GUAM ENVIRONMENTAL PROTECTION AGENCY
EMAS ANALYTICAL PROGRAM

STANDARD OPERATING PROCEDURE

pH by Electrometric Method (SM 4500-H+ B)

Prepared by: _____ 10-26-2015
Edelisa S. Yanit, Chemist II — Date

Peer Reviewed by: _____ 10-26-2015
Rodolfo B. Paulino, Chemist II — Date

Reviewed by: _____ 10/28/15
Jesse T. Cruz, Administrator
EMAS Division — Date

Approved by: _____ 01/15/16
Eric M. Palacios, Administrator
Guam Environmental Protection Agency — Date

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1 SCOPE AND APPLICATION

1.1 This method is applicable to drinking, surface and saline water, domestic and industrial wastewaters and acid rain (atmospheric deposition).

2 METHOD SUMMARY

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

3 INTERFERENCES

3.1 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants or high salinity.

3.2 pH electrodes can become sluggish in their response when coated with oils and suspended solids present in samples. These coatings can usually be removed by gentle wiping or detergent washing, followed by distilled water rinsing. An additional treatment with 0.1M HCl may be necessary to remove any remaining film.

3.3 Temperature effects on the electrometric measurement of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference can be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source is the change of pH inherent in the sample at various temperatures. This error is sample dependent and cannot be controlled, it should therefore be noted by reporting both the pH and temperature at the time of analysis.

4 DEFINITIONS

4.1 pH – the logarithm of the reciprocal of the hydrogen ion concentration in gram atoms per liter, provides a measure on a scale from 0-14 of the acidity or alkalinity of a solution where 7 is neutral, values above 7 are increasingly more basic to 14, and values below 7 are increasingly more acidic to zero.

4.2 Laboratory Duplicate – an aliquot of sample prepared and analyzed separately, using identical procedures. Analyses of a sample and LD indicate precision associated with laboratory procedures, but do not indicate precision in sample collection, preservation or storage procedures.

5 SAMPLE HANDLING AND PRESERVATION

5.1 Samples are collected in clean glass or plastic containers.

5.2 No preservation is indicated for pH samples.

5.3 Samples should be analyzed as soon as possible preferably in the field at the time of sampling.

5.4 High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.

6 EQUIPMENT AND SUPPLIES

6.1 Bench top pH meter accurate to 0.1 pH units (OAKTON PC510 pH and conductivity meter series)

6.2 pH combination electrode

6.3 Automatic Temperature Compensating electrode (ATC)

6.3 Magnetic stirrer

6.4 Teflon coated stir bars

6.5 50-ml beakers for buffers

6.6 150 ml beakers for samples

7 REAGENTS AND STANDARDS

7.1 Reagent Water – Use ASTM Type II reagent water

7.2 Standard buffer solutions – commercially prepared:

pH 4.00 @ 25°C (OAKTON # 05942-22 or equivalent)

pH 7.00 @ 25°C (OAKTON # 05942-42 or equivalent)

pH 10.00 @ 25°C (OAKTON # 05942-62 or equivalent)

7.3 0.1M HCl (for electrode cleaning). To prepare 0.1M HCl, 0.83ml of concentrated HCl is diluted to 100 ml with de-ionized water

7.4 2M - 4M KCL solution (for electrode storage solution). To prepare 3M KCl, dissolve 10.8 g KCl in about 50 ml de-ionized water in 100-ml volumetric flask, dilute to the mark and shake.

8 QUALITY CONROL PROCEDURES

8.1 pH Meter Calibration

8.1.1 The pH meter is calibrated at three points before each use period with pH 4.00, 7.00, and 10.00 standard buffers as recommended by the instrument's manufacturer.

8.1.2 The following section describes the steps for a three-point calibration of Oakton pH meter PC510.

8.1.2.1 Pour fresh aliquots of pH buffers 4, 7, and 10 into their respective 50-ml beakers containing stir bars. On the pH Meter Calibration section of the pH Analytical Results Logbook (GEPA Log: CH-02-01), write the date and initials of analyst in the appropriate spaces.

8.1.2.2 Turn on the pH meter by pressing the "ON/OFF" key.

8.1.2.3 Be sure to remove the protective electrode storage bottle of the probe before calibration or measurement.

8.1.2.4 If necessary, press the MODE key to select pH mode. The pH indicator appears in the upper right hand corner of the display.

8.1.2.5 Rinse the probe thoroughly with de-ionized water. Do not wipe the probe as this causes a build-up of electrostatic charge on the glass surface.

8.1.2.6 Dip the pH probe into the standard calibration buffer (pH 4.00). The end of the probe must be completely immersed into the sample. Dip the ATC probe into the calibration buffer as well.

8.1.2.7 Press CAL/MEAS key to enter pH calibration mode. The CAL indicator will be shown. The primary display will show the measured reading while the smaller secondary reading will indicate the pH standard buffer solution.

8.1.2.8 Wait for the pH value to stabilize. The READY indicator appears when the reading is stable.

8.1.2.9 Press ENTER to confirm calibration. The meter is now calibrated to the current buffer. The lower display automatically scrolls through the remaining buffer options.

8.1.2.10 Rinse the electrode with de-ionized water and place it in the next buffer (pH 7.00).

8.1.2.11 Follow steps (8.1.2.8) to (8.1.2.10) for the third calibration point using pH 10.00.

8.1.2.12 When calibration is complete, press CAL/MEAS to return to pH measurement mode.

NOTE: Do not re-use calibration solutions (pH buffers) after calibration. Contaminants in the solution can affect the calibration, and eventually the accuracy of the measurements.

9.0 ANALYTICAL PROCEDURES

9.1 Rinse the pH electrode with de-ionized water before use to remove any impurities adhering to the probe body. Blot dry with soft tissue paper.

9.2 Press ON to switch meter on.

9.3 If necessary, press the MODE key to select pH measurement mode. The MEAS annunciator appears on the top center of the LCD. The ATC indicator appears in the lower right hand corner to indicate Automatic Temperature Compensation.

9.4 Dip the probe into the sample beaker containing a Teflon coated stir bar. Establish equilibrium between electrodes and sample by stirring the sample to insure homogeneity; stir gently to minimize carbon dioxide entrainment. Since the conductivity cell contains the temperature sensor, make sure it is also immersed in the solution.

9.5 When dipping the probe into the sample, the sensor or the glass bulb of the electrode must be completely immersed into the sample.

9.6 For buffered samples or those of high ionic strength (e.g., tap water, ground and surface waters, especially seawater), condition electrodes after rinsing by dipping them into the sample for one minute. Blot dry, immerse in a fresh portion of the sample, and measure pH.

9.7 With dilute, poorly buffered solutions or those of low ionic strength (e.g., high purity water), equilibrate electrodes by immersing in two or three successive portions of sample. Take a fresh sample to measure pH.

9.8 Allow time for the reading to stabilize. The READY indicator will appear when the reading is stable.

9.9 Record the reading in the pH Analytical Results Logbook (GEPA Log: CH-02-01). pH values should be reported to the nearest 0.1 pH unit.

9.10 Remove the probe from the sample solution and rinse with de-ionized water.

9.11 Repeat for remaining samples.

9.12 Laboratory duplicates should be analyzed on one sample in each batch of ten. The difference between the duplicates must be within $\pm$ 0.1 pH unit.

9.13 In addition, a continuing calibration verification (CCV) is determined using one of the pH standards. Result must be within $\pm$ 0.1 pH unit of the expected value. If value is out of criteria, recalibrate and reanalyze all affected samples.

10 DOCUMENTATION

10.1 When samples are received, the laboratory personnel verify that the chain of custody form is properly filled out. Laboratory personnel may then receive and sign the chain of custody. A copy of the chain of custody form is included in the data package (Appendix A).

10.2 Sample results are recorded in the Analytical Results Logbook (GEPA Log: CH-02-01) (Appendix C), and then entered in the Laboratory Information Management System (LIMS) and analytical results are reported. The Analytical Results Report (LIMS or spreadsheet report) is included in the data package (Appendix B).

10.3 The data package consists of the following:

Appendix A: Chain of Custody Form

Appendix B: Analytical Results Report (LIMS or spreadsheet)
Appendix C: Analytical Results Logbook page

11 REFERENCES

11.1 EPA Method 150.1, Approved for NPDES (Editorial Revision 1982)

11.2 SM 4500-H^+ B, Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998.

11.3 OAKTON PC 510 Bench pH/Conductivity Meter Instruction Manual

Appendix A: Chain of Custody Form

GUAM ENVIRONMENTAL PROTECTION AGENCY

AHENSIAN PRUTEKSION LIINA'LA GUAHAN

P.O. BOX 22439
BARRIGADA, GU 96921
EPA.GUAM.GOV

GUAM ENVIRONMENTAL PROTECTION AGENCY 1973

Chain of Custody Record

PROJECT NAME: _____ **ASSIGNED LAB ID NUMBER** _____

SAMPLERS
(Print name and sign)
_____

ELD SAMPLE ID	DATE	TIME	COMPOSITE	GRAB	STATION LOCATION	# OF CONTAINERS						REMARKS

RELINQUISHED BY (*Print name and sign*)	DATE	TIME	RECEIVED BY (*Print name and sign*)
RELINQUISHED BY (*Print name and sign*)	DATE	TIME	RECEIVED BY (*Print name and sign*)
RELINQUISHED BY (*Print name and sign*)			RECEIVED FOR LAB by(*Print name and sign*)

REMARKS

chain of custody #

Page _____ *of* _____

Appendix B: Analytical Results Report (LIMS or spreadsheet)

GUAM ENVIRONMENTAL PROTECTION AGENCY
EMAS Analytical Program

ANALYTICAL REPORT

Report: pH-02360_02372_02373
Parameter: pH
Method: SM 4500-H^+

Matrix: water

Date of Report: 02/20/2012
Analyst: E. Yanit

GEPA Lab ID	Field Sample ID	Date Collected	Date Analyzed	MDL	Result	Unit	Remarks
02360-018	AGRD	3/2/2011	**	**	**	**	Note: ** Not analyzed for
02360-019	AGRA-3	3/2/2011	**	**	**	**	the parameter
02360-020	AGRF-2	3/2/2011	**	**	**	**	
02360-021	ASRI-2	3/2/2011	**	**	**	**	
02360-022	ASRI-4	3/2/2011	**	**	**	**	
02360-023	ASRM	3/2/2011	**	**	**	**	
02360-024	APRM-1B	3/2/2011	**	**	**	**	
02359-024	APRM-1B - LD	3/2/2011	**	**	**	**	
02372-017	ATRN-2	3/22/2011	3/22/2011	N/A	7.89	std units	
02372-018	ATRTO	3/22/2011	3/22/2011	N/A	7.97	std units	
02372-019	ATRF-3	3/22/2011	3/22/2011	N/A	7.93	std units	
02372-020	ATRT-2	3/22/2011	3/22/2011	N/A	7.85	std units	
02372-021	ULRU-2	3/22/2011	3/22/2011	N/A	7.98	std units	
02372-022	MZRT-2	3/22/2011	3/22/2011	N/A	8.52	std units	
02372-023	MZRG-2	3/22/2011	3/22/2011	N/A	7.96	std units	
02372-020	ATRT-2 - LD	3/22/2011	3/22/2011	N/A	7.87	std units	Lab duplicate
	pH 7 buffer		3/22/2011	N/A	7.02	std units	QC check
02373-013	APM-18	3/23/2011	3/23/2011	N/A	8.15	std units	

Peer Reviewed By: _____
Date Reviewed: 2/23/12

Approved By: _____
Date Approved: 3/12/12

GUAM ENVIRONMENTAL PROTECTION AGENCY
EMAS Analytical Program

ANALYTICAL REPORT

Report: pH-02373_02375_02376
Parameter: pH
Method: SM 4500-H^+

Matrix: water

Date of Report: 02/20/2012
Analyst: E. Yanit/ R. Paulino

GEPA Lab ID	Field Sample ID	Date Collected	Date Analyzed	MDL	Result	Unit	Remarks
02373-014	APMOb	3/23/2011	3/23/2011	N/A	8.00	std units	
02373-015	APM-20	3/23/2011	3/23/2011	N/A	8.13	std units	
02373-016	ATMN	3/23/2011	3/23/2011	N/A	8.03	std units	
02373-017	ATMTO	3/23/2011	3/23/2011	N/A	7.99	std units	
02373-018	ATMS	3/23/2011	3/23/2011	N/A	8.02	std units	
02373-019	ATMP	3/23/2011	3/23/2011	N/A	8.09	std units	
02373-020	ULMUE	3/23/2011	3/23/2011	N/A	8.14	std units	
02373-021	MZMMb	3/23/2011	3/23/2011	N/A	8.21	std units	
02373-017	ATMTO - LD	3/23/2011	3/23/2011	N/A	8.01	std units	Lab duplicate
	pH 7 buffer		3/23/2011	N/A	7.03	std units	QC check
02375-013	INRAGB-3	3/28/2011	3/28/2011	N/A	7.89	std units	
02375-014	INRI-2	3/28/2011	3/28/2011	N/A	7.55	std units	
02375-015	TUETO	3/28/2011	3/28/2011	N/A	7.72	std units	
02375-016	TURTG-1C	3/28/2011	3/28/2011	N/A	7.50	std units	
02375-017	PGMPW bridge	3/28/2011	3/28/2011	N/A	7.71	std units	
02375-015	TUETO - LD	3/28/2011	3/28/2011	N/A	7.74	std units	Lab duplicate
	pH 7 buffer		3/28/2011	N/A	7.03	std units	QC check
02376-012	AGFM-09	3/29/2011	3/29/2011	N/A	8.07	std units	

Peer Reviewed By: _____
Date Reviewed: 2/23/12

Approved By: _____
Date Approved: 3/12/12

Appendix C: Analytical Results Logbook page

GEPA Log: CH-02-01

GUAM EPA LABORATORY
ANALYTICAL RESULTS LOGBOOK
pH Elecrometric Method (SM 4500-H^+ B)

pH Meter ID: OAKTON pH/CON 510 Series

Analyst: _____

pH Meter Calibration:

Analysis Date: _____

pH 4.0 Buffer _____*

pH 7.0 Buffer _____*

pH 10.0 Buffer _____*

#	Date Sampled	Lab Sample ID #	Location	pH	Notes
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
LAB DUP					
QC		pH _____ Buffer			

* **CRITERIA:** Results for pH buffers must be within ± 0.1 pH units of the expected value.

The difference between duplicate results must be 0.1 pH units or less.

ACTION: If value is greater than criteria, recalibrate and reanalyze all affected samples. Inform supervisor.

Revision: 4 - 10/20/2015 esy
File: pHlog.xls