

pH MEASUREMENT

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pH MEASUREMENT

1. PROCEDURE IDENTIFIER: TWS-INC-DP-35

INTRODUCTION

2. PURPOSE

This procedure leads to the accurate determination of the pH or hydrogen ion activity of a solution given by the relationship

$$\text{pH} = -\log(a_{\text{H}^+})$$

3. SCOPE

This procedure may be used to measure the pH of an aqueous solution for any task in the LANL Yucca Mountain Project (YMP).

4. APPLICABLE DOCUMENTS

- a. TWS-QAS-QP-05.2 "Preparation of a Detailed Technical Procedure"
- b. TWS-QAS-QP-07 "Procedure for Technical Review of Publications"
- c. TWS-MSTQA-QP-14 "Research and Development (Experimental) Procedure"
- d. The user's manual (or a copy) for the pH meter and electrode to be used are required and will be kept with the instrument.

5. RESPONSIBILITIES

The PI has the responsibility of organizing and overseeing all operations. He may assign appropriate tasks to personnel trained to this DP. It is the responsibility of the users of this DP to adhere to the procedure. Investigators may direct deviations from the procedure upon approval of the responsible PI. It is the responsibility of the user to

document such deviations in accordance with TWS-MSTQA-QP-14. If change requests are in process it is the responsibility of the user to document the procedure change in the laboratory notebook. It is the responsibility of the users of this DP to report unplanned deviations from this procedure to the responsible PI.

6. PRINCIPLE

The pH of a solution is a parameter used to aid in the understanding of the systems studied in the LANL YMP.

7. DEFINITIONS

a_{H^+} = hydrogen ion activity.

Activity = the effective concentration of a free ion in solution.¹

PROCEDURE

8. ADEQUATE AND APPROPRIATE EQUIPMENT, INSTRUMENTATION, AND SOFTWARE

A pH meter must be used to perform the pH measurement. The instrument must measure the voltage developed between a sensing electrode and a reference electrode placed in a solution. The instrument must be able to convert the voltage developed by a pH electrode to a corresponding pH value. The required reference electrode must provide a constant potential regardless of solution composition. The pH electrode used must respond to hydrogen ion activity. The standards used for calibration of the pH meter must be solutions which are buffered with respect to hydrogen ion activity.¹ Malfunction of the instrument can be easily detected by measuring the pH of the standards used for calibration.

9. PREPARATORY VERIFICATION

Notebook Entries

Identify the pH meter and electrode(s) to be used by model number. Identify the pH buffers to be used for standard calibration of the pH meter.

Perform a standard calibration before any pH measurements are effected. This calibration allows the determination of the sample's hydrogen activity by directly relating the electrode potential in the sample to the potential in a standard. The standards to be used for calibration should encompass the pH range of the unknowns. Standardize the pH meter according to the user's manual. Verify the calibration procedure by measuring and recording (in the laboratory notebook) the pH of one of the standards used for calibration. Measure the pH according to the user's manual. The determined pH of the verification sample should be the same as the pH of the measured standard within the limit of error consistent with the investigator's application. If the measurement is not within the desired limits, re-standardize and re-verify. If the second measurement is not within the acceptable limits, test each individual system component (e.g. electrodes), discarding or repairing any component found to be malfunctioning. The calibration step must be verified before measurements may be taken.

10. SUITABLE AND CONTROLLED ENVIRONMENTAL CONDITIONS

This procedure requires no special environmental conditions as long as the calibration of the pH meter is performed under the same conditions as those of samples.

11. ACCEPTANCE OR REJECTION CRITERIA OF DATA

Follow the instrument operations instructions in the user's manual for the pH meter used in order to determine the pH of the sample(s). The required level of precision and accuracy for the pH measurement should be consistent with the application. An estimate of the pH measurement's precision can be obtained by performing multiple measurements. The accuracy of the measurement can be estimated by determining the pH of a standard buffer. For instance, upon completing the sample's pH measurements, multiple pH measurements of the same standard buffer used for calibration verification can be made. This will allow the calculation of the precision and accuracy of the pH measurements and can be used to accept or reject the data obtained.

12. POTENTIAL SOURCES OF UNCERTAINTY AND ERROR

Malfunctioning of the equipment used can lead to uncertainties and errors in the measurements. Malfunctions can be detected by following the acceptance or rejection criteria in section 11.

13. METHOD OF DATA REDUCTION

No methods of data reduction are employed in this DP.

14. METHODS OF RECORDING AND STORING DATA AND RESULTS

Notebook Entry:

Record the pH of the sample(s) studied directly from the pH meter.

15. SAMPLE/SITE TRACEABILITY

Assign a unique identifier to the sample being measured. If a unique identifier already exists for the sample being measured, this identifier will be kept. Once the sample has been in contact with the electrode(s) used to perform pH measurements, this sample cannot be used as a reference LANL YMP sample.

QUALITY ASSURANCE REQUIREMENTS

16. QUANTITATIVE OR QUALITATIVE ACCEPTANCE CRITERIA FOR DETERMINING THAT ACTIVITIES HAVE BEEN SATISFACTORILY ACCOMPLISHED

Notebook Entries

Record the measurements required by the acceptance/rejection criteria delineated in section 11 in the laboratory notebook.

17. HANDLING, SHIPPING, AND STORAGE REQUIREMENT

No special requirements are necessary for samples used in this procedure.

18. IDENTIFICATION OF QA RECORDS TO BE GENERATED AND THEIR CONTROL

The records produced by this procedure are the laboratory notebooks. The data will be published in accordance with LANL Policy and TWS-QAS-QP-07. Investigators may direct deviations and modifications of the procedure for specific applications. Such actions are documented in notebooks.

19. TRAINING REQUIREMENTS AND METHODS

The PI or his designee will train the investigator assigned to use this DP. The training will require observation of the trainee while he performs a standard calibration of the pH meter and a successful pH measurement following this DP.

20. CALIBRATED INSTRUMENTATION INFORMATION

The calibration procedure required in this DP has been outlined in section 9.

21. PROVISION FOR DOCUMENTATION, REPORTING, AND EVALUATION OF PROCEDURAL DEVIATION

Unplanned deviations from this procedure will be documented in the laboratory notebook. The responsible PI or his designee will make a determination as to whether to accept or reject data affected by the deviation. If a decision to accept the data is made, the justification for the acceptance must also be entered in the laboratory notebook.

22. SUBJECTS REQUIRING VERIFICATION

The calibration verification required for continuation of this procedure has been specified in section 9.

23. APPENDIX AND/OR ATTACHMENTS

Reference Material:

1. "Handbook of Electrode Technology", Orion Research Inc., Boston, MA, 1982.