FLORIDA POWER & LIGHT COMPANY
TURKEY POINT UNITS 3 AND 4
NUCLEAR CHEMISTRY PROCEDURE NC-88
APRIL 13, 1979

1.0 Title:

DETERMINATION OF AMMONIA-ELECTRODE METHOD

2.0 Approval and List of Effective Pages:

2.1 Review and Approval:

Consultant Stephen A. Thompson Date 2-16-79
Consultant David H. Saunders Date 2-7-79
Radiochemist Edward L. Fabian Date 12-6-78
Tech Supv D.W. Haase Date 3-12-79
Plant Supv-Nuc J.K. Hays Date 4/13/79
Approved by Plant Nuclear Safety Committee Date 4/13/79

2.2 List of Effective Pages:

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3.0 Scope:

3.1 Requirement:

In order to determine the amount of free OH^- ions in the steam generators and maintain the proper feedwater and boiler water pH control, it is necessary to determine the amount of ammonium ions in solution.

3.2 Purpose:

The ammonia gas sensing electrode has been determined to be the most acceptable and reliable method at Turkey Point 3 and 4 for the determination of ammonium ions in the feedwater and steam generators. The purpose of this procedure is to provide laboratory personnel with instructions necessary to verify proper electrode response, and to determine ammonium ion concentrations of feedwater and steam generator aqueous samples.

4.0 Instructions:

NOTE: If it is not possible to use the electrode method for determination of ammonia concentration, refer to ASTM D-1426 for the determination of ammonia by the direct Nesslerization method.

4.1 Calibration of the Ammonia Gas Sensing Electrode and Orion Microprocessor.

NOTE: When the electrode is not in use it should be kept immersed in a 0.1M ammonium chloride solution of neutral pH. The microprocessor function switch should be in the stand-by position.

4.1.1 Set the function switch on the processor to the concentration position.

4.1.2 Rinse the electrode thoroughly with demineralized water. Wipe the electrode dry. Do not wipe the membrane itself.

4.1.3 Immerse the electrode into a 4.01 pH buffer solution, stir and let the microprocessor stabilize.

4.1.4 Prepare standards of 1.0ppm and 10ppm from the 100ppm ammonia standard. The volume of the standards should be 100 ml.

4.1.5 Remove the electrode from the 4.01 buffer solution and wipe it dry.

4.1.6 Immerse the electrode in 100 ml of 1.0ppm standard and add 1 ml of 10N sodium hydroxide. Allow the microprocessor time to stabilize. Inspect for air bubbles under the electrode and dislodge any that may be present.

4.1.7 When the microprocessor is stabilized, after approximately 3-4 minutes, depress the "Set Concentration" button. Insure that the concentration read-out remains stable at 1.0ppm.

4.1.8 Remove the electrode from the 1.0ppm solution, rinse thoroughly with demineralized water and wipe dry.

4.1.9 Immerse the electrode in a 4.01 buffer solution and allow the meter to attain a stable reading.

4.1.10 Remove the electrode from the 4.01 buffer solution and wipe dry.

4.1.11 Immerse the electrode in 100 ml of 10ppm standard and add 1 ml of 10N sodium hydroxide. The digital read-out should come up to a reading of 10.0. If not, adjust the slope. The slope value must be 58.16±1 mv.

4.1.12 If the value in 4.1.11 cannot be attained, repeat steps 4.1.3 through 4.1.11. If the proper slope cannot be achieved after repeating steps 4.1.3 through 4.1.11, consult the tech manual for the procedure for changing the electrode membrane. Notify supervisory personnel before changing the membrane.

4.1.13 Remove the electrode from the 10.0ppm solution and rinse thoroughly with demineralized water. Wipe the electrode dry.

4.1.14 Immerse the electrode in a 4.01 buffer solution and allow the meter to attain a stable reading.

4.2 Determination of Ammonia Concentration in Aqueous Solutions

4.2.1 Insure that the Orion microprocessor has been calibrated in accordance with 4.1 above.

4.2.2 Remove the electrode from the 4.01 buffer solution and wipe it dry.

4.2.3 Pour 100 ml of sample into a 150 ml beaker.

4.2.4 Immerse the electrode in the sample and add 1 ml of 10N sodium hydroxide. Insure that no air bubbles are trapped on the bottom of the electrode.

4.2.5 Allow the meter reading to stabilize for 2-3 minutes. Record the reading on the appropriate log sheet.

4.2.6 Remove the electrode from the sample solution and rinse thoroughly with demineralized water. Wipe the electrode dry.

4.2.7 Immerse the electrode in a 4.01 buffer solution and allow the meter to attain a stable reading.

4.2.8 Repeat steps 4.2.2 through 4.2.7 for each sample solution.

4.2.9 When the analyses are completed, move the function switch to stand-by and immerse the electrode in a solution of 25 ml of 0.1M ammonium chloride and 25 ml of demineralized water.

4.3 Checking the Electrode Operation

4.3.1 Remove the electrode from the stand-by solution (step 4.2.9) and rinse it thoroughly with demineralized water. Wipe the electrode dry.

4.3.2 Pour 100 ml of demineralized water in a 150 ml beaker.

4.3.3 Immerse the electrode in the demineralized water and turn the function switch to "Relative M.V.".

4.3.4 Add 1 ml of 10N sodium hydroxide and 1 ml of 0.1M ammonium chloride solution to the demineralized water in the beaker.

4.3.5 Allow the meter reading to stabilize for 2-3 minutes. When the reading is stable, depress the "Set Concentration" button to zero the meter.

4.3.6 Add 10 ml of 0.1M ammonium chloride solution to the beaker. Allow the meter reading to stabilize.

4.3.7 The meter should stabilize at a reading of 58.16±1 mv.

4.3.8 If this value cannot be attained, repeat steps 4.3.1 through 4.3.7. If after repeating steps 4.3.1 through 4.3.7 the value still cannot be attained, consult the tech manual for the procedure for changing the electrode membrane. Notify supervisory personnel before changing the membrane.

4.3.9 Remove the electrode from the solution (step 4.3.6) and rinse it thoroughly in demineralized water. Wipe the electrode dry.

4.3.10 Immerse the electrode in an "overnight solution" (see Note in step 4.1) and set the function switch to stand-by.