

FLORIDA POWER & LIGHT COMPANY  
TURKEY POINT UNITS 3 AND 4  
NUCLEAR CHEMISTRY PROCEDURE NC-82  
OCTOBER 3, 1978

1.0 Title:

DETERMINATION OF CHLORIDE IN AQUEOUS SOLUTIONS - MERCURIMETRIC TITRATION

2.0 Approval and List of Effective Pages:

2.1 Review and Approval:

Consultant	<u>David H. Knutson</u>	Date	<u>9-12-78</u>
Consultant	<u>Paul L. Sealey</u>	Date	<u>9-14-78</u>
Radiochemist	<u>Edward L. LaRue</u>	Date	<u>9-14-78</u>
Tech Supv	<u>D. W. Hearn</u>	Date	<u>10-3-78</u>
Plt Supt Nuc	<u>J. K. Hays</u>	Date	<u>10-12-78</u>
Approved by Plant Nuclear Safety Committee		Date	<u>October 3, 1978</u>

2.2 List of Effective Pages:

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3.0 Scope:

3.1 Requirement:

Technical Specifications Table 4.1-2 requires the monitoring of reactor coolant samples for chloride (5/wk). In addition,  $\text{Cl}^-$  is monitored in both primary and secondary systems and their auxiliaries to insure a high degree of purity and to monitor the effects of transient conditions such as condenser inleakage, etc.

3.2 Purpose:

The purpose of this procedure is to provide laboratory personnel with an approved standardized method for the determination of low level chloride ion concentration in aqueous solutions.

3.3 Acceptance Criteria:

The  $\text{Cl}^-$  limit in the reactor coolant system is 0.15 ppm. The minimum sensitivity for this titration is 0.05 ppm  $\text{Cl}^-$ .

4.0 Instructions:

NOTE: Refer to the lab booklet on preparation of reagents for a listing of reagents used and their preparations.

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DETERMINATION OF CHLORIDE IN AQUEOUS SOLUTIONS - MERCURIMETRIC TITRATION4.1 Use of the reagent blank:

4.1.1 A reagent blank is utilized in this procedure to compensate for the effects and possible minor impurities of the reagents used. The reagent blank consists of a portion of  $\text{Cl}^-$  free water that is otherwise representative of the sample to be analyzed. (i.e., demineralizer water is used as a reagent blank for RCS, makeup water tanks, condensate, steam generators, etc. For CCW, a  $\text{Cl}^-$  free sample of water containing approximately the same concentration of potassium chromate should be used.)

4.1.2 A reagent blank should be titrated just prior to the sample, by the same analyst titrating the sample. However, whenever numerous samples are analyzed for  $\text{Cl}^-$  during the day, it is only necessary to have the reagent blank analyzed once per day by each analyst performing the test. Determine a new reagent blank whenever reagents are renewed or contamination of reagents is suspected.

4.2 Test Procedure -  $\text{Cl}^-$  (0-1.0 ppm):

4.2.1 Transfer 100 ml of colorless sample and 100 ml of colorless reagent blank into a container suitable for titrating.

4.2.2 To each of the containers add 8-10 drops of chloride indicator. (Diphenylcarbazone - bromophenol blue)

4.2.3 Following addition of the indicator, the sample should be a shade of blue, violet, or red. If a yellow or orange color develops, refer to ASTM procedure D-512.

4.2.4 To each container add 0.3% nitric acid dropwise while stirring until a pale yellow color develops, then add approximately 1 ml excess acid.

4.2.5 Carefully, titrate each sample with 0.0141N mercuric nitrate using a microburette graduated in .01 ml increments.

4.2.6 The end point is reached when a pale violet color persists. Both the blank and sample should be titrated to an identical shade of violet.

4.2.7 Note the number of milliliters of mercuric nitrate used for each titration. If the final burette level is between graduations, read to the nearest .005 ml.

4.2.8 Subtract the amount (ml) of mercuric nitrate used to titrate the blank from the amount (ml) of mercuric nitrate used to titrate the sample. Multiply the difference by 5 to obtain the concentration of  $\text{Cl}^-$  in ppm. Report  $\text{Cl}^-$  concentration to a minimum sensitivity of .05 ppm. If less than .05, report as <0.05 ppm.

4.3 Test Procedure -  $\text{Cl}^-$  (>1.0 ppm):

4.3.1 Follow the procedure, 4.2 above except that titration of the sample should be performed rapidly at first. As the endpoint is neared, the titration rate should be slowed. The endpoint is reached when a pale violet color persists.

a. Use of a larger volume burette in place of the microburette (.01 ml graduations) should be considered for obtaining more rapid results.

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4.3.2 For very high concentrations of chloride (>50 ppm), volumetric dilution of the samples should be considered. Multiply the results obtained in step 4.2.8 by the appropriate dilution factor to obtain the final results.

4.4 Removal of Interferences:

4.4.1 Turbid samples should be filtered to remove suspended materials prior to analysis.

4.4.2 Removal of chromium from systems such as component cooling water can be accomplished as follows:

- a. Transfer a quantity of sample and reagent blank into separate containers, such that 100 ml of colorless sample will be available for analysis.
- b. Add two scoops of barium nitrate to each of the containers and mix well.
- c. The resulting barium chromate can now be separated by centrifuging or filtration.
- d. Use the resulting colorless solutions for reagent blank and samples.

4.4.3 For removal of interferences other than those normally encountered, refer to ASTM procedure D-512.

4.5 Cl<sup>-</sup> Concentration in solutions containing concentrated boric acid (i.e., BASTs):

4.5.1 Samples of solutions containing concentrated boric acid are diluted such that crystallization of boric acid will not occur at room temperature.

4.5.2 Immediately after sampling, transfer 25 ml of concentrated boric acid sample into a suitable container. Dilute to 100 ml with demineralized water.

4.5.3 Perform the titration and calculations in accordance with 4.2 or 4.3 above.

4.5.4 Multiply results obtained in 4.5.3 by 4 to account for the dilution factor and to obtain Cl<sup>-</sup> concentration. The minimum sensitivity achievable is 0.2 ppm.