

DUKE POWER COMPANY
PROCEDURE PREPARATION
PROCESS RECORD

(1) ID No: CP/O/B/8100/51
Change(s) 0 to
0 Incorporated

- (2) STATION: Catawba
- (3) PROCEDURE TITLE: Chemistry Procedure for the Determination of Iron -
Furnace AA
- (4) PREPARED BY: R. L. Paine DATE: 11/14/83
- (5) REVIEWED BY: J. W. Egan RHC DATE: 11-14-83
Cross-Disciplinary Review By: (N/R) LDE
- (6) TEMPORARY APPROVAL (IF NECESSARY):
By: _____ (SRO) Date: _____
By: _____ Date: _____
- (7) APPROVED BY: J. W. Egan Date: 11/14/83
- (8) MISCELLANEOUS:
Reviewed/Approved By: _____ Date: _____
Reviewed/Approved By: _____ Date: _____

MASTER FILE

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/8100/51

(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: RJ Hunt DATE: 11/14/83
(7) REVIEWED BY: TD Egan DATE: 11-14-83

(8) Page 1 of 1

DUKE POWER COMPANY
ALARA EVALUATION CHECKLIST

(1) Station Catawba Unit: 1 2 3
Other: Shared

(2) Checklist Applicable to: CP/O/B/8100/51

(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 ALARA considerations were:

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

(5) Prepared by: [Signature] Date 11/14/83

(6) Reviewed by: [Signature] Date 11-14-83

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

1.0 DISCUSSION

1.1 Scope

This procedure describes the determination of iron 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 iron concentrations in the range of approximately 1 ppb to 25 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% HNO₃ 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 Iron 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 Iron Stock Solution (1000 ppm Fe)

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

Alternately, a 1000 ppm iron stock solution can be prepared by adding 2.9044 ± 0.0010 grams of oven dried (~ 1 hr. @ ~ 105°C), ferric chloride (FeCl_3) 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 Iron Standard Solution

3.2.1 1 ppm Iron

Pipet 100 microliters of the 1000 ppm Fe 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 Iron

Pipet 2 ml of 1 ppm Fe 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 Iron

Pipet 500 microliters of 1 ppm Fe 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 optimize lamp alignment per Section 4.1 of CP/O/B/8100/41. The correct wavelength for iron is 248.3 nm and the correct slit setting is 0.2 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 "1200" and "TEMP".
- 4.2.3.7 Press "11" 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 "2500" 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 "2700" 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/O/B/8100/41, Section 4.3, NOTE.

4.2.5 Optical Temperature Sensor

Optimize the Optical Temperature Sensor per Section 4.5 of CP/O/B/8100/41. The atomization temperature is 2500°C. This must be done for each element.

4.2.6 Graphite Tube

Ensure that the graphite tube is free of any residual iron by performing Section 4.4 of CP/O/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 Fe standard solution into an acid washed, thoroughly rinsed, sample cup and place it in the number 1 slot on the sample tray.

4.3.3 On the Autosampler controller:

- 4.3.3.1 Press "40" 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 absorbance is not at least 0.15 absorbance units (15 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:

<u>Position</u>	<u>Standard/Sample</u>
AZ	0.2% HNO ₃ as used to make standards
S1	20 ppb Fe standard solution
1	5 ppb Fe standard solution
2	5 ppb Fe 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 the 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