

10CFR50.59 FORMAT FOR SAFETY EVALUATION

STATION CASALE COUNTY STATION UNIT 0  
SYSTEM PS TEST/PROCEDURE NO. LP1330-20 REV. 1  
TEST/PROCEDURE TITLE POST ACCIDENT ANALYSIS OF BORON  
EQUIPMENT No. N/A

DESCRIPTION OF TEST/PROCEDURE

Method of determining Boron Conc. in Reactor H<sub>2</sub>O following accident.

SAFETY EVALUATION: Answer the following questions with a "yes" or "no", and provide specific reasons justifying the decision:

1. Is the probability of an occurrence or the consequence of an accident, or malfunction of equipment important to safety as previously evaluated in the Final Safety Analysis Report increased? Yes X No, Because:

*No only deleted a reference to a deleted procedure*

2. Is the possibility for an accident or malfunction of a different type than any previously evaluated in the Final Safety Analysis Report created? Yes X No, Because:

*No only deleted reference to a deleted procedure*

3. Is the margin of safety, as defined in the basis for any Technical Specification, reduced? Yes X No, Because:

*No only deleted reference to a deleted procedure*

8308310478 830825  
PDR ADOCK 05000373  
F PDR

Performed By

*Be Knoll*

Date

*2/17/83*

Approved By

*W. H. Hume*

Date

*4/1/80*

X005  
111

PROCEDURE HISTORY AND SAFETY EVALUATION  
(10 CFR 50.59)

Does this procedure/revision constitute a change to the procedure as described in the Final Safety Analysis Report?

Yes ( )

No (X)

Is a change in the Technical Specifications involved?

No ( )

SAFETY EVALUATION: Answer the following questions with a "yes" or "no", and provide specific reasons justifying the decision:

1. Is the probability of an occurrence, the consequence of an accident, or the malfunction of safety-related equipment, as previously evaluated in the Final Safety Analysis Report, increased?

Yes

No (X), because:

2. Is the possibility for an accident or the malfunction of a different type than any previously evaluated in the Final Safety Analysis Report created?

Yes

No (X), because:

3. Is the margin of safety, as defined in the basis for any Technical Specification, reduced?

Yes

No (X), because:

Any Answer \* Yes ( )

All Answers No (X)

Request and receive Nuclear Regulatory Commission authorization for change.

Authorization Received ( )

Initiate Procedure Implementation

\* NOTE:  
Any answer checked "yes" should be reported in the annual report to the NRC.

Performed by

Date

*Ref Knoll*

*2/17/83*

7/06/83

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P 1330-24	DETERMINATION OF REACTOR COOLANT HYDROGEN AND OFF-GAS CONCENTRATIONS AT THE HIGH RADIATION SAMPLING SYSTEM	C2	1/83	03

POST-ACCIDENT ANALYSIS OF BORON  
(Backup Procedure)

A. PURPOSE

The purpose of this procedure is to delineate an alternate method for determining boron following a Regulatory Guide 1.3 or 1.4 release of fission products. The method described by this procedure is referenced to as the Curcumin Spectrophotometric Method.

B. REFERENCES

1. Final Report, Analyses for Dissolved Hydrogen, Dissolved Oxygen, Boron, Chloride, pH, and conductivity under normal Post-Accident Conditions, NUS Corporation, October, 1980.
2. LCP 110-2, "Preparation of Reagents".
3. LCP 210-6, "Operation of the Klett-Summerson Colorimeter".

C. PREREQUISITES

1. Equipment:
  - a. Klett-Summerson Colorimeter.
  - b. Filter No. 54.
  - c. 20 mm X 40 mm cells.
  - d. Graduated cylinders, 10 or 25 ml and 500 ml.
  - e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
  - f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
  - g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
  - h. Beakers 250 ml or 400 ml, wide form.



- i. Tongs, long reach.
  - j. Polyethylene stirring rods, 7.
  - k. Medicine droppers, 7.
  - l. Filter paper, Whatman No. 30, or equivalent.
  - m. Glass or plastic funnel.
  - n. Semi-logarithmic paper, dual cycle.
  - o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.
2. Reagents:
- a. 95% Isopropyl alcohol.
  - b. Curcumin reagent.
  - c. Boron stock standard solution, 1000 ppm boron.
  - d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

C. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. Wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.

4. Pipette 1.0 ml of DD1 water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^\circ\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.

14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis), refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

ATTACHMENT A  
 LASALLE COUNTY STATION  
 Post-Accident Analysis of Boron

LZP 1330-20  
 Revision 1  
 July 26, 1983  
 7

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)



ATTACHMENT B

LZP-1330-20

Revision 1

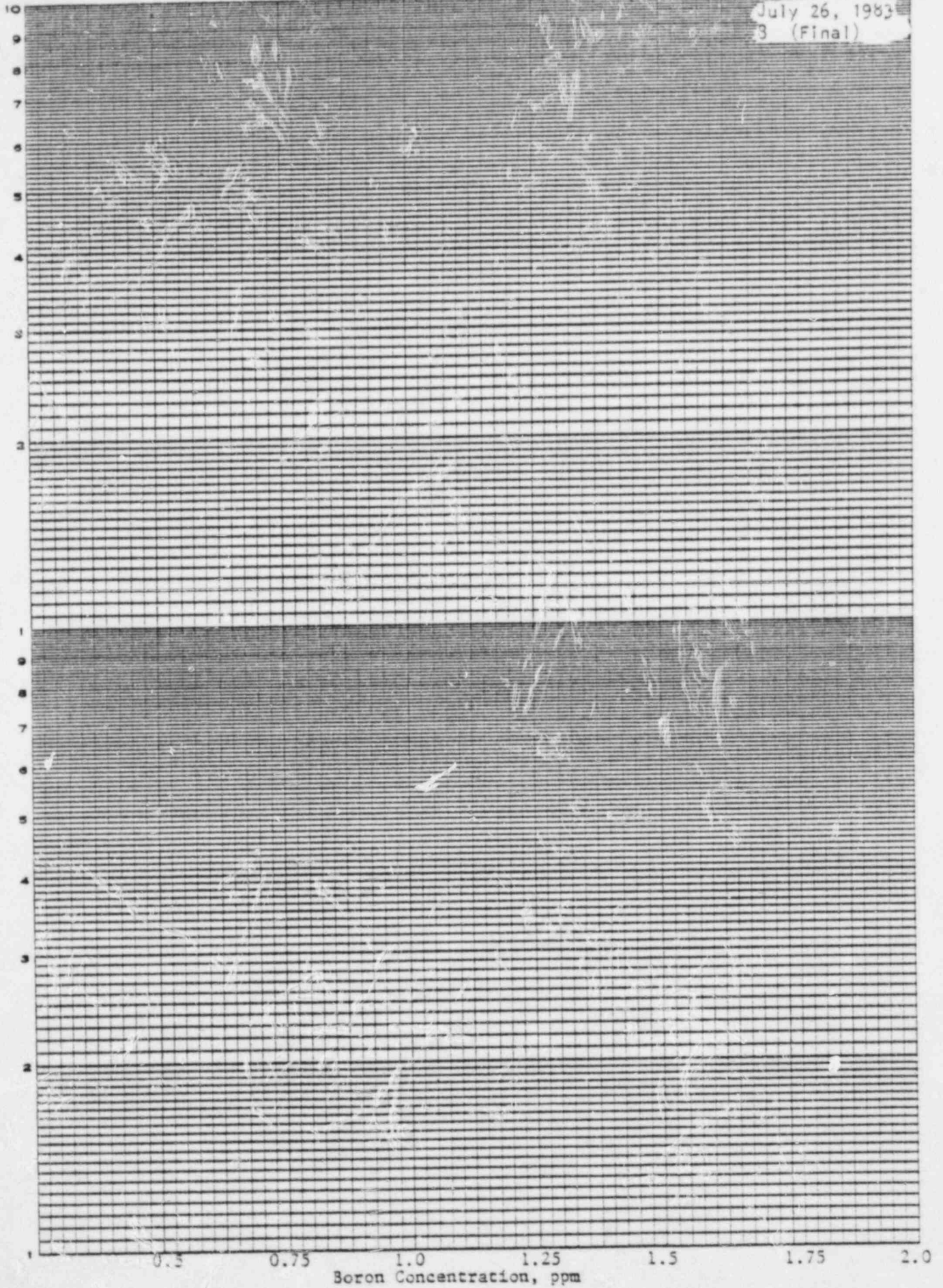
July 26, 1983

3 (Final)

DIETZEN CORPORATION  
MADE IN U.S.A.

NO. 341-L210 DIETZEN GRAPH PAPER  
SEMI-LOGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH

ABSORBANCE



7/08/83

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P 1220-1	EMERGENCY TELEPHONE NUMBER	C1	5/81	01
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A. PURPOSE

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1. Equipment:
  - a. Klett-Summerson Colorimeter.
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  - c. 20 mm X 40 mm cells.
  - d. Graduated cylinders, 10 or 25 ml and 500 ml.
  - e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
  - f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
  - g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
  - h. Beakers 250 ml or 400 ml, wide form.

- i. Tongs, long reach.
- j. Polyethylene stirring rods, 7.
- k. Medicine droppers, 7.
- l. Filter paper, Whatman No. 30, or equivalent.
- m. Glass or plastic funnel.
- n. Semi-logarithmic paper, dual cycle.
- o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.

2. Reagents:

- a. 95% Isopropyl alcohol.
- b. Curcumin reagent.
- c. Boron stock standard solution, 1000 ppm boron.
- d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

C. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. Wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. Wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.



4. Pipette 1.0 ml of DD1 water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
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CAUTION

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6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^{\circ}\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.



14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis), refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

LZP 1330-20  
Revision 1  
July 26, 1983  
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ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)

# ATTACHMENT 3

LZP-1330-20

Revision 1

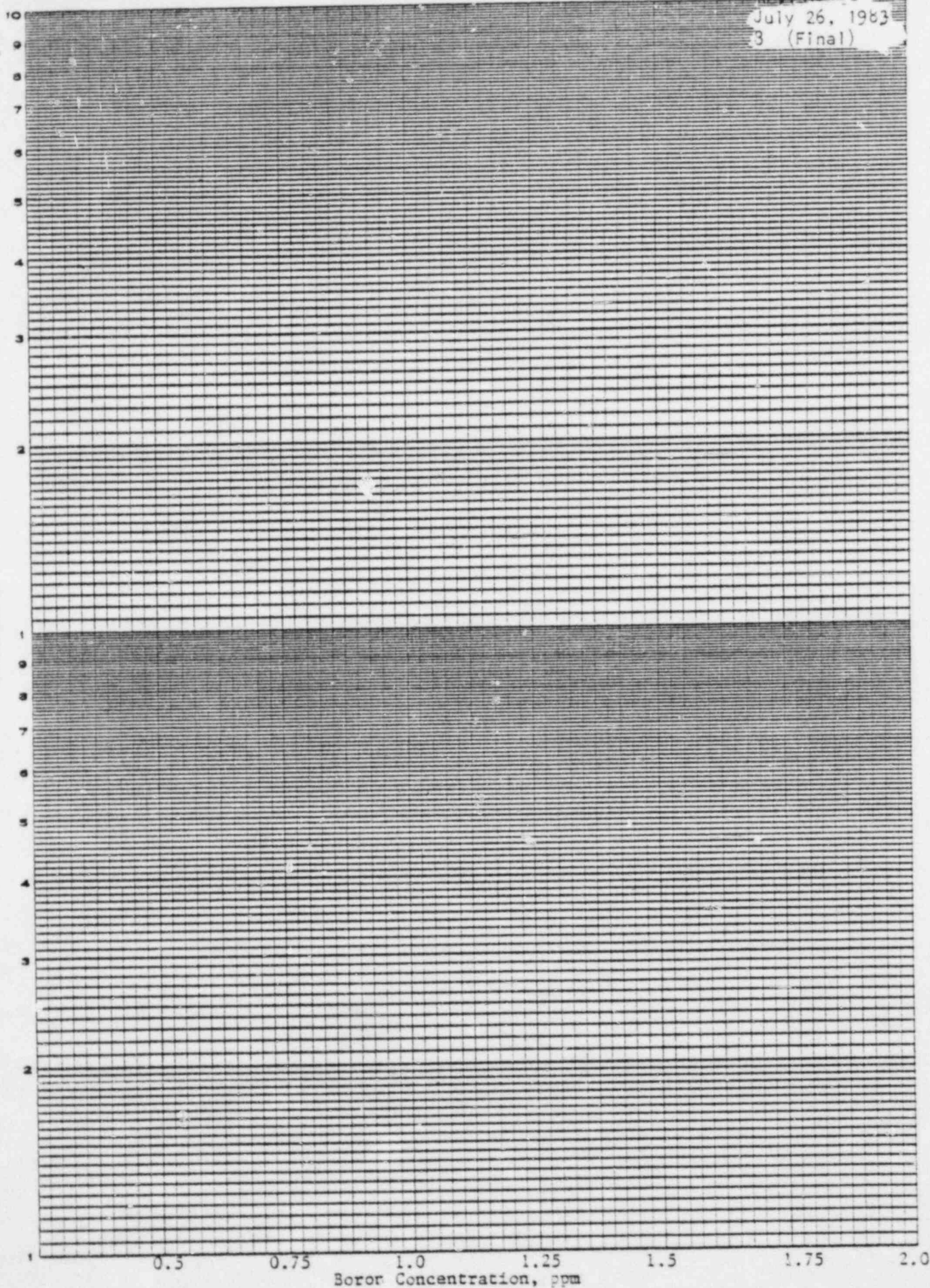
July 26, 1983

3 (Final)

DIETZEN CORPORATION  
MADE IN U.S.A.

NO. 3-1-L21D DIETZEN GRAPH PAPER  
SEMI-LOGARITHMIC  
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7/CB/83

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  - c. 20 mm X 40 mm cells.
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  - e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
  - f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
  - g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
  - h. Beakers 250 ml or 400 ml, wide form.



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- c. Boron stock standard solution, 1000 ppm boron.
- d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

D. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.

4. Pipette 1.0 ml of DDI water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^{\circ}\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.

14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis), refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

LZP 1330-20  
Revision 1  
July 26, 1983  
7

ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)



# ATTACHMENT 3

LZP-1330-20

Revision 1

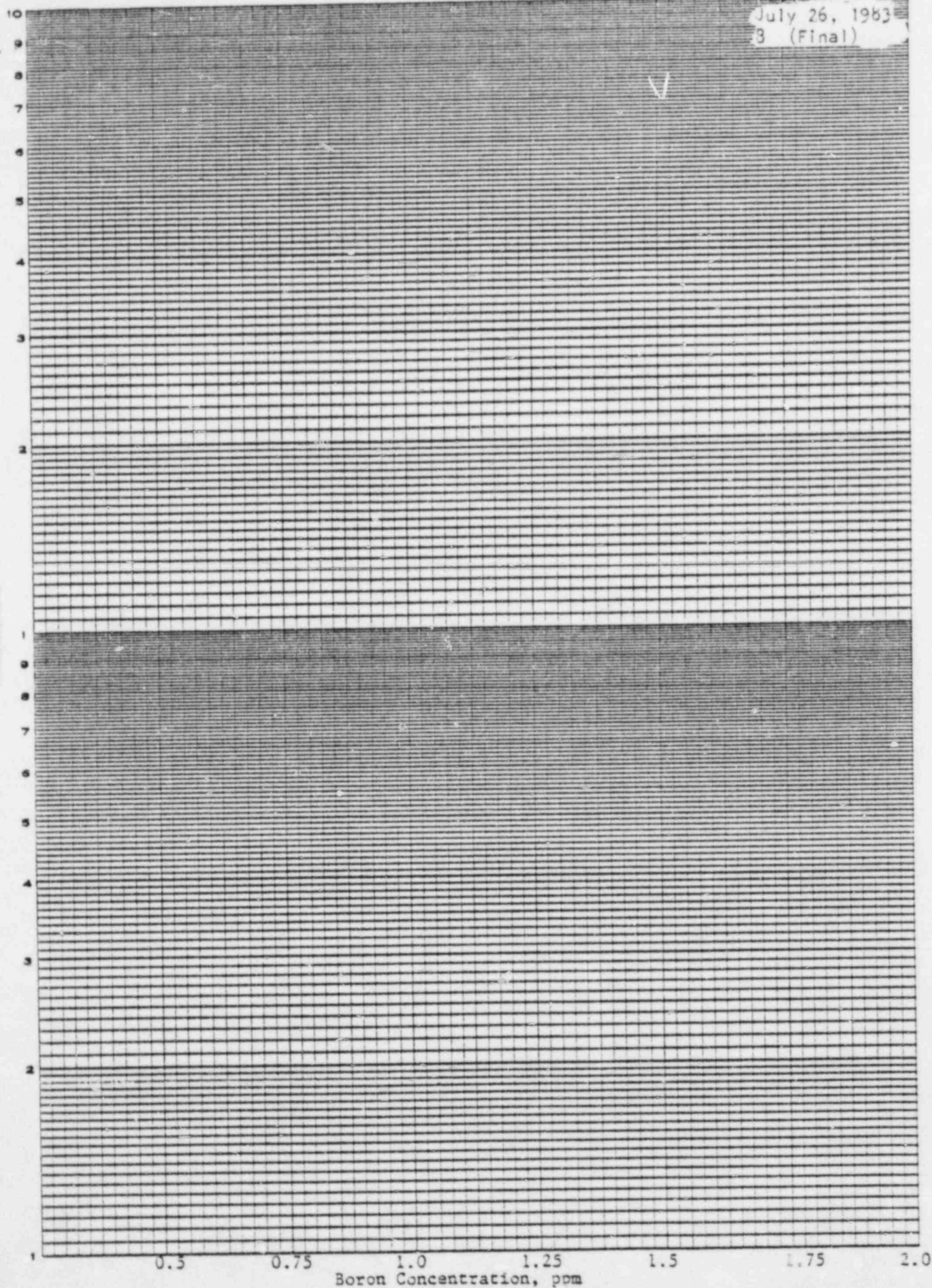
July 26, 1983

3 (Final)

DIETZEN CORPORATION  
MADE IN U.S.A.

NO. 341-L210 DIETZEN GRAPH PAPER  
SEMI-LOGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH

ABSORBANCE



7/08/83

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1P 1220-1	EMERGENCY TELEPHONE NUMBER	C1	5/81	01
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1P 1330-3	DELETED	C3	7/82	03
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1P 1330-22	CALIBRATION OF THE MODEL 10 DIONEX ION CHROMATOGRAPH	C2	4/82	03
1P 1330-23	DETERMINATION OF REACTOR COOLANT PH, CONDUCTIVITY AND DISSOLVED OXYGEN CONCENTRATION AT THE HIGH RADIATION SAMPLE SYSTEM	C2	6/82	03
1P 1330-24	DETERMINATION OF REACTOR COOLANT HYDROGEN AND OFF-GAS CONCENTRATIONS AT THE HIGH RADIATION SAMPLING SYSTEM	C2	1/83	03

POST-ACCIDENT ANALYSIS OF BORON  
(Backup Procedure)

A. PURPOSE

The purpose of this procedure is to delineate an alternate method for determining boron following a Regulatory Guide 1.3 or 1.4 release of fission products. The method described by this procedure is referenced to as the Curcumin Spectrophotometric Method.

B. REFERENCES

1. Final Report, Analyses for Dissolved Hydrogen, Dissolved Oxygen, Boron, Chloride, pH, and conductivity under normal Post-Accident Conditions, NUS Corporation, October, 1980.
2. LCP 110-2, "Preparation of Reagents".
3. LCP 210-6, "Operation of the Klett-Summerson Colorimeter".

C. PREREQUISITES

1. Equipment:
  - a. Klett-Summerson Colorimeter.
  - b. Filter No. 54.
  - c. 20 mm X 40 mm cells.
  - d. Graduated cylinders, 10 or 25 ml and 500 ml.
  - e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
  - f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
  - g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
  - h. Beakers 250 ml or 400 ml, wide form.

- i. Tongs, long reach.
- j. Polyethylene stirring rods, 7.
- k. Medicine droppers, 7.
- l. Filter paper, Whatman No. 30, or equivalent.
- m. Glass or plastic funnel.
- n. Semi-logarithmic paper, dual cycle.
- o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.

2. Reagents:

- a. 95% Isopropyl alcohol.
- b. Curcumin reagent.
- c. Boron stock standard solution, 1000 ppm boron.
- d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

C. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. Wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. Wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.



4. Pipette 1.0 ml of DDI water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^\circ\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.



14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis); refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

LZP 1330-20  
Revision 1  
July 26, 1983  
7

ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)

# ATTACHMENT B

LZP-1330-20

Revision 1

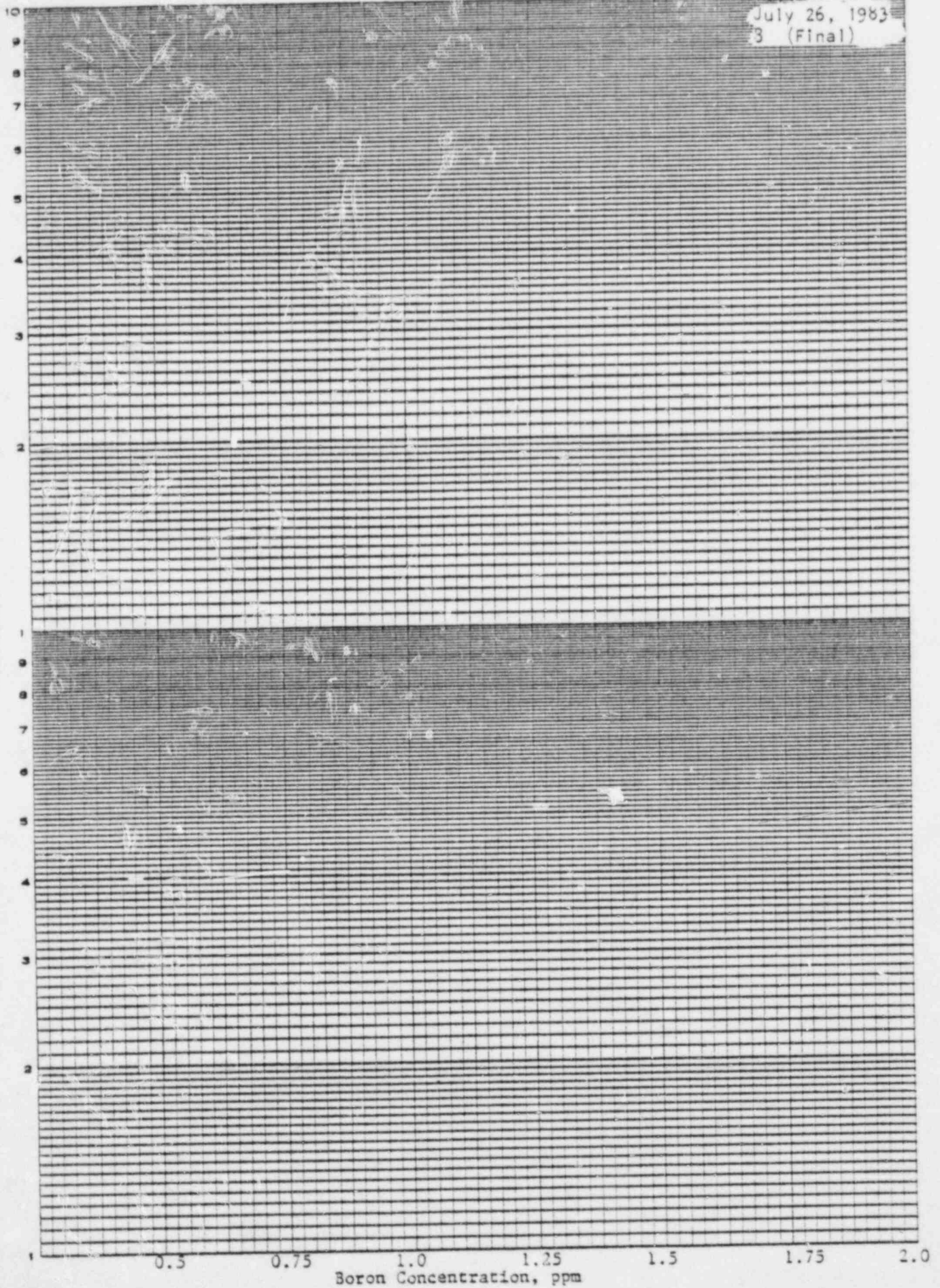
July 26, 1983

3 (Final)

DIETZEN CORPORATION  
MADE IN U.S.A.

THE 471 SEMI-LOGARITHMIC GRAPHING  
BENCH-LEGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH

ABSORBANCE



7/08/83

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LP 1220-1	EMERGENCY TELEPHONE NUMBER	01	5/81	01
LP 1220-4	DELETED CONTAINMENT ATMOSPHERE FOR RADIOIODINES DETERMINATION AND PARTICULATES ALTERNATE MEANS	01	7/81	01
LP 1310-1	NOTIFICATIONS	04	7/83	02
LP 1320-1	AUGMENTATION OF PLANT STAFFING	05	7/83	02
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LP 1330-3	DELETED	03	7/82	03
LP 1330-4	DELETED	02	7/82	03
LP 1330-5	POST-ACCIDENT NOBLE GAS RELEASE RATE DETERMINATION	00	11/80	03
LP 1330-6	DELETED	01	7/81	03
P 1330-7	DELETED	02	7/82	02
P 1330-8	SAMPLING LIQUID PROCESS MONITORS DURING LIQUID MONITOR HIGH RADIATION ALARM CONDITIONS	01	8/82	03
P 1330-10	DELETED	01	1/82	03
P 1330-11	DILUTION AND TRANSFER OF GAS SAMPLES	00	11/81	03
P 1330-20	POST-ACCIDENT ANALYSIS OF BAREN (BACKUP PROCEDURE)	00	3/81	03
P 1330-21	POST-ACCIDENT ANALYSIS OF CHLORIDE	03	6/82	03
P 1330-22	CALIBRATION OF THE MODEL 10 DIONEX ION CHROMATOGRAPH	02	4/82	03
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P 1330-24	DETERMINATION OF REACTOR COOLANT HYDROGEN AND OFF-GAS CONCENTRATIONS AT THE HIGH RADIATION SAMPLING SYSTEM	02	1/83	03



POST-ACCIDENT ANALYSIS OF BORON  
(Backup Procedure)

A. PURPOSE

The purpose of this procedure is to delineate an alternate method for determining boron following a Regulatory Guide 1.3 or 1.4 release of fission products. The method described by this procedure is referenced to as the Curcumin Spectrophotometric Method.

B. REFERENCES

1. Final Report: Analyses for Dissolved Hydrogen, Dissolved Oxygen, Boron, Chloride, pH, and conductivity under normal Post-Accident Conditions, NUS Corporation, October, 1980.
2. LCP 110-2, "Preparation of Reagents".
3. LCP 210-6, "Operation of the Klett-Summerson Colorimeter".

C. PREREQUISITES

1. Equipment:
  - a. Klett-Summerson Colorimeter.
  - b. Filter No. 54.
  - c. 20 mm X 40 mm cells.
  - d. Graduated cylinders, 10 or 25 ml and 500 ml.
  - e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
  - f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
  - g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
  - h. Beakers 250 ml or 400 ml, wide form.



- i. Tongs, long reach.
  - j. Polyethylene stirring rods, 7.
  - k. Medicine droppers, 7.
  - l. Filter paper, Whatman No. 30, or equivalent.
  - m. Glass or plastic funnel.
  - n. Semi-logarithmic paper, dual cycle.
  - o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.
2. Reagents:
- a. 95% Isopropyl alcohol.
  - b. Curcumin reagent.
  - c. Boron stock standard solution, 1000 ppm boron.
  - d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

D. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.

4. Pipette 1.0 ml of DD1 water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^{\circ}\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.

14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis); refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

LZP 1330-20  
Revision 1  
July 26, 1983  
7

ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)



# ATTACHMENT B

LZP-1330-20

Revision 1

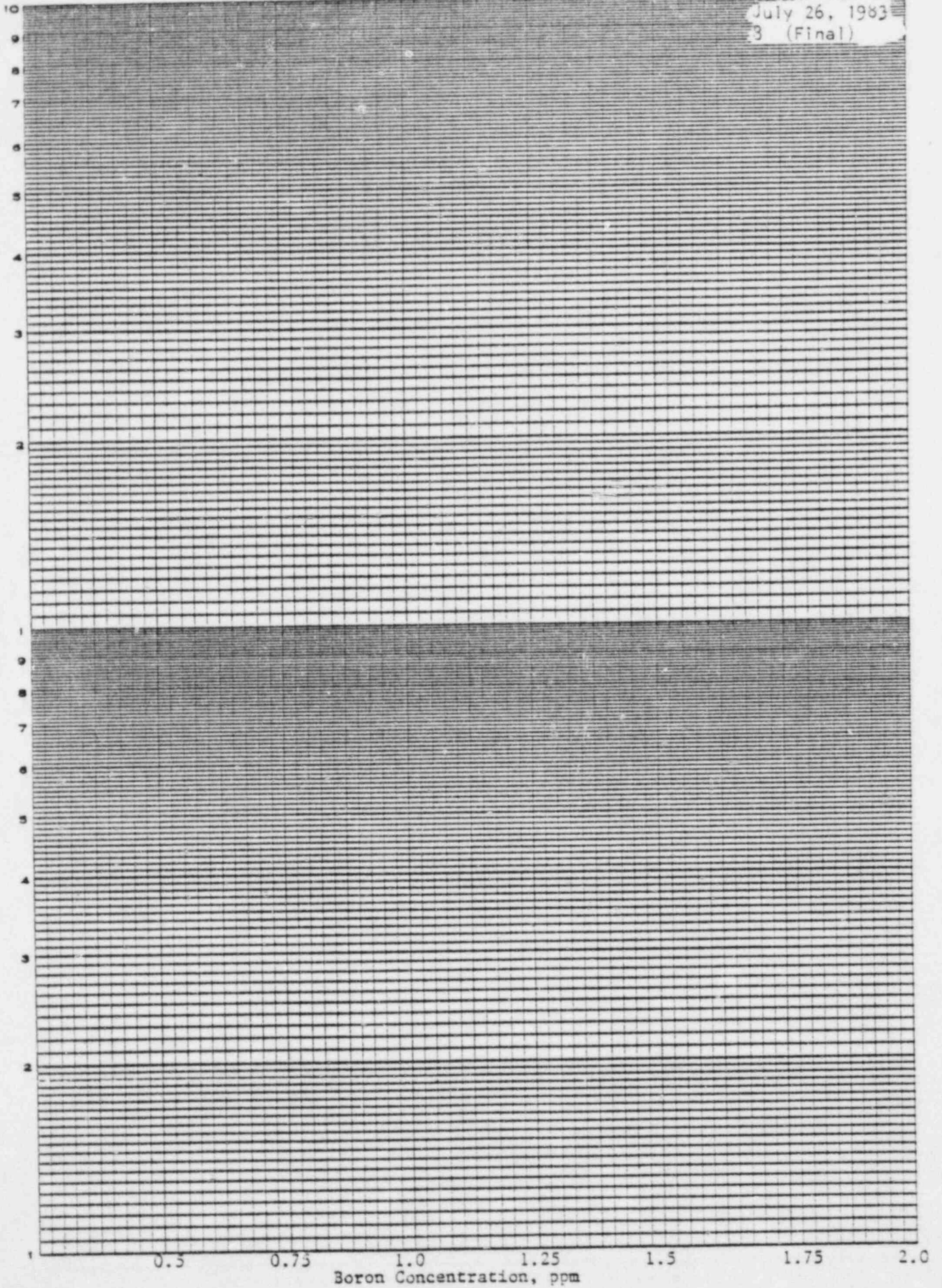
July 26, 1983

3 (Final)

DIETZEN CORPORATION  
MADE IN U.S.A.

NO. 341-1210 DIETZEN GRAPH PAPER  
SEMI-LOGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH

ABSORBANCE



7/06/83

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ZP 1200-4	CLASSIFICATION OF A LIQUID RELEASE	C1	5/82	01
ZP 1210-1	HAZARDOUS MATERIAL INCIDENTS REPORTING	C2	3/83	01
ZP 1220-1	EMERGENCY TELEPHONE NUMBER	C1	5/81	01
ZP 1220-4	DELETED CONTAINMENT ATMOSPHERE FOR RADIOIODINES DETERMINATION AND PARTICULATES ALTERNATE MEANS	C1	7/81	01
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ZP 1320-1	AUGMENTATION OF PLANT STAFFING	05	7/83	02
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ZP 1330-2	DELETED	C2	7/82	03
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ZP 1330-5	POST-ACCIDENT NOBLE GAS RELEASE RATE DETERMINATION	C0	11/80	03
ZP 1330-6	DELETED	C1	7/81	03
ZP 1330-7	DELETED	C2	7/82	02
ZP 1330-8	SAMPLING LIQUID PROCESS MONITORS DURING LIQUID MONITOR HIGH RADIATION ALARM CONDITIONS	C1	8/82	03
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ZP 1330-11	DILUTION AND TRANSFER OF GAS SAMPLES	C0	11/81	03
ZP 1330-20	POST-ACCIDENT ANALYSIS OF BARGN (BACKUP PROCEDURE)	C0	3/81	03
ZP 1330-21	POST-ACCIDENT ANALYSIS OF CHLORIDE	C3	6/82	03
ZP 1330-22	CALIBRATION OF THE MODEL 10 DIONEX ION CHROMATOGRAPH	C2	4/82	03
ZP 1330-23	DETERMINATION OF REACTOR COOLANT PH, CONDUCTIVITY AND DISSOLVED OXYGEN CONCENTRATION AT THE HIGH RADIATION SAMPLE SYSTEM	C2	6/82	03
ZP 1330-24	DETERMINATION OF REACTOR COOLANT HYDROGEN AND OFF-GAS CONCENTRATIONS AT THE HIGH RADIATION SAMPLING SYSTEM	C2	1/83	03

POST-ACCIDENT ANALYSIS OF BORON  
(Backup Procedure)

A. PURPOSE

The purpose of this procedure is to delineate an alternate method for determining boron following a Regulatory Guide 1.3 or 1.4 release of fission products. The method described by this procedure is referenced to as the Curcumin Spectrophotometric Method.

B. REFERENCES

1. Final Report, Analyses for Dissolved Hydrogen, Dissolved Oxygen, Boron, Chloride, pH, and conductivity under normal Post-Accident Conditions, NUS Corporation, October, 1980.
2. LCP 110-2, "Preparation of Reagents".
3. LCP 210-6, "Operation of the Klett-Summerson Colorimeter".

C. PREREQUISITES

1. Equipment:
  - a. Klett-Summerson Colorimeter.
  - b. Filter No. 54.
  - c. 20 mm X 40 mm cells.
  - d. Graduated cylinders, 10 or 25 ml and 500 ml.
  - e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
  - f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
  - g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
  - h. Beakers 250 ml or 400 ml, wide form.

- i. Tongs, long reach.
- j. Polyethylene stirring rods, 7.
- k. Medicine droppers, 7.
- l. Filter paper, Whatman No. 30, or equivalent.
- m. Glass or plastic funnel.
- n. Semi-logarithmic paper, dual cycle.
- o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.

2. Reagents:

- a. 95% Isopropyl alcohol.
- b. Curcumin reagent.
- c. Boron stock standard solution, 1000 ppm boron.
- d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

C. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. Wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. Wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.



4. Pipette 1.0 ml of DDI water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^{\circ}\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.



14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis); refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

LZP 1330-20  
Revision 1  
July 26, 1983  
7

ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)

# ATTACHMENT B

LZP-1330-20

Revision 1

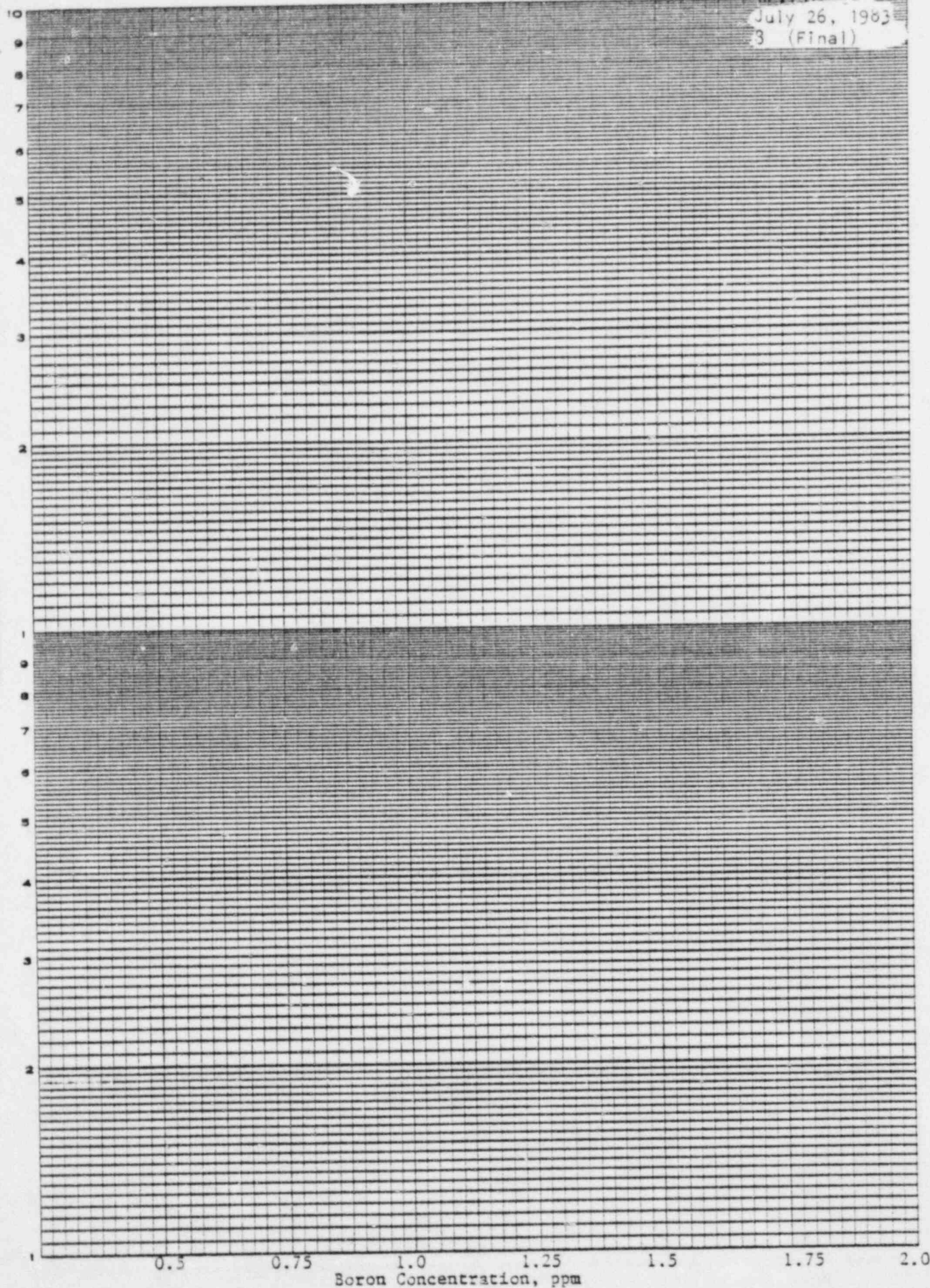
July 26, 1983

3 (Final)

MINI-MAX CORPORATION  
MADE IN U.S.A.

ABSORBANCE

SEMI-LOGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH



7/CB/83

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1P 1220-1	EMERGENCY TELEPHONE NUMBER	C1	5/81	01
1P 1220-4	DELETED CONTAINMENT ATMOSPHERE FOR RADIOIODINES DETERMINATION AND PARTICULATES ALTERNATE MEANS	C1	7/81	01
P 1310-1	NOTIFICATIONS	04	7/83	02
P 1320-1	AUGMENTATION OF PLANT STAFFING	05	7/83	02
P 1330-1	DELETED	C2	7/82	02
P 1330-2	DELETED	C2	7/82	03
P 1330-3	DELETED	C3	7/82	03
P 1330-4	DELETED	C2	7/82	03
P 1330-5	POST-ACCIDENT NOBLE GAS RELEASE RATE DETERMINATION	C0	11/80	03
P 1330-6	DELETED	C1	7/81	03
P 1330-7	DELETED	C2	7/82	02
P 1330-8	SAMPLING LIQUID PROCESS MONITORS DURING LIQUID MONITOR HIGH RADIATION ALARM CONDITIONS	C1	8/82	03
P 1330-10	DELETED	C1	1/82	03
P 1330-11	DILUTION AND TRANSFER OF GAS SAMPLES	C0	11/81	03
P 1330-20	POST-ACCIDENT ANALYSIS OF BARNON (BACKUP PROCEDURE)	C0	3/81	03
P 1330-21	POST-ACCIDENT ANALYSIS OF CHLORIDE	C3	6/82	03
P 1330-22	CALIBRATION OF THE MODEL 10 DIONEX ION CHROMATOGRAPH	C2	4/82	03
P 1330-23	DETERMINATION OF REACTOR COOLANT PH, CONDUCTIVITY AND DISSOLVED OXYGEN CONCENTRATION AT THE HIGH RADIATION SAMPLE SYSTEM	C2	6/82	03
P 1330-24	DETERMINATION OF REACTOR COOLANT HYDROGEN AND OFF-GAS CONCENTRATIONS AT THE HIGH RADIATION SAMPLING SYSTEM	C2	1/83	03



POST-ACCIDENT ANALYSIS OF BORON  
(Backup Procedure)

A. PURPOSE

The purpose of this procedure is to delineate an alternate method for determining boron following a Regulatory Guide 1.3 or 1.4 release of fission products. The method described by this procedure is referenced to as the Curcumin Spectrophotometric Method.

B. REFERENCES

1. Final Report, Analyses for Dissolved Hydrogen, Dissolved Oxygen, Boron, Chloride, pH, and conductivity under normal Post-Accident Conditions, NUS Corporation, October, 1980.
2. LCP 110-2, "Preparation of Reagents".
3. LCP 210-6, "Operation of the Klett-Summerson Colorimeter".

C. PREREQUISITES

1. Equipment:
  - a. Klett-Summerson Colorimeter.
  - b. Filter No. 54.
  - c. 20 mm X 40 mm cells.
  - d. Graduated cylinders, 10 or 25 ml and 500 ml.
  - e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
  - f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
  - g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
  - h. Beakers 250 ml or 400 ml, wide form.



- i. Tongs, long reach.
- j. Polyethylene stirring rods, 7.
- k. Medicine droppers, 7.
- l. Filter paper, Whatman No. 30, or equivalent.
- m. Glass or plastic funnel.
- n. Semi-logarithmic paper, dual cycle.
- o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.

2. Reagents:

- a. 95% Isopropyl alcohol.
- b. Curcumin reagent.
- c. Boron stock standard solution, 1000 ppm boron.
- d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

C. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.

4. Pipette 1.0 ml of DDI water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^{\circ}\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.

14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis), refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

LZP 1330-20  
Revision 1  
July 26, 1983  
7

ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)



# ATTACHMENT 3

LZP-1330-20

Revision 1

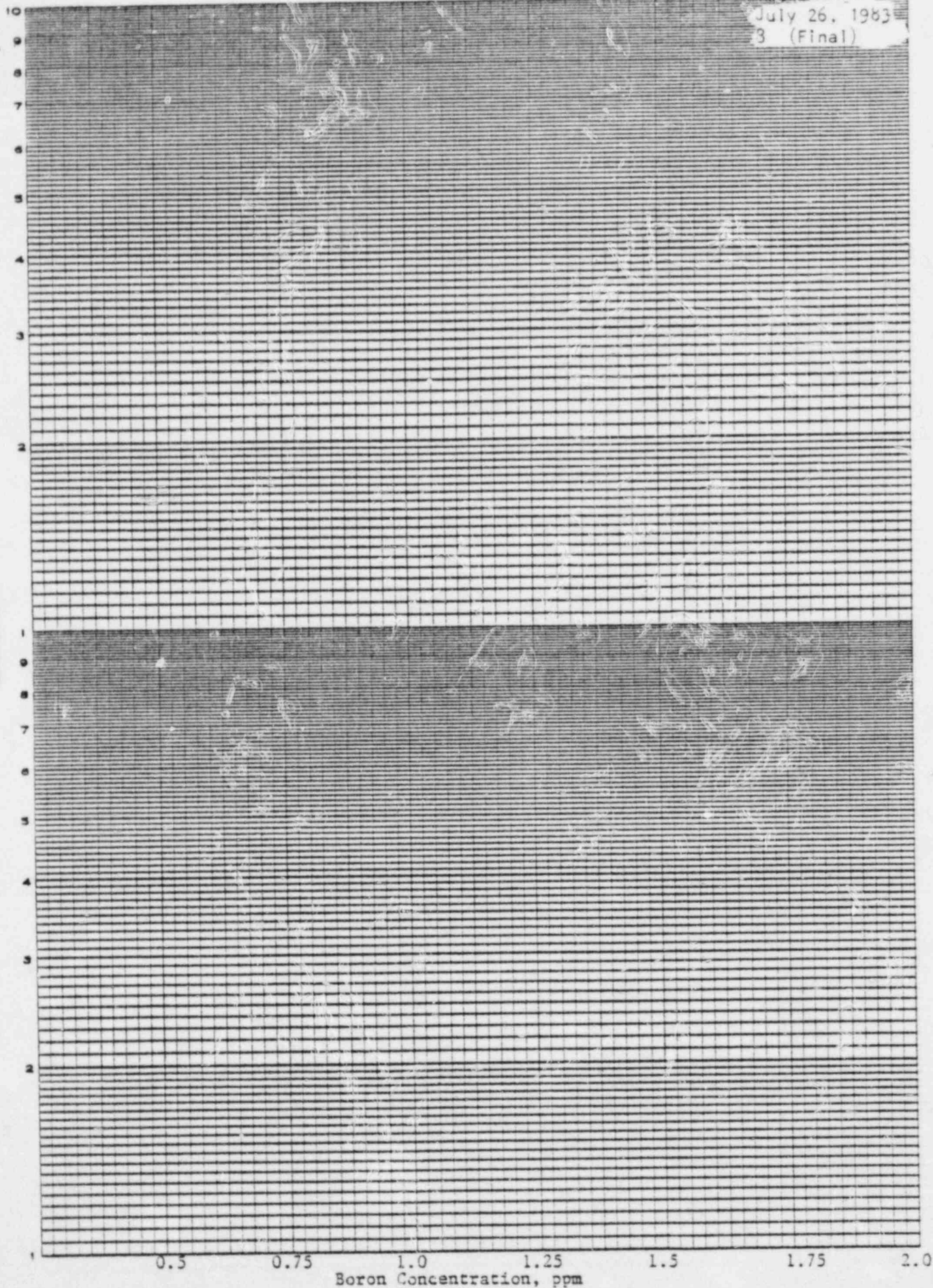
July 26, 1983

3 (Final)

DIETZEN CORPORATION  
MADE IN U.S.A.

2 CYCLES X 10 DIVISIONS PER INCH  
SEMI-LOGARITHMIC  
SCALE

ABSORBANCE



7/08/83

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P 1330-1	DELETED	02	7/82	02
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P 1330-3	DELETED	03	7/82	03
P 1330-4	DELETED	02	7/82	03
P 1330-5	POST-ACCIDENT NOBLE GAS RELEASE RATE DETERMINATION	00	11/80	03
P 1330-6	DELETED	01	7/81	03
P 1330-7	DELETED	02	7/82	02
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P 1330-11	DILUTION AND TRANSFER OF GAS SAMPLES	00	11/81	03
P 1330-20	POST-ACCIDENT ANALYSIS OF BAREN (BACKUP PROCEDURE)	00	3/81	03
P 1330-21	POST-ACCIDENT ANALYSIS OF CHLORIDE	03	6/82	03
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P 1330-24	DETERMINATION OF REACTOR COOLANT HYDROGEN AND OFF-GAS CONCENTRATIONS AT THE HIGH RADIATION SAMPLING SYSTEM	02	1/83	03

POST-ACCIDENT ANALYSIS OF BORON  
(Backup Procedure)

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1. Equipment:
  - a. Klett-Summerson Colorimeter.
  - b. Filter No. 54.
  - c. 20 mm X 40 mm cells.
  - d. Graduated cylinders, 10 or 25 ml and 500 ml.
  - e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
  - f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
  - g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
  - h. Beakers 250 ml or 400 ml, wide form.

- i. Tongs, long reach.
  - j. Polyethylene stirring rods, 7.
  - k. Medicine droppers, 7.
  - l. Filter paper, whatman No. 30, or equivalent.
  - m. Glass or plastic funnel.
  - n. Semi-logarithmic paper, dual cycle.
  - o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.
2. Reagents:
- a. 95% Isopropyl alcohol.
  - b. Curcumin reagent.
  - c. Boron stock standard solution, 1000 ppm boron.
  - d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

D. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.



4. Pipette 1.0 ml of DDI water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^{\circ}\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.



14. within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis), refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

LZP 1330-20  
Revision 1  
July 26, 1983  
7

ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)

ATTACHMENT B

LZP-1330-20

Revision 1

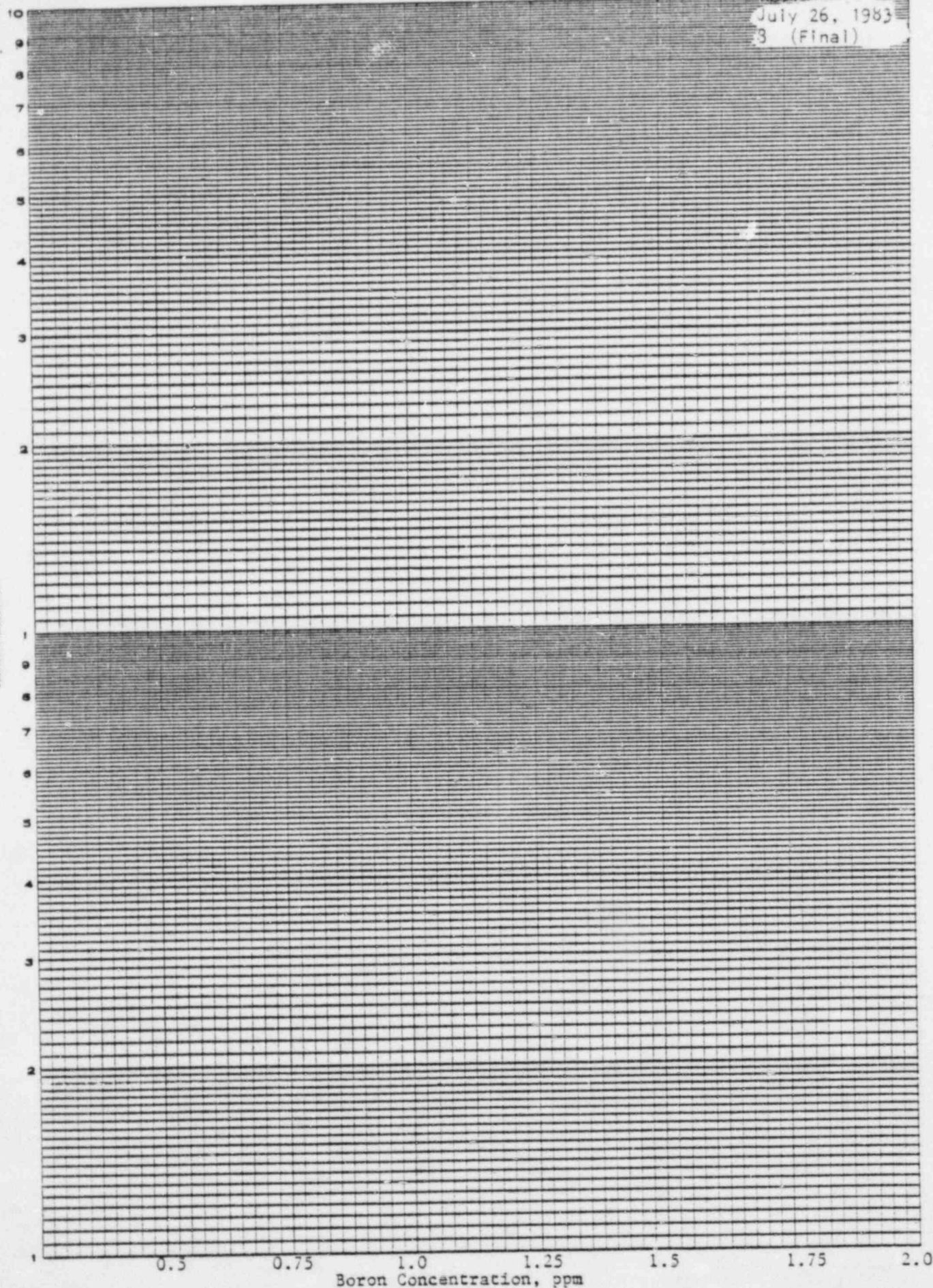
July 26, 1983

3 (Final)

WILSON SURPUNITION  
MADE IN U.S.A.

SEMI-LOGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH

ABSORBANCE



7/C6/83

## LZP INDEX

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P 1220-1	EMERGENCY TELEPHONE NUMBER	C1	5/81	01
P 1220-4	DELETED CONTAINMENT ATMOSPHERE FOR RADIOIODINES DETERMINATION AND PARTICULATES ALTERNATE MEANS	C1	7/81	01
P 1310-1	NOTIFICATIONS	04	7/83	02
P 1320-1	AUGMENTATION OF PLANT STAFFING	05	7/83	02
P 1330-1	DELETED	C2	7/82	02
P 1330-2	DELETED	C2	7/82	03
P 1330-3	DELETED	C3	7/82	03
P 1330-4	DELETED	C2	7/82	03
P 1330-5	POST-ACCIDENT NOBLE GAS RELEASE RATE DETERMINATION	00	11/80	03
P 1330-6	DELETED	01	7/81	03
P 1330-7	DELETED	C2	7/82	02
P 1330-8	SAMPLING LIQUID PROCESS MONITORS DURING LIQUID MONITOR HIGH RADIATION ALARM CONDITIONS	01	8/82	03
P 1330-10	DELETED	01	1/82	03
P 1330-11	DILUTION AND TRANSFER OF GAS SAMPLES	00	11/81	03
P 1330-20	POST-ACCIDENT ANALYSIS OF BARGN (BACKUP PROCEDURE)	00	3/81	03
P 1330-21	POST-ACCIDENT ANALYSIS OF CHLORIDE	03	6/82	03
P 1330-22	CALIBRATION OF THE MODEL 10 DIONEX ION CHROMATOGRAPH	C2	4/82	03
P 1330-23	DETERMINATION OF REACTOR COOLANT PH, CONDUCTIVITY AND DISSOLVED OXYGEN CONCENTRATION AT THE HIGH RADIATION SAMPLE SYSTEM	C2	5/82	03
P 1330-24	DETERMINATION OF REACTOR COOLANT HYDROGEN AND OFF-GAS CONCENTRATIONS AT THE HIGH RADIATION SAMPLING SYSTEM	C2	1/83	03

POST-ACCIDENT ANALYSIS OF BORON  
(Backup Procedure)

A. PURPOSE

The purpose of this procedure is to delineate an alternate method for determining boron following a Regulatory Guide 1.3 or 1.4 release of fission products. The method described by this procedure is referenced to as the Curcumin Spectrophotometric Method.

B. REFERENCES

1. Final Report, Analyses for Dissolved Hydrogen, Dissolved Oxygen, Boron, Chloride, pH, and conductivity under normal Post-Accident Conditions, NUS Corporation, October, 1980.
2. LCP 110-2, "Preparation of Reagents".
3. LCP 210-6, "Operation of the Klett-Summerson Colorimeter".

C. PREREQUISITES

i. Equipment:

- a. Klett-Summerson Colorimeter.
- b. Filter No. 54.
- c. 20 mm X 40 mm cells.
- d. Graduated cylinders, 10 or 25 ml and 500 ml.
- e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
- f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
- g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
- h. Beakers 250 ml or 400 ml, wide form.



- i. Tongs, long reach.
- j. Polyethylene stirring rods, 7.
- k. Medicine droppers, 7.
- l. Filter paper, Whatman No. 30, or equivalent.
- m. Glass or plastic funnel.
- n. Semi-logarithmic paper, dual cycle.
- o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.

2. Reagents:

- a. 95% Isopropyl alcohol.
- b. Curcumin reagent.
- c. Boron stock standard solution, 1000 ppm boron.
- d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

D. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. Wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. Wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.

4. Pipette 1.0 ml of DDI water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^{\circ}\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.

14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis), refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

LZP 1330-20  
Revision 1  
July 26, 1983  
7

ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)



ATTACHMENT B

LZP-1330-20

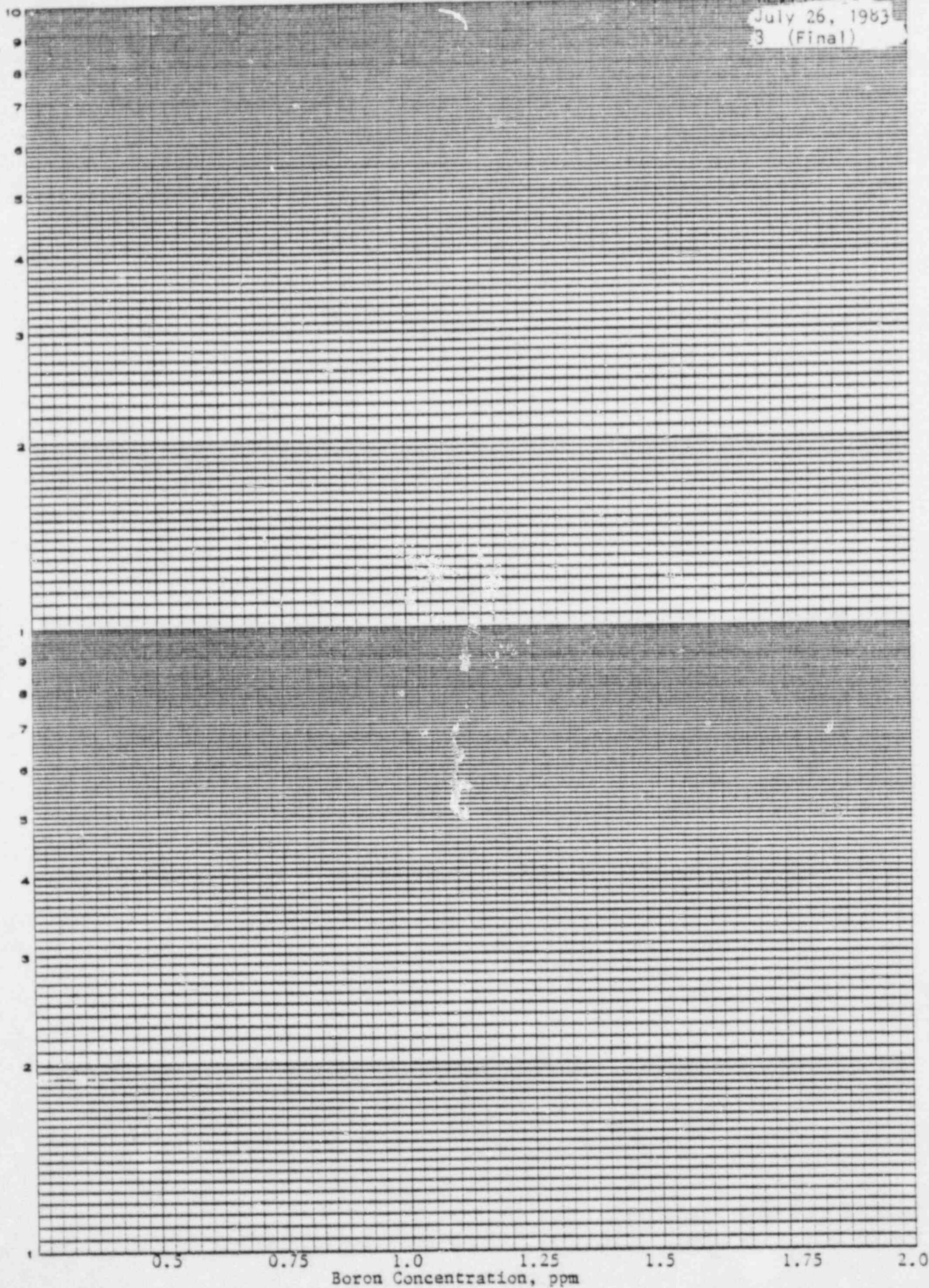
Revision 1

July 26, 1983

3 (Final)

MADE IN U.S.A.

SEMI-LOGARITHMIC  
2 CYCLES X 10 DIVISIONS PER INCH  
ABSORBANCE



7/08/83

## LZP INDEX

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PROC. NO.	TITLE	REV.	REV DATE	GISKETT
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P 1210-1	HAZARDOUS MATERIAL INCIDENTS REPORTING	C2	3/83	01
P 1220-1	EMERGENCY TELEPHONE NUMBER	C1	5/81	01
P 1220-4	DELETED CONTAINMENT ATMOSPHERE FOR RADIOIODINES DETERMINATION AND PARTICULATES ALTERNATE MEANS	C1	7/81	01
P 1310-1	NOTIFICATIONS	04	7/83	02
P 1320-1	AUGMENTATION OF PLANT STAFFING	05	7/83	02
P 1330-1	DELETED	C2	7/82	02
P 1330-2	DELETED	C2	7/82	03
P 1330-3	DELETED	C3	7/82	03
P 1330-4	DELETED	C2	7/82	03
P 1330-5	POST-ACCIDENT NOBLE GAS RELEASE RATE DETERMINATION	C0	11/80	03
P 1330-6	DELETED	C1	7/81	03
P 1330-7	DELETED	C2	7/82	02
P 1330-8	SAMPLING LIQUID PROCESS MONITORS DURING LIQUID MONITOR HIGH RADIATION ALARM CONDITIONS	C1	8/82	03
1330-10	DELETED	C1	1/82	03
1330-11	DILUTION AND TRANSFER OF GAS SAMPLES	C0	11/81	03
1330-20	POST-ACCIDENT ANALYSIS OF BARGON (BACKUP PROCEDURE)	C0	3/81	03
1330-21	POST-ACCIDENT ANALYSIS OF CHLORIDE	C3	6/82	03
1330-22	CALIBRATION OF THE MODEL 10 DIONEX ION CHROMATOGRAPH	C2	4/82	03
1330-23	DETERMINATION OF REACTOR COOLANT PH, CONDUCTIVITY AND DISSOLVED OXYGEN CONCENTRATION AT THE HIGH RADIATION SAMPLE SYSTEM	C2	6/82	03
1330-24	DETERMINATION OF REACTOR COOLANT HYDROGEN AND OFF-GAS CONCENTRATIONS AT THE HIGH RADIATION SAMPLING SYSTEM	C2	1/83	03

POST-ACCIDENT ANALYSIS OF BORON  
(Backup Procedure)

A. PURPOSE

The purpose of this procedure is to delineate an alternate method for determining boron following a Regulatory Guide 1.3 or 1.4 release of fission products. The method described by this procedure is referenced to as the Curcumin Spectrophotometric Method.

B. REFERENCES

1. Final Report, Analyses for Dissolved Hydrogen, Dissolved Oxygen, Boron, Chloride, pH, and conductivity under normal Post-Accident Conditions, NUS Corporation, October, 1980.
2. LCP 110-2, "Preparation of Reagents".
3. LCP 210-6, "Operation of the Klett-Summerson Colorimeter".

C. PREREQUISITES

1. Equipment:

- a. Klett-Summerson Colorimeter.
- b. Filter No. 54.
- c. 20 mm X 40 mm cells.
- d. Graduated cylinders, 10 or 25 ml and 500 ml.
- e. Volumetric flasks, 25 ml, 100 ml and 1000 ml.
- f. Evaporating dishes, 100 to 150 ml capacity, Vycor glass (Corning glass), or equivalent (used for boron determination only).
- g. Oxford pipettes with disposable plastic tips, 1 ml, 4 ml, 5 ml, 15 ml, 20 ml, and 5 ml Mohr pipette, or equivalent.
- h. Beakers 250 ml or 400 ml, wide form.

- i. Tongs, long reach.
- j. Polyethylene stirring rods, 7.
- k. Medicine droppers, 7.
- l. Filter paper, Whatman No. 30, or equivalent.
- m. Glass or plastic funnel.
- n. Semi-logarithmic paper, dual cycle.
- o. Water bath, set at  $55 \pm 2^{\circ}\text{C}$ , in an operating fume hood.

2. Reagents:

- a. 95% Isopropyl alcohol.
- b. Curcumin reagent.
- c. Boron stock standard solution, 1000 ppm boron.
- d. Boron standard solutions, 0.5 ppm, 1.0 ppm, 1.5 ppm, and 2.0 ppm.

D. PRECAUTIONS

- 1. A Regulatory Guide 1.3 or 1.4 release of fission products implies extremely high levels of radioactivity. Dose rates may be high enough to prevent entry into many areas of the plant that are normally habitable. Rad/Chem Supervision should be contacted prior to entry into any area when such a release of fission products is suspected.
- 2. wear radiation dosimetry as recommended by Rad/Chem Supervision.
- 3. wear protective clothing and respiratory protection as recommended by Rad/Chem Supervision.
- 4. Handle the sample container with long reach tongs. Dose rates from a 5 ml sample could range up to 50 R/hr.
- 5. Appropriate survey instruments should be available for monitoring during the processing and analysis of this sample.

6. Carry out all analytical operations behind adequate shielding in an operating fume hood.
7. Be certain any spills are wiped up immediately and properly disposed of.
8. The same geometry evaporating dishes must be used for all standards and samples to assure that evaporative rates are equivalent.
9. The temperature of the bath must be controlled so that all samples dry at the same rate.
10. The curcumin reagent must be stored under refrigeration after preparation and not be more than three days old.
11. Tests show that many variables can affect the color development in this procedure. To minimize these effects, the procedure includes processing standard boron solutions along with the samples.

E. LIMITATIONS AND ACTIONS

1. The diluted reactor water sample should contain between 0.5 and 2 ppm boron.
2. The total analysis time for this procedure is about 120 minutes (2 hours).
3. This procedure may also be used for boron analysis under normal conditions. If so, treat the sample in accordance with practices for routine reactor water.

F. PROCEDURE

1. Clean the immediate work area.
2. Inspect the labware and clean if necessary using approved cleaning techniques.
3. Clean the Klett-Summerson Colorimeter optical surfaces, the filter, and the cells. The outside surface of the cells must be free of dirt, fingerprints, and scratches.



4. Pipette 1.0 ml of DDI water (blank) and 1.0 ml of the 0.5, 1.0, 1.5 and 2.0 ppm boron standards into separate labeled evaporating dishes.
5. Pipette 1.0 ml of the diluted sample solution into two separate evaporating dishes for duplicate analyses.

CAUTION

Carry out all analytical operations behind adequate shielding in an operating fume hood.

6. Pipette 4.0 ml of curcumin reagent into each evaporating dish. Swirl the contents gently to mix, using long tongs to handle the two radioactive samples.
7. Place each evaporating dish on the bottom of the 250 or 400 ml wide-form beakers, using long tongs to handle the radioactive samples. Float the beakers in a water bath set at  $55 \pm 2^\circ\text{C}$ .
8. Evaporate the contents of each evaporating dish to complete dryness and note the time of dryness for each sample.
9. Remove the evaporating dishes from the bath at 15 minutes after the contents appear dry and allow them to cool to room temperature.
10. Add approximately 10 ml of 95 percent isopropyl alcohol to each evaporating dish. Use a polyethylene stirring rod to aid in dissolution of the red-colored reaction product and oxalic acid.
11. Using a medicine dropper, transfer the contents of the evaporating dishes to individual 25 ml volumetric flasks which have been previously rinsed with 95 percent alcohol.
12. Rinse the evaporating dishes with 95 percent alcohol and add the contents to the flasks. Dilute the contents of each flask to 25.0 ml using 95 percent isopropyl alcohol.
13. Zero the Klett-Summerson Colorimeter with deionized water in accordance with Reference 3.



14. Within one hour of dissolution of the dried product (step F.8.), pour the "blank" into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

CAUTION

The waste from steps F.15.-F.17. contains radionuclides; dispose of this waste in accordance with guidances provided by Rad/Chem Supervision.

NOTE

If dose levels warrant, the Klett-Summerson should be put in the fume hood behind the shielding with appropriate precautions taken to prevent contamination of this instrument.

15. Empty and rinse the cell with a small portion of one of the samples, then pour the sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).

NOTE

If a solution appears turbid, filter through a dry filter paper directly into a clean dry cell.

16. Empty and rinse the cell with a small portion of the duplicate sample, then pour that sample into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A).
17. Empty and rinse the cell with a small portion of the 0.5 ppm boron standard, then pour the standard into the cell and measure the absorbance. Record the reading on LRC Form 1088 (Attachment A). Repeat for each of the remaining standards; 1.0, 1.5 and 2.0 ppm boron.
18. Subtract the "blank" from each of the measured samples and standards absorbances. Record the difference on LRC Form 1088 (Attachment A).
19. Plot the corrected absorbance for the standards on the log axis of dual cycle semilogarithmic graph paper against concentration (linear axis), refer to Attachment 8. Draw the best fit straight line through the data points.

20. Determine the ppm boron in each sample from the calibration curve. Record the value on LRC Form 1088 (Attachment A).
21. Calculate the average boron concentration for the duplicate samples in accordance with LRC Form 1088 (Attachment A).
22. Multiply the average boron concentration by the sample dilution factor to determine the sample boron concentration. Refer to LRC Form 1088 (Attachment A).
23. Clean and decontaminate the Klett-Summerson Colorimeter and cells as necessary.
24. Ensure the sampling containers and liquid samples have been properly disposed of in accordance with instructions provided by Rad/Chem Supervision.
25. Survey the work area for radioactive contamination and decontaminate as necessary.
26. Clean the work area and labware as necessary.

G. CHECKLISTS

1. None.

H. TECHNICAL SPECIFICATION REFERENCES

1. None.

ATTACHMENT A  
LASALLE COUNTY STATION  
Post-Accident Analysis of Boron

LZP 1330-20  
Revision 1  
July 26, 1983  
7

Date: \_\_\_\_\_

	Absorbance Measurement	Absorbance-Blank	Concentration from Calibration Curve
BLANK			
Sample 1			
Sample 2			
0.5 ppm Standard			
1.0 ppm Standard			
1.5 ppm Standard			
2.0 ppm Standard			

Ave. boron conc., ppm =  $\frac{\text{Conc. sample \#1} + \text{Conc. sample \#2}}{2}$

2

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Sample boron conc., ppm = (Ave. boron conc., ppm) x Dilution Factor (DF)

ATTACHMENT B

LZP-1330-20

Revision 1

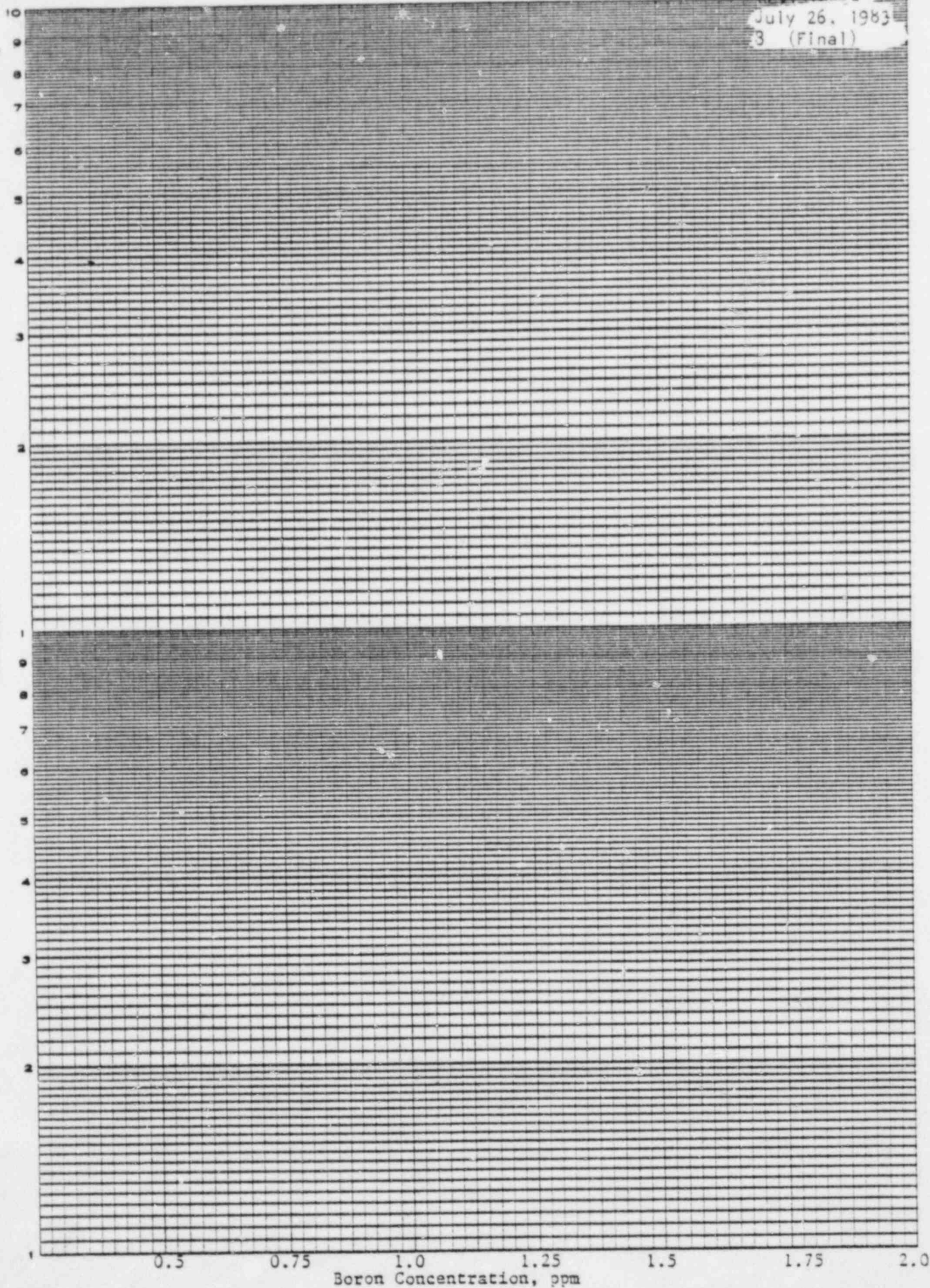
July 26, 1983

3 (Final)

MADE IN U.S.A.

2 CYCLES X 10 DIVISIONS PER INCH

ABSORBANCE



LAP 820-3  
Revision 22  
August 25, 1982  
a FINAL

ATTACHMENT C

OFF-SITE PROCEDURE MODIFICATION FORM

Aug 8, 1983  
DATE

Nuc. Reactor Reg (10)

Please REMOVE the following pages from your controlled copy of the LaSalle County Station LZP 15 Procedures Manual. INSERT the new pages as indicated and REMOVE and DESTROY the superseded pages. SIGN this transmittal form in the space provided for Manual holder below. RETURN this signed sheet to:

Office Supervisor  
LaSalle County Station

R. M. Molycax  
Station Superintendent  
LaSalle County Station

96-105

MANUAL NUMBER

MANUAL HOLDER SIGNATURE \_\_\_\_\_ DATE \_\_\_\_\_  
(IF NEW HOLDER, PLEASE ADVISE)

DOCUMENT	REMOVE/REV.	INSERT/REV/DATE
<u>LZP INDEX</u>	<u>3</u>	<u>3</u> <u>7/83</u>
<u>1330-20</u>	<u>0</u>	<u>1</u> <u>7/83</u>
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