

November 11, 2002

U. S. Nuclear Regulatory Commission  
Document Control Desk  
Washington, D. C. 20555

Subject: Oconee Nuclear Station  
Docket Nos. 50-269, -270, -287  
Emergency Plan Implementing Procedures Manual  
Volume B, Revision 2002-10

Please find attached for your use and review copies of the revision to the Oconee Nuclear Station Emergency Plan:

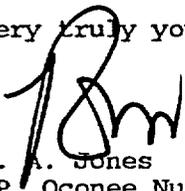
Volume B Revision 2002-10 November 2002

This revision is being submitted in accordance with 10 CFR 50-54(g) and does not decrease the effectiveness of the Emergency Plan or the Emergency Plan Implementing Procedures.

Any questions or concerns pertaining to this revision please call Rodney Brown, Emergency Planning Manager at 864-885-3301.

By copy of this letter, two copies of this revision are being provided to the NRC, Region II, Atlanta, Georgia.

Very truly yours,



R. A. Jones  
VP, Oconee Nuclear Site

xc: (w/2 copies of attachments)  
Mr. Luis Reyes,  
Regional Administrator, Region II  
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Mr. Steven Baggett  
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(w/o Attachments, Oconee Nuclear Station)  
NRC Resident Inspector  
M. D. Thorne, Manager, Emergency Planning

A045

November 11, 2002

OCONEE NUCLEAR SITE

SUBJECT: Emergency Plan Implementing Procedures  
Volume B, Revision 2002-10

Please make the following changes to the Emergency Plan, Volume B  
by following these instructions.

REMOVE

Cover Sheet Rev. 2002-09

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Chemistry Lab - LM/O/P003C  
(11/18/96)

Chemistry Lab - LM-O-P919  
(07/18/02)

ADD

Cover Sheet Rev. 2002-10

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Chemistry Lab - LM/O/P003C  
(11/06/02)

Chemistry Lab - LM O P919  
(10/28/02)

**DUKE POWER**  
**EMERGENCY PLAN**  
**IMPLEMENTING PROCEDURES**  
**VOLUME B**



**APPROVED:**

*W. W. Foster*

W. W. Foster, Manager  
Safety Assurance

11/11/2002

Date Approved

11/11/2002

Effective Date

**VOLUME B**  
**REVISION 2002-10**  
**NOVEMBER 2002**

**VOLUME B**  
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CP/2/A/2002/004C	Operating Procedure For The Post Accident Liquid Sampling System (PALSS)	01/08/02
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Revision 2002-10  
November 2002

Duke Power Company  
Nuclear Generation Department

LABORATORY METHOD PROCESS RECORD

Reference Use  
INFORMATION ONLY

Station: Oconee

LM/O/P003 C

Rev. #: 2 Change: \_\_\_\_\_  
(A, B, or C, etc.)

Title: Determination of Boron by Manual Colorimetric Titration Using Phenolphthalein Indicator

(check applicable boxes)

- This Method is based on General Lab Method LM/G/ \_\_\_\_\_ Rev, \_\_\_ without substantive changes.
- This Method is based on General Lab Method LM/G/ \_\_\_\_\_ Rev, \_\_\_ with substantive changes.
- This Method is not based on a General Lab Method.
- This revision does not invalidate criteria for use of a laboratory method (reference SCM-6, Section 5.1.1) (if any of these criteria are no longer valid, then a Technical Procedure must be used for this activity)
- These changes affect the associated method development package.
- Field testing performed. Date: \_\_\_\_\_ Initials: \_\_\_\_\_

Description of Change: (Attach additional pages if necessary)

- 1 Added Statement Results may affect Reactivity Management
- 2 Removed Burette requirement for 25 ml Burette
- 3 Removed note to use daily pH & mantrol
- 4 Corrected BEF calculation
- 5 removed ref to DC 40 Lab method & added ref. to McGuire Lab method

Basis for Change: (Attach additional pages if necessary)

1. Reports Boron to Control Room
2. Lab Burette is 50 ml
3. No longer keep liquid mantrol on daily basis
4. Calculation had error - Ref LM/M/P903
5. DC-40 method superseded

Field Testing Comments: (Attach additional pages if necessary)

Prepared by Don & Clark Date 10/31/02

Technical Review by Ricky Date 11/6/02

Additional Review by (optional) \_\_\_\_\_ Date \_\_\_\_\_

Approved by Byron J. Ross Date 11/6/02

## Determination of Boron by Manual Colorimetric Titration Using Phenolphthaline Indicator

### 1. Discussion

**NOTE:** A control copy of this procedure shall be routed to the Emergency Preparedness Team within three working days after any approved changes.

#### 1.1 Scope

This procedure describes the determination of boron by a manual colorimetric titration in samples containing multiple forms of boron.

The intent of this procedure is to provide a mechanism to analyze borated samples during an Appendix "R", which has resulted in the loss of our normal analytical instrumentation and when a high degree of accuracy is not required.

#### 1.2 Principle

Boric acid is a very weak, monobasic acid with an ionization constant ( $K_A$ ) of  $6.4 \times 10^{-10}$ . Because it is a very weak acid, the concentration of boric acid in aqueous solutions cannot be potentiometrically determined directly with a strong base. However, boric acid in the presence of mannitol acts as a much stronger acid with a  $K_A \cong 1 \times 10^{-4}$ , and can be directly determined by titration with a strong base. The dramatic increase in the ionization constant of boric acid is due to the formation of a mannitol - borate complex. In this determination, an excess of mannitol is added to the sample to be analyzed, then standard sodium hydroxide is added to the sample until a faint pink color is observed and remains consistent.

#### 1.3 Limits and Ranges

This procedure is applicable to samples containing between 100 and 2500 ppm boron. It is recommended for conservation of time and dose that samples  $> 1000$  ppm should be diluted.

#### 1.4 Precision and Accuracy

The precision should be  $\pm 10$  ppm and the accuracy should be  $\pm 5.0$  %.

## 1.5 Interferences

- 1.5.1 Lithium hydroxide is an interfering factor in low boron concentrations (< 100 ppm B). 1.56 ppm B per ppm  $\text{Li}^+$  should be added to the calculated boron concentration. Also, phosphate, (over 10 ppm), germanium, and tetravalent vanadium react with mannitol like boron and should be absent.
- 1.5.2 Any error from atmospheric carbon dioxide can be minimized by utilizing a drying column attached to the sodium hydroxide titrant bottle.

## 1.6 Precautions

**CAUTION:** Chemical hazards shall be known prior to use. For additional information and first aid requirements, refer to the MSDS Sheets.

- 1.6.1 Results obtained by this lab method may affect Reactivity Management.
- 1.6.2 If failed fuel exceeds 1%, follow instructions given by Radiation Protection and perform analysis according to CSM 5.2.
- 1.6.3 The following PPE shall be worn for:
- 1.6.3.1 8% mannitol
- safety glasses
  - rubber or vinyl gloves
  - lab coat
- 1.6.3.2 0.1005 - 0.0995 N NaOH
- safety glasses
  - rubber or vinyl gloves
  - lab coat
- 1.6.3.3 Boron standard
- safety glasses
  - rubber or vinyl gloves
  - lab coat
- 1.6.3.4 Phenolphthalein indicator
- safety glasses
  - rubber or vinyl gloves
  - lab coat

1.6.3.5 Methanol

- chemical splash goggles
- rubber gloves
- lab coat

## 2. Apparatus

- 2.1 200 mL Tall Form Beaker or a 250 mL Erlenmeyer flask
- 2.2 Burette: with sufficient volume to dispense up to 25 mL NaOH titrant
- 2.3 Pipettes: 10 mL glass class "A" for sample and standard transfer
- 2.4 Graduated Cylinder: 100 mL for mannitol measurement

## 3. Reagents

**NOTE:** It is imperative that certified 0.1 N NaOH and 1000 PPM boron standard be used in this analysis.

3.1 Mannitol Solution, 8% W/V

Dissolve 320 grams  $\pm$  1 gm of d - mannitol per 4 liters of demineralized water and adjust the pH to  $8.5 \pm 0.1$  with dilute NaOH or HCl

Stable for two (2) weeks.

3.2 0.1005 - 0.0995 N (Certified) Sodium Hydroxide:

Use Fisher SS-276 or equivalent. Expiration date as noted on manufacturer's bottle.

3.3 Boron Standard:

Use Fisher SB-155, certified 1000 ppm  $\pm$  1% or equivalent. Expiration date as noted on manufacturer's bottle.

3.4 Phenolphthalein Indicator:

Add 0.1 grams of disodium salt of phenolphthaline to 200 mL of methanol. Shelf life is indefinite.

#### 4. Procedure

**NOTE:** When performing this analysis, two aliquots of the 1000 ppm boron standard must be analyzed with each set of samples or once per day whichever is less frequent.

- Calculations

Boron Equivalency Factor (BEF)

$$\frac{\left(\frac{0.17485\text{mgB}}{\text{mgH}_3\text{BO}_3}\right)\left(\frac{61.83\text{mg H}_3\text{BO}_3}{\text{meq H}_3\text{BO}_3}\right)\left(\frac{1000\text{ppm}}{\text{mgB/mL}}\right)}{10\text{ mL sample vol.}} = 1081$$

where: 0.17485 = fraction B in H<sub>3</sub>BO<sub>3</sub>

61.83 = equivalent weight of H<sub>3</sub>BO<sub>3</sub>

1000 = conversion mg B/mL to ppm

Vol. of Titrant X Normality of Titrant X BEF = ppm Boron

- 4.1 Fill the titration burette with 0.1 N NaOH and record the initial reading of the meniscus.
- 4.2 Using a volumetric pipet, transfer 10.00 ± 0.01 mL of the 1000 ppm standard or sample to an appropriate beaker.
- 4.3 Add 100 mLs ± 1.0 mL of 8% mannitol solution to the sample or standard and mix thoroughly by swirling the beaker.
- 4.4 Add 3 to 7 drops of phenolphthaline to the sample/standard to be analyzed and mix by slowly swirling the beaker.
- 4.5 Using the titration buret, slowly open the stopcock until NaOH begins to flow in a uniform "droplet" pattern. Continue to titrate the solution, swirling the beaker slowly, with the Certified Standard Sodium Hydroxide (0.1 N) until one drop produces a faint, constant pink color.
- 4.6 Carefully record the final burette reading and determine the amount of sodium hydroxide used by subtracting the initial burette reading from the final burette reading.
- 4.7 Perform a duplicate analysis by repeating Steps 4.1 through 4.5. Samples and standards must agree within +/- 0.2 mLs.

4.8 Calculate sample and standard concentration by:

$$(\text{Titration Buret Vol.}) \times (0.1 \text{ N NaOH}) \times (1081) = \text{ppm Boron}$$

4.9 Log standard results in Enclosure 6.1.

## 5. References

5.1 B&W Water Chemistry Manual (1385), Rev. 5

5.2 McGuire Nuclear Station, LM/M/P903, Rev. 1, "Manual Boron Analysis"

## 6. Enclosures

6.1 Boron Manual Titration Performance Check



Duke Power Company  
Nuclear Generation Department

LABORATORY METHOD PROCESS RECORD

Reference Use  
INFORMATION ONLY

Station: Oconee

LM/O/P919

Rev. #: 7 Change: \_\_\_\_\_  
(A, B, or C, etc.)

Title: Boron Analysis by Mettler DL-58 Boron Titrator

(check applicable boxes)

- This Method is based on General Lab Method LM/G/\_\_\_\_ Rev, \_\_\_\_ without substantive changes.
- This Method is based on General Lab Method LM/G/\_\_\_\_ Rev, \_\_\_\_ with substantive changes.
- This Method is not based on a General Lab Method.
- This revision does not invalidate criteria for use of a laboratory method (reference SCM-6, Section 5.1.1) (if any of these criteria are no longer valid, then a Technical Procedure must be used for this activity)
- These changes affect the associated method development package.
- Field testing performed. Date: \_\_\_\_\_ Initials: \_\_\_\_\_

Description of Change: (Attach additional pages if necessary)

*See ATTACHED*

Basis for Change: (Attach additional pages if necessary)

*See ATTACHED*

Field Testing Comments: (Attach additional pages if necessary)

Prepared by Don F Clark Date 10/12/02

Technical Review by Pachelt Date 10/21/02

Additional Review by (optional) \_\_\_\_\_ Date \_\_\_\_\_

Approved by Bryan L. Noy Date 10/28/02

## Changes to LM/O/P919 Boron Analysis By Mettler DL-58 Boron Titrator

Added statement in "Precautions" to bracket samples with appropriate QC, (10 ppm samples  $\leq 100$ , and 1000 ppm  $> 100$  ppm).

Added requirement to obtain Fisher Certificate of Analysis (COA) for each 1000 ppm standard lot number.

Added instructions to prepare 10 ppm Boron standard.

Added notes where applicable: (PIP 4472)

- 2 level teaspoons equates to approximately 5 grams of mannitol
- mannitol may be added anytime between placing it in the sample changer and starting analysis

Clarified during standardization when to choose "Save Entry" or "Modify Entry"

4.4.13 Corrected reference to steps PIP 4841

Enclosure 6.2 Section 4 - Rewrote section to allow for adding any programmed method as an additional sample series to an existing one.

## Boron Analysis by Mettler DL58 Boron Titration

### 1. Purpose

**NOTE:** Seven Control Copies and one Information Only copy of this Lab Method shall be routed to the Emergency Preparedness Team within three (3) working days following any approved changes/modifications.

#### 1.1 Scope

**CAUTION:** This laboratory method describes the automatic titrimetric method for determining boron in aqueous solutions. This laboratory method is applicable for Primary Systems.

#### 1.2 Principle

Boric acid is a weak acid and cannot be titrated directly with a strong base to an accurate endpoint. Mannitol is added to stabilize the anion of boric acid and increase the strength of the acid.

The mannitol-borate complex can then be titrated with a strong base. The concentration of boron is proportional to the amount of standard sodium hydroxide required to titrate the boron to a pH endpoint of 8.3.

The titration is performed by a Mettler DL58 automated titration system. Samples are loaded onto a Mettler ST20A Sample Transport Unit. A digital buret assembly dispenses titrant into the sample. The rate of titrant delivery is slowed near the equivalence point. This allows ample time for thorough mixing and reaction of titrant with the sample until the pH equivalence point is obtained.

#### 1.3 Interferences

1.3.1 Carbon Dioxide is an interferent; however, the interference should be negligible if the following conditions are met:

- Water used for preparing sodium hydroxide is boiled or deaerated.
- Sodium hydroxide reservoir is air tight or equipped with a CO<sub>2</sub> absorption tube filled with drying agent.
- Sample is not over-stirred.
- Sample exposure to air is minimized.

1.3.2 Lithium Hydroxide and Ammonium Hydroxide are potential interferences; however, these interferences have been determined to be negligible as the change in boron concentration that they cause is less than the required accuracy needed.

#### 1.4 Limits

This method is applicable for samples containing between 0.2 - 10,000 ppm boron as boric acid.

## 1.5 Precautions

- 1.5.1 The pH electrode should be stored in a conditioning agent as recommended by the electrode manufacturer when not in use.
- 1.5.2 The sodium hydroxide reservoir should be emptied and refilled rather than "topped off" when the supply is low.
- 1.5.3 Typically, when the titrator has been idle, the first standard/sample will show a high bias result. Therefore, when the titrator has been idle, the first standard/sample of a titration series should be run in duplicate and the first result discarded.
- 1.5.4 If the titrator does not initiate analysis after pressing the [Run] key twice, then the [Run] key may need to be pressed again or the [Start] key may be pressed.
- 1.5.5 If the titrator goes into the Hold mode when adding samples to a series, the [Hold] key may need to be pressed to resume analysis.
- 1.5.6 This laboratory method is related to plant Reactivity Management. Reactor coolant boron directly impacts Reactivity Management. Reactor coolant and support system boron concentrations are determined by this method.
- 1.5.7 When determining the concentration of PALSS boron, results obtained must be multiplied by the PALSS dilution factor found in the appropriate PALSS procedure(s).
- 1.5.8 Wear the following minimum proper personal protective equipment (PPE) when performing this analysis.
- Labcoat
  - Safety glasses
  - Gloves
  - Apron and faceshield or hood sash when handling 50% W/W Sodium Hydroxide or Hydrochloric Acid
- 1.5.9 When boric acid is heated at 100° to 160°C, it gradually decomposes and loses water to form metaboric acid (HBO<sub>2</sub>) then pyroboric acid (H<sub>2</sub>B<sub>4</sub>O<sub>7</sub>), and finally boric anhydride (B<sub>2</sub>O<sub>3</sub>). Hence, in preparing a boron standard, boric acid is neither dried nor desiccated prior to weighing.
- 1.5.10 For error messages and/or malfunctions, refer to Enclosure 6.3.
- 1.5.11 Sample concentration must not exceed a burette delivery volume of 40 ml. See Section 4.4.2 for selecting appropriate sample size.
- 1.5.12 Sodium Hydroxide lines and delivery tip must be kept free of air bubbles at all times. Air bubbles will result in unreliable sample results.
- 1.5.13 Samples should be bracketed with an appropriate QC. For samples ≤ 100 ppm expected use a 10 ppm QC, for > 100 ppm expected a 1000 ppm QC.

## 2. Apparatus

- 2.1 Mettler DL58 Compact Titrator System
  - 2.1.1 DL58 Titrator
  - 2.1.2 20 ml Buret or 10 mL Buret
  - 2.1.3 ST20A Sample Transport
  - 2.1.4 Printer
  - 2.1.5 Electronic Balance with Data Output Module
  - 2.1.6 Combination pH electrode, pH 0-14
- 2.2 Sample cups, 100 mL
- 2.3 Volumetric flasks
- 2.4 Volumetric pipets

## 3. Reagents

**CAUTION:** Chemical hazards shall be known prior to use. For additional information and first aid requirements, refer to MSDS Sheets.

- 3.1 pH Buffers, 7, 9, 10
  - 3.1.1 Fisher buffers or equivalent may be purchased or prepared and used per package instructions.
  - 3.1.2 Use manufacturer stated shelf life OR record shelf life as 3 months.
- 3.2 Electrode Filling Solution
  - 3.2.1 Use solution specified by the manufacturer of the electrode in use.
  - 3.2.2 Use manufacturer stated shelf life.

**NOTE:** Reagents may be prepared in varying quantities as long as the ratios remain unchanged.

- 3.3 Sodium Hydroxide (NaOH), 0.10 N
  - 3.3.1 Purchased NaOH (Fisher SS278-1 or equivalent)
    - 3.3.1.1 Use manufacturer stated shelf life.
    - 3.3.1.2 Store in an air tight container OR a container fitted with a carbon dioxide (CO<sub>2</sub>) absorption tube.
    - 3.3.1.3 Standardize solution once per week or prior to use, whichever is less frequent, per Section 4.3 (NaOH Standardization).

3.3.2 Prepared NaOH, 0.10N

**WARNING:** Sodium Hydroxide is corrosive. Level III PPE (labcoat, safety glasses, gloves, and faceshield or hood sash) is required when handling 50% W/W Sodium Hydroxide.

- 3.3.2.1 Add 32.0 grams (31.5 - 32.5) of liquid 50% W/W NaOH to 3850 (3845 - 3855) mLs of boiled or deaired reagent grade water **AND** mix.
- 3.3.2.2 Store in an air tight container **OR** a container fitted with a carbon dioxide (CO<sub>2</sub>) absorption tube.
- 3.3.2.3 Record shelf life as 1 year.
- 3.3.2.4 Standardize solution once per week or prior to use, whichever is less frequent, per section 4.3 (NaOH Standardization).

3.4 Electrode Storage Solution (0.5 mol/L KCL)

- 3.4.1 Weigh out 37.275g ± 0.1g anhydrous KCl. Add to a 1 L volumetric flask and bring to volume with deionized water.
- 3.4.2 Record shelf life as 3 months.

3.5 Standard Boric Acid Solution (NaOH Standardization), 1000 mg/L

- 3.5.1 Dissolve 5.7194 grams (5.7189 - 5.7199) of Boric Acid (H<sub>3</sub>BO<sub>3</sub>) with reagent grade water in a 1000 mL volumetric flask **AND** dilute to volume.
- 3.5.2 Record shelf life as 1 year.
- 3.5.3 Verify solution concentration per section 4.4 (QC Check/Sample Analysis).

3.6 QC Boric Acid Solution, 1000 mg/L

- 3.6.1 Use purchased 1000 mg/L Boric Acid Standard
- 3.6.2 Use manufacturer stated shelf life.
- 3.6.3 Obtain certificate of analysis (COA) from Fisher for each lot number.

3.7 QC Boric Acid Solution, 10 mg/L

- 3.7.1 Using a volumetric pipet and a volumetric flask, dilute 10 mL of the purchased Fisher standard to 1000 mL with deionized water.
- 3.7.2 Record shelf life as 1 month.

3.8 Mannitol

### 3.9 Hydrochloric Acid (HCl), 0.0025N

**WARNING:** Hydrochloric acid is corrosive. Level III PPE (labcoat, apron, safety glasses, gloves, faceshield or hood sash) is required when handling concentrated Hydrochloric Acid.

- 3.9.1 Add 250  $\mu$ L of concentrated HCl to a 1000 mL volumetric flask partially filled with reagent grade water.
- 3.9.2 Dilute to volume with reagent grade water **AND** mix.
- 3.9.3 Record shelf life as 1 month.

- NOTE:**
1. It is **NOT** the intent of this procedure to perform the sections sequentially but to perform only those section(s) necessary to meet the need at the time.
  2. During troubleshooting specific keystroke functions can deviate from this method in order to identify and correct instrument problems.

The programmed methods in the instrument are:

1. Probe Cal. (pH probe calibration)
2. Measure pH (pH 9 buffer check)
3. Blank (mannitol)
4. Boron NaOH Standardization (titer factor)
5. Boron (density corrected) (QC/Sample analysis)

## 4. Procedure

### 4.1 Initial Setup

- 4.1.1 Ensure power to titrator, balance, sample changer, and printer is on.
- 4.1.2 Ensure sufficient volume of sodium hydroxide and reagent grade water in reservoirs.
- 4.1.3 If needed, refer to Enclosure 6.1 (Sample Changer Manual Operation) to operate the sample changer in the manual mode at any time during the performance of this procedure
- 4.1.4 If needed, refer to Enclosure 6.2 (Sample Series Manipulations) to perform any of the following at any time during the performance of this procedure:
  - Delete a sample from a sample series
  - Add additional samples to end of sample series
  - Insert an urgent sample into a sample series
  - Add additional sample series to existing series.

4.2 Daily pH Electrode Standardization

- 4.2.1 Ensure electrode is clean.
- 4.2.2 Check filling solution level AND fill as necessary.
- 4.2.3 Ensure cap is not covering vent hole.
- 4.2.4 Using approximately 50 mL of each buffer, place cup of pH 7 buffer in 1<sup>st</sup> sample changer position, pH 10 buffer in 2<sup>nd</sup> position, and pH 9 buffer in 3<sup>rd</sup> position.
- 4.2.5 Press or select the following in the order listed:
  - 4.2.5.1 [Sample]
  - 4.2.5.2 [F5] (Add)
  - 4.2.5.3 Select "New Sample Series"
  - 4.2.5.4 [F5] (OK)
  - 4.2.5.5 Select "Number of Samples"
  - 4.2.5.6 [2]
  - 4.2.5.7 Scroll down and select "Method ID"
  - 4.2.5.8 [1]
  - 4.2.5.9 [F5] (OK)
  - 4.2.5.10 [F5] (OK)
  - 4.2.5.11 [F5] (OK)
  - 4.2.5.12 [F5] (Add)
  - 4.2.5.13 Select "New Sample Series"
  - 4.2.5.14 [F5] (OK)
  - 4.2.5.15 Select "Number of Samples"
  - 4.2.5.16 [1]
  - 4.2.5.17 Scroll down and select "Method ID"
  - 4.2.5.18 [2]
  - 4.2.5.19 [F5] (OK)
  - 4.2.5.20 [F5] (OK)
  - 4.2.5.21 [Run]

- 4.2.5.22 [F5] (OK)
- 4.2.5.23 [F5] (START)
- 4.2.5.24 [F5] (OK)

**NOTE:** Measurements will be made and calculations carried out automatically for the first two buffers.

- 4.2.6 After first sample series has completed and calibration results have printed, press [Run] to continue to 2<sup>nd</sup> sample series (pH 9 buffer).
  - 4.2.7 **IF** pH 9 buffer result is outside  $9 \pm 0.1$  pH units, **THEN** repeat Steps 4.2.4 - 4.2.7.
  - 4.2.8 On a daily basis, record slope value in appropriate database or logsheet for instrument performance trending.
- 4.3 NaOH Buret Rinse:
- 4.3.1 Perform this section:
    - When NaOH reservoir is refilled.
  - 4.3.2 Stir NaOH reservoir to thoroughly mix **AND** perform the following:
    - 4.3.2.1 Place empty sample cup in 1<sup>st</sup> position of sample changer and refer to Enclosure 6.1 (Sample Changer Manual Operation) to position electrode assembly in sample cup.
    - 4.3.2.2 Perform the following to rinse buret:
      - A. Press [Burette]
      - B. Select "Dispense"
      - C. Press [F5](OK)
      - D. Ensure the following are selected:
        - 1. Burette drive 2
        - 2. Burette volume per volume of dispenser being used.
        - 3. Volume (mL) 100 for 20 mL dispenser or 50 (ml) for 10 mL dispenser
      - E. Press [F5] (Start)

**NOTE:** The buret will rinse 5 times the volume of the dispenser.

4.3.2.3 Verify there are no air bubbles in the NaOH delivery line or tip.

4.3.2.4 IF air bubbles are present, repeat Steps 4.3.2.1 to 4.3.2.3.

#### 4.4 NaOH Standardization

4.4.1 Perform this section:

- Once per week OR prior to use, whichever is less frequent.
- WHEN NaOH reservoir has been refilled and buret rinsed per Step 4.3.

4.4.2 Analyze a Blank as follows:

**NOTE:**  $\approx$  5 grams of mannitol equates to 2 level teaspoons.

4.4.2.1 Place cup in 1<sup>st</sup> position of sample changer AND add  $\approx$  5 grams (4 - 6) of Mannitol to empty sample cup.

4.4.2.2 Press or select the following in the order listed:

- [Sample]
- [F5] (Add)
- Select "New Sample Series"
- [F5] (OK)
- Select "Number of Samples"
- [1]
- Scroll down and select "Method ID"
- [3]
- [F5] (OK)
- [F5](OK)
- [Run]
- [F5] (OK)
- [F5] (START)
- [F5] (OK)

- NOTE:**
- The blank will run, results will be entered and printed out automatically.
  - A high blank value > 0.0300 mL will skew the results and should be verified by additional blank analysis by repeating Step 4.4.

4.4.3 Determination of titer factor with standard boric acid solution (1000 ppm)

4.4.3.1 Press or select the following in the order listed:

- A. [Sample]
- B. [F5] (Add)
- C. Select "New Sample Series"
- D. [F5] (OK)
- E. Select "Number of Samples"
- F. [2]
- G. Scroll down and select "Method ID"
- H. [4]
- I. [F5] (OK)

4.4.4 Place a sample cup on balance pan AND tare.

4.4.5 Transfer 20 g (18.0 - 22.0) of Standard Boric Acid Solution (NaOH Standardization) into sample cup.

4.4.6 When balance reading stabilizes, press [F4] (Balance).

4.4.7 Press [F5] (OK) twice

4.4.8 IF weight is accepted, select "Save Entry".

4.4.9 IF you choose to reweigh the sample select "Modify Entry" and repeat Steps 4.4.4 to 4.4.9.

4.4.10 Press [F5] (OK)

4.4.11 Place sample cup containing Standard Boric Acid Solution in sample changer.

- NOTE:**
1.  $\approx$  5 grams of mannitol equates to 2 level teaspoons.
  2. Mannitol may be added at anytime once the sample has been placed in the sample changer prior to starting the analysis (Step 4.4.14).

- 4.4.12 Add  $\approx$  5 grams (4 - 6) Mannitol to each sample cup.
- 4.4.13 Repeat Steps 4.4.4 to 4.4.12 once.
- 4.4.14 Press [Run]
- 4.4.15 Press [F5] (OK)
- 4.4.16 Press [F5] (START)
- 4.4.17 Press [F5] (OK)

**NOTE:** Boric Acid Standard Solution will run, results will be entered and printed out automatically.

- 4.4.18 Analyze a QC check per Step 4.5 (QC Check/Sample Analysis) to verify that the standardization is acceptable.
- 4.5 QC Check/Sample Analysis
- 4.5.1 **IF** analyzing samples containing borax with pH >6.4 (following post accident conditions), **THEN** adjust sample pH to 6.2 (6.0 - 6.4) using 0.0025N HCl.
- 4.5.2 Select appropriate sample size:

Approximate Boron Concentration (ppm)	Approximate Sample Size (grams)
0 - 500	25
500 - 2000	10
2000 - 10,000	5

- 4.5.3 Press or select the following in the order listed:
- 4.5.3.1 [Sample]
- 4.5.3.2 [F5] (Add)
- 4.5.3.3 Select "New Sample Series"
- 4.5.3.4 [F5] (OK)
- 4.5.3.5 Select "Number of Samples"

- 4.5.3.6 [Enter number of samples in sample set, i.e. 6]
- 4.5.3.7 Scroll down and select "Method ID"
- 4.5.3.8 [5]
- 4.5.3.9 [F5] (OK)
- 4.5.4 **IF** entering sample ID is desired, press [^] to select "Sample ID" **AND** using the keyboard enter sample name, **THEN** press [v] to select "weight [g]".
- 4.5.5 Place sample cup on balance pan **AND** tare.
- 4.5.6 Transfer appropriate amount of sample into the sample cup **AND** weigh.
- 4.5.7 When balance reading stabilizes, press [F4] (Balance).
- 4.5.8 Press [F5] (OK) twice.
- 4.5.9 Place sample cup in sample changer.

**NOTE:**

1.  $\approx$  5 grams of mannitol equates to 2 level teaspoons.
2. Mannitol may be added at anytime once the sample has been placed in the sample changer prior to starting the analysis (Step 4.5.12).

- 4.5.10 Add  $\approx$  5 grams (4 - 6) Mannitol to each sample cup.
- 4.5.11 **IF** analyzing more than one sample, **THEN** repeat Steps 4.5.4 - 4.5.10 for each additional sample.
- 4.5.12 Press [Run]
- 4.5.13 Press [F5] (OK)
- 4.5.14 Press [F5] (Start)
- 4.5.15 Press [F5] (OK)
- 4.6 Instrument Standby
  - 4.6.1 Place sample cup of conditioning agent in last available position in sample changer.
  - 4.6.2 Place red marker in hole next to sample cup of conditioning agent.

**NOTE:** The sample changer will recognize the marked sample cup as the last in the sample set and place the electrodes in it automatically.

#### 4.7 Calculations

**NOTE:** These calculations are performed automatically by the instrument.

##### 4.7.1 Boron Result

$$\text{ppm Boron} = \text{Const} \times \frac{1}{\text{Weight}} \times V \times \text{Conc}$$

$$\text{Where: Const} = \frac{\frac{10.811 \text{ g Boron}}{\text{mol H}_3\text{BO}_3} \times 1000}{1 \text{ eq / mol}}$$

Weight = Sample weight in grams

$$V = \text{ml} \times \frac{1}{\text{total buret vol (ml)}}$$

V = titrant consumption at the equivalence point in buret units.

$$\text{Conc} = \frac{1}{\text{Const Reag}} \times \frac{\text{Weight}}{V}$$

Conc = titrant concentration in milli-equivalents per buret volume

$$\text{Const Reag} = \frac{61.83 \text{ g H}_3\text{BO}_3 / \text{eq}}{1000}$$

##### 4.7.2 Density Correction

$$[\text{B}]_{\text{mg/L}} = [\text{B}]_{\text{mg/kg}} \cdot (0.99707 + (1.96082\text{E}^{-6} \cdot [\text{B}]_{\text{mg/kg}}))$$

Where:  $1.96082\text{E}^{-6}$  = factor based on linear regression of boric acid solution densities.

$0.99707$  = density of water at 25°C.

## 5. References

- 5.1 Mettler Toledo DL58 Titrator Reference Handbook.
- 5.2 Nuclear Generation Department Analytical Quality Control Program.

## 6. Enclosures

- 6.1 Sample Changer Manual Operation
- 6.2 Sample Series Manipulations
- 6.3 Error Messages, Malfunctions, and Troubleshooting
- 6.4 Routine Maintenance for the DL-58

## 1. Change Lift Position

- 1.1 Press or select the following in the order listed:
  - 1.1.1 [Changer] or [F1] (Esc) to return to/bring up sample changer menu.
  - 1.1.2 Select "Change lift position"
  - 1.1.3 [F5] (OK)
  - 1.1.4 [F4] (Modify) to select "Top", "Bottom", or "Middle"
  - 1.1.5 [F5] (Start)
  - 1.1.6 [F1] (Esc) to return to menu.

## 2. Rotate Turntable

- 2.1 Press or select the following in the order listed:
  - 2.1.1 [Changer] or [F1] (Esc) to return to/bring up sample changer menu.
  - 2.1.2 Select "Rotate turntable"
  - 2.1.3 [F5] (OK)
  - 2.1.4 Select "Direction"
  - 2.1.5 [F4] (Modify) to select "Forward" or "Backward"
  - 2.1.6 Select "Number of Positions"<sup>4</sup>
  - 2.1.7 Enter the number of positions to move from the numeric keypad
  - 2.1.8 [F5] (Start)
  - 2.1.9 [F1] (Esc) to return to menu.

### 3. Rinse Electrodes with Demineralized Water

3.1 Press or select the following in the order listed:

3.1.1 [Changer] or [F1] (Esc) to return to/bring up sample changer menu.

3.1.2 Select "Dispense/rinse"

3.1.3 [F5] (OK)

3.1.4 [F4] (Modify) to select "Rinse"

**NOTE:** The "Dose" and "Dispense" options are not used.

3.1.5 [F5] (Start)

**NOTE:** Water will rinse electrodes until [F5] is pressed again.

3.1.6 [F5] (Stop)

3.1.7 [F1] (Esc) to return to menu.

## 1. Delete a Sample from a Sample Series

- 1.1 Press or select the following in the order listed:
  - 1.1.1 [Sample]
  - 1.1.2 Select sample to be deleted
  - 1.1.3 [F2] (Delete)
  - 1.1.4 [OK].
- 1.2 Remove deleted sample from sample changer **AND** move any remaining samples forward one position.
- 1.3 Press [Run] to return to measured values screen.

## 2. Add Additional Samples to Sample Series

- 2.1 Press or select the following in the order listed:
  - 2.1.1 [Sample]
  - 2.1.2 Select last sample in series
  - 2.1.3 [F5] (Add)
  - 2.1.4 Select "Sample to Series"
  - 2.1.5 [F5] (OK).
- 2.2 Perform Steps 4.5.4 - 4.5.10 of the procedure.
- 2.3 Repeat Steps 2.1 - 2.2 for each additional sample.
- 2.4 Press [Run]

## 3. Insert an Urgent Sample into Sample Series

<b>NOTE:</b> Active sample will continue to run while adding urgent sample.
---

- 3.1 Press or select the following in the order listed:
  - 3.1.1 [Sample]
  - 3.1.2 Select active sample
  - 3.1.3 [F5] (Add)
  - 3.1.4 Select "Urgent sample"

- 3.1.5 [F5] (OK)
- 3.1.6 Select "Method ID"
- 3.1.7 [5]
- 3.1.8 [F5] (OK)
- 3.2 Repeat Steps 4.5.4 - 4.5.8 of the procedure.
- 3.3 Place sample cup in appropriate position in sample changer **AND** move remaining samples as needed.
- 3.4 Add  $\approx$  5 grams (4 - 6) Mannitol to sample cup (equivalent to 2 level teaspoons).
- 3.5 When active sample completes and results print, press "Run" to start urgent sample.
- 3.6 When urgent sample completes and results print, press [F5] (OK) to continue with original sample series.

#### **4. Add Additional Sample Series to Existing Series**

<b>NOTE:</b> The last sample in the current series must be selected prior to adding the new series (F5(Add)).
---

- 4.1 At anytime up to 2 additional sample series/methods may be loaded into the titrator program along with the current method being run. This can be accomplished as follows:
  - 4.1.1 Refer to the section associated for the preferred method and perform as written:
    - Method 1 / Method 2 pH calibration / 9 Buffer check Section 4.2
    - Method 3 Blank Analysis Section 4.4.2
    - Method 4 Titer Factor Section 4.4.3
    - Method 5 QC/sample analysis Section 4.5
  - 4.1.2 After each sample series is complete, press [Run] to start the next programmed series.
  - 4.1.3 Repeat at 4.1.1 as desired as long as no more than 3 sample series are programmed to run at one time.

**Enclosure 6.3  
Error Messages, Malfunctions  
and Troubleshooting**

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**1. Malfunctions not reported by the DL-58 Titrator**

Malfunction	Potential Cause	Action
No display on titrator	Titrator not connected to power supply	Connect to power supply, if fault persists contact METTLER TOLEDO Service
Several points of the display missing		Contact METTLER TOLEDO Service
Display does not match the pressed key		Contact METTLER TOLEDO Service
Stirrer does not rotate	Stirrer not properly assembled or sensors block it at the titration stand	Check stirrer and seating of the sensors
Transfer error to attached peripheral	Peripheral faulty or switched off	Check attached device is functioning properly
Device (printer, balance, terminal) at Centronics or RC interface do not react	Device not switched on Wrong settings Configuration (switch settings) wrong	Switch on device Settings and configuration must match (see Section 2.7)
Burette does not move to zero position when switched on	Burette drive faulty	Contact METTLER TOLEDO Service
Wrong potential or pH values	Electrode faulty Calibration data wrong Faulty cable	Check electrode (see electrode data sheet) Check defined data Use new electrode Replace cable
No dispensing, the titrant is discharged from stopcock or piston	Burette tip clogged Follower cam on burette mounted wrongly	Clean burette tip Insert follower cam correctly (see Section 10.2.3)

**Enclosure 6.3  
Error Messages, Malfunctions  
and Troubleshooting**

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**2. Error Messages generated by the DL-58 Titrator**

Message	Potential Problem	Action
EPROM or RAM Test Failed	Memory is faulty	Contact Staff or METTLER TOLEDO Support
Internal Error	Potential Hardware Failure has occurred	Contact Staff or METTLER TOLEDO Support
Memory Faulty	Parts of User Memory is faulty	Contact Staff or METTLER TOLEDO Support
Faulty Data Deleted	Stored Methods have been partially or totally deleted	Reload Methods manually or from memory card
Storage Not Possible	Memory capability of processor has been exceeded	Notify Staff

**3. Common Problems and Troubleshooting**

**3.1**

Message	Potential Problem	Action
pH 9 Buffer Out of Control	Bad pH probe (more likely problem)	Replace probe
	Faulty cable	Replace cable
Observe jumpy pH readings with NaOH additions	Faulty cable (more likely problem)	Replace cable
	Faulty probe	Replace probe
Insufficient stirring of mannitol	Broke propeller blades on stirrer	Replace stirrer
Insufficient water dispensed	Worn pump tubing	Replace small tubing inside pump cover
OOQ QC and/or samples not trending	<ul style="list-style-type: none"> <li>- any of above</li> <li>- poor standardization</li> <li>- air bubbles in NaOH dispensing line</li> <li>- clogged dispensing line</li> <li>- worn buret and/or piston (visible leakage)</li> <li>- noticeable temperature shift in lab</li> </ul>	<ul style="list-style-type: none"> <li>- see related action</li> <li>- recalibrate and/or restandardize</li> <li>- clear line of air bubbles</li> <li>- check line &amp; change if needed</li> <li>- inspect, clean or replace as needed</li> <li>- write R005 for air handling problems and recalibrate &amp; standardize</li> </ul>
Sample Changer skips sample	<ul style="list-style-type: none"> <li>Sensor is dirty</li> <li>Electronics confused</li> </ul>	Clean Sample Changer and Sensor Reset Electronics by switching DL58 and Sample Changer OFF then back ON

**Enclosure 6.3**  
**Error Messages, Malfunctions**  
**and Troubleshooting**

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- 3.2 **IF** normal troubleshooting within the shift fails to correct the problem, the instrument should be declared out-of-service and documented per the QC Program.
- 3.3 **IF** both titrators are declared out-of-service at the same point of time, actions shall be taken (i.e. reagent preparation) to be ready to perform a Manual Boron Analysis per LM/O/P003C.

**Enclosure 6.4**  
**Routine Maintenance for the DL-58**

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**NOTE:** All daily, weekly, and monthly routine maintenance will be logged in the comments section of the Chemdesk QC Chart. Major maintenance activities will be logged in the Maintenance Log.

**1. Weekly Maintenance**

- 1.1 Check probe.
- 1.2 Change probe fill solution.
- 1.3 Empty NaOH dispensing line and refill (during buret flush).
- 1.4 Empty, rinse, and refill water container.

**2. Monthly Maintenance**

- 2.1 Change drying tube.
- 2.2 Clean Titrator table.
- 2.3 Inspect stirrer arm.
- 2.4 Clean buret and piston.

**3. Bi-Annual Maintenance**

- 3.1 Change small tubing inside cover of water pump.