FLORIDA POWER AND LIGHT COMPANY
TURKEY POINT UNITS 3 AND 4
NUCLEAR CHEMISTRY PROCEDURE NC-84
OCTOBER 8, 1981

1.0 Title:

DETERMINATION OF Cu, Fe, Na, Cr, Ca, K, and Mg
BY THE ATOMIC ABSORPTION FLAME METHOD

2.0 Approval and List of Effective Pages:

2.1 Approval:

Change Dated 10/8/81 Reviewed by Plant Nuclear Safety Committee: 81-67

and Approved by Plant Manager - Nuclear: 10/8/81

2.2 List of Effective Pages:

Page	Date	Page	Date	Page	Date	Page	Date
1	10/8/81	4	10/8/81	7	10/8/81	10	10/8/81
2	10/8/81	5	10/8/81	8	10/8/81		
3	10/8/81	6	10/8/81	9	10/8/81		

3.0 Scope:

3.1 Requirement:

3.1.1 Excess copper (Cu) and/or iron (Fe) in the feedwater indicates corrosion in the secondary system. Excess sodium (Na) along with free hydroxide in the steam generators can lead to caustic embrittlement of steam generator components. It is, therefore, necessary that the Cu and Fe concentrations in the feedwater, and Na concentration in the steam generators be routinely monitored.

3.1.2 Detectable chromium (Cr) concentrations in the RHR system indicates inleakage from the component cooling water system. The RHR system should be checked prior to putting it in service and periodically while it is in service.

3.1.3 Calcium (Ca) and magnesium (Mg) can react with aluminum silicates to form insoluble compounds. Sodium (Na) and potassium (K) can react with aluminum silicates to form soluble compounds. These insoluble and soluble compounds, known as zeolites, may be deposited on the fuel rod cladding and cause unequal neutron flux and local "hot spots". For this reason, the concentrations of Ca, Mg, Na and K are monitored in various plant systems. Aluminum (Al) and silicate (SiO_2) concentrations are also monitored using "wet chemistry" techniques rather than the atomic absorption flame method.

3.2 Purpose:

The purpose of this procedure is to provide laboratory personnel with an approved method for the determination of low level concentrations of Cu, Fe, Na, Cr, Ca, K, and Mg.

3.3 Acceptance Criteria:

3.3.1 Concentrations of the following elements in the feedwater should be below Westinghouse recommendations for AVT chemistry control:

1. Cu < 0.005 ppm
2. Fe < 0.010 ppm
3. Na < 0.100 ppm

3.3.2 Concentrations of Cr in the RHR system should be less than detectable, i.e. < 0.1 ppm.

3.3.3 Concentrations of Ca, K, Mg, and Na have the following limits in the listed systems.

SYSTEM	PWST	RWST	RCS	RHR	SFP	BAST
Ca Limit (ppm)	< 0.02	< 0.08	< 0.05	< 0.08	< 0.10	< 0.66
K Limit (ppm)	< 0.01	*	*	*	*	*
Mg Limit (ppm)	< 0.02	< 0.08	< 0.05	< 0.08	< 0.10	< 0.66
Na Limit (ppm)	< 0.01	*	*	*	*	*

*Analysis is not required.

4.0 Instructions:

4.1 Copper (Cu) Analysis:

4.1.1 Select or prepare 2 copper standards with at least one within the optimal range of instrument operation (0.04 - 7 ppm). One standard should be of a value near that expected for the samples. Normally a 100 ppb standard is used for feedwater samples. When operating in a range below 100 ppb, a 25 ppb standard should be prepared just prior to use.

4.1.2 Turn instrument power on.

4.1.3 Rotate the copper hollow cathode lamp into position.

4.1.4 Set the instrument as follows:

1. Hollow cathode switch to A.
2. Mode switch to SB-A.
3. Copper hollow cathode lamp current to 5 mA.
4. Curve correct both fully counterclockwise.

4.1.5 Check burner alignment and make adjustments if necessary. The light beam should pass directly over the center of the slot of the single slot burner. Use the burner alignment tool for this purpose.

4.1.6 Make the following instrument adjustments:

1. Burner height adjusted by burner alignment tool.
2. Slit width - 320 μm
3. I/Io switch - I

4.1.7 Dial wavelength to 324.7 nm. Optimization is achieved when a maximum deflection is observed on the intensity meter. Optimization should occur between 2 and 8 on the intensity meter. To achieve this, the lamp current or H.V. on the photomultiplier tube should be adjusted.

4.1.8 Focus the light beam on the photomultiplier tube using the zoom lens. Optimize as in 4.1.7.

4.1.9 Go to step 4.8.

4.2 Iron (Fe) Analysis:

4.2.1 Select or prepare 2 iron standards with at least one within the optimal range of instrument operation (0.1 - 11.0 ppm). One standard should be of a value near that expected for the samples. Normally a 100 ppb standard is used for the feedwater samples. When operating in a range below 100 ppb, a 25 ppb standard should be prepared just prior to use.

4.2.2 Turn instrument power on.

4.2.3 Rotate the iron hollow cathode lamp into position.

4.2.4 Set instrument as follows:

1. Hollow cathode switch to A.
2. Mode switch to SB-A
3. Iron hollow cathode lamp current to 10 mA
4. Curve correct both fully counterclockwise

4.2.5 Check burner alignment and make adjustments if necessary. The light beam should pass directly over the center of the slot of the single slot burner. Use burner alignment tool for this purpose.

4.2.6 Make the following instrument adjustments:

1. Burner height adjusted by burner alignment tool.
2. Slit width - 80 μm
3. I/Io switch - I

4.2.7 Dial wavelength to 248.3 nm. Optimization is achieved when a maximum deflection is observed on the intensity meter. Optimization should occur between 2 and 8 on the intensity meter. If not, lamp current or H.V. on PM tube should be adjusted.

4.2.8 Focus the light beam on the photomultiplier tube using the zoom lens. Optimize as in 4.2.7.

4.2.9 Go to step 4.8.

4.3 Sodium (Na) Analysis:

4.3.1 Select or prepare a sodium standard within the optimal range of instrument operation (0.008 - 0.9 ppm). The standard should be of a value near that expected for the samples.

4.3.2 Turn instrument power on.

4.3.3 Place the sodium hollow cathode lamp into position.

4.3.4 Set the instrument as follows:

1. Hollow cathode switch to A
2. Mode switch to SB-A
3. Sodium hollow cathode lamp current to 8 mA
4. Curve correct both fully counterclockwise

4.3.5 Check burner alignment and make adjustments if necessary. The light beam should pass directly over the center of the slot of the single slot burner. Use the burner alignment tool for this purpose.

4.3.6 Make the following instrument adjustments:

1. Burner height adjusted by burner alignment tool
2. Slit width - 160 μm
3. I/Io switch - I

4.3.7 Dial wavelength to 589.0 nm. Optimization is achieved when a maximum deflection is observed on the intensity meter. Optimization should occur between 2 and 8 on the intensity meter. To achieve this, lamp current or H.V. on photomultiplier tube should be adjusted.

4.3.8 Focus the light beam on the photomultiplier tube using the zoom lens.

4.3.9 Go to step 4.8.

4.4 Chromium (Cr) Analysis:

4.4.1 Select or prepare a chromium standard within the optimal range of instrument operation (0.100 - 16.0 ppm). The standard should be of a value near that expected for the samples.

4.4.2 Turn instrument power on.

4.4.3 Rotate the chromium hollow cathode lamp into position.

4.4.4 Set the instrument as follows:

1. Hollow cathode switch to A
2. Mode switch to SB-A
3. Chromium hollow cathode lamp current to 7 mA
4. Curve correct both fully counterclockwise

4.4.5 Check burner alignment and make adjustments if necessary. The light beam should pass directly over the center of the slot of the single slot burner. Use the burner alignment tool for this purpose.

4.4.6 Make the following instrument adjustments:

1. Burner height adjusted by burner alignment tool
2. Slit width - 160 μm
3. I/Io switch - I

4.4.7 Dial wavelength to 357.9 nm. Optimization is achieved when a maximum deflection is observed on the intensity meter. Optimization should occur between 2 and 8 on the intensity meter. To achieve this, lamp current or H.V. on photomultiplier tube should be adjusted.

4.4.8 Focus the light beam on the photomultiplier tube using the zoom lens. Optimize as in 4.4.7.

4.4.9 Go to step 4.8.

4.5 Calcium (Ca) Analysis:

4.5.1 Standards and Blanks: Boron concentrations, as high as 2000 ppm have been shown to have no effect on the detection of calcium at the 1.0 ppm level. A 1.0 ppm standard should be prepared by diluting a 1000 ppm calcium standard 1 ml to 1 liter. When magnesium is to be determined at the same time, the standard can contain both Ca and Mg.

4.5.2 Turn on power to the atomic absorption spectrophotometer.

4.5.3 Set the following instrument conditions:

1. Hollow cathode switch to A
2. Mode switch to SB-A
3. Calcium hollow cathode lamp in place with current at 7 mA
4. Curve correct both fully counterclockwise
5. I/Io switch to I
6. Burner height adjusted by burner alignment tool
7. Slit width at 320 μm

4.5.4 The single slot burner head should be in place with the beam from the hollow cathode lamp passing over the center of the slot. The burner alignment tool may be used to check this.

4.5.5 Set the wavelength to 422.7 nm. Optimum sensitivity is achieved when the deflection of the intensity meter is at a maximum. If the intensity meter is not in the range between 2 and 8, adjust the high voltage on the photomultiplier tube and/or the lamp current to get into this range.

4.5.6 Focus the beam on the PM tube, optimizing as described in 4.5.5

4.5.7 Go to step 4.8.

4.6 Potassium (K) Analysis:

4.6.1 Standards and Blanks: A 100 ppb standard should be prepared prior to analysis, by diluting a 1000 ppm K standard, 0.1 ml to 1 liter.

4.6.2 Turn on power to the atomic absorption spectrophotometer.

4.6.3 Set the following instrument conditions:

1. Hollow cathode switch to A
2. Mode switch to SB-A
3. Potassium hollow cathode lamp in place with current at 7 mA

4. Curve correct both fully counterclockwise

5. I/Io switch to I

6. Burner height adjusted with burner alignment tool

7. Slit width at 320 μm

4.6.4 The single slot burner head should be in place with the beam from the hollow cathode lamp passing over the center of the slot. The burner alignment tool may be used to check this.

4.6.5 Set the wavelength to 766.5 nm. Optimum sensitivity is achieved when the deflection of the intensity meter is at a maximum. If the intensity meter is not in the range between 2 and 8, adjust the high voltage on the photomultiplier tube and/or the lamp current to get into this range.

4.6.6 Focus the beam on the PM tube, optimizing as described in 4.6.5.

4.6.7 Go to step 4.8.

4.7 Magnesium (Mg) Analysis:

4.7.1 Standards and Blanks: Boron concentrations, as high as 2000 ppm have been shown to have no effect on the detection of magnesium at the 100 ppb level. Standards should be prepared prior to analysis. When calcium is to be determined at the same time, the standard may contain both Ca and Mg.

4.7.2 Turn on power to the atomic absorption spectrophotometer.

4.7.3 Set the following instrument conditions:

1. Hollow cathode switch to "A"

2. Mode switch to SB-A

3. Magnesium hollow cathode lamp in place with current at 4 mA

4. Curve correct both fully counterclockwise

5. I/Io switch to I

6. Burner height adjusted by burner alignment tool.

7. Slit width at 320 μm

4.7.4 The single slot burner head should be in place with the beam from the hollow cathode lamp passing over the center of the slot. The burner alignment tool may be used to check this.

4.7.5 Set the wavelength to 285.2 nm. Optimum sensitivity is achieved when the deflection of the intensity meter is at a maximum. If the intensity meter is not in the range between 2 and 8, adjust the high voltage on the photomultiplier tube and/or the lamp current to get into this range.

4.7.6 Focus the beam on the PM tube, optimizing as described in 4.7.5.

4.7.7 Go to step 4.8.

4.8 Operation of the Atomic Absorption Spectrophotometer (AA)

4.8.1 Flame ignition

1. Open air valves to instrument (AA)
2. Open acetylene valve to instrument (AA).
3. Set gas control selector switch to "fuel" position.
4. Depress pilot button (some adjustment of fuel and air flow rates may be necessary before ignition occurs).

4.8.2 Upon ignition, maintain <u>continuous</u> aspiration using deionized water. Aspiration rate should be between 2 and 4 mls/min.

4.8.3 Perform the following:

1. Adjust the air flow rate to approximately 14-18 SCFH
2. Adjust fuel flow to achieve a lean, blue flame
3. Allow approximately 10 minutes warm up time before standardizing and running samples.

4.8.4 After warm up is achieved:

1. Select the integration switch to auto or manual (if manual mode is chosen, each integration must be initiated by depression of the integration button).
2. Set the integration period switch to 1, 4, or 16 seconds.
3. Aspirate a deionized water blank. Zero the instrument by depression of the auto zero button.
4. Aspirate the standard. Set the readout to the desired concentration using the mA/count adjustment, and the "decimal" set button.
5. Recheck and adjust blank if required.

4.8.5 Aspirate samples

NOTE: Return to blank between each sample and zero if necessary.

NOTE: Dilute samples if values are above the optimal range. If dilution is required, multiply result by dilution factor.

4.8.6 Record values in appropriate log books, and/or worksheet (Appendix 1).

4.8.7 Standard and blank should be checked again before turning off the AA.

4.9 Instrument Shutdown:

4.9.1 Shut off the acetylene valve and allow the flame to go out and the pressure gauge to return to zero.

4.9.2 Shut off the air valve and allow the pressure gauge to return to zero.

4.9.3 Return the burner control to "off".

4.9.4 Turn off current to hollow cathode lamp. If necessary, remove lamp and replace it with the lamp that was originally in its place. For example, the Cr, Ca, K, and Mg lamps are usually removed from the AA after use. Place the removed lamps back in their storage containers.

4.9.5 Shut off power to the AA.

4.10 Report any values which are out of specification to the Lab Supervisor.

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APPENDIX 1

MONTHLY ZEOLITE ANALYSIS

MONTH:_____ ANALYST_____

SYSTEM	PWST Unit 3	PWST Unit 4	RWST Unit 3	RWST Unit 4	RCS/(RHR) Unit 3	RCS/(RHR) Unit 4	SFP Unit 3	SFP Unit 4	BAST TANK NUMBER A	BAST TANK NUMBER B	BAST TANK NUMBER C
Mg Limit (PPM)	0.02		0.08		0.05 (0.08)		0.10		0.66		
Mg Test Results											
Ca Limit (PPM)	0.02		0.08		0.05 (0.08)		0.10		0.66		
Ca Test Results											
SiO_2 Limit	0.10		0.30		0.20 (0.30)		Not Req'd		2.00		
SiO_2 Test Results							XXX	XXX			
Al Limit (PPM)	0.02		0.08		0.05 (0.08)		Not Req'd		0.66		
Al Test Results							XXX	XXX			
Na Limit (PPM)	0.01										
Na Test Results											
K Limit (PPM)	0.01										
K Test Results											

Comments:_____