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R. A. JONES
Vice President

Duke Power
29672 / Oconee Nuclear Site
7800 Rochester Highway
Seneca, SC 29672

864 885 3158
864 885 3564 fax

April 12, 2004

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 2004-04

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

Volume B Revision 2004-04 April 2004

This revision is being submitted in accordance with 10 CFR 50-54(q) 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
U. S. Nuclear Regulatory Commission
61 Forsyth St., SW, Suite 24T23
Atlanta, Georgia 30303

w/copy of attachments
Mr. James R. Hall
Rockville, Maryland

(w/o Attachments, Oconee Nuclear Station)
NRC Resident Inspector
J. R. Brown, Manager, Emergency Planning

A045

April 12, 2004

OCONEE NUCLEAR SITE

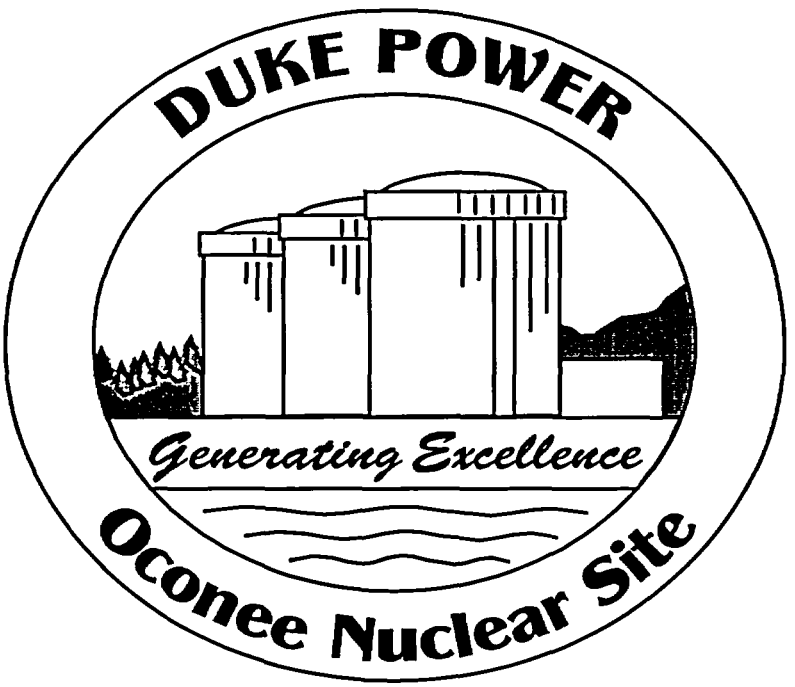
SUBJECT: Emergency Plan Implementing Procedures
Volume B, Revision 2004-04

Please make the following changes to the Emergency Plan Implementing Procedures Volume B.

REMOVE	INSERT
Cover Sheet Rev. 2004-03	Cover Sheet Rev. 2004-04
Table of Contents, page 1,2,3 & 4	Table of Contents page 1,2,3, & 4
Laboratory Method Process Record LM/O/P919 - 02/15/04	Laboratory Method Process Record LM/O/P919 - 03/29/04

DUKE POWER

EMERGENCY PLAN
IMPLEMENTING PROCEDURES
VOLUME B



APPROVED:

Larry E. Nicholson, Manager
Safety Assurance

4/12/04
Date Approved

04/12/2004
Effective Date

VOLUME B
REVISION 2004-04
APRIL 2004

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Volume B
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DELETED PROCEDURES

DELETED PROCEDURES

Duke Power Company
Nuclear Generation Department

LABORATORY METHOD PROCESS RECORD

Reference Use

Station: Oconee LM/O/P919

Rev. #: 9 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/6919 Rev, 1 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: 3/27/04 Initials: LB

Description of Change: (Attach additional pages if necessary)
Clarified Precaution 1.5.14 to refer to sample of unknown matrix. Included "buffering capacity" and replaced "will" with "may"
4.5 Added related note. 4.5.2 Revised steps to determine need to pH adjust.

Basis for Change: (Attach additional pages if necessary)
PIP O-04-00882

Field Testing Comments: (Attach additional pages if necessary)

Prepared by Ron Clark Date 2/25/04

Technical Review by Amanda Bullard Date 3/3/04

Additional Review by (optional) _____ Date _____

Approved by George A. Barber Date 3/29/04

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_2 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.

1.3.3 Matrix interference has been found to occur due to possible inputs into the Reactor Building Normal Sump (RBNS) samples. Therefore, these samples should be pH'd prior to analysis (Step 4.5.2). All RBNS sample results are reported as Information Only and designated with an "N".

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_2) then pyroboric acid ($H_2B_4O_7$), and finally boric anhydride (B_2O_3). Hence, in preparing a boron standard, boric acid is neither dried nor desiccated prior to weighing.

1.5.10 For error messages, malfunctions, and/or troubleshooting refer to Enclosure 6.3.

1.5.11 Sample concentration must not exceed a burette delivery volume of 40 mL. See Step 4.5.3 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.

1.5.14 Samples of unknown matrix with pH > 7 and buffering capacity may give skewed results if not pH adjusted prior to analysis (Step 4.5.2).

1.5.15 Large temperature swings in the lab will affect the analysis, requiring restandardization.

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 and Static Ionizers

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_2) 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 dearated reagent grade water AND mix.

3.3.2.2 Store in an air tight container OR a container fitted with a carbon dioxide (CO_2) 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_3BO_3) 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

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 Hydrochloric Acid (HCl), 0.0025N

3.9.1 Add 250 µL 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.

3.10 Hydrochloric Acid (HCl) 50% V/V

3.10.1 Adding the acid to reagent grade water, combine 50 mLs of reagent grade water and 50 mLs concentrated HCl.

3.10.2 Within a closed container, mix by inverting.

3.10.3 Record shelf life as 6 months.

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 NaOH Burret Rinse:

4.2.1 Perform this section:

- When NaOH reservoir is refilled.

4.2.2 Stir NaOH reservoir to thoroughly mix **AND** perform the following:

4.2.2.1 Place empty sample cup in 1st position of sample changer and refer to Enclosure 6.1 (Sample Changer Manual Operation) to position electrode assembly in sample cup by placing cup in middle position.

4.2.2.2 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.

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

4.2.2.4 **IF** air bubbles are present, repeat Steps 4.2.2.1 to 4.2.2.3.

4.3 Daily pH Electrode Standardization

4.3.1 Ensure electrode is clean.

4.3.2 Check filling solution level **AND** fill as necessary.

4.3.3 Ensure cap is not covering vent hole.

4.3.4 Using approximately 50 mL of each buffer, place cup of pH 7 buffer in 1st sample changer position, pH 10 buffer in 2nd position, and pH 9 buffer in 3rd position.

4.3.5 Press or select the following in the order listed:

4.3.5.1 [Sample]

4.3.5.2 [F5] (Add)

4.3.5.3 Select "New Sample Series"

4.3.5.4 [F5] (OK)

4.3.5.5 Select "Number of Samples"

4.3.5.6 [2]

4.3.5.7 Scroll down and select "Method ID"

4.3.5.8 [1]

4.3.5.9 [F5] (OK)

4.3.5.10 [F5] (OK)

4.3.5.11 [F5] (OK)

4.3.5.12 [F5] (Add)

4.3.5.13 Select "New Sample Series"

4.3.5.14 [F5] (OK)

4.3.5.15 Select "Number of Samples"

4.3.5.16 [1]

4.3.5.17 Scroll down and select "Method ID"

4.3.5.18 [2]

4.3.5.19 [F5] (OK)

4.3.5.20 [F5] (OK)

4.3.5.21 [Run]

4.3.5.22 [F5] (OK)

4.3.5.23 [F5] (START)

4.3.5.24 [F5] (OK)

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

4.3.6 After first sample series has completed and calibration results have printed, press [Run] to continue to 2nd sample series (pH 9 buffer).

4.3.7 **IF** pH 9 buffer result is outside 9 ± 0.1 pH units, **THEN** repeat Steps 4.3.4 - 4.3.7.

4.3.8 On a daily basis, record slope value in appropriate database or logsheet for instrument performance trending.

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 4.2.

4.4.2 Analyze a Blank as follows:

NOTE: ≈ 5 grams of mannitol equates to 2 level teaspoons.

4.4.2.1 Place cup in 1st position of sample changer **AND** add ≈ 5 grams
(4 - 6) of Mannitol to empty sample cup.

4.4.2.2 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. [1]

G. Scroll down and select "Method ID"

H. [3]

I. [F5] (OK)

J. [F5](OK)

K. [Run]

L. [F5] (OK)

M. [F5] (START)

N. [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 Section 4.4.2.

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 Press [F5] (OK)

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

NOTE: 1. ≈ 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).

4.4.10 Add ≈ 5 grams (4 - 6) Mannitol to each sample cup.

4.4.11 Repeat Steps 4.4.4 to 4.4.10 once.

4.4.12 Press [Run]

4.4.13 Press [F5] (OK)

4.4.14 Press [F5] (START)

4.4.15 Press [F5] (OK)

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

4.4.16 Analyze a QC check per Step 4.5 (QC Check/Sample Analysis) to verify that the standardization is acceptable.

NOTE: Samples of unknown matrix or requiring pH adjustment shall be reported as Info. Only unless results are documented as accurate, based on spiking data.

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 IF analyzing samples of unknown matrix or with a suspected pH > 7, THEN perform the following:

4.5.2.1 Determine the pH.

4.5.2.2 IF the pH is < 7, continue to step 4.5.3.

4.5.2.3 IF the pH is > 7,

A. Add 2 even scoops of mannitol to approximately 25 mls of sample.

B. Recheck pH of the sample following addition of mannitol.

C. IF pH < 7, continue to Step 4.5.3.

D. IF pH > 7, THEN adjust sample pH to a range of 6.0 - 6.4 using 50% HCl in 10 µl increments.

4.5.2.4 Using the pH adjusted sample, continue to Step 4.5.3.

4.5.3 Select appropriate sample size:

Boron Concentration (ppm) ± 50 ppm	Sample Size (grams) ± 2 g
0 – 500	25
500 – 2000	10
2000 - 10,000	5

4.5.4 Press or select the following in the order listed:

4.5.4.1 [Sample]

4.5.4.2 [F5] (Add)

4.5.4.3 Select "New Sample Series"

4.5.4.4 [F5] (OK)

4.5.4.5 Select "Number of Samples"

4.5.4.6 [Enter number of samples in sample set, i.e. 6]

4.5.4.7 Scroll down and select "Method ID"

4.5.4.8 [5]

4.5.4.9 [F5] (OK)

4.5.5 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]".

4.5.6 Place sample cup on balance pan AND tare.

4.5.7 Transfer appropriate amount of sample into the sample cup AND weigh.

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

4.5.9 Press [F5] (OK) twice.

4.5.10 Place sample cup in sample changer.

NOTE: 1. ≈ 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 4.5.13).

4.5.11 Add ≈ 5 grams (4 - 6) Mannitol to each sample cup.

4.5.12 IF analyzing more than one sample, THEN repeat Steps 4.5.5 - 4.5.11 for each additional sample.

4.5.13 Press [Run]

4.5.14 Press [F5] (OK)

4.5.15 Press [F5] (Start)

4.5.16 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

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

Where:

$$\text{Const} = \frac{\frac{10.811 \text{ g Boron}}{\text{mol } H_3BO_3} \times 1000}{1\text{eq / mol}}$$

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

$$\text{Const Reag} = \frac{61.83 \text{ g } H_3BO_3 / \text{eq}}{1000}$$

4.7.2 Density Correction

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

Where: $1.96082E^{-6}$ = 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 Operation of Primary Lab Temperature / Humidity Monitor

6.5 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"

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 4.5.5 - 4.5.11 of the procedure.

2.3 Repeat Steps 2.1 - 2.2 of this enclosure for each additional sample.

2.4 Press [Run]

3. Insert an Urgent Sample into Sample Series

NOTE:	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 4.5.5 - 4.5.9 of the procedure.

3.3 Place sample cup in appropriate position in sample changer AND move remaining samples as needed.

3.4 Add ≈ 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

NOTE:	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 procedure section associated for the preferred method and perform as written:

- Method 1 / Method 2 pH calibration / 9 Buffer check Section 4.3
- 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.

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 stopcook or piston	Burette tip clogged Follower cam on burette mounted wrongly	Clean burette tip Insert follower cam correctly (see Section 10.2.3)

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

Message	Potential Problem	Action
pH 9 Buffer Out of Control	Bad pH probe (more likely problem) Faulty cable	Replace probe Replace cable
Observe jumpy pH readings with NaOH additions	Faulty cable (more likely problem) Faulty probe	Replace cable 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
OOC QC and/or samples not trending	- any of above - poor standardization - air bubbles in NaOH dispensing line - clogged dispensing line - worn buret and/or piston (visible leakage) - noticeable temperature shift in lab	- see related action - recalibrate and/or restandardize - clear line of air bubbles - check line & change if needed - inspect, clean or replace as needed - write R005 for air handling problems and recalibrate & standardize
Sample Changer skips sample	Sensor is dirty Electronics confused	- Clean Sample Changer and Sensor Reset Electronics by switching DL58 and Sample Changer OFF then back ON - Ensure Black sample spacers used on sampler changer
Instrument lost contact with computer	Lab x is not open or computer is locked up	Check associate computer & open program or unlock computer Press OK - Titrator should restart, if not press reset & continue analysis by pressing run.

3.1 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.

3.2 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.

Operation of Primary Lab Temperature / Humidity Monitor

NOTE: The following allows you to pull data from the Temperature/Humidity monitor, print the data, save the data, reset the device and restart the device.

1. Ensure PC-87 (Temperature/Humidity Monitor) is connected via cable to the lab computer.
2. From desktop, open MadgeTech 2.00 program icon
3. From Main Menu, Click "Device"
4. Select "Read Device Data". This will download data from the device.
5. **WHEN** download of data is complete, select "Summary Tab".
6. Record the following to comments on Enclosure 6.5:
   - Dates in service
   - Minimum Temperature
   - Maximum Temperature
   - Average Temperature
7. Print a hard copy of the "Summary" for staff review.
8. Compare the average to the previous week's data.
9. **IF** > 5 °C difference between the minimum and maximum for the current time frame or between the current and previous weeks mean, then contact staff.
10. To save the file perform the following:
    - 10.1 Select "Data" tab
    - 10.2 Select "File"
    - 10.3 Select "Save As"
    - 10.4 Select "Sections Drive"
    - 10.5 Select "Chem"
    - 10.6 Select "Primary"
    - 10.7 Select "Instrument Backup Files"

10.8 Select "Madge tech lab temp_humidity"

10.9 Save using a format of datemonthyear for the file name

Example: 20jan04.csv

CAUTION: The following will clear all readings

11. WHEN complete reset and restart the device:

11.1 Select "Device" from the main menu.

11.2 Select "Reset Device".

11.3 WHEN prompted to continue, select "yes"

11.4 WHEN prompted that the device is reset, select "OK".

11.5 From main menu select the following:

11.5.1 "Device"

11.5.2 "Start Device"

11.6 WHEN prompted to continue, select "yes"

11.7 Verify on the "Start Device" screen the following:

- "Start Now" is checked
- User ID is "RHTemp"
- Reading Rate is 5 minutes

11.8 WHEN prompt screen appears, select "Start Device" button

11.9 Verify a message appears "Device Started"

11.10 WHEN pop up screen shows device started, select "OK".

12. IF you want to check device status perform the following:

    12.1 Select "Device" from main menu.

    12.2 Select " Identify Device and Read Status "

    12.3 Verify from pop up screen Device Status Info.

    12.4 Select "OK"

13. Close program when complete.

Enclosure 6.5

Routine Maintenance for the DL-58

NOTE: Daily pH results shall be logged on the Chemdesk QC Chart for the appropriate instrument (Slope under "Raw Data", Zero Point / pH 9 results under "Comments".) Also recorded weekly on the appropriate QC Chart under "Comments" are the Blank data and Titer Factor. Major maintenance activities will be logged in the Maintenance Log.

WEEKLY

		Date / Initials Performed					
Wipe down instrument							
Change pH probe filling Sol'n							
Empty NaOH dispensing line & refill with buret flush							
Standardize w/1000 ppm NIST Boron Standard							
Record Lab temp from Madge Tech Program (Enc. 6.4)	Date Min Max Mean						

		Date / Initials Performed					
Wipe down instrument							
Change pH probe filling Sol'n							
Empty NaOH dispensing line & refill with buret flush							
Standardize w/1000 ppm NIST Boron Standard							
Record Lab temp from Madge Tech Program (Enc. 6.4)	Date Min Max Mean						

		Date / Initials Performed					
Wipe down instrument							
Change pH probe filling Sol'n							
Empty NaOH dispensing line & refill with buret flush							
Standardize w/1000 ppm NIST Boron Standard							
Record Lab temp from Madge Tech Program (Enc. 6.4)	Date Min Max Mean						

Place in Maintenance Log when complete.

MONTHLY

	January	February	March	April	May	June
Clean Instrument & Sample Changer Table						
Inspect and Clean Stirrer Propellers						
Inspect Burets for Leakage & Change Piston as Required (Sect. 10.2 of Manual)						
Clean Buret and Piston (Section 10.2.5 of Manual)						
Change Out Drying Tubes						

	July	August	September	October	November	December
Clean Instrument & Sample Changer Table						
Inspect and Clean Stirrer Propellers						
Inspect Burets for Leakage & Change Piston as Required (Sect. 10.2 of Manual)						
Clean Buret and Piston (Section 10.2.5 of Manual)						
Change Out Drying Tubes						

Place in Maintenance Log when complete.

BIANNUAL

Date / Initials Performed

Change Internal Tube of Water Pump _____ _____

ANNUALLY

Replace Static Ionizers

PC-73	Control #	Date Rec'd
	_____	_____
	_____	_____
	_____	_____
	_____	_____

PC-74	Control #	Date Rec'd
	_____	_____
	_____	_____
	_____	_____
	_____	_____

AS NEEDED

	Date / Initials Performed							
Clean under Balance Pan								
Replace pH Probe								
Replace Buret								
Replace pH Probe Cable								
Clean/Replace Buret Valve								

Place in Maintenance Log when complete.