50-255

Date Entered: Jan 12, 2001

TO: USNRC/WASHINGTON
JMCKNIGHT Copy Number: 145

TRANSMITAL NUMBER: **173319**

PROCEDURE NUMBER: EI-7.5

TITLE: BORON; CHLORIDE ION CHROMATOGRAPHY METHOD POST ACCIDENT

TRANSMITTAL: LISTED BELOW ARE NEW/REVISED PROCEDURES WHICH MUST BE IMMEDIATELY INSERTED INTO OR DISCARDED FROM YOUR PROCEDURE MANUAL.

Action Required	Section or Description
REMOVE AND DESTROY	EI-7.5, R/10, ENTIRE PROCEDURE
REPLACE WITH	EI-7.5, R/10, ENTIRE PROCEDURE
	EDITORIAL

SIGN, DATE, AND RETURN THE ACKNOWLEDGEMENT FORM WITHIN 10 DAYS TO THE PALISADES PLANT DOCUMENT CONTROL.

SIGNATURE OR INITIALS _____

DATE _____

A045

PALISADES NUCLEAR PLANT
EMERGENCY IMPLEMENTING PROCEDURE

TITLE: BORON; CHLORIDE ION CHROMATOGRAPHY METHOD POST ACCIDENT

Michael Sullivan 10/08/01
Procedure Sponsor Date

ILGallagher 8/5/97
Technical Reviewer Date

LFJohns 8/4/97
User Reviewer Date

TITLE: BORON; CHLORIDE ION CHROMATOGRAPHY METHOD POST ACCIDENT

Table of Contents

e

TITLE: BORON; CHLORIDE ION CHROMATOGRAPHY METHOD POST ACCIDENT

Table of Contents

ATTACHMENTS

TITLE: BORON; CHLORIDE ION CHROMATOGRAPHY METHOD POST ACCIDENT

USER ALERT
REFERENCE USE PROCEDURE

Refer to the procedure periodically to confirm that all procedure segments of an activity will be or are being performed. Where required, sign appropriate sign-off blanks to certify that all segments are complete.

1.0 PERSONNEL RESPONSIBILITY

The OSC Chemistry Supervisor shall implement this procedure.

2.0 PURPOSE

This analytical procedure is applicable to determination of boron and chloride in undiluted post accident reactor water.

3.0 INITIAL CONDITIONS AND/OR REQUIREMENTS

3.1 This procedure shall be implemented per Emergency Implementing Procedure EI-7.2, "Emergency Post Accident Analysis."

3.2 An undiluted post accident sample collected requiring a boron and chloride analysis. Gray lab ion chromatograph screen on program manager display.

4.0 REFERENCES

4.1 SOURCE DOCUMENTS

4.1.1 Dionex Application Note #56, July 1988, "Determination of Trace Anions and Key Organic Acids in High Purity, Ammoniated, and Borated Waters Found in Steam Cycle Power Plants" | e

4.1.2 Technical Specifications Chapter 5, Section 5.5.3, "Post Accident Sampling Program" | e

TITLE: BORON; CHLORIDE ION CHROMATOGRAPHY METHOD POST ACCIDENT

4.2 **REFERENCE DOCUMENTS**

4.2.1 Emergency Implementing Procedure EI-7.2, "Emergency Post Accident Analysis"

4.2.2 Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheets"

4.2.3 Palisades Administrative Procedure 10.46, "Plant Records"

5.0 **PROCEDURE**

USER ALERT
REFERENCE USE PROCEDURE

Refer to the procedure periodically to confirm that all procedure segments of an activity will be or are being performed. Where required, sign appropriate sign-off blanks to certify that all segments are complete.

NOTE: The steps described in this procedure outline basic methods of performing tasks. Advanced users may consult the vendor manuals for additional methods for performing these same tasks.

5.1 **EQUIPMENT NEEDED**

5.1.1 Dionex Model 2010i Ion Chromatograph

5.1.2 Dionex Advanced Computer Interface

5.1.3 Dionex System Computer and Printer

5.2 **REAGENTS NEEDED**

5.2.1 2000 ppm Boron Standard Solution, 1.0 ppm Chloride (Functional Check)

5.2.2 70 Millimolar Sodium Tetraborate

5.2.3 500 ppm Boron Standard Solution, 50 ppb Chloride

5.2.4 2000 ppm Boron Standard Solution, 1.0 ppm Chloride

5.2.5 4000 ppm Boron Standard Solution, 10.0 ppm Chloride

5.2.6 6000 ppm Boron Standard Solution, 20.0 ppm Chloride

5.2.7 100 ppm Chloride Standard Solution (Calibration)

5.2.8 100 ppm Chloride Standard Solution (Functional Check)

5.2.9 1000 ppm Chloride Standard Solution

6.0 PRECAUTIONS AND LIMITATIONS

6.1 PERSONNEL SAFETY

6.1.1 The sample is expected to be highly radioactive. Strict attention must be given to minimize radiation exposure by using shielding and controlling personnel time of exposure and distance.

6.1.2 Sample bottle should not be handled directly. All liquid discharges from the Ion Chromatograph shall be directed and rinsed to the lab drain.

7.0 WORKING RANGE

500 ppm to 6000 ppm for Boron, 50 ppb to 20 ppm for Chloride

7.1 SAMPLE PRETREATMENT

The sample to be analyzed shall be an undiluted PCS PASM sample.

7.2 **STANDARDIZATION**

NOTE: Attachment 4, "Dionex Method Parameters PASMCL.MET," is a copy of the PASMCL method used for analysis.

7.2.1 Prime the sample loading pump per Attachment 5. Sample pump is located in the grey lab behind the Advanced Computer Interface.

7.2.2 Setup Ion Chromatograph for PASM Analysis by:

a. Remove Dionex autosampler discharge fitting from Bulkhead Port #3, System 2.

b. Attach sample pump discharge fitting to Bulkhead Port #3, System 2.

c. Verify open pressurized water supply to SRS to start flow.

d. Verify pump switch on and pump stroke to highest setting.

7.2.3 Perform a standardization prior to boron analysis using four calibration standards by:

a. Prepare 500 ppm Boron, 50 ppb Chloride; 2000 ppm Boron, 1.0 ppm Chloride; and 4000 ppm Boron, 10 ppm Chloride, 6000 ppm Boron, 20 ppm Chloride standards. See Attachment 3, "Standard and Reagent Preparation," for reagent preparation.

b. Place sample pickup tube into first calibration standard (500 ppm Boron, 50 ppb Chloride).

c. Place mouse arrow on "RUN" icon in Dionex AI-450 window under Program Manager and press left clicker twice rapidly. An hour glass should appear (wait).

d. Place arrow on "LOAD" and press left clicker once.

e. Place arrow on "Schedule" and press left clicker once.

f. Place arrow on "PASM1.SCH" and press left clicker twice.

g. Place arrow on "OK" and press left clicker once.

h. Place arrow on "RUN" and press left clicker once.

i. Place arrow on "START" and press left clicker once.

j. Place arrow on "OK" and press left clicker once. (This starts sample run which will take about 20 minutes.)

k. Repeat Steps 7.2.3b through 7.2.3j for PASM2.SCH (2000 ppm Boron, 1.0 ppm Chloride standard).

l. Repeat Steps 7.2.3b through 7.2.3j for PASM3.SCH (4000 ppm Boron, 10.0 ppm Chloride standard).

m. Repeat Steps 7.2.3b through 7.2.3j for PASM4.SCH (6000 ppm Boron, 20.0 ppm Chloride standard).

n. Edit completed chromatograph as necessary per Attachment 2. Editing of completed chromatograph may be performed while analysis is continuing.

7.2.4 Enter calibration data into calibration table by:

a. Place the mouse arrow on the "Calplot" icon under program manager window and click left clicker twice.

b. Place mouse arrow on file and click left clicker once.

c. Place mouse arrow on "Open Method" and press left clicker once.

d. Place mouse arrow on "PASMCL.MET" and press left clicker twice.

e. Place mouse arrow on "Edit" and press left clicker once.

f. Place mouse arrow on "Component Table" and press left clicker once.

g. Place mouse arrow on component name that calibration data will be entered for and press left clicker once.

h. Place mouse arrow on peak area's to be entered and press left clicker once.

i. Enter new peak area for concentration level.

j. Repeat Steps 7.2.4h and 7.2.4i for each new area.

k. Repeat Steps 7.2.4g through 7.2.4j for the second component.

l. Place mouse arrow on "Exit" and press left clicker once.

m. Place mouse arrow on "File" and press left clicker once.

n. Place mouse arrow on "Save" and press left clicker once.

o. Place mouse arrow on top left space bar and press left clicker twice. Program Manager window or icon should now be displayed.

7.3 **FUNCTIONAL CHECK**

7.3.1 Functionally check calibration curve prior to sample analysis.

7.3.2 Prepare 2000 ppm Boron, 1.0 ppm Chloride standard from a separate reagent manufacturer than that used for calibration.

7.3.3 Place sample pickup tube into functional check standard.

7.3.4 Load "PASMFC.SCH" into IC computer as follows: (2000 ppm Boron, 1.0 ppm Chloride Functional Check.)

a. Place mouse on "RUN" icon in Dionex AI-450 window under Program Manager and press left clicker twice rapidly. An hour glass should appear (wait).

b. Place arrow on "LOAD" and press left clicker once.

c. Place arrow on "SCHEDULE" and press left clicker once.

d. Place arrow on "PASMFC.SCH" and press left clicker twice.

e. Place arrow on "OK" and press left clicker once.

f. Place arrow on "RUN" and press left clicker once.

g. Place arrow on "START" and press left clicker once.

h. Place arrow on "OK" and press left clicker once. (This starts sample run which will take about 20 minutes.)

i. Edit completed chromatograph as necessary per Attachment 2.

7.3.5 Functional check results shall be 1900 ppm to 2100 ppm for Boron, 0.9 ppm to 1.1 ppm for the Chloride Functional Check. If a functional check fails, repeat it. If the functional check fails after three (3) times, notify OSC Supervisor.

7.3.6 Record functional check in accordance with Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheets."

7.4 **SAMPLE ANALYSIS**

7.4.1 Place a weight on sink water foot pedal to allow flow of flush water to sink.

7.4.2 Load "PASM.SCH" into IC as follows:

a. Place arrow on "LOAD" in run window and press left clicker once.

b. Place arrow on "SCHEDULE" and press left clicker once.

c. Place arrow on "PASM.SCH" and press left clicker twice.

d. Place arrow on "OK" and press left clicker once.

e. Place arrow on "RUN" and press left clicker once.

f. Place arrow on "START" and press left clicker once.

7.4.3 Insert sample pickup tube into the undiluted PASM Cask and puncture the sample septum with the pickup needle.

7.4.4 Make sure pickup needle is fully inserted to bottom of sample vial by rotating needle one full turn while maintaining downward pressure.

7.4.5 Place arrow on "OK" and press left clicker once. (This starts sample run which will take about 25 minutes.)

TITLE: BORON; CHLORIDE ION CHROMATOGRAPHY METHOD POST ACCIDENT

WARNING

Failure to leave the laboratory during PASM analysis may result in overexposure.

7.4.6 Leave area while IC analyzes sample to reduce exposure.

7.4.7 Record results on Attachment 1 of Emergency Implementing Procedure EI-7.2, "Emergency Post Accident Analysis."

7.5 **SAMPLE FLUSH**

NOTE: Attachment 6, "Dionex Method Parameters - RINSE.MET," is a copy of the rinse method.

7.5.1 Remove sample pickup tube from undiluted PASM cask.

7.5.2 Obtain the 5 cc shielded aliquator with needle.

7.5.3 Draw 5 cc of DI water into aliquator syringe.

7.5.4 Inject 5 cc of DI water into undiluted sample vial in PASM cask.

7.5.5 Repeat process to inject an additional 5 cc of DI water for a total of 10 cc into PASM sample vial.

7.5.6 Insert sample pickup tube into the undiluted PASM cask and puncture the sample septum with the pickup needle.

7.5.7 Make sure pickup needle is fully inserted to bottom of sample vial by rotating needle one full turn while maintaining downward pressure.

7.5.8 Load and run "RINSE.MET" as follows:

a. Place arrow on "Load" in run window and press left clicker once.

b. Place arrow on "Method" and press left clicker once.

c. Place arrow on "RINSE.MET" and press left clicker twice.

d. Place arrow on "Ok" and press left clicker once.

e. Place arrow on "Run" and press left clicker once.

f. Place arrow on "Start" and press left clicker once.

g. Place arrow on "Ok" and press left clicker once. This starts the sample load pump and runs for 5 minutes. Sample will be pumped out to waste sink.

h. Verify sample pump primed and operational by observing discharge hose for flow. Prime pump per Attachment 5 as necessary.

i. Leave the area while sample is being pumped out to waste to reduce exposure.

7.5.9 Repeat Steps 7.5.1 through 7.5.8 as directed by Radiation Services to reduce dose rates prior to sample vial removal from cask.

7.5.10 Remove vial from cask (use of remote handling tools may be necessary as directed by Radiation Services).

7.5.11 Place vial in shielded storage area.

7.5.12 Edit completed chromatograph for PASM sample as necessary per Attachment 2.

7.6 **ACCEPTANCE CRITERIA**

Functional Check result shall be 1900 ppm to 2100 ppm for Boron, 0.9 ppm to 1.1 ppm for the Chloride Functional Check Standard.

TITLE: BORON; CHLORIDE ION CHROMATOGRAPHY METHOD POST ACCIDENT

8.0 ATTACHMENTS AND RECORDS

8.1 ATTACHMENTS

8.1.1 Attachment 1, "PASM Schedules"

8.1.2 Attachment 2, "Editing Chromatographs (Optimize)"

8.1.3 Attachment 3, "Standard and Reagent Preparation"

8.1.4 Attachment 4, "Dionex Method Parameters - PASMCL.MET"

8.1.5 Attachment 5, "Priming Sample Load Pump"

8.1.6 Attachment 6, "Dionex Method Parameters - RINSE.MET"

8.2 RECORDS

Records generated by this procedure shall be filed in accordance with Palisades Administrative Procedure 10.46, "Plant Records."

9.0 SPECIAL REVIEWS

None

PASM SCHEDULES

DIONEX SCHEDULE - C:\DX\SCHEDULE\PASM1.SCH

Inj#	Sample Name	Method	Data File		Vol	Dil	Int Std
1	AUTOCAL1		..\PASMCL.m	..\PASM	1	1	1
2			..\HALT.met	..\PASM	1	1	1

DIONEX SCHEDULE - C:\DX\SCHEDULE\PASM2.SCH

Inj#	Sample Name	Method	Data File		Vol	Dil	Int Std
1	AUTOCAL2		..\PASMCL.m	..\PASM	1	1	1
2			..\HALT.met	..\PASM	1	1	1

DIONEX SCHEDULE - C:\DX\SCHEDULE\PASM3.SCH

Inj#	Sample Name	Method	Data File		Vol	Dil	Int Std
1	AUTOCAL3		..\PASMCL.m	..\PASM	1	1	1
2			..\HALT.met	..\PASM	1	1	1

DIONEX SCHEDULE - C:\DX\SCHEDULE\PASM4.SCH

Inj#	Sample Name	Method	Data File		Vol	Dil	Int Std
1	AUTOCAL4		..\PASMCL.m	..\PASM0291.D01	1	1	1
2			..\HALT.met	C:\DX\DATA\PASM	1	1	1

DIONEX SCHEDULE - C:\DX\SCHEDULE\PASM.SCH

Inj#	Sample Name	Method	Data File		Vol	Dil	Int Std
1	PASM F.C.		..\PASMCL.m	..\PASM	1	1	1
2			..\HALT.met	..\PASM	1	1	1

DIONEX SCHEDULE - C:\DX\SCHEDULE\PASM.SCH

Inj#	Sample Name	Method	Data File		Vol	Dil	Int Std
1	PASMBORON,CHLORIDE		..\PASMCL.m	..\PASM0191.DO1	1	1	1
2	RINSE		..\RINSE.me	..\PASM0192.DO1	1	1	1
3			..\HALT.met	C:\DX\DATA\PASM	1	1	1

EDITING CHROMATOGRAPHS (OPTIMIZE)

1.0 Check each chromatograph to determine if editing is necessary by:

a. Check each peak of interest to see if baselines start at beginning of the peak and end at end of the peak. If baseline editing is desired, go to Section 2.0.

b. Check peaks of interest to see each is correctly identified by retention time. If a peak is misidentified or identified but not named, go to Section 3.0 to edit retention times.

c. Check all peaks to see if they are identified as actual peaks. Each peak should have an associated peak number printed on the chromatograph. If there are peaks present but no peak numbers given, go to Section 4.0 to edit peak integration.

2.0 EDITING BASELINES

2.1 To open "OPTIMIZE.EXE," place arrow on the "OPTIMIZE" icon in Dionex AI-450 window under Program Manager and press left clicker twice.

2.2 Place arrow on "FILE" and press left clicker once.

2.3 Place arrow on "OPEN DATA FILE..." and press left clicker once.

2.4 Place arrow on [-C-] and press left clicker twice. Sample data and standards data is stored in "C" drive.

2.5 Find data file to be edited. The file number is located at top of chromatograph printout.

2.6 Place arrow on data file to be edited and press left clicker once.

2.7 Place arrow on "OK" and press left clicker once. Data file will appear on screen.

2.8 Place arrow on "OPERATIONS" and press left clicker once.

2.9 Place arrow on "SET BASELINES MANUALLY" and press left clicker once.

2.10 Place arrow on peak number to be edited (located in top left window), and press left clicker twice. This will cause desired peak to appear in the right window.

EDITING CHROMATOGRAPHS (OPTIMIZE)

2.11 Place the mouse cursor on desired start of peak and press right clicker once.

2.12 Place arrow on "MOVE PEAK START" under "OPERATIONS" and press left clicker once.

2.13 Place cursor on desired end of peak and press right clicker once.

2.14 Place arrow on "MOVE PEAK END" under "OPERATIONS" and press left clicker once.

2.15 Repeat Steps 2.10 through 2.14 until all desired baseline changes for that chromatograph are completed.

2.16 Place arrow on "FILE" and press left clicker once.

2.17 Place arrow on "VIEW REPORT..." and press left clicker once; this will show the edited report. Corrected results can be taken from the concentration column.

2.18 Exit by pressing left clicker on top Control Menu Box twice.

2.19 Exit editing by pressing left clicker on top Control Menu Box twice.

2.20 Exit Optimize by pressing left clicker on top Control Menu Box twice.

2.21 Place arrow on "YES" and press left clicker once to save chromatograph changes.

2.22 Place arrow on "OK" and press left clicker once to save data. Program Manager should be displayed.

3.0 EDITING RETENTION TIMES

3.1 To open "OPTIMIZE.EXE" place arrow on the "OPTIMIZE" icon in Dionex AI-450 window under Program Manager and press left clicker twice.

3.2 Place arrow on "FILE"" and press left clicker once.

3.3 Place arrow on "OPEN DATA FILE..." and press left clicker once.

3.4 Place arrow on [-C-] and press left clicker twice. Sample data and standards data is stored in "C" drive.

EDITING CHROMATOGRAPHS (OPTIMIZE)

3.5 Find data file to be edited. The file number is located at top of chromatograph printout.

3.6 Place arrow on data file to be edited and press left clicker once.

3.7 Place arrow on "OK" and press left clicker once. Data file will appear on screen.

3.8 Place arrow on "EDIT" and press left clicker once.

3.9 Place arrow on "COMPONENTS" and press left clicker once.

3.10 Place arrow on retention time for component to be changed and press left clicker once.

3.11 Key in new retention time and press "ENTER" on keyboard.

3.12 Continue with Steps 3.10 and 3.11 until all desired retention times are entered.

3.13 Place arrow on "EXIT" and press left clicker once.

3.14 Place arrow on "FILE" and press left clicker once.

3.15 Place arrow on "VIEW REPORT" and press left clicker once. Report will be displayed with new retention times. Results can be obtained from concentration column or printed.

3.16 Place arrow on top Control Menu Box and press left clicker twice.

3.17 Place arrow on top Control Menu Box and press left clicker twice.

4.0 EDITING PEAK INTEGRATION

4.1 To open "OPTIMIZE.EXE," place arrow on the "OPTIMIZE" icon in Dionex AI-450 window under Program Manager and press left clicker twice.

4.2 Place arrow on "FILE" and press left clicker once.

4.3 Place arrow on "OPEN DATA FILE..." and press left clicker once.

4.4 Place arrow on [-C-] and press left clicker twice. Sample data and standards data is stored in "C" drive.

EDITING CHROMATOGRAPHS (OPTIMIZE)

4.5 Place arrow on data file number located in upper box that editing is desired. The data file number is located at top of chromatograph. Press the left clicker once.

4.6 Place arrow on "OK" and press the left clicker once.

4.7 Place arrow on "OPERATIONS" and press left clicker once.

4.8 Place arrow on "ADJUST INTEGRATION" and press left clicker once.

4.9 Placing arrow on wider or narrower in peak width box and pressing left clicker will change peak width.

4.10 Placing arrow on "RE-INTEGRATE" will cause reintegration with the new peak width. After reintegration, view bottom chromatograph to see if unidentified peak is now identified.

4.11 Repeat Steps 4.9 and 4.10 until unidentified peak is identified and numbered.

4.12 Place arrow on "FILE" and press clicker once.

4.13 Place arrow on "VIEW REPORT..." and press left clicker once. If peak is identified correctly, results can be obtained from concentration column.

4.14 Place arrow on Control Menu Box at top of window and press left clicker twice.

4.15 Place arrow on Control Menu Box and press left clicker twice.

4.16 Place arrow on Control Menu Box and press left clicker twice.

4.17 A note will come on the screen asking if you want to save the changed method, place arrow on "NO" and click once.

4.18 A note will come on the screen asking if you want to save changes made to file, place arrow on "YES" and click once. (Program Manager should be at the bottom left of the screen.)

STANDARD AND REAGENT PREPARATION

1.0 **Standard Anion Eluent Gradient System With Self-Regenerating Suppressor (0.07M $Na_2B_4O_7$)**

Into a 1 liter volumetric flask containing approximately 500 ml DDW, transfer 17.3 g Boric Acid (H_3BO_3) and 7.3 mls of 50% Sodium Hydroxide solution. Dilute to mark with DDW and mix by inversion. Label in accordance with Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheets." Shelf life is one year.

2.0 **1000 ppm Chloride Standard**

Dissolve 1.6484 g (1.6482 g to 1.6486 g) Sodium Chloride (NaCl) in a one liter container containing approximately 500 g DI water. Dilute to 1000 g total weight with DI water. Mix by inversion. Label in accordance with Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheets." Shelf life is one year.

NOTE: Use of an antistatic device will aid in the weighing of the boric acid.

3.0 **100 ppm Chloride Standard**

Weigh 100 g of the 1000 ppm Chloride Standard into a one liter container containing approximately 500 g DI water. Dilute to 1000 g total weight and mix by inversion. Label in accordance with Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheets." Shelf life is one year.

4.0 **500 ppm Boron Standard, 50 ppb Chloride Standard (Auto Cal 1)**

Dissolve 2.8595 g (2.8309 g to 2.8880 g) Boric Acid (H_3BO_3) in a one liter container containing approximately 500 g DI water. Pipet 0.5 g of the 100 ppm Chloride standard into the one liter container. Dilute to 1000 g total weight with DI water. Mix by inversion. Label in accordance with Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheet." Shelf life is one day.

STANDARD AND REAGENT PREPARATION

5.0 **2000 ppm Boron Standard, 1.0 ppm Chloride Standard (Auto Cal 2 and Functional Check)**

Dissolve 11.438 g (11.3236 g to 11.5524 g) Boric Acid (H_3BO_3) in a one liter container containing approximately 500 g DI water. Pipet 10 g of the 100 ppm Chloride standard into the one liter container. Dilute to 1000 g total weight with DI water. Mix by inversion. Label in accordance with Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheet." Shelf life is one month.

6.0 **4000 ppm Boron Standard, 10 ppm Chloride Standard (Auto Cal 3)**

Dissolve 22.876 g (22.6472 g to 23.1048 g) Boric Acid (H_3BO_3) in a one liter container containing approximately 500 g DI water. Pipet 10 g of the 1000 ppm Chloride standard into the one liter container. Dilute to 1000 g total weight with DI water. Mix by inversion. Label in accordance with Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheet." Shelf life is one month.

7.0 **6000 ppm Boron Standard, 20 ppm Chloride Standard (Auto Cal 4)**

Dissolve 34.314 g (33.9708 g to 34.6572 g) Boric Acid (H_3BO_3) in a one liter container containing approximately 500 g DI water. Pipet 20 g of the 1000 ppm Chloride standard into the one liter container. Dilute to 1000 g total weight with DI water. Mix by inversion. Label in accordance with Chemistry Procedure CH 1.5, "Operational Chemistry Logs, Records, Graphs, Labels, and Data Sheet." Shelf life is one month.

DIONEX METHOD PARAMETERS
PASMCL.MET

Method Comment:
Column ID:
Analyst ID:

System Parameters

System Name: Anions/Tetraborate System

Number of Detectors	1
Run Time (minutes)	8.50
Sampling Rate (seconds)	0.50
Detector 1 Type	CDM-2
Detector 1 Real Time Plot Scale Maximum (uS)	30.00
Minimum	-3.00
Save Data File	Yes

Data File Name: C:\DX\DATA\PASM.DXX

-- DETECTOR 1 PARAMETERS --

Report Options

Create ASCII Report File	No
Print Report	Yes
Print All Components	Yes
Print Components Found	No
Print Missing Components	No
Print All Peaks	Yes
Print Unknown Peaks	No
Print Chromatogram	Yes
Autoscale Chromatogram Maximum	Yes
Autoscale Maximum Value Delay (minutes)	0.0
Autoscale Chromatogram Minimum	Yes
Autoscale Minimum Value Delay (minutes)	0.0
Fill peaks with Color	No
Draw Grid Lines on Chromatogram	No
Show Component Fraction Numbers	No
Label with Peak Number	No
Label with Retention Times on Chromatogram	Yes
Label with Component Name	Yes

Format File Name: C:\DX\METHOD\DEFAULT.PRF

Integration Parameters

Starting Peak Width (seconds)	5.0
Peak Threshold	0.200
Peak Area Reject	5
Area Reject for Reference Peaks	0

DIONEX METHOD PARAMETERS
PASMCL.MET

Data Events

Time	Description
0.00	Force baseline at start of all peaks

Calibration Parameters

Number of Levels for Calibration	4
Force Calibration Curve Through Origin	No
Calibration Fit Type	Quadratic
Replace or Average Calibrations	Replace
External or Internal Calibration	External
Calculate Unknowns by Area or Height	Area
Default Sample Volume	1.0
Default Dilution Factor	1.0
Default Response Factor for Unknown Peaks	0.0
Calibration Standard Volume	1.0
Internal Standard Amount in Samples	1.0
Amount Units	ppm

DIONEX METHOD PARAMETERS
PASMCL.MET

Component Table -- Last Modified: 14:33 on Thu, 06 June 1996

Component #1 BORON Retention Time 1.18
Reference Comp none Window Size 10.00%
Amount = K0 + K1*Area
K0 = 4.39531E+002
K1 = 8.25493E-006

Level	Amount	Area	Height
1	5.00000E+002	28709195	458954
2	2.00000E+003	178717115	4412541
3	4.00000E+003	399996575	12551485
4	5.00000E+003	679308810	23167358

Component #2 CHLORIDE Retention Time 6.56
Reference Comp none Window Size 10.00%
Amount = K0 + K1*Area
K0 = -7.55744E-001
K1 = 1.27953E-009

Level	Amount	Area	Height
1	1.00000E-001	149141900	18478590
2	1.00000E+000	1927711550	117800175
3	1.00000E+001	8460031850	289706788
4	2.88000E+001	16131423825	406937831

Timed Events File: C:\DX\METHOD\PASMCL.TE

Step	Time	Description
Init		CDM-2 AutoOffset Off
Init		CDM-2 Recorder Mark OFF
Init		CDM-2 Temp Comp = 1.7 / Deg C
Init		CDM-2 Recorder Range = 1.00 uS
Init		CDM-2 Cell ON
Init		CHA Heater = 25 / Deg C
Init		Valve A ON
Init		Valve B ON
Init		Inject Valve OFF
Init		ACI Autosmp OFF
Init		ACI RLY 3 OFF
Init		ACI RLY 4 OFF
Init		ACI as SRS ON
Init		ACI PUMP OFF
Init		GPM Start

DIONEX METHOD PARAMETERS
PASMCL.MET

Init		GPM Hold Gradient Clock
Init		GPM Reset ON
1	0.1	GPM Run Gradient Clock
1	0.1	GPM Reset OFF
2	8.1	ACI PUMP ON
3	8.7	ACI PUMP OFF
4	9.2	CDM-2 AutoOffset ON
4	9.2	Start Samplin

Lo Pressure Limit = 20
Hi Pressure Limit = 2000
Eluent 1 - 0.07 M Tetraborate
Eluent 2 - Double Deionized Water
Eluent 3 -
Eluent 4 -
V5 Off - Off
V5 On - On
V6 Off - Off
V6 On - On

Time	Flow	%1	%2	%3	%4	V5	V6	Comment
0.0	2.0	40	60	0	0	1	0	Start Of Column Flush
3.0	2.0	40	60	0	0	1	0	End Of Column Flush
3.1	2.0	0	100	0	0	1	0	Rinse Separator Column
7.8	2.0	0	100	0	0	0	0	Start Loop Load
9.1	2.0	0	100	0	0	1	0	Inject Start Gradient
19.6	2.0	40	60	0	0	1	0	Gradient
29.6	2.0	40	60	0	0	1	0	Gradient

PRIMING SAMPLE LOAD PUMP

1. Toggle sample load pump off.
2. Unplug sample load pump from ACI switched outlet #2.
3. Plug sample load pump into a power outlet strip.
4. Remove sample needle from suction line.
5. Attach a luer loc fitting to suction line.
6. Ensure discharge tube is routed to a waste container.
7. Attach a 5 cc syringe filled with DI water to suction side luer loc fitting.
8. Apply pressure to syringe plunger and toggle pump on.
9. Observe discharge tube for water flow. When discharge flow is observed, toggle pump switch off.
10. Remove syringe from luer loc fitting. Pump is now primed.
11. Unplug pump from power outlet strip.
12. Remove luer loc fitting from suction line and reattach sample needle.
13. Plug pump into ACI switched outlet #2.
14. Toggle pump power switch to on.

DIONEX METHOD PARAMETERS
RINSE.MET

Method Comment:
Column ID:
Analyst ID:

System Parameters

System Name: Anions/Tetraborate System

Number of Detectors	1
Run Time (minutes)	6.00
Sampling Rate (seconds)	0.50
Detector 1 Type	CDM-2
Detector 1 Real Time Plot Scale Maximum (uS)	30.00
Minimum	-3.00
Save Data File	Yes

Data File Name: C:\DX\DATA\PASM.DXX

-- DETECTOR 1 PARAMETERS --

Report Options

Create ASCII Report File	No
Print Report	Yes
Print All Components	Yes
Print Components Found	No
Print Missing Components	No
Print All Peaks	Yes
Print Unknown Peaks	No
Print Chromatogram	Yes
Autoscale Chromatogram Maximum	Yes
Autoscale Maximum Value Delay (minutes)	0.0
Autoscale Chromatogram Minimum	Yes
Autoscale Minimum Value Delay (minutes)	0.0
Fill peaks with Color	No
Draw Grid Lines on Chromatogram	No
Show Component Fraction Numbers	No
Label with Peak Number	No
Label with Retention Times on Chromatogram	Yes
Label with Component Name	Yes

Format File Name: C:\DX\METHOD\DEFAULT.PRF

Integration Parameters

Starting Peak Width (seconds)	0.0
Peak Threshold	0.000
Peak Area Reject	0
Area Reject for Reference Peaks	0

DIONEX METHOD PARAMETERS
RINSE.MET

Calibration Parameters

Number of Levels for Calibration	1
Force Calibration Curve Through Origin	No
Calibration Fit Type	Quadratic
Replace or Average Calibrations	Replace
External or Internal Calibration	External
Calculate Unknowns by Area or Height	Area
Default Sample Volume	1.0
Default Dilution Factor	1.0
Default Response Factor for Unknown Peaks	0.0
Calibration Standard Volume	1.0
Internal Standard Amount in Samples	1.0
Amount Units	ppb

Timed Events File: C:\DX\METHOD\PASMCL.TE

Step	Time	Description
Init		CDM-2 AutoOffset Off
Init		CDM-2 Recorder Mark OFF
Init		CDM-2 Temp Comp = 1.7 / Deg C
Init		CDM-2 Recorder Range = 1.00 uS
Init		CDM-2 Cell OFF
Init		CHA Heater = 25 / Deg C
Init		Valve A ON
Init		Valve B ON
Init		Inject Valve OFF
Init		ACI Autosmp OFF
Init		ACI RLY 3 OFF
Init		ACI RLY 4 OFF
Init		ACI as SRS OFF
Init		ACI PUMP OFF
Init		GPM Start
Init		GPM Hold Gradient Clock
Init		GPM Reset ON
1	0.1	ASI PUMP ON
2	5.0	GPM Reset OFF
2	5.0	GPM Stop
2	5.0	GPM Reset OFF
4	9.2	CDM-2 AutoOffset ON

DIONEX METHOD PARAMETERS
RINSE.MET

Lo Pressure Limit = 20
Hi Pressure Limit = 2000
Eluent 1 - 0.07 M Tetraborate
Eluent 2 - Double Deionized Water
Eluent 3 -
Eluent 4 -
V5 Off - Off
V5 On - On
V6 Off - Off
V6 On - On

Time	Flow	%1	%2	%3	%4	V5	V6	Comment
0.0	0.0	0	100	0	0	1	0	Start Of Column Flush