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W. R. McCollum, Jr.  
Vice President

November 16, 1999

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 99-10

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


Volume B Revision 99-10 November, 1999

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 Mike Thorne, Emergency Planning Manger at 864-885-3210.

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

Very truly yours,



W. R. McCollum, Jr.  
VP, Oconee Nuclear Site

xc: (w/2 copies of attachments)  
Mr. Luis Reyes,  
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A045

POW APOU

November 16, 1999

OCONEE NUCLEAR SITE

SUBJECT: Emergency Plan Implementing Procedures  
Volume B, Revision 99-10

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

REMOVE

Cover Sheet Rev. 99-09

Table of Contents page 1, 2, & 3

LM-O-P919 - (07/01/99)

DELETE - DTA-1 Rev. 8  
ESS Maintenance

ADD

Cover Sheet Rev. 99-10

Table of Contents page 1, 2, & 3

LM-O-P919 - (10/26/99)

If you have any questions regarding this revision please contact Mike  
Thorne at ext. 885-3210.

# DUKE POWER

## EMERGENCY PLAN IMPLEMENTING PROCEDURES VOLUME B



**APPROVED:**

  
\_\_\_\_\_

**W. W. Foster, Manager  
Safety Assurance**

11/16/99  
\_\_\_\_\_

**Date Approved**

11/16/99  
\_\_\_\_\_

**Effective Date**

**VOLUME B  
REVISION 99-10  
NOVEMBER 1999**

Chemistry Lab LM-O-P-003A	Determination of Boron Using The Mettler DL40GP - (06/18/98)
Chemistry Lab LM-O-P003C	Determination Of Boron By Manual Colorimetric Titration - (11/18/96)
Chemistry Lab LM-O-P919	Boron Analysis by Mettler DL 58 Boron Titration – (10/26/99)
CP/1/A/2002/004C	Operating Procedure for the Post Accident Liquid Sampling System (PALSS) - (09/17/99)
CP/1&2/A/2002/005	Post Accident Caustic Injection into the Low Pressure Injection System - (08/04/99)
CP/2/A/2002/004C	Operating Procedure for the Post Accident Liquid Sampling System (PALSS) - (09/17/99)
CP/3/A/2002/004C	Operation Procedure for Operation of the Post-Accident Liquid Sampling System (PALSS) - (10/13/99)
CP/3/A/2002/005	Post Accident Caustic Injection into the Low Pressure Injection System - (03/17/99)
HP/0/B/1009/009	Procedure for Determining The Inplant Airborne Radioiodine Concentration During Accident Conditions - (12/03/97)
HP/0/B/1009/012	Distribution of Potassium Iodide Tablets In The Event Of A Radioiodine Release - (06/15/99)
HP/0/B/1009/015	Procedure for Sampling and Quantifying High Level Gaseous Radioiodine And Particulate Radioactivity - (06/16/99)
HP/0/B/1009/016	Procedure for Emergency Decontamination of Personnel and Vehicles On-Site And From Off-Site Remote Assembly Area - (12/29/97)
HP/1/A/1009/017	Operating Procedure For Post-Accident Containment Air Sampling System - (08/30/99)
HP/2/A/1009/017	Operating Procedure For Post-Accident Containment Air Sampling System - (08/30/99)
HP/3/A/1009/017	Operating Procedure For Post-Accident Containment Air Sampling System - (08/30/99)
RP/O/B/1000/011	Planned Emergency Exposure - (02/01/94)
RP/0/B/1000/025	Operational Support Center Manager Procedure - (09/02/99)
RP/0/B/1000/027	Re-Entry Recovery Procedure - (04/07/97)

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Maintenance Directive 9.2	Emergency Plan For Members Of The Work Control Group - (07/30/94)
OMP 1-7	Operations Emergency Response Organization - (09/28/99)
Radiation Protection Manual 11.1	Radiation Protection Emergency Response - (09/01/98)
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CP/0/A/2003/02B	Determination of Failed Fuel - (04/03/86) DELETED
CP/0/A/2004/02A	Post Accident Determination of Boron Concentration Using the Orion Fluoroborate Electrode - (03/28/85) DELETED
CP/0/A/2004/02F	Determination of Boron for High pH Samples Following Caustic - (12/12/94) - DELETED
CP/0/A/2004/09D	Post Accident Determination of PH - (03/28/85) DELETED
CP/0/A/2004/037	Determination of Boron by Manual Colorometric Titration Using Phenolphthaline Indicator - (12/12/94) DELETED
CP/0/A/2005/2D	Post Accident Determination of Gamma Isotopic Activity - (07/09/82) DELETED
CP/0/B/2001/05A	Post Accident Analytical Procedure Guidelines- (06/14/85) DELETED
CP/0/B/2005/09	Determination of Failed Fuel - (10/05/90) DELETED
CP/0/B/4003/01	Procedure for Environmental Surveillance Following a Large Unplanned Release of Gaseous Radioactivity - (07/25/85) DELETED
CP/0/B/4003/02	The Determination of Plume Direction and Sector(s) to be Monitored Following a Large Unplanned Release of Gaseous Activity DELETED
HP/0/B/1009/10	Procedure for Quantifying Gaseous Releases Through Steam Relief Valves Under Post-Accident Conditions - (10/30/85) DELETED
HP/0/B/1009/11	Projection of Offsite Dose from the Uncontrolled Release of Radioactive Materials Through a Unit Vent - (05/24/85) DELETED
HP/0/B/1009/14	Project Offsite Dose from Releases other than Through a Vent - (02/12/85) DELETED
IP/0/A/0050/001	Procedure to Provide Emergency Power to an HPI Pump Motor from the ASW Switchgear - (10/05/92) DELETED
IP/0/B/0050/004	Emergency Power - Telephone System - (03/30/87) DELETED
DTA-1	Site Assembly (ESS - Maintenance Division) - (11/07/95) DELETED
DTA-2	Station Support During a Site Assembly - (03/26/92) DELETED
Integrated Sched.	Integrated Scheduling Group Directive 6.0 - (10/26/89) DELETED
STA. SVCS. 3.1.6	Industrial Safety, Health, and/Fire Protection Section - (09/18/89) DELETED
Commodities & Facilities CF 1-10	Site Assembly CF 1-10 - (11/01/94) - DELETED
Commodities & Facilities Functional Area Directive 102	Station Support During a Site Assembly Functional Area Directive 102 - (07/14/97) - DELETED from Volume B, moved to Volume C on 06/15/98 Rev. 98-04

# INFORMATION ONLY

Duke Power Company  
Nuclear Generation Department

## LABORATORY METHOD PROCESS RECORD

### Reference Use

LM- 0 - P919

Title: BORON ANALYSIS BY METTLER DL-58 TITRATION

Revision #: 4 Change: \_\_\_\_\_  
(A, B, or C, etc.)

Description of Change: (Attach additional pages if necessary)

- 1- Section 1.5.8 – deleted portable shield option.
- 2- Added section 1.5.10 – Addresses new enclosure 6.3 Error & Malfunctions.
- 3- Added section 1.5.11 – Addresses precautions related to 40 ml burette limitations.
- 4- Added section 1.5.12 – Precaution to verify no air bubbles are in NaOH delivery line.
- 5- Added Caution Box to Section 3. Relating to chemical hazards and knowledge expectations.
- 6- Moved NOTE under section 3. To 3.2.2.
- 7- Corrected typo on section 3.3.3.1.
- 8- Added two new sections 4.2.5.23 and 4.2.5.24 to eliminate multiple actions in one step.
- 9- Added instruction clarification to steps 4.2.5.7, 4.2.5.17, 4.3.3.2 section (G) (L) and (M), 4.3.4.1 section (G), and 4.4.3.7 with the words “Scroll down”
- 10- Corrected errors in numbered sequences under section 4.3.
- 11- Corrected errors in numbered sequences under section 4.3.2.
- 12- Added section 4.3.2.3 to verify that there are no air bubbles in NaOH lines.
- 13- Added section 4.3.2.4 to direct action to be taken if air bubbles are detected.
- 14- Added section 4.3.3.2 part (N) to eliminate multiple actions in one step.
- 15- Added two new sections 4.3.15 and 4.3.16 to eliminate multiple actions in one step.
- 16- Added section 4.4.13 to clarify keypad stroke requirements.
- 17- Added Enclosure 6.3 “Error Messages and Malfunctions” to assist in troubleshooting.

#### Basis for Change:

To upgrade method to comply with procedures writers guide and to incorporate operating experience incurred operating the equipment.

Prepared by Ray O Smith Date 9/26/99

Technical Review by (optional) Mike G Date 9/29/99

Approved by [Signature] Date 10/26/99

## Boron Analysis by Mettler DL58 Boron Titration

### 1. Purpose

**NOTE:** Seven Control Copies and one Information Only copy of this CSM 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 ( $\text{HBO}_2$ ) then pyroboric acid ( $\text{H}_2\text{B}_4\text{O}_7$ ), and finally boric anhydride ( $\text{B}_2\text{O}_3$ ). 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.

## 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, 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

**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).
- 3.3.3 Electrode Storage Solution (0.5 mol/L KCL)
  - 3.3.3.1 Weigh out 37.275g ± 0.1g anhydrous KCl. Add to a 1 L volumetric flask and bring to volume with deionized water.
  - 3.3.3.2 Record shelf life as 3 months.
- 3.4 Standard Boric Acid Solution (NaOH Standardization), 1000 mg/l
  - 3.4.1 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.
  - 3.4.2 Record shelf life as 1 year.
  - 3.4.3 Verify solution concentration per section 4.4 (QC Check/Sample Analysis).
- 3.5 QC Boric Acid Solution, 1000 mg/l
  - 3.5.1 Use purchased 1000 mg/l Boric Acid Standard
  - 3.5.2 Use manufacturer stated shelf life.
- 3.6 Mannitol
- 3.7 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.

- 3.7.1 Add 250 µl of concentrated HCl to a 1000 ml volumetric flask partially filled with reagent grade water.
- 3.7.2 Dilute to volume with reagent grade water AND mix.
- 3.7.3 Record shelf life as 1 month.

## 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.
- 4.1.4 If needed, refer to Enclosure 6.2 (Sample Series Manipulations) to perform any of the following:
  - 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 7 buffer in 1<sup>st</sup> sample changer position, 10 buffer in 2<sup>nd</sup> position, and 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 (9 buffer).
- 4.2.7 **IF** 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 Standardization

- 4.3.1 Perform this section:

- Once per week **OR** prior to use, whichever is less frequent.
- When NaOH reservoir is refilled.

- 4.3.2 **IF** NaOH reservoir has been refilled, **THEN** 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.
- 4.3.3 Analyze a Blank as follows:
- 4.3.3.1 Add  $\approx$  5 grams (4 - 6) of Mannitol to empty sample cup **AND** place cup in 1<sup>st</sup> position of sample changer.
- 4.3.3.2 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.

- 4.3.4 Determination of titer factor with standard boric acid solution (1000 ppm)
  - 4.3.4.1 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)
  - 4.3.5 Place a sample cup on balance pan AND tare.
  - 4.3.6 Transfer 20 mls (18.0 - 22.0) of Standard Boric Acid Solution (NaOH Standardization) into sample cup.
  - 4.3.7 When balance reading stabilizes, press [F4] (Balance).
  - 4.3.8 Press [F5] (OK) twice
  - 4.3.9 Select "Save Entry" or "Modify Entry".
  - 4.3.10 Press [F5] (OK)
  - 4.3.11 Place sample cup containing Standard Boric Acid Solution in sample changer.
  - 4.3.12 Repeat Steps 4.3.5 to 4.3.11.
  - 4.3.13 Add  $\approx$  5 grams (4 - 6) Mannitol to each sample cup.
  - 4.3.14 Press [Run]
  - 4.3.15 Press [F5] (START)

4.3.16 Press [F5] (OK)

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

4.3.17 Analyze a QC check per Section 4.4 (QC Check/Sample Analysis) to verify that the standardization is acceptable.

4.4 QC Check/Sample Analysis

4.4.1 **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.

4.4.2 Select appropriate sample size:

Approximate Boron Concentration (ppm)	Approximate Sample Size (grams)
0 – 500	25
500 – 2000	10
2000 - 10,000	1

4.4.3 Press or select the following in the order listed:

4.4.3.1 [Sample]

4.4.3.2 [F5] (Add)

4.4.3.3 Select “New Sample Series”

4.4.3.4 [F5] (OK)

4.4.3.5 Select “Number of Samples”

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

4.4.3.7 Scroll down and select “Method ID”

4.4.3.8 [5]

4.4.3.9 [F5] (OK)

4.4.4 **IF** entering sample ID is desired, press [^] to select “Sample ID” **AND** enter sample name, **THEN** press [v] to select “weight [g]”.

4.4.5 Place sample cup on balance pan **AND** tare.

4.4.6 Transfer appropriate amount of sample into the sample cup **AND** weigh.

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

4.4.8 Press [F5] (OK) twice.



- 4.4.9 Place sample cup in sample changer.
  - 4.4.10 **IF** analyzing more than one sample, **THEN** repeat Steps 4.4.4 - 4.4.9 for each additional sample.
  - 4.4.11 Add  $\approx$  5 grams (4 - 6) Mannitol to each sample cup.
  - 4.4.12 Press [Run]
  - 4.4.13 Press [F5] (OK)
  - 4.4.14 Press [F5] (Start)
  - 4.4.15 Press [F5] (OK)
- 4.5 Instrument Standby
- 4.5.1 Place sample cup of conditioning agent in last available position in sample changer.
  - 4.5.2 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.

#### 4.6 Calculations

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

##### 4.6.1 Boron Result

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

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

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

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

##### 4.6.2 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 and Malfunctions

## **1. Change Lift Position**

- 1.1 Press or select the following in the order listed:
  - 1.1.1 [Changer]
  - 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]
  - 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

3.1 Press or select the following in the order listed:

3.1.1 [Changer]

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 4.4.4 - 4.4.9.
- 2.3 Repeat steps 2.1 - 2.2 for each additional sample.
- 2.4 Add  $\approx$  5 grams (4 - 6) Mannitol to sample cup.
- 2.5 Press [Run]

## 3. Insert an Urgent Sample into Sample Series

<b>NOTE:</b> Active sample will continue to run while adding urgent sample.
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- 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 4.4.4 - 4.4.8.
- 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.
- 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**

- 4.1 Press or select the following in the order listed:
  - 4.1.1 [Sample]
  - 4.1.2 Select last sample in series
  - 4.1.3 [F5] (Add)
  - 4.1.4 Select "New Sample Series"
  - 4.1.5 [F5] (OK).
  - 4.1.6 Select "Number of Samples"
  - 4.1.7 [Enter number of samples in sample set, i.e. 6]
  - 4.1.8 Select "Method ID"
  - 4.1.9 [5]
  - 4.1.10 [F5] (OK)
- 4.2 Perform steps 4.4.4 - 4.4.11.
- 4.3 Repeat steps 4.1 - 4.2 for each additional sample series.
- 4.4 After initial series is complete, press [Run] to start additional sample series.

**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	Check electrode (see electrode data sheet) Check defined data Use new electrode
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 40.2.3)



**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