Form 34731 (10-81)
(Formerly SPD-1002-1)

DUKE POWER COMPANY
PROCEDURE PREPARATION
PROCESS RECORD

(1) ID No: CP/O/B/8100/13
Change(s) 0 to
0 Incorporated

(2) STATION: Catawba

(3) PROCEDURE TITLE: Chemistry Procedure for the Determination of Copper - Furance AA

(4) PREPARED BY: R L Pinto DATE: 11/14/83

(5) REVIEWED BY: T D Dan RHC DATE: 11-14-83

Cross-Disciplinary Review By: _____ (N/R:) TDE

(6) TEMPORARY APPROVAL (IF NECESSARY):

By: _____ (SRO) Date: _____

By: _____ Date: _____

(7) APPROVED BY: J W. Luf Date: 11/14/83

(8) MISCELLANEOUS:

Reviewed/Approved By: _____ Date: _____

Reviewed/Approved By: _____ Date: _____

MASTER FILE

Form 34634 (4-81)

SPD-1001-2

DUKE POWER COMPANY
NUCLEAR SAFETY EVALUATION CHECK LIST

(1) STATION: Catawba UNIT: 1 _____ 2 _____ 3 _____
OTHER: Shared

(2) CHECK LIST APPLICABLE TO: CP/O/B/8100/13

(3) SAFETY EVALUATION - PART A

The item to which this evaluation is applicable represents:

Yes ____ No ✓ A change to the station or procedures as described in the FSA or a test or experiment not described in the FSAR?

If the answer to the above is "Yes", attach a detailed description of the item being evaluated and an identification of the affected section(s) of the FSAR.

(4) SAFETY EVALUATION - PART B

Yes ____ No ✓ Will this item require a change to the station Technical Specifications?

If the answer to the above is "Yes," identify the specification(s) affected and/or attach the applicable pages(s) with the change(s) indicated.

(5) SAFETY EVALUATION - PART C

As a result of the item to which this evaluation is applicable:

Yes ____ No ✓ Will the probability of an accident previously evaluated in the FSAR be increased?

Yes ____ No ✓ Will the consequences of an accident previously evaluated in the FSAR be increased?

Yes ____ No ✓ May the possibility of an accident which is different than any already evaluated in the FSAR be created?

Yes ____ No ✓ Will the probability of a malfunction of equipment important to safety previously evaluated in the FSAR be increased?

Yes ____ No ✓ Will the consequences of a malfunction of equipment important to safety previously evaluated in the FSAR be increased?

Yes ____ No ✓ May the possibility of malfunction of equipment important to safety different than any already evaluated in the FSAR be created?

Yes ____ No ✓ Will the margin of safety as defined in the bases to any Technical Specification be reduced?

If the answer to any of the preceding is "Yes", an unreviewed safety question is involved. Justify the conclusion that an unreviewed safety question is or is not involved. Attach additional pages as necessary.

(6) PREPARED BY: RE Rents DATE: 11/14/83

(7) REVIEWED BY: HB Barron DATE: 11-14-83

(8) Page 1 of 1

Form 18855 (3-80)

DUKE POWER COMPANY

ALARA EVALUATION CHECKLIST

(1) Station Catawba Unit: 1____ 2____ 3____

Other: Shared

(2) Checklist Applicable to: CP/O/B/8100/13

(3) ALARA Evaluation

Check those items below which were considered applicable during the preparation and review of this document.

_____ Flushing and draining were used to minimize source - strength and contamination levels prior to performing an operation.

_____ Permanent and/or movable shielding was specified for reduction of levels.

_____ Use of permanent or temporary local exhaust ventilation systems was used for control of airborne contamination.

_____ Operation was designed to be completed with the least practicable time spent in the radiation field.

_____ Appropriate tools and equipment were specified for the operation to be performed.

_____ The operation was designed considering the minimum number of people necessary for safe job completion.

_____ Remote handling equipment and other special tools were specified to reduce external dose.

_____ Contamination - control techniques were specified.

_____ The operation was designed to be conducted in areas of as low an exposure as practicable.

_____ Additional ALAPA considerations were:

_____

_____

_____

✓ ALARA Principles were not considered since the procedure did not involve work in a radiation area.

(5) Prepared by: R F Rainey Date 11/10/83

(6) Reviewed by: F D Davis Date 11-14-83

DUKE POWER COMPANY
CATAWBA NUCLEAR STATION
CHEMISTRY PROCEDURE FOR THE DETERMINATION
OF COPPER - FURNACE AA

1.0 DISCUSSION

1.1 Scope

This procedure describes the determination of copper by furnace atomic absorption spectroscopy.

1.2 Principle

Refer to CP/0/B/8100/41.

1.3 Precision and Interferences

1.3.1 The precision and accuracy of this procedure will be determined by Quality Control Chart data.

1.3.2 This procedure is applicable for copper concentrations in the range of approximately 0.5 ppb to 40 ppb. Samples of higher concentration should be diluted into this range with cation-polished water or Super-Q water.

1.4 Limits and Precautions

1.4.1 The limits and precautions given in CP/0/B/8100/41 should be followed.

1.4.2 Every effort should be made to minimize contamination when analyzing metals in the ppb range.

1.4.3 All volumetric flasks, pipet tips, and sample cups should be stored in ~ 10% HNO_3 and rinsed thoroughly with cation-polished water or Super-Q water immediately prior to use.

2.0 APPARATUS

2.1 Perkin-Elmer Model 4000 Atomic Absorption Spectrophotometer with HGA 500 Furnace and AS-40 Autosampler

2.2 Copper hollow cathode lamp

2.3 Nalgene volumetric flasks

2.4 Eppendorf pipets

2.5 Sample cups for autosampler

2.6 Argon

3.0 REAGENTS

3.1 Copper Stock Solution (1000 ppm Cu)

3.1.1 1000 ppm Copper Reference Standard Solution (e.g. Fisher Atomic Absorption Reference Standard Solution)

Alternately, a 1000 ppm copper stock solution can be prepared by adding 2.5119 ± 0.0010 grams of oven dried (~ 1 hr. @ ~ 105°C), cupric sulfate ($CuSO_4$) plus 2 ml of concentrated nitric acid (HNO_3) to a 1000 ml volumetric flask and diluting to volume with cation-polished water or Super-Q water.

3.2 Copper Standard Solution

3.2.1 10 ppm Copper

Pipet 1 ml of the 1000 ppm Cu stock solution (Section 3.1) into a 100 ml, acid washed, nalgene volumetric flask. Add 0.2 ml of concentrated nitric acid (HNO_3). Dilute to volume with cation-polished water or Super-Q water. This standard should be prepared weekly.

3.2.2 20 ppb Copper

Pipet 200 microliters of 10 ppm Cu standard solution into a 100 ml, acid washed, nalgene volumetric flask. Add 0.2 ml of concentrated nitric acid (HNO_3). Dilute to volume with cation-polished water or Super-Q water. This sample should be prepared daily.

3.2.3 5 ppb Copper

Pipet 50 microliters of 10 ppm Cu standard solution into a 100 ml, acid washed, nalgene volumetric flask. Add 0.2 ml of concentrated nitric acid (HNO_3). Dilute to volume with cation-polished water or Super-Q water. This standard should be prepared daily.

3.3 Cation-Polished Water and Super-Q Water

Cation-polished water and Super-Q water should have a resistance in excess of 13 megohms.

4.0 PROCEDURE

4.1 Sample Collection

4.1.1 Samples should be collected in nalgene bottles which have been stored filled with ~ 10% HNO_3. The sample bottles must be rinsed thoroughly with cation-polished water or Super-Q water. Also, immediately upon returning to the lab, add enough concentrated nitric acid (HNO_3) to make the final sample ~ 0.2% HNO_3 (e.g. 1 ml HNO_3 in a 500 ml sample bottle). Shake the sample.

4.1.2 The sample should be analyzed as soon as possible after collection but within 6 hours.

4.1.3 The sample should be shaken well immediately prior to pouring it into the sample cup.

4.2 Instrument Setup

4.2.1 Spectrophotometer

Turn on instrument and optomize lamp alignment per Section 4.1 of CP/0/B/8100/41. The correct wavelength for copper is 324.7 nm and the correct slit setting is 0.7 nm.

4.2.2 Autosampler

4.2.2.1 Press "STANDBY" to take the autosampler out of the standby mode.

4.2.3 Furnace

4.2.3.1 Press "STANDBY" to take the furnace out of standby.

4.2.3.2 Press "130" and "TEMP".

4.2.3.3 Press "6" and "RAMP TIME".

4.2.3.4 Press "20" and "HOLD TIME".

4.2.3.5 Press "STEP" to advance to Step 2.

4.2.3.6 Press "900" and "TEMP".

4.2.3.7 Press "10" and "RAMP TIME".

4.2.3.8 Press "20" and "HOLD TIME".

4.2.3.9 Press "STEP" to advance to Step 3.

4.2.3.10 Press "2250" and "TEMP".

4.2.3.11 Press "0" and "RAMP TIME".

4.2.3.12 Press "5" and "HOLD TIME".

4.2.3.13 Press "-5" and "REC".

4.2.3.14 Press "-1" and "READ".

4.2.3.15 Press "50" and "INT FLOW".

4.2.3.16 Press "STEP" to advance to Step 4.

4.2.3.17 Press "2600" and "TEMP".

4.2.3.18 Press "1" and "RAMP TIME".

4.2.3.19 Press "2" and "HOLD TIME".

4.2.3.20 If you wish to check your entries, press "CHECK". Then press the appropriate step number and "STEP". Then press the key for the parameter you wish to check.

EXAMPLE: If you wish to check the entry you made for ramp time on Step 2, press "CHECK", "2", "STEP", and "RAMP TIME".

To exit the check mode, press "CHECK". Then press "1", "STEP" to return to Step 1.

4.2.4 Ensure that a standard graphite tube is in the furnace. See CP/0/B/8100/41, Section 4.3, NOTE.

4.2.5 Optical Temperature Sensor

Optomize the Optical Temperature Sensor per Section 4.5 of CP/0/B/8100/41. The atomization temperature is 2250°C. This must be done for each element.

4.2.6 Graphite Tube

Ensure that the graphite tube is free of any residual copper by performing Section 4.4 of CP/0/B/8100/41.

4.3 Sensitivity Check

4.3.1 Fill an acid washed, thoroughly rinsed sample cup with cation polished or Super-Q water and place it in the AZ slot on the sample tray.

4.3.2 Pour a portion of 20 ppb Cu standard solution into an acid washed, thoroughly rinsed, sample cup and place it in the number 1 slot on the tray.

4.3.3 On the Autosampler controller:

4.3.3.1 Press "60" and "SAMPLE VOLUME".

4.3.3.2 Press "1" and "LAST SAMPLE".

4.3.3.3 Press "RESET" and allow the tray to reset.

4.3.3.4 Press "START/STOP".

4.3.4 At the end of the atomization cycle, for the 20 ppb standard observe the strip chart recorder. If the absorbence is not at least 0.20 absorbence units (20 small chart divisions), stop the analysis. Review Sections 4.2 and 4.3 and/or call the responsible Chemistry Supervisor.

4.4 Quality Control Chart Data and Sample Analysis

4.4.1 Load the sample tray as follows:

Position	Standard/Sample
AZ	0.2% HNO_3 as used to make standards
S1	20 ppb Cu standard solution
1	5 ppb Cu standard solution
2	5 ppb Cu standard solution
3→	Samples (3 cups for each sample)

4.4.2 Press "RESET" on the Autosampler controller.

4.4.3 Press "AZ" on the spectrophotometer.

4.4.4 Press "MANUAL" on the Autosampler controller and allow the Autosampler to sample the AZ position twice. Then press "MANUAL" again to stop sampling.

4.4.5 On the spectrophotometer:

4.4.5.1 Press "CONC" and "PEAK HEIGHT".

4.4.5.2 Press "20.0" and "S1"

4.4.5.3 Press "5" and "t"

4.4.6 On the Autosampler Controller:

4.4.6.1 Press the number corresponding to the last sample and press "LAST SAMPLE".

4.4.6.2 Press "1" and "# STDS".

4.4.7 Press "AZ" on the spectrophotometer.

4.4.8 If the recorder is being used, press "REC MAN", re-zero the recorder (to 10 chart divisions) and press "REC MAN" again.

4.4.9 On the Autosampler controller, press "START/STOP" to initiate the analyses.

NOTE: If the analysis has to be repeated, press "ABS", "CONT", and "AZ". Then repeat Steps 4.4.2, 4.4.5, 4.4.7, 4.4.8 and 4.4.9.

4.4.10 Read and record the results from tne display on the spectrophotometer. The results from samples 1 and 2 will be the Quality Control Chart data. Two of the three results for each sample must agree with each other within the limits of the current Quality Control Charts.

4.4.11 If the results are higher than the upper limit of the linear range given in 1.3.2, dilute with cation-polished or Super-Q water and multiply the results by the dilution factor. If the results are less than the lower limit in 1.3.2, report the results as less than that number.

4.4.12 This analysis is subject to environmental contamination within the lab. If the results of this analysis are higher than expected, repeat the analysis using the same sample from the original sample bottle but loading it into a different sample cup.

5.0 REFERENCES

5.1 Perkin-Elmer Model 4000 Atomic Absorption Spectrophotometer Operator's Manual

5.2 Perkin-Elmer AS-40 Autosampler Operator's Manual

5.3 Perkin-Elmer HGA-500 Graphite Furnace Operator's Manual

5.4 Perkin-Elmer Analytical Methods for Graphite Furnace A.A.S.

6.0 ENCLOSURES

None