FLORIDA POWER & LIGHT COMPANY
TURKEY POINT UNITS 3 AND 4
NUCLEAR CHEMISTRY PROCEDURE NC-83
NOVEMBER 21, 1979

1.0 Title:

DETERMINATION OF DISSOLVED OXYGEN CONCENTRATION IN WATER

2.0 Approval and List of Effective Pages:

2.1 Review and Approval:

Consultant _____ Date 11-2-79
Consultant _____ Date 10-30-79
Radiochemist _____ Date 10-30-79
Tech Supv _____ Date 11/20/79
Plant Supt-Nuc _____ Date 11/21/79
Approved by Plant Nuclear Safety Committee Date 11/21/79

2.2 List of Effective Pages:

Page	Date	Page	Date	Page	Date
1	11/21/79	3	11/21/79	5	11/21/79
2	11/21/79	4	11/21/79		

3.0 Scope:

3.1 Requirement:

Determination of dissolved oxygen in the Reactor Coolant System is required in accordance with Technical Specifications, Table 4.1-2. In addition, O_2 concentration is monitored in certain primary and secondary systems because control of dissolved oxygen concentration is essential in controlling fluid system corrosion.

3.2 Purpose:

The purpose of this procedure is to provide personnel with the following approved methods of determining dissolved oxygen concentration:

Method A - Chemetric Comparison Method - Low Range (0-100 ppb)
Method B - Chemetric Comparison Method - High Range (0-12 ppm)
Method C - Indigo Carmine Comparison Method - Low Range (0-100 ppb)
Method D - Winkler Titration (>300 ppb)

3.3 Acceptance Criteria:

Dissolved oxygen in the Reactor Coolant System will be maintained below 0.10 ppm (100 ppb) whenever the coolant temperature is above 250°F. Reference: Technical Specifications 3.1.5.

4.0 Instructions:

4.1 Method A - Chemetric Comparison Method - Low Range (0-100 ppb)

NOTE: Method A is a simple, rapid test for the analysis of dissolved oxygen. It is the recommended method for the determination of dissolved oxygen concentration when in the range of 0-100 ppb.

4.1.1 Sample collection may be performed by either of the following methods:

4.1.1.1 Method 1:

4.1.1.1.a Insure there are no breaks in the tubing from the sample source.

4.1.1.1.b Initiate sample flow by opening the sample valve and adjusting flow to approximately 500 to 1000 ml/minute.

4.1.1.1.c After the sample tubing has been purged of entrapped air, insert a chemet approximately halfway into the end of the tubing, tapered tip first.

4.1.1.1.d Agitate the chemet inside the tubing to release any air bubbles.

4.1.1.1.e Using moderate pressure press downward and sideways on the tip of the chemet to break it.

4.1.1.1.f Go on to step 4.1.2.

4.1.1.2 Method 2:

4.1.1.2.a Insure that the bottom of the sample tube is not filled with broken chemet tips.

4.1.1.2.b Connect the tubing from the sample source to the bottom of the plastic sample tube.

4.1.1.2.c Initiate sample flow by opening the sample valve and adjusting flow to approximately 1000 ml/min.

4.1.1.2.d After the sample tube has been purged of entrapped air, insert the snapper tool into the sample stream through the opening at the top of the sample tube.

4.1.1.2.e Insert a chemet into the barrel of the snapper tool, tapered tip downward.

4.1.1.2.f Agitate the chemet and snapper tool slightly to release any air bubbles from the surface of the chemet.

4.1.1.2.g Using moderate pressure, press downward on the chemet to break the tip.

4.1.2 When the chemet has filled, remove it from the sample tube or tubing and immediately position a finger over the opening to prevent air contamination. The chemet should be removed within five seconds of breaking the tip.

4.1.3 Holding the chemet with your finger over the opening in the tip, invert it repeatedly, each time allowing the inert gas bubble to travel from end to end. Continue until uniform coloration in the chemet is achieved.

4.1.4 Wipe the outside of the chemet and place it in the center position of either the 0-40 ppb or 0-100 ppb comparator.

4.1.5 Viewing the comparator against a light source, determine the dissolved oxygen concentration by comparison with the labeled standards.

4.1.6 Record the dissolved oxygen concentration.

4.2 Method B - Chemetric Comparison Method - High Range (0-12 ppm)

NOTE: Method B is a convenient and rapid method for the determination of high range dissolved oxygen concentration, i.e. 0-12 ppm. It is a particularly useful method for determining oxygen concentration in the reactor coolant system when the system has been depressurized and opened to the atmosphere.

4.2.1 Obtain at least 25 ml of sample, taking all necessary precautions for excluding air from the sample.

4.2.2 Immediately after opening sample container, fill the sample cup to 25 ml.

4.2.3 Add three drops of neutralizer solution.

4.2.4 Place a high range chemet inside the barrel of the snapper tool, and insert into the sample cup.

4.2.5 Stir the sample with the chemet for one or two seconds.

4.2.6 After stirring, wait approximately 15 seconds, then press down on the chemet to snap off the tip.

4.2.7 When the chemet has filled, remove it from the sample. Place a finger over the exposed tip, and invert repeatedly while allowing the inert gas bubble to travel from end to end. Continue to invert until uniform coloration in the chemet is achieved.

4.2.8 After about two minutes, compare the chemet against the comparator kit.

NOTE: The comparator kit should be illuminated by a strong white light behind the translucent backing. Store the comparator kit in the dark when not in use.

4.2.9 Record the dissolved oxygen concentration.

4.3 Method C - Indigo Carmine Comparison Method (0-100 ppb)

NOTE: This method is somewhat more complex to perform than Method A; however, it may be used in place of Method A when the situation warrants.

4.3.1 Collect the sample to be analyzed in a 250 ml McLeans's tube. The sample inlet is connected to the bottom of the tube and the overflow is from the top.

4.3.2 Agitate the McLean's tube during filling to dislodge any air bubbles from the inside of the tube.

4.3.3 Purge the tube sufficiently until a representative sample is obtained.

4.3.4 Isolate the sample in the tube by closing the inlet and outlet stopcocks.

4.3.5 Withdraw about 3.5 ml of Indigo Carmine - Potassium Hydroxide working solution into a syringe.

4.3.6 Insure that no air is trapped in the syringe before performing step 4.3.7.

4.3.7 Insert the needle into the injection port of the McLean's tube and inject 3.5 ml of solution into the sample, while venting the McLean's tube from the top.

4.3.8 Remove the syringe from the injection port.

4.3.9 Invert the McLean's tube several times until a uniform color is achieved.

4.3.10 Compare the color developed with the prepared standards.

4.3.11 Record the oxygen level.

4.4 Method D - Winkler Titration (>300 ppb)

NOTE: Method D is a more precise measurement of oxygen where dissolved concentrations are greater than 300 ppb.

4.4.1 Collect the sample to be analyzed in a 500 ml McLean's tube. The sample inlet is connected to the bottom of the tube, and the overflow is from the top.

4.4.2 Flush the tube sufficiently to collect a representative air free sample.

4.4.3 Isolate the sample by closing the inlet and outlet stopcocks.

4.4.4 Using a syringe, inject 2 ml manganous sulfate reagent into the McLean's tube through the injection port, while venting the tube from the top.

4.4.5 Invert tube to mix uniformly.

4.4.6 Using a second syringe, inject two ml of alkaline iodide reagent into the tube in the same manner as step 4.4.4 and invert the tube to mix.

4.4.7 Allow the sample to sit for ten minutes.

4.4.8 Using a third syringe, inject the tube with 2 ml of 27N sulfuric acid in the same manner as step 4.4.4.

4.4.9 Mix sample well and allow to cool to 65-70°F before titrating.

4.4.10 Drain about 50 ml of sample from the bottom of the McLean's tube and discard.

4.4.11 Collect 250 ml of sample from the tube and transfer to a container suitable for titrating.

4.4.12 If the treated sample is dark brown, omit step 4.4.13. Titrate with N/100 sodium thiosulfate until the brown color almost disappears, then add approximately 2 ml of starch solution. The addition of starch will turn the sample blue. Go on to step 4.4.14.

4.4.13 If the treated sample is not dark brown, add approximately 2 ml of starch solution. Upon the addition of starch, the sample should turn blue. If it does not turn blue with the starch addition, the sample contains no appreciable oxygen.

4.4.14 Titrate the sample with N/100 sodium thiosulfate until the sample becomes clear. Note that the endpoint is abrupt.

4.4.15 Determine the oxygen concentration in ppm as follows:

$$\text{ml (sodium thiosulfate)} \times 0.317 = \text{ppm oxygen}$$

4.4.16 Record results.