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TO: 145  
USNRC/WASHINGTON/

PROCEDURE NUMBER: EI-7.8  
TITLE: PH; HYDROGEN SPECIFIC ION ELECTRODE

TRANSMITTAL NUMBER: 563238

TRANSMITTAL: LISTED BELOW ARE NEW/REVISED PROCEDURES WHICH MUST BE  
IMMEDIATELY INSERTED INTO OR DISCARDED FROM YOUR  
PROCEDURE MANUAL.

Action Required

Remove and Destroy EI-7.8, R/0 (ENTIRE PROCEDURE)

Replace with EI-7.8, R/0 (ENTIRE PROCEDURE -  
EDITORIAL CHANGES)

SIGN, DATE AND RETURN THE ACKNOWLEDGEMENT FORM WITHIN 10 DAYS TO THE  
PALISADES PLANT DOCUMENT CONTROL.

SIGNATURE OR INITIALS

*[Handwritten Signature]*

DATE

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PALISADES NUCLEAR PLANT  
EMERGENCY IMPLEMENTATION PROCEDURE  
Revision and Approval Summary

TITLE: pH; HYDROGEN SPECIFIC ION ELECTRODE

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84-44 6/26/86  
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Periodic

7. ~~Biennial~~ Review

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TCN

MRN

EI-91-009

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1.0 PERSONNEL RESPONSIBILITY

See EI-7.2, "Emergency Post Accident Analysis".

2.0 PURPOSE

This method is used for pH determinations for the Primary Coolant System during post accident analysis.

3.0 INITIAL CONDITIONS AND/OR REQUIREMENTS

See EI-7.2, "Emergency Post Accident Analysis".

4.0 REFERENCES

4.1 REFERENCE DOCUMENTS

4.1.1 Emergency Implementation Procedure EI-7.2, "Emergency Post Accident Analysis"

4.2 SOURCE DOCUMENTS

4.2.1 Chemistry Procedure CH 4.1, "pH; Hydrogen Specific Ion Electrode"

4.2.2 ASTM, Volume 11.01, D1293-78, Method A, 1983

4.2.3 Methods for Chemical Analysis of Water and Wastes, Method 150.1, EPA-600/4-79-020, March 1983

5.0 EQUIPMENT AND REAGENTS

5.1 EQUIPMENT AND GLASSWARE

5.1.1 pH Meter, laboratory or field model.

5.1.2 Micro-Combination glass pH probe.

5.1.3 Electrode filling solutions, as specified by electrode manufacturer.

5.1.4 Thermometer, general purpose, 1°C subdivision, range sufficient to measure sample temperature, such as 0°C to 100°C.

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5.2 REAGENTS

5.2.1 Secondary standard buffer solutions, such as pH 4.00, 7.00 and 9.00 (or 10.00). A minimum of three buffer solutions are required with current shelf lives. Secondary standard buffer solutions may be prepared from NBS salts or salts purchased from commercial vendors as tablets or powders. The use of commercially available solutions, that have been validated by comparison to NBS standards, is recommended for routine use.

5.2.2 Water shall be double deionized (DDI).

6.0 PRECAUTIONS AND LIMITATIONS

6.1 PERSONAL SAFETY

See special precautions listed in the Precaution and Limitations Section of EI-7.2, "Emergency Post Accident Analysis".

6.2 INTERFERENCES

6.2.1 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping, detergent or solvent washing, such as alcohol, followed by distilled water rinsing. Immersion in hydrochloric acid (approximately one part acid to nine parts water) for ten minutes may be necessary to remove any remaining film.

6.2.2 The sample can absorb carbon dioxide from the atmosphere if left over a period of time. Dissolved carbon dioxide will cause the sample pH to appear lower than it should read. Other acid vapors such as HCl can also have this effect and ammonia vapor can have the reverse effect.

6.2.3 Temperature exerts two effects on pH measurements. The first is caused by the change in electrode output at various temperatures. This can be controlled with instruments having temperature compensation or by calibration of the electrode-instrument system at the temperature of the samples. The second effect is the change of ionization in the sample at various temperatures. This error is sample dependent and can only be eliminated by making both the standardization and the measurement at the standard temperature for pH measurement which is 25°C.

6.2.4 The tips of the electrodes should be immersed in solution for a minimum of three hours prior to use or in accordance with the electrode manufacturer's instructions.

6.2.5 After each series of measurements, the electrodes should be stored with the tips immersed in a pH 7.00 buffer.

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6.3 WORKING RANGES

0 - 14.00 pH Units

7.0 PROCEDURE

7.1 SAMPLE PRETREATMENT

For sample pretreatment see pH Section in EI-7.2, "Emergency Post Accident Analysis".

7.2 METER STANDARDIZATION

7.2.1 Standardization of the pH meter should be performed daily or prior to use, whichever is less frequent, using the following general procedure. Follow the instrument manufacturer's instructions for instrument operation.

7.2.2 Standardization should be checked every two hours of operation by using one pH buffer which is at a temperature close to that of the sample. Reading should be  $\pm 0.1$  pH units of correct value. If not, restandardize pH meter and remeasure samples run since last satisfactory check, if possible.

7.2.3 Connect electrodes to pH meter and connect pH meter to power supply if necessary.

7.2.4 Place pH meter in standby condition and allow it to warm up thoroughly (about two minutes).

7.2.5 Remove the electrodes from the water and rinse with water.

7.2.6 Place a pH seven (7) buffer solution in a clean beaker using sufficient volume to cover the sensing elements of the electrodes and to give adequate clearance for the magnetic stirring bar.

7.2.7 Place a stirring bar in the beaker and immerse the electrodes into the solution. Turn on the stirrer and adjust the rate of stirring to provide homogeneity, but slow enough to prevent splashing and air entrainment.

7.2.8 Measure the buffer solution temperature and set the temperature compensator on the pH meter to this temperature.

7.2.9 Set the meter to read and adjust the meter to read the specified pH of the buffer solution, at the measured temperature, by use of the calibration/standardization knob.

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- 7.2.10 Place the meter in standby condition, remove the electrodes from the buffer solution and rinse the electrodes with water.
- a. If the slope setting cannot be adjusted to obtain the correct reading within the allowable range, the pH measuring system is not operating properly and shall be removed from service.

- 7.2.11 Repeat the above standardization using a buffer solution two or more pH units different from the first. Obtain the listed value of this buffer by adjusting the slope setting.

7.3 FUNCTIONAL CHECK

Check the above standardization, as appropriate, using a third buffer solution two or more pH units different from the previously used buffer solutions, but do not move the standardization knob nor adjust the slope setting. The meter should read  $\pm 0.1$  pH units of the listed value of this buffer. Record standardization of pH meter in the Chemistry Logbook.

7.4 SAMPLING PREPARATION

Sample preparation is listed in EI-7.2, "Emergency Post Accident Analysis".

7.5 ANALYSIS

- 7.5.1 Using the pH probe and meter standardized in Paragraph 7.2, insert probe into the undiluted sample cask top until it cannot be inserted any further.
- 7.5.2 Ensure probe has been inserted through septum on sample vial. A slight resistance will be felt as probe passes through septum and comes to rest against stop.
- 7.5.3 When the pH meter has stabilized, record the PCS undiluted liquid pH on Attachment 1 of EI-7.2, "Emergency Post Accident Analysis".
- 7.5.4 Slowly withdraw the pH probe from the undiluted sample cask and place it in shielded probe holder. Rinse the pH probe in the laboratory sink.

7.6 DISPOSAL OF REAGENT, ANALYTE, GLASSWARE

All standards and non-radioactive samples should be disposed by using normal sample disposal methods. Radioactive samples should be stored in a shielded area and saved for  $\text{Cl}^-$  analysis.



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7.7 CALCULATIONS

None

8.0 ACCEPTANCE CRITERIA

Functional check for the buffer solution should be within  $\pm 0.1$  pH units of correct value for LED readout meters and  $\pm 0.2$  pH units for meter display pH meters.

9.0 RECORDS AND ATTACHMENTS

9.1 ATTACHMENTS

None

9.2 RECORDS

Functional Checks of the pH meter should be recorded in the Chemistry Logbook.