FLORIDA POWER & LIGHT COMPANY
TURKEY POINT UNITS 3 AND 4
NUCLEAR CHEMISTRY PROCEDURE NC-93
NOVEMBER 21, 1979

1.0 Title:

DETERMINATION OF FREE HYDROXIDE CONCENTRATION

2.0 Approval and List of Effective Pages:

2.1 Approval:

Consultant David K. Knutson Date 11-8-79
Consultant Jennifer A. Chase Date 11-15-79
Radiochemist Edward L. Letsinger Date 11-15-79
Tech Supv [signature] Date 11/20/79
Plant Supt-Nuc [signature] Date 11/21/79
Approved by Plant Nuclear Safety Committee Date 11/21/79

2.2 List of Effective Pages:

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				4	11/21/79

3.0 Scope:

3.1 Requirements:

Monitoring of free hydroxides is important to determine the potential for caustic embrittlement in the steam generators. The free hydroxide test will be performed on designated days when the following criterion is met:

$$[Na^+]ppm \geq 0.05 \times \text{S.G. Cation Conductivity } (\mu mhos)$$

3.2 Purpose:

The purpose of this procedure is to provide laboratory personnel with an approved method for determining free hydroxide concentration.

3.3 Acceptance Criteria:

The free hydroxide concentration should be maintained below the Westinghouse recommended limit for AVT chemistry control.

4.0 Instructions:

4.1 Apparatus:

To insure that the necessary accuracy is obtained in this procedure certain specialized apparatus are required:

4.1.1 Digital pH meter using dual electrodes.

4.1.2 Piston Buret (See NC-91 part 4.0 for operating instructions).

4.1.3 A titration beaker with a large stopper having openings for electrodes and an inlet and outlet for purge gas (argon).

4.2 Standardization of H_2SO_4

4.2.1 Prepare approximately one liter of ≈0.002N sulfuric acid as described in "Preparation of Reagents" Nuclear Chemistry Turkey Point.

4.2.2 Standardize the boric acid titration pH meter as described in NC-20.

4.2.3 Pipette 50 ml of the ≈0.002N H_2SO_4 into a 100 ml beaker.

4.2.4 Titrate with the NaOH standard (used in boric acid titrations) to pH 7.0 ±.05. Stir the solution during the titration with a magnetic stirrer.

4.2.5 Use the following calculations to determine the N of H_2SO_4.

4.2.5.1 $$\text{Normality of NaOH} = \frac{\text{Normality of } H_3BO_3 \times \text{Volume } H_3BO_3}{\text{Volume NaOH}}$$

The normality of 100 ppm H_3BO_3 = 0.009547 for boric acid titration using mannitol

$$\text{Normality of NaOH} = \frac{(0.009547) \times (25 \text{ ml } H_3BO_3)}{\text{Volume NaOH}}$$

4.2.5.2 $$\text{Normality of } H_2SO_4 = \frac{\text{ml NaOH titrant} \times \text{Normality of NaOH}}{\text{Volume } H_2SO_4 \text{ (50 ml)}}$$

4.3 Preparation of pH meter to be used in the hydroxide titration

4.3.1 Soak the electrodes overnight in deionized water or in a buffer solution with pH 4.01.

4.3.2 Standardize the pH meter with pH 5.0 buffer solution (see NC-20).

4.3.3 Introduce the argon blanket into the titration beaker using a continuous purge from the argon line located in the cold lab.

4.4 Sampling

4.4.1 Purge each 200 ml volumetric flask to be used for sampling with argon by inserting a piece of tubing from the argon gas line to the bottom of the flask. Purge the flask with argon for ≈30 seconds. Withdraw the tubing and immediately stopper the flask. Proceed to the sample location.

4.4.2 Fill the volumetric flask to the top with sample and stopper immediately to minimize air contamination. Also draw a second sample for NH_3 and N_2H_4 analyses in an appropriate polypropylene container.

4.4.3 Allow the samples to cool to room temperature.

4.4.4 Adjust the volume in the flask to exactly 200 ml and pour into the titration beaker. Continue the argon purge as in step 4.3.3.

4.4.5 Note the temperature and adjust the pH meter accordingly.

4.4.6 While stirring with the magnetic stirring apparatus, titrate the sample using the piston burette. Add titrant in 1 ml increments.

4.4.7 Begin recording the pH for each incremental addition of the acid upon achieving a pH of ≈5.0. Continue titration until the pH is approximately 4.35. Three to six data points between pH 5.0 and pH 4.35 should have been recorded.

4.4.8 Plot the data points recorded in 4.4.7 on the semi-antilog pH paper supplied by Westinghouse. Back extrapolate with a straight edge to 7.0 to determine the titration endpoint. See Appendix I for an example data sheet.

4.4.9 Analyze the other samples obtained in step 4.4.2 for ammonia and hydrazine as described in NC-88 and NC-97.

4.5 Calculations

4.5.1 Calculate the total hydroxide concentration as follows:

THC ppm = (Normality of H_2SO_4) x (ml H_2SO_4) x 85.0

The free hydroxide in ppm hydroxide can then be calculated by subtracting the amine contribution from the total hydroxide concentration (THC).

Free hydroxide, ppm = THC - (ppm NH_3) - (0.53 x ppm N_2H_4)

4.5.2 Record the results in the appropriate log books.

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APPENDIX I

Reduced copy for example only

mls