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

DUKE POWER COMPANY
PROCEDURE PREPARATION
PROCESS RECORD

(1) ID No: CP/0/B/8100/17
Change(s) 0 to
0 Incorporated

(2) STATION: Catawba

(3) PROCEDURE TITLE: Chemistry Procedure for the Determination of Hydrazine

(4) PREPARED BY: Cynthia S. Dickey DATE: 9/28/83

(5) REVIEWED BY: LD Evans RHC DATE: 9/28/83

Cross-Disciplinary Review By: ____ N/R: LDE

(6) TEMPORARY APPROVAL (IF NECESSARY):

By: ____ (SRO) Date: ____

By: ____ Date: ____

(7) APPROVED BY: M.S. Tuckman /RHC Date: 9-30-83

(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/0/B/8100/17

(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 FS or a test or experiment not described in the FSAR?

If the answer to the above is "Yes", attach a detailed description of the ite 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: Cynthia S. Dickey DATE: 9/28/83

(7) REVIEWED BY: LD Evans DATE: 9-28-83

Form 18855 (3-80)

DUKE POWER COMPANY

ALARA EVALUATION CHECKLIST

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

Other: Shared

(2) Checklist Applicable to: CP/0/B/8100/17

(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: Cynthia S. Dickey Date 9/28/83

(6) Reviewed by: [signature] Date 9-28-83

DUKE POWER COMPANY
CATAWBA NUCLEAR STATION
CHEMISTRY PROCEDURE FOR THE DETERMINATION OF HYDRAZINE

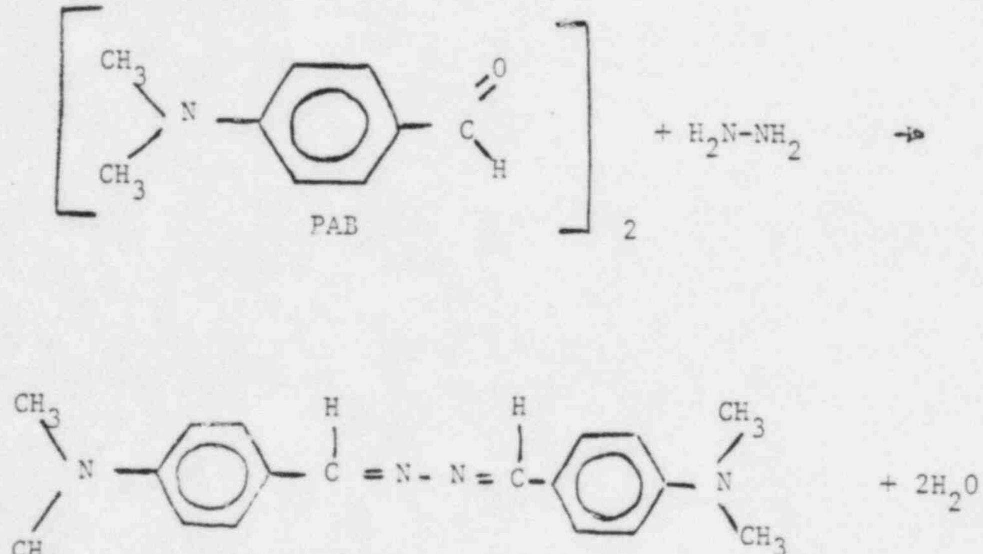
1.0 DISCUSSION

1.1 Scope

This procedure describes the colorimetric method for the determination of hydrazine in water.

1.2 Principle

Para-dimethylaminobenzaldehyde produces a specific yellow condensation reaction with hydrazine as shown:



para-dimethylaminobenzaldehyde azine (yellow)

The intensity of the yellow color is proportional to the amount of hydrazine in the sample within the specified concentration range.

The hydrazine is used primarily as an oxygen scavenger according to the following reaction:

$N_2H_4 + O_2 \rightarrow 2\ H_2O + N_2$

Hydrazine does not react appreciably with oxygen below 150°F, but decomposes rapidly over 400°F.

The thermal decomposition of hydrazine goes by two reactions:

$$3\ N_2H_4 \xrightarrow{\Delta} 2\ NH_3 + 2\ N_2 + 3\ H_2$$

$$3\ N_2H_4 \xrightarrow{\Delta} 4\ NH_3 + N_2$$

Since ammonia is produced by the decomposition, the pH is raised.

An additional benefit is the reduction of red iron oxide to magnetite

$$6\ Fe_2O_3 + N_2H_4 \rightarrow 4\ Fe_3O_4 + 2\ H_2O + N_2$$

The magnetite protects metal surfaces from further corrosion. Normally, a hydrazine residual of 0.010 to 0.015 ppm is sought in the final feedwater sample for condensate system protection. Wet layup concentrations range from 50 to 500 ppm.

1.3 Precision and Interference

1.3.1 The precision and accuracy of this method will be determined by Q-Sum data.

1.3.2 The substances usually present in condensate and reactor coolant do not interfere with the test; however, the hydrazine content may be diminished by oxidizing agents.

1.4 Limits and Precautions

1.4.1 Sample concentration should be between 0.005 and 0.25 ppm hydrazine. Higher concentrations must be proportionally diluted prior to analysis. The lower limit of detectable hydrazine is 0.005 ppm.

1.4.2 Eye protection and rubber gloves shall be worn when working with paradimethylaminobenzaldehyde.

1.4.3 Hydrazine is a primary skin irritant and a potential skin sensitizer; therefore, precautions must be taken to avoid skin contact. Eye protection, lab coat, and rubber gloves will be worn when working with hydrazine dihydrochloride and hydrazine containing solutions and samples. If skin contact occurs, wash the affected area immediately with soap and water.

2.0 APPARATUS

2.1 Spectrophotometer, with wavelength set at 458 nm. NOTE: Allow spectrophotometer to warm up for 30 minutes before use.

2.2 Erlenmeyer flasks, 125 ml

2.3 Graduated cylinder, 50 ml

2.4 The following Eppendorf pipets with required tips:

2.4.1 10 ul

2.4.2 25 µl

2.4.3 50 µl

2.4.4 100 µl

2.4.5 250 µl

2.5 Pipet, 5 ml

2.6 Two matched 1.0 cm cuvettes

3.0 REAGENTS

3.1 Hydrazine Stock Solution (100 ppm)

Dissolve 0.2280 ± 0.0001 grams of hydrazine dihydrochloride (N_2H_4 . 2HCl) in 100 ± 10 ml of Super-Q water and add 10 ± 0.5 ml of concentrated HCl. In a volumetric flask, dilute this solution to 1 liter with Super-Q water and mix. This solution is stable for 6 months.

3.2 Hydrazine Reagent (PAB)

Dissolve 8.0 ± 0.1 grams para-dimethylaminobenzaldehyde in 400 ± 10 ml of methyl alcohol and 40 ± 0.5 ml of concentrated HCl. Store in an amber bottle. This solution is stable indefinitely.

3.3 Hydrochloric Acid Solution (1% V/V)

In a volumetric flask, dilute 10 ± 0.5 ml concentrated HCl to 1 liter with Super-Q water.

4.0 PROCEDURE

4.1 Standard and Sample Preparation

NOTE: Generation of a standard curve is not required if method is in current use; however, two 100 ppb standards are to be run daily for Q-Sum. A new standard curve should be prepared at least once per year.

4.1.1 Prepare a series of hydrazine standards by diluting measured volumes of hydrazine stock solution (Section 3.1) with 1% V/V HCl (Section 3.3) to produce 100 ml solutions of the desired concentrations as follows:

µl of 100 ppm stock solution	diluted to 100 ml = ppm N_2H_4
10 µl	0.010 ppm
25 µl	0.025 ppm
50 µl	0.050 ppm
100 µl	0.100 ppm
250 µl	0.250 ppm

4.1.2 Transfer 50 ± 1.0 ml of each standard, Super-Q water blank, and unknown sample to separate Erlenmeyer flasks.

4.1.3 Add 5.0 ± 0.5 ml of para-dimethylaminobenzaldehyde solution (Section 3.2) to each flask containing either standard, blank, or unknown sample and mix by swirling the flasks. Let the mixture stand 10 minutes, but not more than 100 minutes.

4.2 Instrument Calibration

4.2.1 Adjust the spectrophotometer to the 458 nm wavelength.

4.2.2 Place the reagent blank in the spectrophotometer in a 1.0 cm cuvette.

4.2.3 Set zero absorbance.

4.3 Determination of Unknown Concentrations

4.3.1 Transfer the standards and/or samples into the 1.0 cm cuvette in appropriate order and read the absorbance on the spectrophotometer checking the zero with the reagent blank before each measurement. Standard concentration measurements are to be made in order of increasing concentration. Rinse the cuvette with the subsequent sample before each measurement.

4.3.2 Prepare a standard curve by plotting the absorbance of the standard versus the concentration of the standards.

4.3.3 Determine the hydrazine concentration of the sample by comparing the sample absorbance against the standard curve.

5.0 <u>REFERENCES</u>

5.1 American Society for Testing and Materials, 1978 Annual Book of ASTM Standards, Part 31, D 1385-78, Page 354-357.

5.2 Westinghouse Electric Corporation, May 1974, Chemical Analysis Procedures, Pittsburgh, PA. Page 4-45 to 4-47.

5.3 McGuire Nuclear Station Chemistry Procedure CP/0/B/8100/12.

5.4 Steam Production Department System Power Chemistry Procedure CP/40.

5.5 DPC Hazardous Materials Exposure Control Manual.

6.0 <u>ENCLOSURES</u>

None