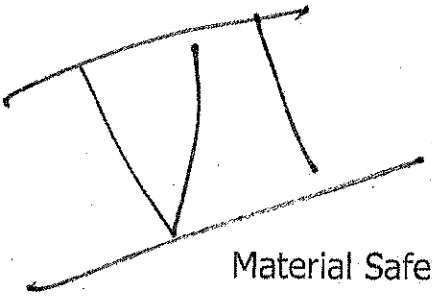
Elemental iodine is not a natural constituent of natural waters. Iodine may be added to potable and swimming pool waters as a disinfectant. For wastewaters, iodine has had limited application. Use of iodine generally is restricted to personal or remote water supplies where ease of application, storage 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

* Approved by Standard Methods Committee, 1997.

RICCA CHEMICAL COMPANY

MSDS



For RICCA, SpectroPure, Red Bird, and Solutions Plus Brands
Emergency Contact (24 hr) – CHEMTREC®
Domestic: 800-424-9300
International: 703-527-3887

BUFFER, REFERENCE STANDARD, pH 5.80 - 7.00, pH 7.20 - 8.000

Material Safety Data Sheet

Section 1: Chemical Product and Company Identification

Catalog Number:

1510, 1513, 1530, 1532, 1539, 1540, 1540.5, 1550, 1563, 1565, 1568, 1572, 1576, 1577, 1580, 1582, B-270, B-280, B-290, B-295, B017640, B017680, B017720, B017800, BX-915, R1509000, R1550100, SB017640, SB017680, SB017720, SB017800

Product Identity:
BUFFER, REFERENCE STANDARD, pH 5.80 - 7.00, pH 7.20 - 8.000

Manufacturer's Name: RICCA CHEMICAL COMPANY LLC	**Emergency Contact(24 hr) -- CHEMTREC®** Domestic: 800-424-9300 International: 703-527-3887
CAGE Code: 0V553	
Address: 448 West Fork Dr Arlington, TX 76012	**Telephone Number For Information:** 817-461-5601
Date Prepared: 6/16/98	**Revision:** 10 Last Revised: 03/19/2007 Date Printed: 06/15/2007 4:49:42 pm

Section 2. Composition/Information on Ingredients

Component	CAS Registry #	Concentration	ACGIH TLV	OSHA PEL
Sodium Phosphate, Dibasic	7558-79-4	< 1	Not Available Not Available	Not Available Not Available
Water, Deionized	7732-18-5	Balance	Not Available Not Available	Not Available Not Available
Potassium Phosphate, Monobasic	7778-77-0	< 1	Not Available Not Available	Not Available Not Available
Preservative (No Mercury compounds or Formaldehyde)	Proprietary	< 0.1	Not Available Not Available	Not Available Not Available

Section 3: Hazard Identification

Emergency Overview: Non-flammable, non-corrosive, non-toxic. Does not present any significant health hazards. May cause irritation. Wash areas of contact with water.

Target Organs: eyes, skin

Eye Contact: May cause slight irritation.

Inhalation: May cause allergic respiratory reaction to those allergic to phosphates.

Skin Contact: May cause slight irritation to those allergic to phosphates.

Ingestion: Large doses may cause stomach upset.

Chronic Effects/Carcinogenicity: None
IARC - No.
NTP - No.
OSHA - No.
Reproductive Information: Not Applicable.
Teratology (Birth Defect) Information: Not Applicable.

Section 4: First Aid Measures - In all cases, seek qualified evaluation.

Eye Contact: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.
Inhalation: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.
Skin Contact: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.
Ingestion: Dilute with water or milk. Call a physician if necessary.

Section 5: Fire Fighting Measures

Flash Point: Not Available.
Method Used: Not Available.
LFL: Not Available.
UFL: Not Available.
Extinguishing Media: Use any means suitable for extinguishing surrounding fire.
Fire & Explosion Hazards: Not considered to be a fire or explosion hazard.
Fire Fighting Instructions: Use normal procedures/instructions.
Fire Fighting Equipment: Use protective clothing and breathing equipment appropriate for the surrounding fire.

Section 6: Accidental Release Measures

Absorb with suitable material (vermiculite, clay, etc.) and dispose of in accordance with local regulations.
Check with local agencies for the proper disposal of phosphate containing solutions.

Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage.
Safety Storage Code: General

Section 8: Exposure Control/Personal Protection

Engineering Controls: No specific controls are needed. Normal room ventilation is adequate.
Respiratory Protection: Normal room ventilation is adequate.
Skin Protection: Chemical resistant gloves.
Eye Protection: Safety glasses or goggles.

Section 9: Physical and Chemical Properties

Appearance: Clear, Colorless Liquid
Odor: Odorless
Solubility in Water: Infinite
Specific Gravity: Approximately 1
pH: 5.8 - 8
Boiling Point(°C): Approximately 100
Melting Point(°C): Approximately 0
Vapor Pressure: Not Applicable.

Section 10: Stability and Reactivity

Chemical Stability: Stable under normal conditions of use and storage.
Incompatibility: None Identified.
Hazardous Decomposition Products: Phosphorus oxides may form when heated to decomposition
Hazardous Polymerization: Will not occur.

Section 11. Toxicological Information

LD50, Oral, Rat: (Sodium Phosphate Dibasic) 17 gm/kg; LD50, Dermal, Rabbit: (Potassium Phosphate Monobasic) >4640 mg/kg; details of toxic effects not reported other than lethal dose value.

Section 12. Ecological Information

Ecotoxicological Information: No information found
Chemical Fate Information: No information found

Section 13. Disposal Considerations

Dilute with water, then flush to sewer if local regulations allow for the flushing of phosphate containing solutions. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information

Part Numbers:

This product is not regulated.

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA Status: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.
TSCA Status: All components of this solution are listed on the TSCA Inventory or are mixtures (hydrates) of items listed on the TSCA Inventory.

Sara Title III:

Section 302 Extremely Hazardous Substances: Not Applicable.
Section 311/312 Hazardous Catagories: No
Section 313 Toxic Chemicals: Not Applicable.

California: None Reported.
Pennsylvania: Sodium Phosphate, Dibasic is listed as an Environmental Hazard on the state's Hazardous Substances List.
RCRA Status: Not Applicable.
CERCLA Reportable Quantity: Sodium Phosphate, Dibasic - 5,000 pounds.

WHMIS: Not Applicable.

Section 16. Other Information

NFPA Ratings:

Health: 1	Flammability: 0	Reactivity: 0	Special Notice Key:None

HMIS Ratings:

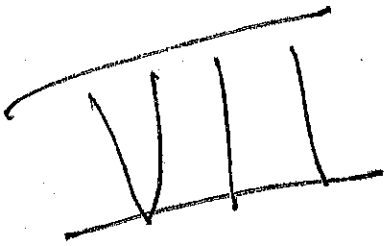
Health: 1	Flammability: 0	Reactivity: 0	Protective Equipment:B (Protective Eyewear, Gloves)

Rev 1, 10-19-98: (Section 1) Specified catalog numbers in title; (Section 16) Removed periods from NFPA and HMIS, Added® to HMIS.
Rev 2, 4-27-2000: Reformatted from WordPerfect® to Microsoft Word®; (Section 1) added catalog number 1540.5, revised emergency telephone number to CHEMTREC® 800-424-9300; (Section 3) Added "May cause...water." in emergency overview; (Section 4) removed note to physician; (Section 7) added storage code; (Section 10) added decomposition products; (Section 11) added toxicological data; (Section 15) added Pennsylvania listing.
Rev 3, 8-21-2000: (Section 1) removed catalog number 1560 due to customer formulation change.
Rev 4, 10-09-2001: Reformatted to electronic data format.
Rev 5, 12-05-2002: (Section 1) added Solutions Plus catalog number B017640.
Rev 6, 07-30-2003: (Section 1) added Solutions Plus catalog numbers B017680, B017720 and B017800 effective 9-1-03.
Rev 7, 10-29-2003: (Section 1) added catalog numbers 1513, 1532 and 1572.
Rev 8, 11-19-2003: (Section 1) added catalog number R1550100.
Rev 9, 03-21-2006: (Section 1) revised title, added catalog numbers R1509000 and 1576; (Section 9) revised pH range from 6 - 8.
Rev 10, 03-19-2007: (Section 1) added catalog number 1539.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

El Dorado Irrigation District
Standard Operating Procedure

pH



METHODS

Water and Wastewater.....Based Upon (20th Edition SM 4500-H^+ B)

SCOPE AND APPLICATION

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. 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 is pH dependant. 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 the water and usually are expressed as milligrams CaCO3 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 liter sample by 1 unit. pH as defined by Sorenson is $-\log[H^+]$; it is the intensity factor of acidity. The pH range for most environmental samples can be expected to measure between 5 and 9 pH units.

DETECTION LIMIT

pH ranges 1 - 14

DEFINITIONS

pH as defined by Sorenson is $-\log[H^+]$; it is the intensity factor of acidity.

SUMMARY

The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode. 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.

SAMPLE PRESERVATION AND STORAGE

A minimum of 100 ml of sample should be collected in a glass or plastic container. Start pH determinations within 15 minutes after sampling.

SAFETY

1. Read all MSDS pertaining to this test prior to initiating analysis.
2. Wear safety glasses at all times in the laboratory.
3. Wear appropriate clothing at all times in the laboratory.
4. Wear appropriate gloves when working with samples and/or reagents.
5. If skin is exposed to sample, reagents, or acids, flush for 15 minutes with water, then wash with a neutralizing solution. Notify a supervisor as soon as possible.

POLLUTION PREVENTION AND WASTE MANAGEMENT

Follow manufacture disposal procedures

APPARATUS

1. Beakers
2. pH meter, capable of reading to 0.05 pH unit with a range of 0 to 14
3. Magnetic Stirrer Plate and Stirring Bar

El Dorado Irrigation District
Standard Operating Procedure

pH

METHODS

Water and Wastewater.....Based Upon (20th Edition SM 4500-H^+ B)

REAGENTS AND STANDARD SOLUTIONS

1. pH buffer: 4.00
2. pH buffer: 7.00
3. pH buffer: 8.00
4. pH buffer: 10.00
5. Deionized Water

INTERFERENCES

Dissolved gases contributing to acidity or alkalinity, such as CO_2, hydrogen sulfide or ammonia may be lost or gained during sampling, storage, or titration. Minimize such efforts by measuring the pH promptly after opening container, avoiding vigorous shaking or mixing. 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. This error can be reduced with the use of a "low sodium error" electrode. The 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.

INSTRUMENT (DEER CREEK WASTEWATER TREATMENT PLANT)

1. Expandable Ion Analyzer model EA 940 and pH electrode (81728 BN WP) in conjunction with an Orion automatic temperature compensation (ATC) probe

PROCEDURE

(Deer Creek Wastewater Treatment Plant)

CALIBRATION

1. Calibration of the pH meter/probe must be once each day of use.
2. Remove filling hole cover on electrode.
3. Press SPEED 0.
4. Meter Reads: "Cal pH?," press YES
5. Meter Reads "Enter # of Buffers (1-3)," press 3.
6. Meter Reads "Do Automatic Cal?," press YES.
   a) Add buffer 4 and a stir bar to a beaker.
7. Meter Reads "pH Electrode Placed in Buffer 1?," place electrode in pH 4.00 buffer then press YES
   a) Meter Reads "Cal as 4.00?," press 2nd function and the mV/temp button.
   b) Record the mV value.
   c) Press the ? button to temperature.
   d) If it says anything other than 4.00, i.e."Cal as 7.01" or 4.00, press NO.
   e) When it reads 4.00, press YES.
8. Meter Reads "pH Electrode Placed in Buffer 2?,"
   a) Place electrode in pH buffer 7 then press YES
9. Meter Reads "Cal as 10.00?,"
   a) Press 2nd function and then the mV/temp button
   b) Record the mV value
   c) Press the ? button to return to the previous screen and press YES.
10. Press the ? button to return to the previous screen and press YES.
    a) Record slope value at top of pH Log Book. Acceptable slope range is 92-102%.

METHODS

Water and Wastewater.....Based Upon (20th Edition SM 4500-H^+ B)

11. Rinse the electrode with distilled water and shake and blot dry. Gently stir the pH QC check solution with the electrode. Record this QC number as the first sample.

ANALYSIS

1. Allow all samples to come to room temperature.
2. Remove filling hole cover on electrode.
3. Rinse the electrode probe with Deionized water and blot dry.
4. Pour a fresh aliquot of sample into beakers with a stir bar.
5. Gently stir the sample with the electrode
6. Record the display and temperature of each measurement after it stabilizes.
7. Return to step 2 for remaining samples.
8. When finished, store electrode in storage solution or in 200ml pH 7 buffer with approximately 1.0g KCL. Replace filling hole cover.

CALIBRATION CHECK

1. After every 10 samples and at the end, use pH Buffer 8.00 for the calibration check
2. Record measurement in the logbook

DUPLICATE

Analyze one or more duplicate samples for each set of ten samples analyzed. The relative percent difference is calculated as follows:

$$RPD = \{D1-D2)/ [(D1+D2/2]\} x100$$

Where, RPD = Relative Percent Difference
D1 = First Sample Value
D2 = Second Sample Value (duplicate)

REPORTING

1. Record the results to the nearest 1/10 of a pH unit (three significant figures).
2. Use two significant figures when reporting results

CALCULATIONS

Instrument calculates the pH and displays the value on the screen.

QUALITY CONTROL

Type	Minimum Frequency	Acceptance Limits
Duplicate	1 / batch	RPD $\leq$ 10 %
Calibration Check	1 / batch	90-110%
Yearly Blind Sample	1 / year	External Evaluation

A batch is 10 samples or less.

CONTINGENCIES FOR OUT OF CONTROL DATA

METHODS

Water and Wastewater.....Based Upon (20th Edition SM 4500-H^+ B)

1. The analyst is allowed to rerun the sample one time then request the help of a Supervisor.
2. If the data cannot be brought into control, corrective action will be taken as directed by the Supervisor or an outside consultant.

RECORD MANAGEMENT

For Information on data archival procedures, refer to the procedure titled "Data Recording and Record Management."

REFERENCES

1. "Standard Methods for the Examination of Water and Wastewater," SM 4500-H^+ B, 20th Edition,1998, American Health Association, American Water Works Association, Water Pollution Control Federation.

APPROVED BY

Wastewater Supervisor Date

Revisions

Effective Date	Revisions Made	Date / Initials	

pH

SM 20th Ed 4500-H^+ B

Acceptable slope range is (92 to 102).

Meter Calibration	Slope		
3 point	4.00	7.00	10.00
mV			
Calibrate as			
Buffer Code			

Analysis Date:

Date Collected is the same as the Analysis Date, unless otherwise indicated.

Analyst:

Reclaim pH range (6.0-9.0) Effluent pH range (6.5-8.5)

	Date Collected	Time Collected	Time Started	Sample Site	Lab #	Instrument Result	RPD%	Collected Y/N	Temp °C	Temp °F	
1				pH Buffer 8 Lot #:							1
2											2
3											3
4											4
5											5
6											6
7											7
8											8
9											9
10				pH Buffer 8 Lot #:							10

Comments:

pH

SM 20th Ed 4500-H^+ B

Acceptable slope range is (92 to 102).

Meter Calibration	Slope		
3 point	4.00	7.00	10.00
mV			
Calibrate as			
Buffer Code			

Analysis Date:

Date Collected is the same as the Analysis Date, unless otherwise indicated.

Analyst:

Reclaim pH range (6.0-9.0) Effluent pH range (6.5-8.5)

	Date Collected	Time Collected	Time Started	Sample Site	Lab #	Instrument Result	RPD %	Collected Y/N	Temp °C	Temp °F	
1				pH Buffer 8 Lot #:							1
2											2
3											3
4											4
5											5
6											6
7											7
8											8
9											9
10				pH Buffer 8 Lot #:							10

Comments:

Certificate of Completion

Sierra Foothill Laboratory, Inc.
Training Organization

"Analysis of pH" 20th Edition Standard Method

THIS IS TO CERTIFY THAT

Jerry I. Tamura

(Name)

Has Completed the Above Training/Educational Program and Has Earned

One Laboratory Contact Hour

Date Issued: March 2, 2011

Signature of Trainer
Jerry I. Tamura
Sierra Foothill Laboratory, Inc.
255 Scottsville Blvd.
Jackson, California 95642
Email: jerry@sierralab.com pH: (209) 223 2800

This Certificate of Completion is issued by the training organization listed above.