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Omaha NE 68102-2247

September 6, 2006
LIC-06-0105

U. S. Nuclear Regulatory Commission
Attn: Document Control Desk
Washington, DC 20555-0001

- References:
1. Docket No. 50-285
 2. Letter from Jeffrey A. Reinhart (OPPDP) to Document Control Desk (NRC) dated August 21, 2006, Fort Calhoun Station, Unit No. 1 License Amendment Request (LAR) "Change of Containment Building Sump Buffering Agent from Trisodium Phosphate to Sodium Tetraborate" (LIC-06-0088) (ML062340039)

SUBJECT: Response to Request for Additional Information Related to the License Amendment Request on Change of Containment Building Sump Buffering Agent from Trisodium Phosphate to Sodium Tetraborate

Reference 2 provided the Omaha Public Power District's request for a license amendment to support changing the containment building sump buffering agent from Trisodium Phosphate to Sodium Tetraborate. Attachment 1 to this letter provides additional information requested in an email dated August 31, 2006. Attachment 1 contains a copy of WCAP-16596, "Evaluation of Alternative Emergency Core Cooling System Buffering Agents." This report is non-proprietary.

I declare under penalty of perjury that the foregoing is true and correct. (Executed on September 6, 2006.)

No commitments are made to the NRC in this letter. If you have additional questions, or require further information, please contact Thomas R. Byrne at (402) 533-7368.

Sincerely,

Jeffrey A. Reinhart
Site Director
Fort Calhoun Station

JAR/TRB/trb

Attachment:

1. Response to Request for Additional Information Related to the License Amendment Request on Change of Containment Building Sump Buffering Agent from Trisodium Phosphate to Sodium Tetraborate
- c: Director of Consumer Health Services, Department of Regulation and Licensure, Nebraska Health and Human Services, State of Nebraska

ATTACHMENT 1

**Response to Request for Additional Information Related to the License Amendment
Request on Change of Containment Building Sump Buffering Agent from Trisodium
Phosphate to Sodium Tetraborate**

**WCAP-16596, "Evaluation of Alternative Emergency Core
Cooling System Buffering Agents"**

Westinghouse Non-Proprietary Class 3

WCAP-16596-NP
Revision 0

July 2006

Evaluation of Alternative Emergency Core Cooling System Buffering Agents



Westinghouse

WCAP-16596-NP, Revision 0

Evaluation of Alternative Emergency Core Cooling System Buffering Agents

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**Electronically approved records are authenticated in the Electronic Document Management System*

This work performed under PWROG Project Number PA-SEE-0285.

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		Yes	No
AmerenUE	Callaway (W)	X	
American Electric Power	D.C. Cook 1&2 (W)	X	
Arizona Public Service	Palo Verde Unit 1, 2, & 3 (CE)	X	
Constellation Energy Group	Calvert Cliffs 1 & 2 (CE)	X	
Constellation Energy Group	Ginna (W)	X	
Dominion Connecticut	Millstone 2 (CE)	X	
Dominion Connecticut	Millstone 3 (W)	X	
Dominion Kewaunee	Kewaunee (W)	X	
Dominion VA	North Anna 1 & 2, Surry 1 & 2 (W)	X	
Duke Energy	Catawba 1 & 2, McGuire 1 & 2 (W)		X
Duke Energy	Oconee 1, 2, 3 (B&W)	X	
Entergy Nuclear Northeast	Indian Point 2 & 3 (W)	X	
Entergy Operations South	Arkansas 2, Waterford 3 (CE), Arkansas 1 (B&W)	X	
Exelon Generation Co. LLC	Braidwood 1 & 2, Byron 1 & 2 (W), TMI 1 (B&W)	X	
FirstEnergy Nuclear Operating Co	Beaver Valley 1 & 2 (W), Davis-Besse (B&W)	X	
Florida Power & Light Group	St. Lucie 1 & 2 (CE)	X	
Florida Power & Light Group	Turkey Point 3 & 4, Seabrook (W)	X	
Nuclear Management Company	Prairie Island 1&2, Pt. Beach 1&2 (W)	X	
Nuclear Management Company	Palisades (CE)	X	
Omaha Public Power District	Fort Calhoun (CE)	X	
Pacific Gas & Electric	Diablo Canyon 1 & 2 (W)	X	
Progress Energy	Robinson 2, Shearon Harris (W), Crystal River 3 (B&W)	X	
PSEG - Nuclear	Salem 1 & 2 (W)	X	
Southern California Edison	SONGS 2 & 3 (CE)	X	
South Carolina Electric & Gas	V.C. Summer (W)	X	
So. Texas Project Nuclear Operating Co.	South Texas Project 1 & 2 (W)	X	

Southern Nuclear Operating Co.	Farley 1 & 2, Vogtle 1 & 2 (W)	X	
Tennessee Valley Authority	Sequoyah 1 & 2, Watts Bar (W)		X
TXU Power	Comanche Peak 1 & 2 (W)	X	
Wolf Creek Nuclear Operating Co.	Wolf Creek (W)	X	

*** This is a list of participants in this project as of the date the final deliverable was completed. On occasion, additional members will join a project. Please contact the PWROG Program Management Office to verify participation before sending documents to participants not listed above.**

PWR Owners Group
International Member Participation* for PWROG Project / Task PA-SEE-0285

Utility Member	Plant Site(s)	Participant	
		Yes	No
British Energy	Sizewell B	X	
Electrabel (Belgian Utilities)	Doel 1, 2 & 4, Tihange 1 & 3	X	
Kansai Electric Co., LTD	Mihama 1, Ohi 1 & 2, Takahama 1 (W)	X	
Korea Hydro & Nuclear Power Corp.	Kori 1, 2, 3 & 4 Yonggwang 1 & 2 (W)	X	
Korea Hydro & Nuclear Power Corp.	Yonggwang 3, 4, 5 & 6 Ulchin 3, 4 & 5 (CE)	X	
Nuklearna Elektrarna KRSKO	Krsko (W)	X	
Nordostschweizerische Kraftwerke AG (NOK)	Beznau 1 & 2 (W)	X	
Ringhals AB	Ringhals 2, 3 & 4 (W)	X	
Spanish Utilities	Asco 1 & 2, Vandellos 2, Almaraz 1 & 2 (W)	X	
Taiwan Power Co.	Maanshan 1 & 2 (W)	X	
Electricite de France	54 Units	X	

* This is a list of participants in this project as of the date the final deliverable was completed. On occasion, additional members will join a project. Please contact the PWROG Program Management Office to verify participation before sending documents to participants not listed above.

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EXECUTIVE SUMMARY

The PWR Owners Group (PWROG) has commissioned this program to evaluate candidate replacement buffering agents for trisodium phosphate (TSP) and sodium hydroxide (NaOH) in PWR Emergency Core Cooling Systems (ECCS). The results of this evaluation identify recommended alternative buffering agents for plants now using TSP and NaOH for post-accident buffering in order to reduce the risk of sump screen plugging due to the formation of chemical products. A reduction in the potential for chemical precipitate formation may allow plants to more easily demonstrate design margin for new or existing sump screens, which will assist plants in meeting the December 2007 deadline for corrective actions set by the NRC in Nuclear Regulatory Commission (NRC) Generic Letter (GL) 2004-02.

The primary goal of this program is to identify suitable buffering agents to reduce precipitate generation under post-accident conditions. In addition, the properties of the candidate buffers must be comparable to those of the buffers currently in use. In order to qualify the alternative buffers, the candidates were subjected to a selection process which involved both evaluation of literature data and a series of tests. Specifically, the report presents the following conclusions from this evaluation process.

Selection of Candidates

Candidate buffering agents were selected for testing based on the criteria established in Section 2.3. Sodium tetraborate decahydrate (NaTB) and sodium metaborate tetrahydrate (NaMB) were selected because of the advantage that NaTB is already in use in Ice Condenser PWR plants. Sodium tripolyphosphate (NaTPP) and sodium gluconate (NaGlu) were also selected for testing because of their ability to act as sequestering agents, which may serve to inhibit precipitation.

Test Phase Results

Seven phases of tests were conducted to determine the acceptability of the selected candidates. Sodium gluconate was eliminated due to its inability to achieve a target pH of 8.0. Although sodium tripolyphosphate provided the best results in the precipitation testing phase, NaTPP formed the least amount of precipitate with the addition of aluminum and calcium, the corrosion testing showed that NaTPP may cause significant release of iron and aluminum from containment structural materials. In the environmental effects testing, in which the candidates were exposed to elevated temperature for 30 days, sodium tetraborate maintained its form as did TSP, while the sodium metaborate formed a liquid.

Conclusions and Recommendations

The sodium tetraborate and sodium metaborate buffers were determined from the test phases to be the most comparable alternatives to TSP and NaOH. Due to the results of the environmental effects test, in which sodium metaborate dissolved, sodium tetraborate would be recommended as the best alternative to TSP. However, sodium metaborate in solution form would be a suitable replacement for NaOH solution. The testing confirmed that TSP is an excellent buffering agent for plants with a low loading of calcium-bearing materials.

1.0 REPORT OVERVIEW

The purpose of this report is to evaluate alternative buffering agents for use in PWR Emergency Core Cooling Systems (ECCS) which may decrease chemical precipitate formation post-LOCA. The information contained in this report supports the resolution of GSI-191 through replacement of the buffering agent in order to reduce potential post-accident chemical effects in containment sump fluids.

Section 2 of this report presents an introduction to the alternate buffer program, the background of the chemical effects issue, the buffer properties of concern to be evaluated for each candidate, and also provides the objective of this program.

Section 3 describes the review of literature data performed in order to identify candidate buffers for the test program based on the criteria for buffer properties.

Section 4 outlines the Test Plan for comparison of the candidate buffers with the buffering agents currently employed in US PWRs.

Section 5 presents the results of the bench scale acceptability tests performed according to the Test Plan.

Section 6 performs an evaluation of the buffering agent criteria provided in Section 2.3. For each candidate buffer, precipitation under post-LOCA conditions, the quantity required to adjust the pH to > 8.0 , the dissolution rate, affordability and availability, corrosiveness, the effect on boric acid solubility, environmental stability, habitability concerns, and the potential for oxide/CRUD release are discussed.

Section 7 presents the conclusions and recommendations developed from the results of the test phases in Section 5 and the evaluation of the buffering agent criteria performed in Section 6.

Finally, Section 8 discusses considerations for replacement of the buffering agent with a suggested alternative, including pH requirements and necessary evaluations.

2.0 INTRODUCTION

In February 2006, the PWR Owners' Group (PWROG) authorized testing to evaluate alternative buffering agents for use in the Emergency Core Cooling System (ECCS) at PWRs (Reference 2.4-1). The purpose of the testing is to identify candidate replacements for trisodium phosphate (TSP) and sodium hydroxide (NaOH) currently used in this application at most plants. Laboratory testing to support this program is being conducted at the Fauske and Associates facility in Burr Ridge, IL under an approved test plan (Reference 2.4-2).

A replacement for TSP is desirable due to the potential interaction of phosphate with metals dissolved from containment materials under accident conditions, particularly calcium, to produce amorphous metal phosphate precipitates. A replacement for NaOH is desirable due to the enhanced corrosion of aluminum alloys in NaOH solutions, and resultant generation of amorphous aluminum-containing precipitates, including aluminum oxyhydroxide and metal aluminosilicates. Past testing has shown that such amorphous precipitates do not settle readily and are difficult to filter, and thus are likely to contribute to sump strainer head loss under accident conditions (References 2.4-3-2.4-6).

The primary goal of the alternative buffering agent test program is to identify suitable buffering agents to minimize precipitate generation under post-accident conditions. This goal may be accomplished by selecting buffering agents that do not react directly with dissolved species to produce precipitate, as well as selecting buffering agents that reduce the dissolution/corrosion of containment materials or maintain dissolved metals in solution to prevent precipitation. In addition to minimizing precipitate generation, the candidate buffer properties must be comparable to those of the chemicals currently used.

2.1 BACKGROUND

The conditions in Pressurized Water Reactor (PWR) containment buildings are designed to both contain radioactive materials releases and facilitate core cooling in the event of a Loss of Coolant Accident (LOCA). The cooling process requires water discharged from the break and containment spray to be collected in a sump for recirculation by the Emergency Core Cooling System (ECCS) and Containment Spray System (CSS). Typically, a containment sump contains one or more screens in series that protect the components of the ECCS and CSS from debris that could be washed into the sump. Debris generated by the action of the discharged water and the latent containment debris inside containment may be transported to the containment sump and downstream of the sump to the ECCS and CSS components when the ECCS and CSS are realigned from injecting water from the Refueling or Borated Water Storage Tank (RWST or BWST). There is a concern that this debris may form a debris bed at the sump screen that would sufficiently impede the recirculating flow as to challenge long-term core cooling requirements.

The NRC identified its concern regarding maintaining adequate long-term core cooling in Generic Safety Issue (GSI) 191 (Reference 2.4-7). Generic Letter (GL) 2004-02 (Reference 2.4-8), issued in September 2004, identified actions that utilities must take to address the sump blockage issue. The NRC's position is that plants must be able to demonstrate that

debris transported to the sump screen after a LOCA will not lead to unacceptable head loss for the recirculation pumps, will not impede flow through the ECCS and CSS, and will not adversely affect the long-term operation of either the ECCS or the CSS. Generic Letter 2004-02 also identifies that all mitigating actions by plants be implemented by the end of December 2007 if required to enable licensees to demonstrate acceptable ECCS and CSS performance.

A major concern in evaluating the effects of the debris transported to the sump screen is the chemical products which may form in a post-LOCA sump environment. Materials present in containment may dissolve or corrode when exposed to the reactor coolant and spray solutions. This behavior would result in oxide particulate corrosion products and the potential for the formation of precipitates due to changes in temperature and reactions with other dissolved materials. These chemical products may become another source of debris loading to be considered in sump screen performance and downstream effects.

The results of the Integrated Chemical Effects Test (ICET) program (Reference 2.4-9) and the PWROG sponsored Chemical Effects Bench Testing (Reference 2.4-5) indicate two main contributors to precipitate formation following a LOCA. The high pH of the NaOH buffered sump solution post-LOCA may cause significant metal corrosion, resulting in oxide particulate corrosion products and the potential for formation of metal silicates. Also, the reaction of TSP with dissolved metals may form metal phosphate precipitates. Recent tests performed at Argonne National Laboratory (ANL) (Reference 2.4-6) demonstrate that chemical products, specifically calcium phosphate precipitates, may contribute significantly to head loss across simulated sump screen debris beds.

2.2 PROGRAM OBJECTIVE

The objective of this program is to evaluate candidate buffering agents as potential alternatives to trisodium phosphate (TSP) or sodium hydroxide (NaOH) used in Emergency Core Cooling Systems (ECCS) in operating Pressurized Water Reactors (PWR). Replacement of the existing buffering agent may reduce the quantity of precipitate formation and the resulting impact on head loss of the chemical precipitates. The information contained in this report may be utilized by plants considering replacement of the buffering agent as part of their GSI-191 resolution.

2.3 BUFFER PROPERTIES

This program is being conducted in order to identify alternative buffering agents which minimize the potential for chemical precipitate formation following a LOCA. Thus, possible reactions between materials in containment and the buffer candidates will be evaluated as part of the program. In addition to minimizing precipitate generation, the following properties must be comparable to those of the chemicals currently in use (Reference 2.4-1):

1. Quantity required to adjust pH to target value
2. Dissolution rate in water at post-LOCA sump temperatures
3. Affordability and ready availability

4. No demonstrated deleterious effects, e.g., corrosion to key containment structural materials
5. Does not adversely affect the solubility of boric acid, or lead to an increase in boric acid precipitation on structures
6. Resistant to degradation from radiation, elevated temperatures and humidity, i.e., long storage life in containment environment
7. Non-hazardous material, i.e., does not create habitability concerns during storage or handling
8. Will not cause significant release of metal oxide deposits from the fuel or primary coolant system surfaces

The candidates for this test program were selected considering both potential precipitate formation and the criteria listed above. Their properties were evaluated through the testing given in the Test Plan provided in Section 4.0 and literature research results provided in Section 3.0.

2.4 REFERENCES

- 2.4-1 PWROG PA-SEE-0285, "Alternate Buffer for ECCS," February 2006.
- 2.4-2 LTR-CDME-06-65, "Test Plan: Evaluation of Alternative Buffering Agents for ECCS Application," March 2006.
- 2.4-3 LA-UR-05-0124, "Integrated Chemical Effects Test Project: Test #1 Data Report," June 2005.
- 2.4-4 LA-UR-05-6996, "Integrated Chemical Effects Test Project: Test #3 Data Report," October 2005.
- 2.4-5 WCAP-16530-NP, Revision 0, "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191," February 2006.
- 2.4-6 Oras, J., et al., "Chemical Effects/Head Loss Testing Quick Look Report, Tests ICET 3-4 to 11," January 20, 2006.
- 2.4-7 Generic Safety Issue 191 (GSI-191), "Assessment of Debris Accumulation on Pressurized Water Reactor (PWR) Sump Performance."
- 2.4-8 NRC Generic Letter 2004-02, "Potential Impact of Debris Blockage on Emergency Recirculation During Design Basis Accidents at Pressurized-Water Reactors, September 13, 2004.
- 2.4-9 Test Plan: Characterization of Chemical and Corrosion Effects Potentially Occurring Inside a PWR Containment Following a LOCA, Revision 13, July 20, 2005.

3.0 SELECTION OF CANDIDATE BUFFERING AGENTS

A review of literature data was performed to identify candidate alternative buffering agents. This review identified three primary classes of potential alternatives:

1. Borates: this class includes salts of tetraborate, metaborate, and octaborate. A distinct advantage of this class is that sodium tetraborate is already in use in Ice Condenser plants.
2. Polymeric (condensed) phosphates: this class includes salts of tripolyphosphate, pyrophosphates and metaphosphates (e.g., sodium hexametaphosphate).
3. Sequestering (chelating) agents: this class includes chemicals capable of sequestering metal ions dissolved from containment materials, thereby inhibiting precipitation, such as salts of citrate and ethylenediaminetetraacetate (EDTA). It should be noted that some polymeric phosphates are excellent sequestering agents for calcium and magnesium.

Other potential classes of materials that were considered include carbonates, amines, and salts of other strong oxyacids or weak acids. Carbonates were dismissed due to the potential for precipitation of metal carbonates (Reference 3.1-1). Amines were dismissed due primarily to habitability concerns and the fact that most commonly used amines are volatile liquids, so that storage and handling would be challenging. Salts of other strong oxyacids (e.g., sodium nitrate) or other weak acids (e.g., sodium acetate) could be effective buffers, but offer no benefits relative to the borates.

Any of the sodium or potassium borate salts would be suitable candidates as alternatives to sodium hydroxide or trisodium phosphate. Sodium tetraborate decahydrate (Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) has been selected for testing since it is currently used in nuclear plant applications. Sodium metaborate tetrahydrate ($\text{NaBO}_2 \cdot 4\text{H}_2\text{O}$) has been selected since it provides a higher pH than other borates (Reference 3.1-2).

A number of polymeric (or condensed) phosphates are readily available commercially. These include sodium pyrophosphate, sodium trimetaphosphate, sodium tripolyphosphate and sodium hexametaphosphate. All of the condensed phosphates are good sequestering agents for alkali earth metals (e.g., calcium and magnesium), and are used for this purpose in detergents (Reference 3.1-1). However, they may slowly hydrolyze to produce orthophosphate (PO_4^{3-}), and are thus chemically equivalent to TSP, i.e., may interact with metals to form metal phosphate precipitates. Neither sodium hexametaphosphate (Calgon) nor sodium trimetaphosphate will produce the desired final buffered pH (the pH of 1% solutions is ~7), and therefore neither is a good candidate. Sodium pyrophosphate decomposes at 94°C (201°F) and is therefore not a good candidate. Therefore, sodium tripolyphosphate has been the only condensed phosphate selected for testing.

A wide variety of other sequestering agents are available for consideration. However, most available sequestering agents are not specific to calcium and may mobilize radionuclides (e.g.,

cobalt-60) from the oxides on the fuel and other surfaces within the primary coolant system. For example, citrate and EDTA are used for the chemical decontamination of primary systems (Reference 3.1-3). Of the sequestering agents commonly used for calcium, sodium gluconate may serve as an effective buffering agent and would not result in significant release of radionuclides.

The candidates selected for testing as potential alternatives to sodium hydroxide or trisodium phosphate as ECCS buffering agents are summarized in Table 3-1 with their properties (Reference 3.1-4).

Table 3-1: Properties of Candidate ECCS Buffering Agents

Buffering Agent	pH (1% solution)	Molecular Weight (g/mol)	Decomposition Temperature (°C)
Sodium tetraborate decahydrate (NaTB)	9.2	381.4	>600
Sodium metaborate tetrahydrate (NaMB)	11.0	137.9	>900 ⁽¹⁾
Sodium tripolyphosphate (NaTPP)	8.0	367.9	622
Sodium gluconate (NaGlu)	10.0	218.1	206
Sodium hydroxide (NaOH)	>14	40.0	>1000
Trisodium phosphate dodecahydrate (TSP)	12.0	380.1	>1000 ⁽²⁾

(1) Above 52°C the waters of hydration may be liberated.

(2) Above 100°C the waters of hydration may be liberated.

3.1 REFERENCES

- 3.1-1 Newell, S. B., *Chemistry: An Introduction*, Little, Brown and Company, 1977.
- 3.1-2 U. S. Borax Product Profile, "Sodium Metaborate, 8 Mol," February 2002.
- 3.1-3 *Recent Developments in Decontamination Technology*, EPRI Radiation Control Seminar, Seattle, WA, August 1999.
- 3.1-4 Dean, J. A. (ed.), *Lange's Handbook of Chemistry*, 15th Edition, McGraw-Hill, 1999.

4.0 TEST PLAN

A test plan was developed in order to compare the candidate buffer properties to those of the buffering agents currently in use (Reference 4.8-1). Some of the buffer properties compared through testing include the buffer's dissolution behavior, potential for precipitate generation, resistance to temperature and humidity, and impact on boric acid solubility. The test program has been arranged in phases so that only candidates that meet the criteria established for each phase are tested in subsequent phases. Note, if specific criterion for a given test phase is not met, testing of candidate buffers may continue at the test director's discretion.

4.1 PHASE 1: DISSOLUTION TESTING

During this phase, the rate of dissolution in water at 150°F will be determined for a 1 weight percent solution of each candidate buffering agent. The dissolution rate of trisodium phosphate will also be determined for comparison. Sodium hydroxide is added as a solution, so comparison testing is not applicable.

Perform the following steps for each candidate buffering agent and for trisodium phosphate:

1. Heat 1 liter of demineralized water to 150±5°F (66±3°C).
2. Add 10 grams of the test chemical.
3. Record the time required for complete dissolution (determined visually).
4. Allow the solution to cool to 25°C.
5. Measure pH and verify solution is free of precipitates.

Acceptance criteria:

1. Complete dissolution in less than 2 hours or less than three times the amount of time required for dissolution of trisodium phosphate dodecahydrate, whichever is less;
2. pH ≥ 8.0 after cooling; and
3. no visible precipitates after cooling.

4.2 PHASE 2: PH ADJUSTMENT OF BORIC ACID SOLUTIONS

During this phase, the quantity of each candidate buffering agent required to adjust boric acid solutions to a range of pH values will be determined. Three different boric acid concentrations will be used.

1. Prepare 1 liter of stock boric acid solutions containing 2000 ppm, 2500 ppm and 3000 ppm boron by dissolving 11.4294 g, 14.2994 g and 17.1592 g of boric acid (H_3BO_3) in demineralized water; measure and record the pH of the stock solutions.

Perform the following steps for each candidate buffering agent and for trisodium phosphate and sodium hydroxide:

2. Prepare 250 milliliters of titrant solution containing X ppm boron and 50 g/L of the test chemical. The addition quantities will be:

Boric acid = $(250 \text{ mL}/1000 \text{ mL/L}) * X \text{ ppm}/1000 * (61.83 \text{ g H}_3\text{BO}_3/10.81 \text{ g B})$

Test Chemical = $50 \text{ g/L} * (250 \text{ mL}/1000 \text{ mL/L})$

Where X is the boron concentration being tested, 2000, 2500 or 3000.

3. Titrate 50 milliliter aliquots of the appropriate stock boric acid solution with the corresponding test solution; after the pH exceeds 7.0, measure and record pH after each 2 milliliter addition; terminate the titration when pH reaches a maximum or reaches 10.0, whichever occurs first.
4. Perform steps 2 and 3 for each boron concentration.
5. For each boric acid solution, determine the quantity (in grams) of each test chemical required to reach pH values of 8.0, 8.5, 9.0, 9.5 and 10.0. This will be:

$\text{mL titrant required} * 0.05 \text{ g/mL}$

6. Note the formation of any precipitates during the titrations.

Acceptance Criteria:

1. Ability to achieve a maximum pH of at least 8.0 in all three boric acid solutions;
2. quantity required to achieve a pH of 8.0 no more than two times that required using trisodium phosphate dodecahydrate (on a weight basis); and
3. no visible precipitates formed during titrations.

4.3 PHASE 3: DISSOLUTION IN BORIC ACID AS A FUNCTION OF TEMPERATURE

During this phase, the dissolution rate of the candidate buffering agents in a nominal boric acid solution will be determined as a function of temperature.

1. Prepare 1 liter of stock 2500 ppm boron solution using boric acid (see step 1 of Phase 2); measure and record the pH of the stock solution.

Perform the following steps for each candidate buffering agent and for trisodium phosphate:

2. Heat 100 milliliters of the boric acid solution to $100 \pm 5^\circ\text{F}$ ($38 \pm 3^\circ\text{C}$).
3. Add the quantity of the test chemical required to achieve a pH of 8.0, as determined in Phase 2 testing.
4. Record the time required for complete dissolution (determined visually).
5. Allow the solution to cool to 25°C and verify it is free of precipitates.
6. Repeat steps 2 to 4 at $150 \pm 5^\circ\text{F}$ ($66 \pm 3^\circ\text{C}$) and at $200 \pm 5^\circ\text{F}$ ($93 \pm 3^\circ\text{C}$).

Acceptance criteria:

1. At each temperature, complete dissolution in less than 2 hours or less than three times the amount of time required for dissolution of trisodium phosphate, whichever is less;
2. no visible precipitates observed either immediately after dissolution or during cooling.

4.4 PHASE 4: ALUMINUM AND CALCIUM ADDITION

During this phase, calcium and aluminum salts will be added to solutions containing the candidate buffers to determine whether precipitates form.

1. Prepare 1 liter of stock 2500 ppm boron solution using boric acid (see step 1 of Phase 2); measure and record the pH of the stock solution.
2. Prepare 100 milliliter stock solutions containing 10000 ppm aluminum (Al) and 10000 ppm calcium (Ca). Any soluble salts may be used, such as aluminum nitrate for aluminum or calcium chloride for calcium. The addition quantities are determined as:

$$\text{Al or Ca salt (g)} = (1/\text{fraction Al or Ca in salt}) * 0.1 \text{ L} * 10 \text{ g/L}$$

Perform the following steps for each candidate buffering agent and for trisodium phosphate and sodium hydroxide:

3. Heat 95 milliliters of the stock boric acid solution to $150 \pm 5^\circ\text{F}$ ($66 \pm 3^\circ\text{C}$).
4. Add the quantity of the test chemical required to achieve a pH of 8.5, as determined in Phase 2 testing.
5. Add 5 milliliters of stock Al or Ca solution; note the formation of any precipitates.
6. Transfer the solution to a 100 mL graduated cylinder.
7. Allow the solution to cool to 25°C ; note the formation of any precipitates during cooling.
8. After 1, 2 and 24 hours, record the volume of any precipitates formed.
9. Perform steps 3 to 8 using both Al and Ca.

Acceptance criteria:

1. For Al, the volume of precipitate generated is no more than generated using sodium hydroxide or trisodium phosphate; and
2. no precipitate generated on addition of Ca.

Phase 4A&B: Determination of Threshold Values for Precipitate Formation in NaTB and NaMB

The initial results of Phase 4 testing showed that precipitates formed on addition of calcium and aluminum to solutions containing NaTB and NaMB. This result was contrary to the results of previous testing conducted in both the PWROG program (Reference 4.8-2) and the ICET program (Reference 4.8-3). Specifically, at lower levels of dissolved calcium and aluminum, and at lower pH, no precipitates were observed with NaTB in the presence of either calcium or aluminum. Therefore, follow-on testing was performed to determine threshold values for precipitate formation, and to examine the effect of pH.

During this phase, calcium and aluminum salts were added at various concentrations to solutions containing the candidate buffers to determine whether precipitates would form. In Phase 4A, 75 ppm solutions were tested, and in Phase 4B, solutions of varying concentrations were tested.

1. 1 liter of stock 2,500 ppm boron solution was prepared using boric acid (see step 1 of Phase 2); the pH of the stock solution was measured and recorded. Additional stock boron solution was prepared as needed.
2. 50 milliliter stock solutions containing 5,000 ppm aluminum, 7,500 ppm aluminum, 5,000 ppm calcium, and 7,500 ppm calcium were prepared. Note, 10,000 ppm solutions were used for initial Phase 4 testing. Aluminum nitrate was used for aluminum and calcium chloride was used for calcium. The addition quantities were determined as:

$$\text{Salt (g)} = (1/\text{fraction Al or Ca in salt}) * 0.05 \text{ L} * 5 \text{ g/L [for 5,000 ppm]}$$

$$\text{Salt (g)} = (1/\text{fraction Al or Ca in salt}) * 0.05 \text{ L} * 7.5 \text{ g/L [for 7,500 ppm]}$$

The following steps were performed for sodium tetraborate and sodium metaborate:

3. 100-x milliliters of the stock boric acid solution were heated to 150±5°F (66±3°C) using a hot plate with stirring, where x was the volume of stock Al or Ca solution added in step 5.
4. The quantity of the test chemical required to achieve a pH of 8.5 was added, as determined in Phase 2 testing.
5. Sufficient stock 5,000 ppm or 7,500 ppm Al or Ca solution was added to achieve varying quantities of dissolved Al or Ca; the formation of any precipitates was noted. Concentrations ranged from 75 ppm to 476 ppm for Al and from 75 ppm to 380 ppm for Ca.
6. The solution was transferred to a 100 mL graduated cylinder.
7. The solution was allowed to cool to 25°C; the formation of any precipitates during cooling was noted.
8. After 1, 2 and 24 hours, the volume of any precipitates formed was recorded.
9. Steps 3 to 8 were performed using both Al and Ca.
10. Steps 3 to 9 were repeated, adjusting the metal concentration as required to find the precipitate threshold concentration.

Phase 4C: Determination of pH Effects on Precipitation in NaTB and NaMB

During this phase, the steps in Phase 4 were repeated with pH adjusted to 7.0, 7.5 and 8.0, rather than 8.5.

4.5 PHASE 5: CORROSION TESTING

During this phase, the effects of the buffering agents on key structural materials will be determined. The materials to be tested are A508 carbon steel and commercially pure aluminum metal (A1100).

1. Prepare 5 liters of stock 2500 ppm boron solution using boric acid (71.497 g); measure and record pH.

Perform the following steps for each candidate buffering agent and for trisodium phosphate and sodium hydroxide:

2. Weigh and dimension duplicate A508 and commercially pure aluminum corrosion coupons.

3. To 500 milliliters of stock solution, add sufficient test chemical to achieve a pH 8.0 solution.
4. Transfer 250 milliliters of the test solutions to two test vessels; the test vessels should be fitted with lids to reduce liquid loss due to evaporation during the test.
5. Heat the test solutions to $150\pm 5^{\circ}\text{F}$ ($66\pm 3^{\circ}\text{C}$).
6. Insert duplicate A508 coupons in one test vessel and duplicate aluminum coupons in the other test vessel.
7. Maintain the vessel temperature at $150\pm 5^{\circ}\text{F}$ ($66\pm 3^{\circ}\text{C}$) for two weeks.
8. Cool the test solutions and measure pH and dissolved iron or aluminum, as applicable.
9. Remove the corrosion specimens
10. Rinse with demineralized water, followed by acetone or ethanol, dry and weigh.
11. Descale the corrosion coupons and repeat step 10.
12. Determine weight loss as a function of coupon surface area (scaled and descaled).

Acceptance criteria:

1. General corrosion no greater than observed with sodium hydroxide or trisodium phosphate;
2. No indication of localized corrosion, such as pitting.

4.6 PHASE 6: ENVIRONMENTAL EFFECTS TESTING

During this phase, the effects of elevated temperature during storage will be evaluated. Note, this test phase may be initiated in parallel with Phase 1 testing. Dissolution testing (step 6) need only be performed on candidates that meet criteria established for Phases 1 to 5.

Perform the following steps for each candidate buffering agent and for trisodium phosphate:

1. Place 10 grams of each candidate buffer in individual glass beakers and cover with a watch glass.
2. Place the beakers in an oven set at $150\pm 5^{\circ}\text{F}$ ($66\pm 3^{\circ}\text{C}$).
3. Maintain oven temperature for 30 days.
4. After 30 days exposure, remove the candidate buffers and physically examine.
5. Repeat Phase 1 dissolution tests.

Acceptance criteria:

1. Complete dissolution in less than 2 hours or less than three times the amount of time required for dissolution of trisodium phosphate, whichever is less;
2. $\text{pH} \geq 8.0$ after cooling; and
3. no visible precipitates after cooling.

4.7 PHASE 7: BORIC ACID SOLUBILITY TESTING

The purpose of this test phase is to determine the solubility limit of boric acid in the presence of the candidate buffering agents. For consistency, the solubility limit will be determined at an initial pH value of 7.0 in a solution with an initial boron concentration of 2500 ppm.

1. Prepare 1 liter of stock 27.5% boric acid solution (275 g boric acid + 725 g demineralized water); measure and record pH.

Perform the following steps for each candidate buffering agent and for trisodium phosphate and sodium hydroxide:

2. Using phase 2 data, determine the quantity of the test chemical required to achieve a pH of 7.0 in 100 mL of 2500 ppm B solution.
3. Heat the solution to boiling (expected to be near 103°C).
4. Add 19.23 (i.e., 27.5%/1.43%) times the quantity of test chemical determined in step 3 to the test solution; this will simulate concentration during core boiling.
5. Maintain solution at or near boiling, and add boric acid in ~0.5 g increments to determine solubility limit.

Acceptance criteria:

1. Presence of the buffering agent does not decrease the solubility of boric acid.

4.8 REFERENCES

- 4.8-1 LTR-CDME-06-65, Revision 1, "Test Plan: Evaluation of Alternative Buffering Agents for ECCS Application," April 2006.
- 4.8-2 WCAP-16530-NP, Revision 0, "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191," February 2006.
- 4.8-3 LA-UR-05-9177, "Integrated Chemical Effects Test Project: Test #5 Data Report," January 2006.

5.0 TEST RESULTS

Bench scale acceptability tests were performed at Fauske & Associates, Inc. in May 2006 according to the Test Plan presented in Section 4 above. The results of each of the test phases are provided below (Reference 5.8-1).

5.1 PHASE 1: DISSOLUTION TESTING RESULTS

This test phase determined the dissolution time for each buffer candidate. The results are presented in Table 5-1 below. The rate of dissolution for each buffer candidate was comparable to that for TSP. All of the tested buffers met the requirement established for complete dissolution in less than 3 times the dissolution time for TSP. The shortest time to achieve complete dissolution was 2:20 minutes for TSP, while the longest time was 5:17 min for sodium tripolyphosphate. In addition, with the exception of sodium gluconate, all of the buffers met the requirement for a pH greater than or equal to 8.0 upon cooling. Although the regulatory requirement for minimum pH is 7.0, a value of 8.0 was used in evaluation of the buffering agents since many plants target higher ECCS coolant pH values.

Table 5-1: Phase 1 Dissolution Testing Results

Buffering Agent	Mass [g]	Temperature of Water [°C]	Time for Dissolution [min]	pH of Solution @ 25°C
Sodium tetraborate decahydrate	10.0016	66.9	2:21	9.22
Sodium metaborate tetrahydrate	10.0513	68.2	2:32	10.20
Sodium tripolyphosphate	10.0169	68.8	5:17	9.24
Sodium gluconate	10.0240	67.5	3:22	6.93
Trisodium phosphate dodecahydrate	10.0344	67.4	2:20	11.90

Sodium gluconate was eliminated as a candidate from future testing due to failure to meet the pH requirement.

5.2 PHASE 2: PH ADJUSTMENT OF BORIC ACID SOLUTIONS RESULTS

During this test phase, titrations were performed to determine the quantity of each buffering agent required to adjust the boric acid solution to a pH of 8.0, 8.5, 9.0, 9.5, and 10.0 for boric acid concentrations of 2000 ppm, 2500 ppm, and 3000 ppm boron. The detailed test results for this phase are given in Appendix A. Figure 5.1 presents the mass of each buffer required to obtain pH values between 6.0 and 9.0 for a boric acid solution concentration of 2500 ppm.

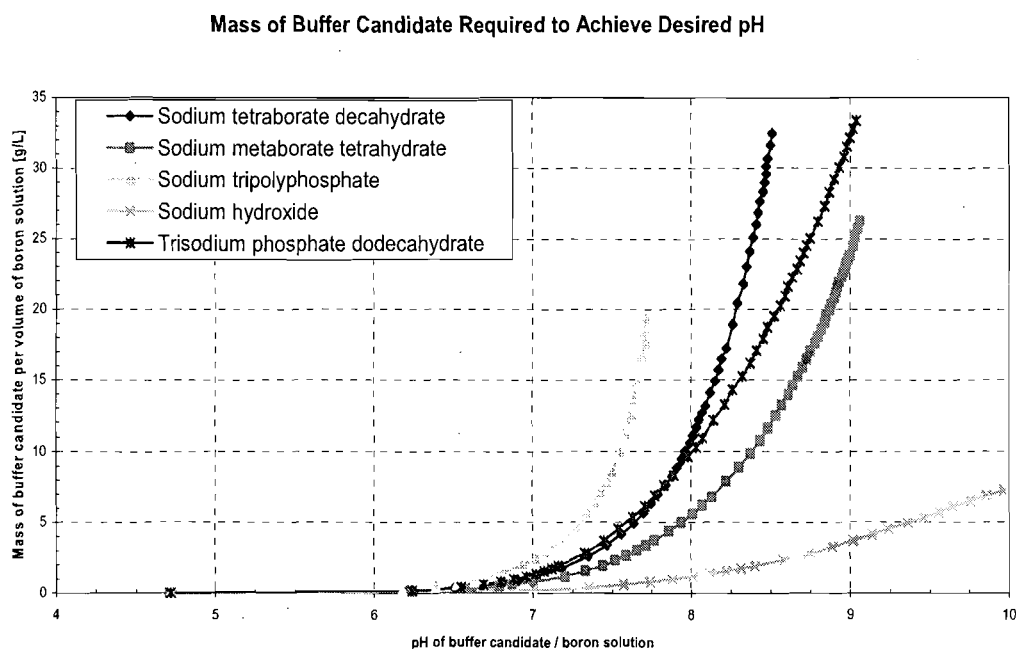
Figure 5-1: Mass of Buffer Candidate Required to Achieve Desired pH

Table 5-2 provides the mass of each buffering agent required to achieve a pH of 7.5, 8.0, and 8.5 for a boric acid concentration of 2500 ppm.

Table 5-2: Phase 2 pH Adjustment of 2500 ppm Boric Acid Solution Results

Buffering Candidate	Mass required to achieve a pH of 7.5 [g/L]	Mass required to achieve a pH of 8.0 [g/L]	Mass required to achieve a pH of 8.5 [g/L]
Sodium tetraborate decahydrate	3.669	10.89	31.63
Sodium metaborate tetrahydrate	2.219	5.543	11.99
Sodium tripolyphosphate	8.597	na	na
Sodium hydroxide	0.5301	1.156	2.176
Trisodium phosphate dodecahydrate	4.177	9.939	19.14

From the results above, a similar quantity of sodium metaborate, sodium tetraborate and trisodium phosphate are required to adjust the pH of the boric acid solution, while more sodium tripolyphosphate and much less sodium hydroxide are required to achieve the same target pH values. Because the pH values of 8.0 and 8.5 were not able to be achieved in the 2500 ppm boric acid solution with sodium tripolyphosphate; this buffer did not meet the requirement for a pH of 8.0 established in the Test Plan. However, sodium tripolyphosphate was maintained in the following test phases, as a minimum target pH of 7.0 may be sufficient for some plants.

5.3 PHASE 3: DISSOLUTION IN BORIC ACID AS A FUNCTION OF TEMPERATURE RESULTS

This test phase determined the dissolution rate of the buffering agents in a 2500 ppm boric acid solution at temperatures of 100°F (38°C), 150°F (66°C), and 200°F (93°C). Similar to the first test phase, the dissolution rates for the buffer candidates were found to be comparable to those for TSP, so all buffers met the acceptance criterion and were included in the next test phase. Appendix B contains the detailed results of this testing, while Table 5-3 below compares times for dissolution of the candidates at each temperature.

Table 5-3: Phase 3 Dissolution Testing Results as a Function of Temperature

Buffering Agent	Time for Dissolution (min)		
	Temperature = 100°F (38°C)	Temperature = 150°F (66°C)	Temperature = 200°F (93°C)
Sodium tetraborate decahydrate (NaTB)	1:40	0:44	0:29
Sodium metaborate tetrahydrate (NaMB)	1:37	1:06	0:37
Sodium tripolyphosphate (NaTPP)	2:24	1:42	0:49
Trisodium phosphate dodecahydrate (TSP)	2:27	1:55	0:42

5.4 PHASE 4: ALUMINUM AND CALCIUM ADDITION RESULTS

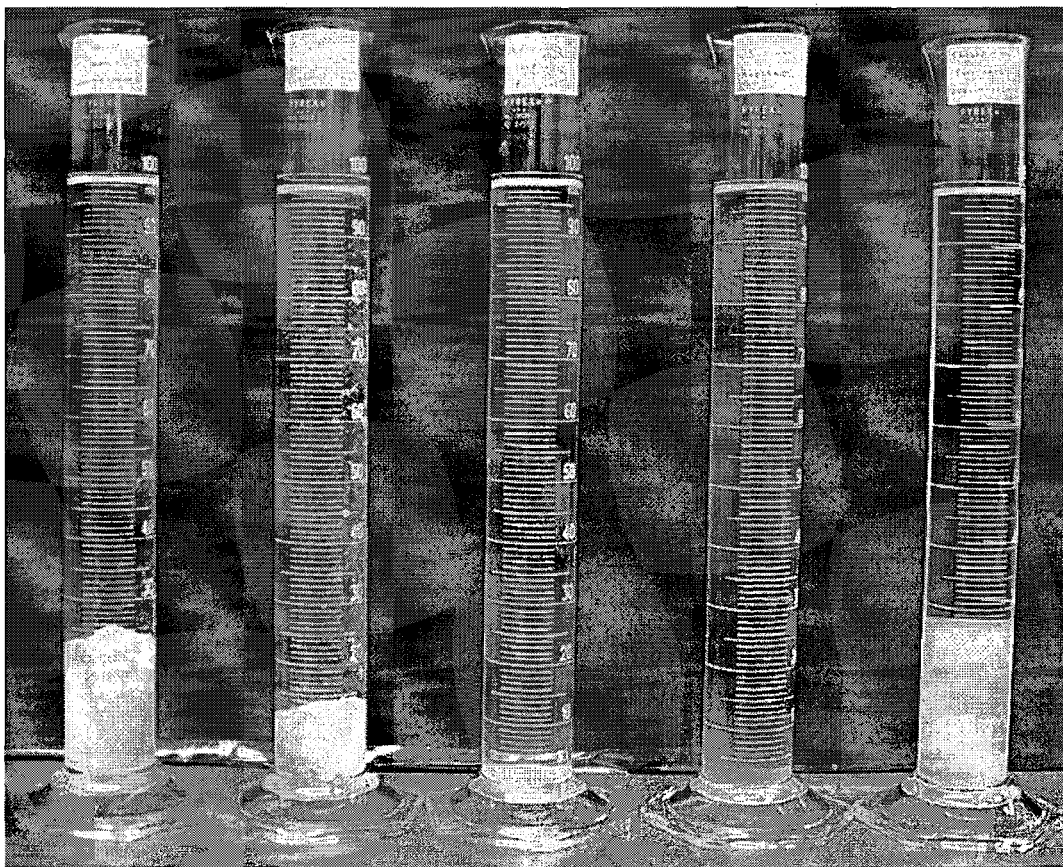
WCAP-16530 (Reference 5.8-2) demonstrated that the main metals that contribute to precipitate formation in PWR containment sumps are aluminum and calcium. Thus, aluminum and calcium salts were combined with 2500 ppm boric acid solutions and each candidate buffering agent in this test phase in order to determine whether precipitates would form. Although silicate is also a significant contributor to precipitate formation, no precipitates have been identified that contain both buffering agent anions and silicate.

5.4.1 Phase 4 Testing Results

For the addition of 500 ppm dissolved calcium from calcium chloride, a significant amount of precipitate formed with TSP, while very little precipitate formed with sodium tripolyphosphate and sodium hydroxide. A crystalline-appearing precipitate formed with sodium tetraborate and sodium metaborate. However, the volume of precipitate formed was less than observed for TSP. See Table 5-4 for the quantity of precipitate formation for each buffer. Figure 5-2 shows the precipitate formation approximately 48 hours after the addition of calcium.

Table 5-4: Phase 4 Calcium Addition Precipitate Formation

Buffering Candidate	Volume of Precipitate After 1 Hour [ml]	Volume of Precipitate After 2 Hours [ml]	Volume of Precipitate After 24 Hours [ml]	pH of Liquid After Ca Addition	Liquid Appearance
Sodium tetraborate decahydrate	29	29	24	8.44	Suspended precipitate particles
Sodium metaborate tetrahydrate	13	13	12	8.44	Suspended precipitate particles
Sodium tripolyphosphate	0	0	1	7.32	Slightly cloudy
Sodium hydroxide	0	0	<1	8.47	Slightly cloudy
Trisodium phosphate dodecahydrate	43	39	31	8.44	Clear

Figure 5-2: Precipitate Formation with Calcium Addition

Pictured above from left to right are sodium tetraborate, sodium metaborate, sodium tripolyphosphate, sodium hydroxide, and trisodium phosphate.

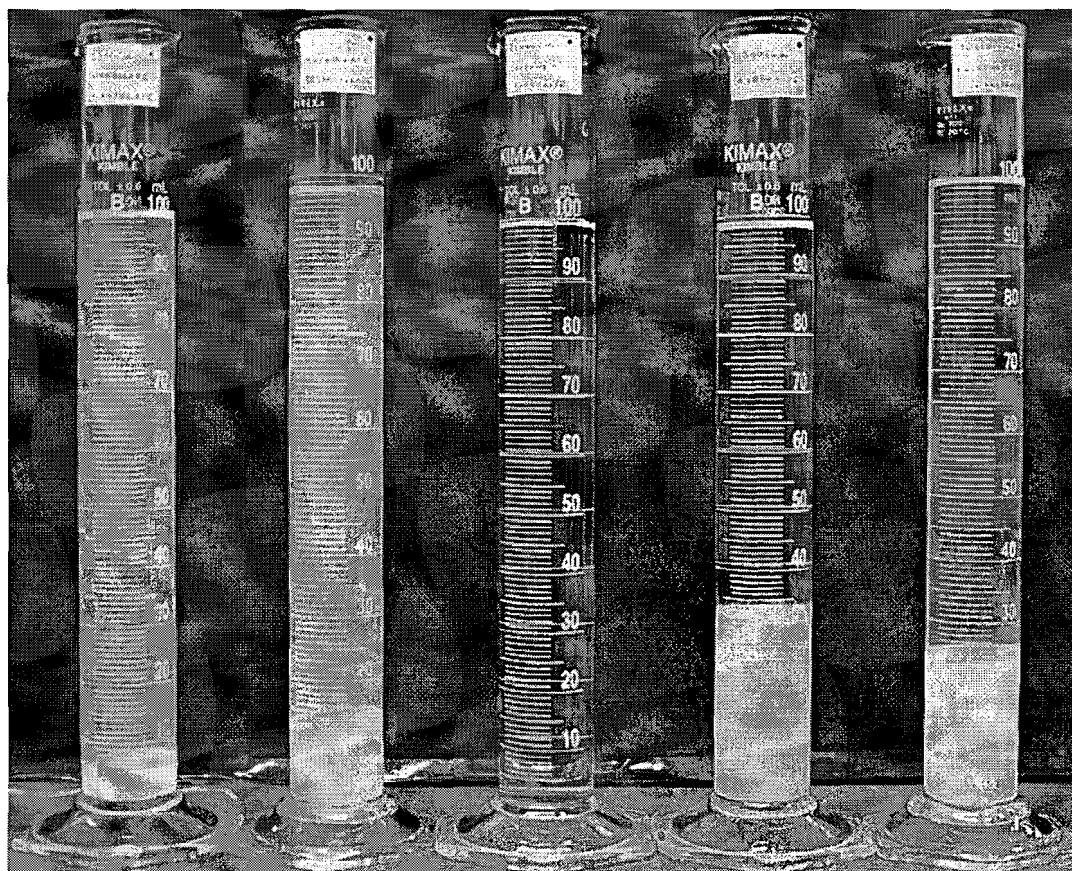
With the addition of 500 ppm of dissolved aluminum from aluminum nitrate, the most amount of precipitate formed with sodium hydroxide, while slightly less formed with trisodium phosphate. Cloudy precipitate formation was observed for sodium tetraborate and sodium metaborate, with

the quantities being less than those observed for TSP. No observable precipitate formed with sodium tripolyphosphate. Table 5-5 gives the amount of precipitate formed with each buffer, and Figure 5-3 presents the precipitate formation approximately 48 hours after aluminum addition.

Table 5-5: Phase 4 Aluminum Addition Precipitate Formation

Buffering Candidate	Volume of Precipitate After 1 Hour [ml]	Volume of Precipitate After 2 Hours [ml]	Volume of Precipitate After 24 Hours [ml]	pH of Liquid After Al Addition	Liquid Appearance
Sodium tetraborate decahydrate	10	9	7	7.98	Cloudy
Sodium metaborate tetrahydrate	21	19	15	7.70	Cloudy
Sodium tripolyphosphate	0	0	0	6.02	Clear
Sodium hydroxide	48	42	34	6.93	Clear
Trisodium phosphate dodecahydrate	32	28	23	7.97	Slightly Cloudy

Figure 5-3: Precipitate Formation with Aluminum Addition



Pictured above from left to right are sodium tetraborate, sodium metaborate, sodium tripolyphosphate, sodium hydroxide, and trisodium phosphate.

5.4.2 Phase 4A, B, and C Testing Results

Additional testing was performed with sodium tetraborate and sodium metaborate at lower calcium and aluminum concentrations (see Appendix C for test report) and at lower pH. Testing at elevated pH (initial value of 8.5) and lower concentrations of calcium and aluminum showed that precipitation did not occur before threshold values of >177 ppm (NaTB) or 75 ppm (NaMB) for aluminum and >254 ppm for calcium (both NaTB and NaMB). In the case of calcium, a reduction in the solution pH resulted in dissolution of the precipitate. At lower pH values (8.0 and lower), precipitation did not occur upon addition of calcium up to the maximum concentration of 700 ppm tested. For the additional testing with aluminum, pH had no discernible effect on precipitate formation.

5.4.3 Precipitate Characterization

Settling and filterability tests, similar to those conducted in Reference 5.8-2, were performed on the precipitates generated from the aluminum and calcium metal additions to the alternate buffer solutions as described in Section 5.4.1 (Reference 5.8-3). These tests provide a measure of the potential impact of the generated precipitates on containment sump screen head loss. In general, a higher settling rate and higher filter cake coefficient indicate that the precipitate will be less detrimental in causing a high pressure drop across the screen.

Precipitates with high settling rates may settle to the bottom of containment instead of remaining in suspension, and thus would not contribute to sump screen head loss. Also, the settling rate provides some measure of the particle size and/or morphology which may contribute to the pressure drop across the screen. In addition, high filter cake coefficients indicate that a precipitate in suspension will easily pass through a filter, avoiding contributing to head loss at the sump screen.

Table 5-6 below presents the measured settling volume of the precipitates formed in the Phase 4 testing and the calculated average settling rates. Little or no precipitate formed when aluminum was added to the NaTPP solution or when calcium was added to the NaOH solution, so these combinations are not presented below.

Table 5-6: Measured Settling Rates of Aluminum and Calcium Precipitates

Buffering Agents		Volume of PPT (ml)					Average Settling Rate (mm/hr)
		15 min	1 hr	2 hr	3 hr	4 hr	
Candidates with Al	NaMB w/ Al	10.3	10.3	10.3	10.3	10.3	ND ⁽¹⁾
	NaTB w/ Al	10.3	10.3	10.3	10.3	10.3	ND
Candidates with Ca	NaMB w/ Ca	0.8	0.7	0.6	0.6	0.6	4 ⁽²⁾
	NaTB w/ Ca	1.4	0.9	0.8	0.8	0.7	69
	NaTPP w/ Ca	10.4	9.9	5.7	4.2	2.1	9
Current Buffers with Al or Ca	NaOH w/ Al	10.1	10.1	10.0	9.9	9.9	0.5
	TSP w/ Al	10.4	10.4	10.4	10.4	10.4	ND
	TSP w/ Ca	9.7	8.2	6.7	5.4	4.3	10

- (1) ND = No measurable settling occurred over the four hour observation period.
- (2) The initial settling was instantaneous for the precipitates formed when calcium was added to the NaMB solution. An average settling rate of 4 mm/hr was calculated after this initial settling occurred.

Table 5-7 presents the calculated filter cake coefficients from the filterability testing. In the Phase 4 testing, no precipitate was visible from the addition of aluminum to the NaTPP solution. However, this solution did cause a pressure drop across the test filter in the filterability testing, so a filter cake coefficient was determined. A filter cake coefficient was not calculated for the precipitate formed from the combination of NaMB and calcium because no pressure drop occurred when the precipitate was added to the test loop.

Table 5-7: Calculated Filter Cake Coefficients

PPT Run	Sample Name	Mass PPT (g)	K_{fx}
1	NaMB w/ Al	0.0078	0.652
2	NaMB w/ Al	0.0073	0.883
3	NaMB w/ Al	0.0074	0.985
4	NaOH w/ Al	0.1390	0.100
6	NaOH w/ Al	0.0123	0.099
7	NaOH w/ Al	0.0095	1.572
10	NaTB w/ Al	0.0030	0.838
11	NaTPP w/ Al	0.0064	7.385
13	TSP w/ Al	0.0070	0.052
14	TSP w/ Al	0.0038	0.217
17	NaOH w/ Ca	0.0005	0.352
18	NaTB w/ Ca	0.0044	2.009
20	NaTPP w/ Ca	0.0005	0.043
22	TSP w/ Ca	0.0020	0.338

Overall, the precipitates formed from the addition of aluminum to the buffer solutions did not appear to settle over the four hour observation period. The calcium precipitates settled more quickly, with the calcium precipitates formed in the NaTB and NaMB solutions settling faster than those in the TSP and NaTPP solutions. An average settling rate of 69 mm/hr was calculated for the NaTB with calcium precipitate, and the initial settling of the NaMB with calcium precipitate occurred prior to the first volume measurement. In contrast, the calculated settling rates for the calcium precipitates formed in the TSP and NaTPP solutions were 10 mm/hr and 9 mm/hr, respectively.

In the filterability testing, the filter cake coefficients for the aluminum precipitates formed from the candidate buffers, NaTB, NaMB and NaTPP, appeared higher than those calculated for TSP and NaOH. Also, the filter cake coefficients for the precipitates generated from the calcium addition to the TSP, NaOH, and NaTPP solutions were lower than those determined for NaTB and NaMB. The calcium precipitate formed in NaTB had a filter cake coefficient of ~2.0 while the calcium precipitate formed in NaMB caused no discernible head loss.

The settling and filterability characteristics of the precipitates formed from the alternate buffers were examined in this section to provide a comparison among the buffer and metal combinations tested. These results do not provide an accurate measure of the precipitate contribution to sump screen head loss.

5.4.4 Buffering Capacity

Although not the primary intent of this testing, behavior of the buffering agents on addition of acidic aluminum nitrate solution provides data on the relative buffering capacity of the candidates. Examination of the pH values after liquid addition in Table 5-5 show that NaTB and TSP have nearly identical buffering capacity, with a pH change from 8.5 to about 8.0 after aluminum nitrate addition. The pH change for NaMB was slightly greater (8.5 to 7.7). As expected due to the limited buffering capacity of NaOH, the pH change was much greater (8.5 to 6.9). The initial pH of the NaTPP solution was lower (7.5), so a direct comparison of the buffering capacity of this agent is not possible.

To further examine the buffering capacity of NaTB relative to TSP, stock solutions of 1500 ppm boric acid were prepared and the pH was adjusted to 7.5 with either NaTB or TSP. In one test, solid boric acid (a weak acid) was added in 300 ppm increments and the resulting pH was measured. In a second test, hydrochloric acid (a strong acid) was added in 73 ppm increments. The results of this testing showed that NaTB and TSP have comparable buffering capacity (see Figures 5-4 and 5-5).

Figure 5-4: Buffering Capacity of NaTB and TSP for Boric Acid Addition

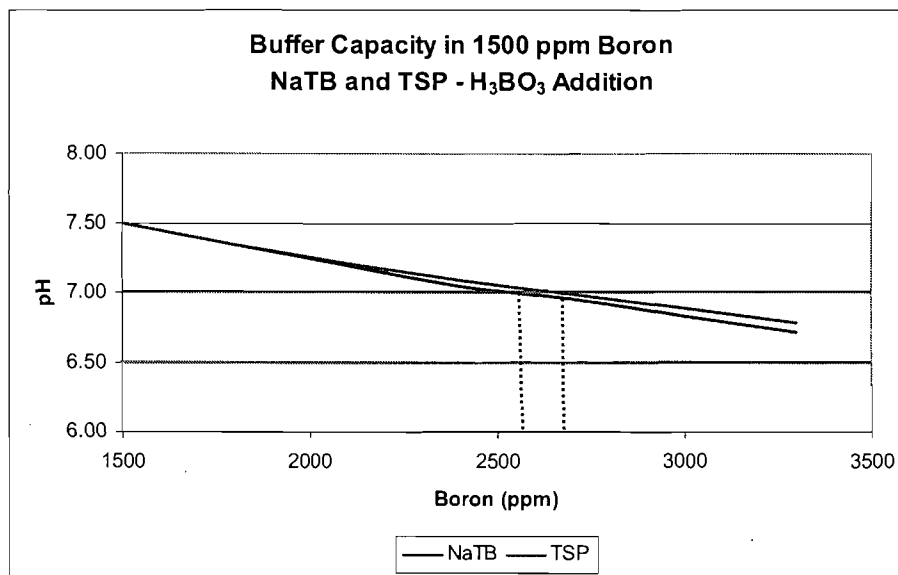
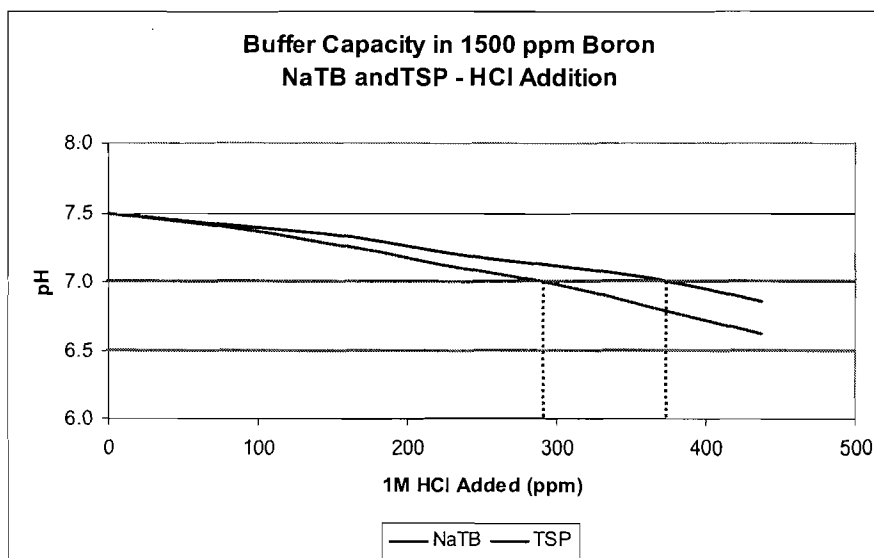


Figure 5-5: Buffering Capacity of NaTB and TSP for Hydrochloric Acid Addition

5.5 PHASE 5: CORROSION TESTING RESULTS

In the corrosion testing the effects of the buffering agents on key structural materials, which are typically comprised of aluminum or carbon steel, were determined. A508 carbon steel and commercially pure aluminum (A1100) metal coupons were purchased for this test phase. The coupons were submerged for two weeks at an elevated temperature in a 2500 ppm boric acid solution buffered to a pH value of 8.0 with each buffering agent, with the exception of sodium tripolyphosphate which was tested at an initial pH of 7.5. ICP (Inductively Coupled Plasma) analysis was then performed on the solutions to measure the quantity of iron or aluminum dissolved from the metal coupons.

Appendix C presents the detailed results from this test phase including the aluminum and carbon steel coupon mass loss and images of the metal coupons after two weeks of exposure to the buffering agent and boric acid solutions. Table 5-8 below presents the average coupon mass loss.

Table 5-8: Phase 5 Corrosion of Aluminum and Carbon Steel Metal Coupons

Buffering Agent	Steel Corrosion ⁽¹⁾ (g/m ²)	Aluminum Corrosion ⁽¹⁾ (g/m ²)
Sodium tetraborate decahydrate (NaTB)	1.03	35.1
Sodium metaborate tetrahydrate (NaMB)	0.62	9.49
Sodium tripolyphosphate (NaTPP)	54.8	24.5
Trisodium phosphate dodecahydrate (TSP)	1.00	2.92
Sodium hydroxide (NaOH)	1.04	14.8

(1) Average descaled weight loss of two specimens

From the results presented above, sodium tripolyphosphate caused the greatest corrosion to steel. For the remaining buffering agents, material loss was very low (1 g/m² or less). Aluminum corrosion was higher in the presence of sodium tetraborate and sodium tripolyphosphate than observed with TSP, while aluminum corrosion was lower in the presence of sodium metaborate than observed with sodium hydroxide.

The fact that dissolved silicate will also inhibit corrosion of submerged aluminum should be considered in the evaluation of the significance of the increased corrosion of submerged aluminum in the presence of sodium tetraborate and sodium tripolyphosphate relative to TSP. It should be noted that the inhibitory effects of phosphate and silicate are not included in the PWROG chemical model since these effects have not been quantified for all chemistry conditions.

The initial acceptance criteria established for this test phase was general corrosion no greater than observed with TSP or sodium hydroxide and no indication of localized corrosion. Sodium tripolyphosphate did not satisfy the criteria for either steel or aluminum. Sodium tetraborate and sodium metaborate satisfied both criteria for corrosion to carbon steel, and sodium metaborate satisfied both criteria for aluminum. While no indication of localized corrosion to aluminum was observed with sodium tetraborate, general corrosion was higher than observed with either TSP or sodium hydroxide. Based on the acceptable performance in other test phases, acceptable corrosion behavior with steel, and on consideration of silicate passivation of submerged aluminum, sodium tetraborate was retained in the test program.

5.6 PHASE 6: ENVIRONMENTAL EFFECTS TESTING

In this phase of testing, the effects of elevated temperature and humidity during storage on the buffering agents were determined by placing samples in an oven at 150°F (66°C) and 30% relative humidity. After 30 days of exposure at these conditions, the buffers were subjected to dissolution testing similar to that conducted in Phase 1.

The TSP was observed to be clumped together at the end of the 30 days but still appeared granular. The sodium tetraborate and sodium tripolyphosphate buffers formed a solid clump but were able to be removed from the test beaker. The sodium metaborate also formed a solid clump which appeared more crystalline and did not separate easily from the beaker. In addition, a significant decrease in the volume of the sodium metaborate was observed.

The time for dissolution after exposure to the elevated temperature and humidity for 30 days slightly increased for TSP from 2 minutes, 20 seconds for fresh product to 2 minutes, 37 seconds. The time for dissolution of the remaining buffering agents met the acceptance criterion of less than three times the time required for dissolution of TSP. Also, for all buffers tested, the pH of the solution after cooling was greater than 8.0 and no visible precipitates were observed. Table 5-9 summarizes the results of the environmental effects testing.

An initial test was performed at 100% humidity by placing a beaker of water in the oven. This condition was found to be too severe. After 2 weeks, the sodium tetraborate remained a solid, but both the trisodium phosphate and sodium metaborate had dissolved. Upon cooling, the trisodium phosphate solidified, but the sodium metaborate did not.

Table 5-9: Phase 6 Environmental Effects Testing Results

Buffer Candidate	Mass of Dissolution Sample [g]	Temperature of Water at Addition [°C]	Dissolution Time [min]	pH of Resulting Solution	Observations
Sodium tetraborate decahydrate	7.6877	67.9	3:29	9.21	Most of clump broke up when added to the water
Sodium metaborate tetrahydrate	7.4978	67.1	4:39	10.07	All of the loose sample was dissolved in 2:59 while sample that was still stuck to glass from the beaker took 4:39
Trisodium phosphate dodecahydrate	4.8797	66.9	2:37	11.39	Dissolved in the same fashion as in phase 1
Sodium tripolyphosphate	10.119	66.6	2:47	8.91	Most of clump broke up when added to the water

5.7 PHASE 7: BORIC ACID SOLUBILITY TESTING RESULTS

The impact on boric acid solubility of the candidate buffers was determined in this test phase. Table 5-10 demonstrates that all of the buffer candidates, in addition to the TSP and NaOH buffers, increase the solubility of boric acid.

Table 5-10: Phase 7 Determination of Buffer Impact on Boric Acid Solubility

Test Chemical	Mass of Test Chemical [g]	Initial Weight % Boric Acid	Mass of Boric Acid Dissolved [g]	Final Weight % Boric Acid	% Increase in Boric Acid Solubility
Sodium tetraborate decahydrate	2.1907	27.73	30.1687	29.85	7.65
Sodium metaborate tetrahydrate	1.5415	27.55	31.1107	30.37	10.24
Sodium tripolyphosphate	4.5097	27.47	35.9081	33.53	22.06
Sodium hydroxide	0.3994	27.58	30.7523	30.63	11.06
Trisodium phosphate dodecahydrate	2.5050	27.56	33.5439	32.07	16.36

5.8 REFERENCES

- 5.8-1 FAI/06-48, Revision 0, "Evaluation of Alternative Buffering Agents for ECCS Application: Bench Scale Acceptability Tests," May 2006.
- 5.8-2 WCAP-16530-NP, Rev. 0, "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191," February 2006.
- 5.8-3 LTR-CSA-06-52, "Settling and Filterability Tests on Precipitates from Alternate Buffer Testing," July 2006.

6.0 EVALUATION OF BUFFERING AGENT CRITERIA

This section provides the evaluation of the buffering agents for the criteria presented in Section 2.3. In the evaluation, the buffering agents are rated as EXCELLENT, GOOD or POOR for each criterion. A designation of EXCELLENT indicates the buffering agent is markedly superior to the other agents evaluated for the parameter of interest. A designation of POOR indicates that the performance of the buffering agent is markedly inferior for that attribute. The results of this evaluation are summarized in Table 6-1.

6.1 PRECIPITATION UNDER POST-LOCA CONDITIONS

Sodium Hydroxide (NaOH): due to the transient high pH conditions that may exist when NaOH is used, large quantities of dissolved aluminum and silicon may be released from containment materials (Reference 6.10-1). These dissolved species will eventually precipitate as sodium aluminum silicate and aluminum oxyhydroxide. Thus, sodium hydroxide is rated as POOR for precipitate formation.

Trisodium Phosphate (TSP): most metal phosphates are insoluble at the range of pH and temperature that would exist in the containment sump post-LOCA. However, in the presence of trisodium phosphate, aluminum would preferentially precipitate as an aluminum silicate or oxyhydroxide, and release of most other metals is low in the presence of trisodium phosphate. The exception is release of calcium from calcium-bearing materials such as calcium silicate insulation. In the presence of these calcium-bearing materials, release of calcium will result in formation of calcium phosphate (References 6.10-1 and 6.10-2). Thus, TSP is rated as GOOD for precipitate formation in the absence of calcium-bearing minerals, but POOR in the presence of such materials.

Sodium Tetraborate (NaTB): sparingly soluble metal borates may form at elevated pH and elevated levels of dissolved aluminum or calcium (see Section 5.4 and Reference 6.10-3). However, pH is easily maintained at 8.0 or less using NaTB, and in most cases dissolved aluminum/calcium concentrations will not exceed the threshold values. Thus, NaTB is rated as GOOD for precipitate formation.

Sodium Metaborate (NaMB): NaMB has the same chemical behavior as NaTB with respect to precipitate formation (References 6.10-4 and 6.10-5), and is therefore rated as GOOD for precipitate formation.

Sodium Tripolyphosphate (NaTPP): NaTPP is a good sequestering agent for most metals, particularly calcium and aluminum which are the predominant metals present in the coolant under post-LOCA conditions. Sequestration inhibits formation of precipitates, as a result, no metal precipitates would form in the presence of NaTPP. Therefore, NaTPP is rated as EXCELLENT for precipitate formation.

Sodium Gluconate (NaGlu): similar to NaTPP, NaGlu is a good sequestering agent for calcium and aluminum and would therefore be expected to be rated as EXCELLENT for precipitate formation. However, precipitate formation in the presence of NaGlu was not evaluated due to inability of NaGlu to establish a pH>7.0 in the presence of boric acid.

6.2 QUANTITY REQUIRED TO ADJUST PH >8.0

Sodium Hydroxide: as a strong base, very little NaOH is required to buffer boric acid solutions to a pH>8.0, relative to the other buffering agents tested. However, this effect also results in correspondingly lower buffering capacity when NaOH is used as the buffering agent. Therefore, plants using NaOH generally have higher target pH values than those using other buffering agents. Overall, NaOH is rated as EXCELLENT based solely on the minimal quantity required to rapidly raise pH.

Trisodium Phosphate: a manageable quantity of TSP is required to adjust the pH of boric acid solutions to >8.0. In this program, TSP is used as the yardstick by which the other buffering agents are rated. Thus, TSP is rated as GOOD for quantity required to adjust pH>8.0.

Sodium Tetraborate: the quantity of NaTB required to adjust the pH of the boric acid solutions tested (2000, 2500 and 3000 ppm) is comparable to the quantity of TSP required, up to pH 8.0. Above this value, relatively more NaTB is required to achieve an equivalent pH. Thus, NaTB is rated as GOOD for quantity required to adjust pH>8.0.

Sodium Metaborate: at a boron concentration of 2500 ppm, the quantity of NaMB required to adjust pH to 8.0 is about half of the quantity of TSP required. Thus, NaMB is rated as EXCELLENT for quantity required to adjust pH>8.0. Note, as an alternative to sodium hydroxide in liquid form, a comparable volume of NaMB solution would be required to achieve an equivalent pH.

Sodium Tripolyphosphate: at a boron concentration of 2500 ppm, the highest pH achievable using NaTPP is 7.6, and more than twice as much NaTPP as TSP is required to reach a pH value of 7.5 in the presence of 2500 ppm boron. Thus, NaTPP is rated as POOR for quantity required to adjust pH>8.0. NaTPP would be rated as GOOD if the nominal pH target were reduced to 7.5.

Sodium Gluconate: it was not possible to adjust the pH of boric acid solutions tested to >7.0 using NaGlu. Thus, NaGlu is rated as POOR for quantity required to adjust pH>8.0.

6.3 DISSOLUTION RATE

All of the buffering agents tested dissolved in boric acid solutions in six minutes or less, even after exposure to simulated containment conditions for 30 days. Thus, all buffering agents are rated as GOOD with respect to dissolution rate. Note, this parameter is not applicable to sodium hydroxide since NaOH is applied as a solution, rather than as a solid material.

6.4 AFFORDABILITY AND AVAILABILITY

Affordability and ready availability were considered in the initial selection of the buffering agents for testing. NaOH, TSP and NaTB are rated as EXCELLENT since they are already in use in PWR applications. All other buffering agents are rated as GOOD.

6.5 CORROSIVENESS

Sodium Hydroxide: under the transient high pH and high temperature conditions that may exist during the first several hours of a LOCA event, corrosion to aluminum metal may be elevated in the presence of NaOH. However, overall corrosion to aluminum and other metals when NaOH is used as the buffering agent is comparable to other agents tested. Thus, NaOH is rated as GOOD for corrosiveness.

Trisodium Phosphate: when TSP is used as the buffering agent, corrosion to steel is low and comparable to other agents tested. However, corrosion to aluminum is markedly lower. Thus, TSP is rated as EXCELLENT for corrosiveness.

Sodium Tetraborate: compared to the results obtained with other buffering agents, corrosion to submerged aluminum is higher in the presence of NaTB, but not excessive. Corrosion to steel is comparable. Thus, NaTB is rated as GOOD for corrosiveness.

Sodium Metaborate: corrosion to both steel and aluminum is low in the presence of NaMB, and comparable to other agents tested. Thus, NaMB is rated as GOOD for corrosiveness.

Sodium Tripolyphosphate: corrosion to steel is more than 50 times higher in the presence of NaTPP than observed with any of the other buffering agents tested. Corrosion to aluminum is also higher, but the difference is not as marked. Thus, NaTPP is rated as POOR for corrosiveness.

Sodium Gluconate: due to the inability to provide the required pH adjustment, corrosiveness of NaGlu was not evaluated.

6.6 EFFECT ON BORIC ACID SOLUBILITY

All of the buffering agents tested increased the solubility of boric acid, and thus all are rated as GOOD. The increase in solubility ranged from 7.65% for NaTB to 22.06% for NaTPP. At a consistent initial pH of 7.0, the relative ranking of the buffering agents was NaTPP > TSP > NaOH > NaMB > NaTB.

6.7 ENVIRONMENTAL STABILITY

Sodium Hydroxide: since NaOH is provided as a liquid, the environmental stability of this buffering agent was not tested. However, since NaOH is already in use, the environmental stability is considered GOOD.

Trisodium Phosphate: slight clumping of TSP occurred during exposure to simulated storage conditions for 30 days. However, the clumped product readily dissolved and provided the expected pH adjustment. As demonstrated by years of use in a PWR environment, TSP has good radiation stability. Thus, TSP is rated as GOOD for environmental stability.

Sodium Tetraborate: as with TSP, clumping of NaTB occurred during exposure to simulated storage conditions for 30 days. However, the clumped product readily dissolved and provided the expected pH adjustment. As demonstrated by years of use in a PWR environment, NaTB has good radiation stability. Thus, NaTB is rated as GOOD for environmental stability.

Sodium Metaborate: during initial tests at 100% humidity, NaMB completely and irreversibly dissolved. Under more moderate conditions, some dissolution of NaMB occurred, and recrystallization resulted in a solid clump that could not be mechanically removed from the test vessel. However, the solid material dissolved readily and provided the expected pH adjustment. Borates have good radiation stability, as demonstrated by long-term use of NaTB and sodium pentaborate in PWR and BWR applications (Reference 6.10-6). Overall, NaMB is rated as POOR for environmental stability when used as a solid. However, NaMB is highly soluble and could be used in liquid form in the same manner as NaOH. Under this usage condition NaMB would be rated as GOOD for environmental stability.

Sodium Tripolyphosphate: as with TSP and NaTB, clumping of NaTPP occurred during exposure to simulated storage conditions for 30 days. However, the clumped product readily dissolved and provided the expected pH adjustment. Based on the chemical similarity to TSP, NaTPP is expected to have good radiation stability (References 6.10-4 and 6.10-5). Thus, NaTPP is rated as GOOD for environmental stability.

Sodium Gluconate: due to the inability to provide the required pH adjustment, the environmental stability of NaGlu was not evaluated.

6.8 HABITABILITY CONCERNS

None of the buffering agents evaluated are considered hazardous during storage, or produce hazardous vapors. Concentrated sodium hydroxide solution is highly corrosive and extreme care must be taken in handling (Reference 6.10-7). Borates are poisonous at elevated concentration, so care must be taken in handling NaTB and NaMB during product transfer (Reference 6.10-8 and 6.10-9). TSP, NaTPP and NaGlu are relatively benign, and present minimal hazards during handling, e.g., dusts are irritants to the eyes and respiratory tract (References 6.10-10, 6.10-11, and 6.10-12).

6.9 OXIDE/CRUD RELEASE

With the exception of the sequestering agents, NaTPP and NaGlu, the buffering agents evaluated will not increase mobilization of radionuclides from the primary coolant system components or fuel. Therefore, NaOH, TSP, NaTB and NaMB are rated as GOOD for oxide/CRUD release. For NaOH, NaTB and NaMB, this conclusion is based on the fact that borates are present in the coolant during operation (Also note that the addition of NaOH to boric acid produces sodium

borate). For TSP, this conclusion is based on the experience with use of TSP as a water treatment chemical in PWR applications for many years. Exposure of the contaminated oxides to NaTPP or NaGlu may result in some mobilization of radionuclides. However, at elevated pH this effect would be expected to be minimal. In the absence of specific test data, NaTPP and NaGlu are rated as POOR for oxide/CRUD release.

While minimization of oxide/CRUD release is not a regulatory requirement for the buffering agent, it should be considered since the primary side components (including fuel) typically contain in excess of 10,000 curies of gamma-emitting radioisotopes, so a significant release of oxide/CRUD would result in elevated dose rates throughout containment.

Table 6-1: Summary of Characteristics of Candidate ECCS Buffering Agents

Criterion	Buffering Agent					
	NaOH	TSP	NaTB	NaMB	NaTPP	NaGlu
Precipitate Formation	Poor ⁽¹⁾	Good ⁽²⁾	Exc	Good	Exc	No Data
Quantity Required to Adjust pH >8.0	Exc	Good	Good	Exc	Poor	Poor
Dissolution Rate	N/A ⁽³⁾	Good	Good	Good	Good	Good
Affordability/Availability	Exc	Exc	Exc	Good	Good	Good
Corrosiveness	Good	Exc	Good	Good	Poor	No Data
Effect on Boric Acid Solubility	Good	Good	Good	Good	Good	No Data
Environmental Stability	Good	Good	Good	Poor ⁽⁴⁾	Good	No Data
Habitability Concern	Good	Exc	Good	Good	Exc	Exc
Oxide/CRUD Release	Good	Good	Good	Good	Poor	Poor

- (1) NaOH rated "Poor" due to elevated Al production.
- (2) TSP rated "Poor" under elevated Ca conditions.
- (3) NaOH provided as a 50% solution.
- (4) NaMB rated "Poor" based on behavior of solid material; "Good" rating as a liquid solution.

6.10 REFERENCES

- 6.10-1 WCAP-16530-NP, Rev. 0, "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191," February 2006.
- 6.10-2 LA-UR-05-6996, "Integrated Chemical Effects Test Project: Test #3 Data Report," October 2005.
- 6.10-3 U. S. Borax Product Profile, "Borax Decahydrate," January 2002.
- 6.10-4 Greenwood, N. N. and Earnshaw, A., Chemistry of the Elements (2nd Edition), Elsevier, 1998.
- 6.10-5 EPRI Report NP-5558-SL, Rev. 1, "Boric Acid Application Guidelines for Intergranular Corrosion Inhibition," December 1990.
- 6.10-6 Eagle Picher Technical Bulletin EP-SSGC-NAPB, Rev. 3, "Enriched Sodium Pentaborate," July 2002.
- 6.10-7 Mallinckrodt-Baker MSDS Number S4037, "Sodium Hydroxide Solutions (more than 10% NaOH)," October 2004.
- 6.10-8 U. S. Borax MSDS, "Borax Decahydrate," May 2000.
- 6.10-9 U. S. Borax MSDS, "Sodium Metaborate, 8 Mol," May 2000.
- 6.10-10 Mallinckrodt-Baker MSDS Number S4770, "Sodium Phosphate, Tribasic, 12-Hydrate," November 2005.
- 6.10-11 Arcos Organics MSDS ACC-01559, Rev. 5, "Sodium Tripolyphosphate," March 2003.
- 6.10-12 Hydrochem Industrial Services MSDS M097, "Sodium Gluconate," December 2000.

7.0 CONCLUSIONS AND RECOMMENDATIONS

The results of the evaluation presented in this report, as summarized in Section 6, indicate that sodium tetraborate decahydrate (Borax) is the best alternative to trisodium phosphate for plants with high loadings of calcium-bearing materials in containment. For plants without a high loading of calcium-bearing materials, TSP is determined to be the superior candidate based on the rankings in Table 6-1. For high-calcium plants, use of sodium tetraborate would eliminate concerns with precipitation of calcium phosphate. Calculations show that changing from TSP to NaTB buffering would reduce the total precipitate formation by more than 40 percent for a plant with a high loading of calcium-bearing materials. Additional considerations to support replacement of TSP with NaTB are:

- NaTB provides a comparable buffering capacity to TSP with a comparable quantity of buffering agent, so that no modification to the existing buffer delivery scheme would be required;
- No new types of precipitates would form at a target pH of 8.0 or less, irrespective of the calcium loading;
- Corrosion to steel structural materials would be comparable to that expected with TSP;
- As with TSP, NaTB addition increases the solubility of boric acid, and thereby provides contingency against boric acid precipitation under post-accident conditions;
- Sodium tetraborate has been evaluated for other potential chemical effects as part of the PWROG Program (Reference 7.1-1) and the ICET program (Reference 7.1-2); and
- Sodium tetraborate is already in use at ice condenser plants.

The results of this evaluation also show that sodium metaborate, in solution form, would be a suitable replacement for sodium hydroxide solution. The room temperature solubility of NaMB is >40% (Reference 7.1-3). Calculations show that changing from NaOH to NaMB buffering, combined with a target pH change from 8.5 to 7.5, would reduce the total precipitate formation by about 50 percent. The primary advantages of replacement of NaOH with NaMB are:

- The more moderate pH of NaMB solutions would reduce aluminum corrosion during the first several hours post-LOCA (per Reference 7.1-3, the pH of an 18% NaMB solution is 12.0, compared to >14.0 for 18% NaOH);
- In combination with boric acid, NaMB has a higher buffering capacity than NaOH, thereby allowing sufficient margin to allow a lower target pH for the final sump chemistry conditions and a concomitant decrease in release of aluminum and silica from containment materials;

- NaMB and NaOH provide a nearly identical increase in the solubility of boric acid, and thereby provides comparable contingency against boric acid precipitation under post-accident conditions;
- Under more moderate sump conditions, long-term corrosion of aluminum is less in the presence of NaMB than that experienced when NaOH buffering is used, while corrosion to carbon steel is low and essentially identical to NaOH; and
- Once dissolved in the containment sump fluid, the chemistry of the boric acid/NaMB buffer system is similar to that of the boric acid/NaTB system, thus existing evaluations of NaTB may be used to evaluate post-accident chemical effects.

7.1 REFERENCES

- 7.1-1 WCAP-16530-NP, Rev. 0, "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191," February 2006.
- 7.1-2 LA-UR-05-9177, "Integrated Chemical Effects Test Project: Test #5 Data Report," January 2006.
- 7.1-3 U. S. Borax Product Profile, "Sodium Metaborate, 8 Mol," February 2002.

8.0 CONSIDERATIONS FOR BUFFER REPLACEMENT

If a plant elects to replace their existing buffering agent, there are several major considerations, including the pH requirements for the equilibrium sump solution post-accident, the quantity of the replacement buffer necessary to meet these requirements, and the potential for additional evaluations to determine the replacement buffer impact.

8.1 PH REQUIREMENTS

A major concern in determining both the type and quantity of buffering agent required to mitigate post-accident consequences is the target pH of the sump solution. A minimum pH is required for fission product removal and to prevent stress corrosion cracking; however, as the pH increases, so does the total precipitate formation (Reference 8.3-1). In addition, the concern for hydrogen generation due to corrosion of aluminum increases with pH.

NRC Branch Technical Position MTEB 6-1 (Reference 8.3-2) provides a minimum pH criteria of 7.0 to avoid stress corrosion cracking based on a Westinghouse report (Reference 8.3-3). This report demonstrates that with a solution pH of 7.0 the time for initiation of cracking of stainless steel is 7.5 and 10 months for sensitized and non-sensitized specimens, respectively. Reference 8.3-2 also discusses an Oak Ridge National Laboratory report in which a minimum pH of 6.5 is recommended in order to reduce the likelihood of stress corrosion cracking (Reference 8.3-4).

In addition, Standard Review Plan Section 6.5.2 (Reference 8.3-5) provides a requirement for the pH of the sump solution to be sufficiently high to prevent significant long-term iodine reevolution. Thus, the equilibrium sump solution pH, after mixing and dilution with the primary coolant and ECCS injection should be equal to or greater than that required to prevent iodine reevolution. A minimum pH of 7.0 is specified in Reference 8.3-5 to provide long-term iodine retention and is conservatively based on Reference 8.3-6 which shows that at a pH of 6.0 or higher there is minimal conversion of iodine compounds to the volatile form of iodine.

8.2 RECOMMENDED EVALUATIONS

8.2.1 Chemical Model Reanalysis

If a plant decides to pursue replacement of the existing TSP or NaOH buffer with one of the recommended buffer candidates, the first step would be to perform the chemical model evaluation presented in Reference 8.3-1 to determine the impact on the chemical precipitate formation. The main concern for precipitate formation with the trisodium phosphate buffer is calcium phosphate, which forms due to the reaction of calcium-bearing materials with phosphate. For a change from TSP to sodium tetraborate, the quantity of chemical products which form will be reduced because the calcium phosphate precipitate will not form. In order to evaluate this change using the chemical model, the selection for TSP, which allows the calcium release to be assumed to form calcium phosphate, should not be made.

The elevated pH values which may be reached post-accident with the sodium hydroxide buffer lead to a significant amount of aluminum precipitate formation (Reference 8.3-1). Thus, a reduction in the final sump pH would aid in mitigating the precipitate formation; however, due to the limited buffering capacity of NaOH, it is not desirable to reduce the quantity of NaOH used to buffer the sump solution. Sodium metaborate is recommended as a suitable replacement for sodium hydroxide for plants which need to reduce the sump pH in order to alleviate the formation of chemical products and would prefer to maintain the current buffer delivery system. Again, the chemical model may be used to determine the precipitate formation for sodium metaborate. First, the reduced pH transient for the replacement buffer must be determined. Then, because sodium metaborate once dissolved behaves sufficiently similar to both TSP and sodium tetraborate, the dissolution testing results used to develop the chemical model in Reference 8.3-1 will also apply to NaMB.

8.2.2 Determination of Replacement Buffer Quantity

If a plant elects to replace the buffering agent, an essential step is the calculation of the amount of the replacement buffering agent required post-LOCA to achieve a desired pH. The determination of the required quantity is dependent upon the target pH, the ECCS boron concentration, and the sump volume. Equations are provided below for a target pH of 7.5 for replacement buffers sodium tetraborate and sodium metaborate.

For boron concentrations of 2000 to 3000 ppm, the amount of sodium tetraborate decahydrate required to achieve a target pH of 7.5 may be determined as:

$$\text{NaTB (kg/m}^3\text{)} = 6.62 - 0.0076 [\text{B}] + 2.56 \text{ E-}06 [\text{B}]^2,$$

where [B] is the boron concentration in the ECCS in ppm.

For example, for a boron concentration of 2500 ppm, 3.62 kg/m³ NaTB would be required to achieve a pH of 7.5. For a sump volume of 1,415 m³ (50,000 ft³), this equates to 5122 kg (11,282 pounds) of NaTB.

For boron concentrations of 2000 to 3000 ppm, the amount of sodium metaborate tetrahydrate required to achieve a target pH of 7.5 may be determined as:

$$\text{NaMB (kg/m}^3\text{)} = 9.34 - 0.0077 [\text{B}] + 1.94 \text{ E-}06 [\text{B}]^2,$$

where [B] is the boron concentration in the ECCS in ppm.

For example, for a boron concentration of 2500 ppm, 2.22 kg/m³ NaMB would be required to achieve a pH of 7.5. For a sump volume of 1,415 m³ (50,000 ft³), this equates to 3,141 kg (6,927 pounds) of NaMB. This equates to 6,382 liters (1,686 gallons) of 40 weight percent solution.

8.2.3 Evaluation of Buffer Replacement Impact

This test program was designed to qualify alternative buffers by comparing the candidate buffer properties to those of the buffers currently in use. Thus, the replacement of the existing buffer with an evaluated alternative is intended to have minimal impact on plant analyses.

Buffering agents are used to buffer the boric acid solution in order to meet the pH requirements discussed in Section 8.1. The areas of concern for pH include radiological consequences, stress corrosion cracking, hydrogen generation, and Equipment Qualification (EQ) concerns. The evaluation of the replacement buffer impact on these areas should be minor as long as the equilibrium sump solution pH remains unchanged and the time to reach this pH is similar. Additional analyses may be required if a plant desires to change their target pH or if the time for complete dissolution of the replacement buffer is significantly longer.

8.3 REFERENCES

- 8.3-1 WCAP-16530-NP, Rev. 0, "Evaluation of Post-Accident Chemical Effects in Containment Sump Fluids to Support GSI-191," February 2006.
- 8.3-2 Branch Technical Position MTEB 6-1, "pH for Emergency Coolant Water for PWRs," Revision 2, July 1981.
- 8.3-3 D.D. Whyte and L.F. Picone, "Behavior of Austenitic Stainless Steel in Post Hypothetical Loss of Coolant Environment," WCAP-7798-L, Westinghouse Nuclear Energy Systems, November 1971.
- 8.3-4 J.C. Griess and E.E. Creek, "Design Considerations of Reactor Containment Spray Systems – Part X, the Stress Corrosion Cracking of Types 304 and 316 Stainless Steel in Boric Acid Solutions," ORNL-TM-2412, Part X, Oak Ridge National Laboratory, May 1971.
- 8.3-5 NUREG-0800, US Nuclear Regulatory Commission Standard Review Plan, Office of Nuclear Reactor Regulation, Section 6.5.2 Containment Spray as a Fission Product Cleanup System, Revision 3, December 2005.
- 8.3-6 C. C. Lin, "Chemical Effects of Gamma Radiation on Iodine in Aqueous Solutions," *Journal of Inorganic and Nuclear Chemistry*, 42, pp. 1101-1107.

Appendix A: Detailed Results of Phase 2 Testing

Test Chemical: Sodium tetraborate decahydrate

Boron Concentration: 2000 ppm

Preparation of 1 liter of stock 2000 ppm boron solution:

Mass of H_3BO_3 : 11.4178 g

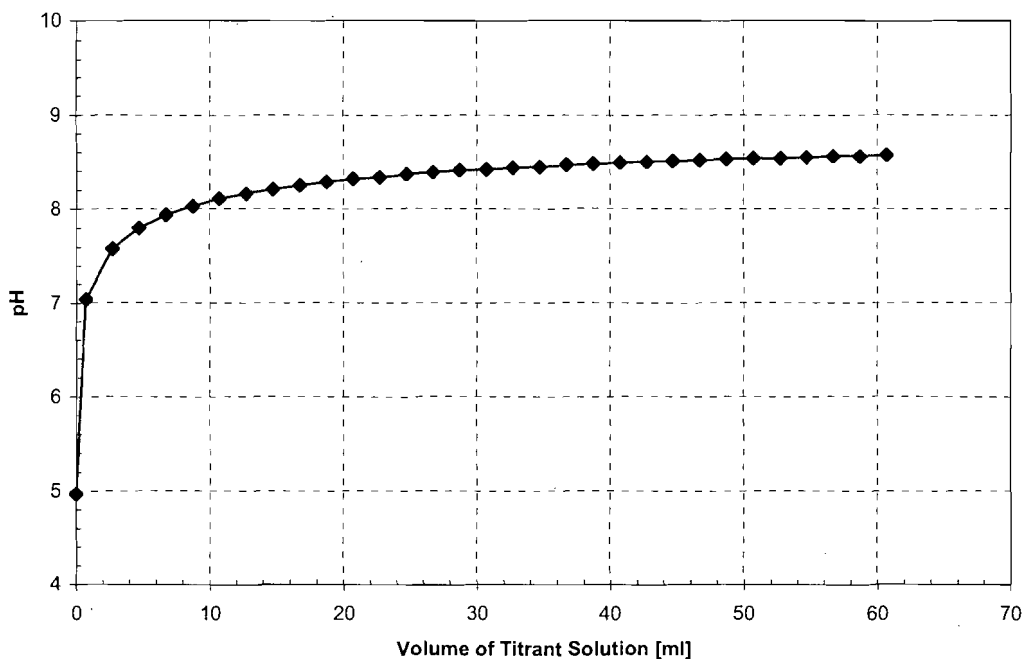
pH of solution: 4.90

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 2.8619 g

Mass of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$: 12.5622 g *

pH of solution: 8.80



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	8.0	0.40
8.5	42.7	2.15

* - did not completely dissolve

Test Chemical: Sodium tetraborate decahydrate

Boron Concentration: 2000 ppm¹

Preparation of 1 liter of stock 2000 ppm boron solution:

Mass of H_3BO_3 : 11.4178 g

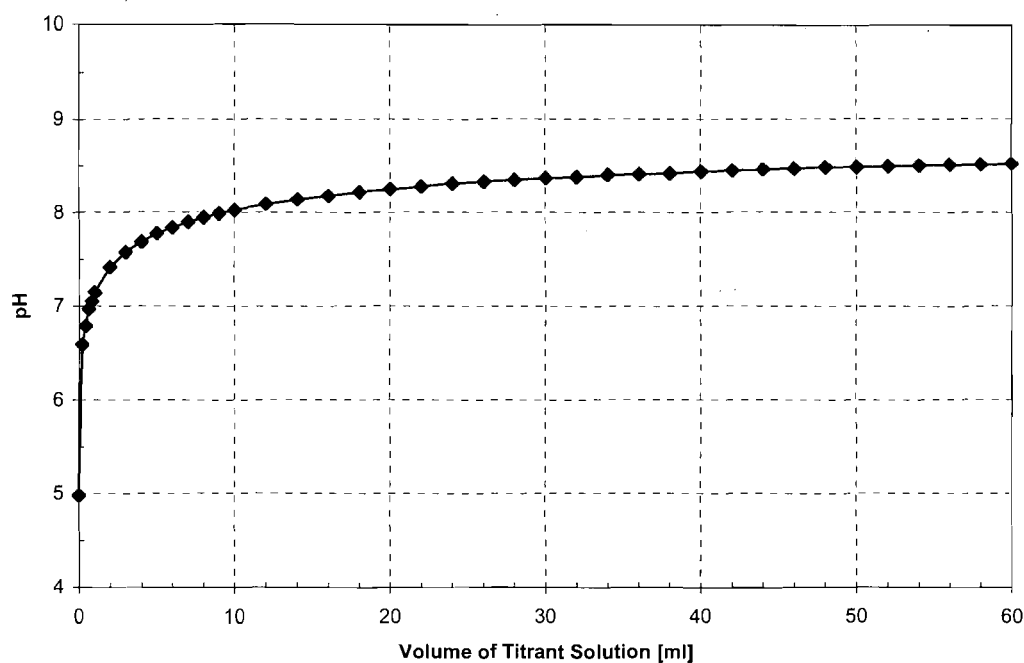
pH of solution: 4.90

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 2.8571 g

Mass of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$: 10.9644 g

pH of solution: 8.76



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	9.3	0.41
8.5	54	2.4

¹ Repeated previous test with less sodium tetraborate decahydrate to assure it completely dissolved.

Test Chemical: Sodium tetraborate decahydrate

Boron Concentration: 2500 ppm

Preparation of 1 liter of stock 2500 ppm boron solution:

Mass of H_3BO_3 : 14.3040 g

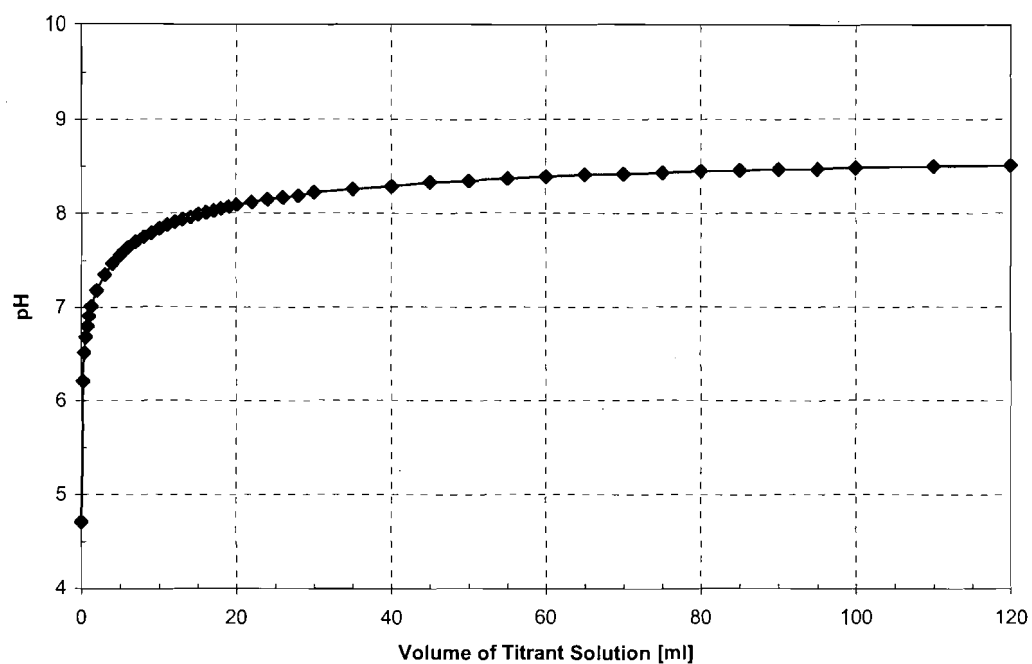
pH of solution: 4.67

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 3.5769 g

Mass of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$: 11.5024 g

pH of solution: 8.67



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	15.5	0.713
8.5	110	5.1

Test Chemical: Sodium tetraborate decahydrate

Boron Concentration: 3000 ppm

Preparation of 1 liter of stock 3000 ppm boron solution:

Mass of H_3BO_3 : 17.1472 g

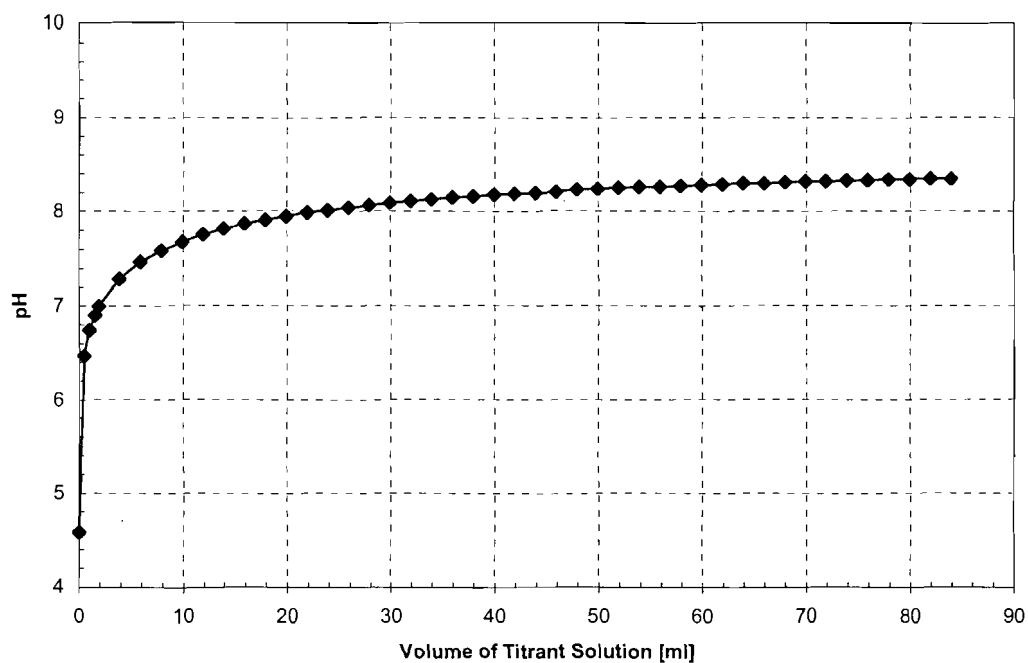
pH of solution: 4.55

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 4.2892 g

Mass of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$: 12.5208 g

pH of solution: 8.60



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	22.9	1.15

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium metaborate tetrahydrate

Boron Concentration: 2000 ppm

Preparation of 1 liter of stock 2000 ppm boron solution:

Mass of H_3BO_3 : 11.4178 g

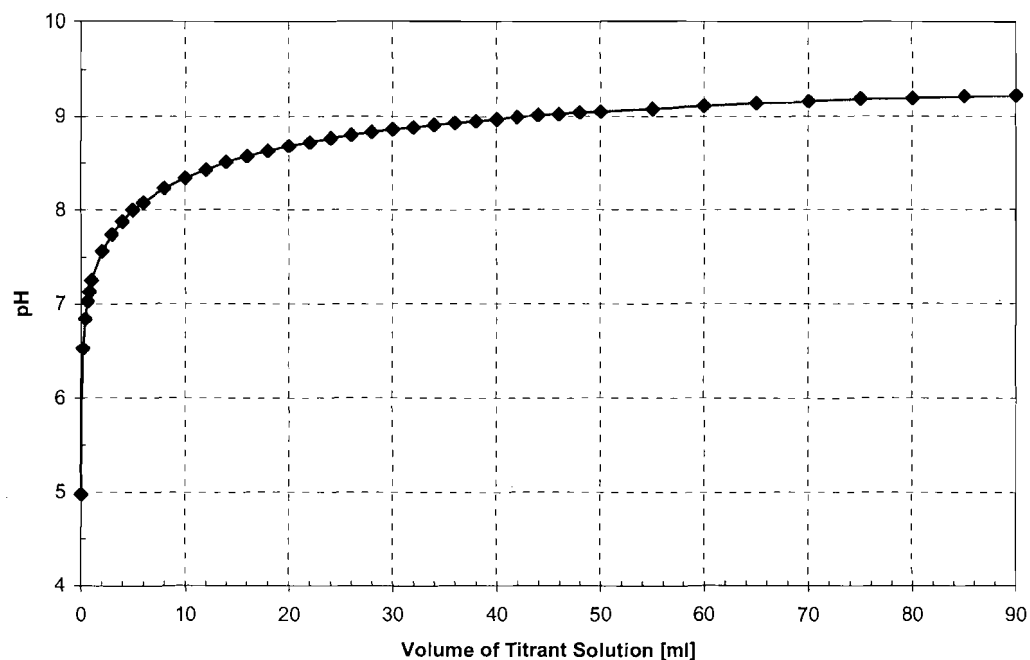
pH of solution: 4.90

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 2.8593 g

Mass of $\text{NaBO}_2 \cdot 4\text{H}_2\text{O}$: 10.2493 g

pH of solution: 9.55



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	5.0	0.20
8.5	13.8	0.566
9.0	43	1.8

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium metaborate tetrahydrate

Boron Concentration: 2500 ppm

Preparation of 1 liter of stock 2500 ppm boron solution:

Mass of H_3BO_3 : 14.3040 g

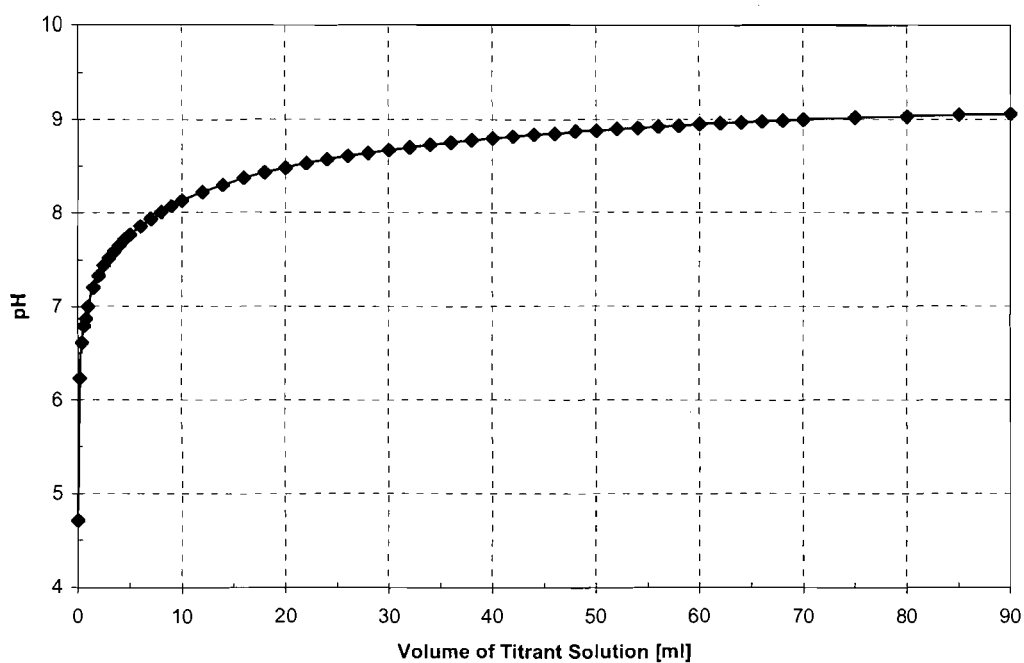
pH of solution: 4.67

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 3.5810 g

Mass of $\text{NaBO}_2 \cdot 4\text{H}_2\text{O}$: 10.2034 g

pH of solution: 9.41



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	7.9	0.32
8.5	21	0.85
9.0	70	3

Test Chemical: Sodium metaborate tetrahydrate

Boron Concentration: 3000 ppm

Preparation of 1 liter of stock 3000 ppm boron solution:

Mass of H_3BO_3 : 17.1472 g

pH of solution: 4.55

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 4.2806 g

Mass of $\text{NaBO}_2 \cdot 4\text{H}_2\text{O}$: 12.5170 g *

pH of solution: 9.44

* - did not dissolve

Test Chemical: Sodium metaborate tetrahydrate

Boron Concentration: 3000 ppm

Preparation of 1 liter of stock 3000 ppm boron solution:

Mass of H_3BO_3 : 17.1472 g

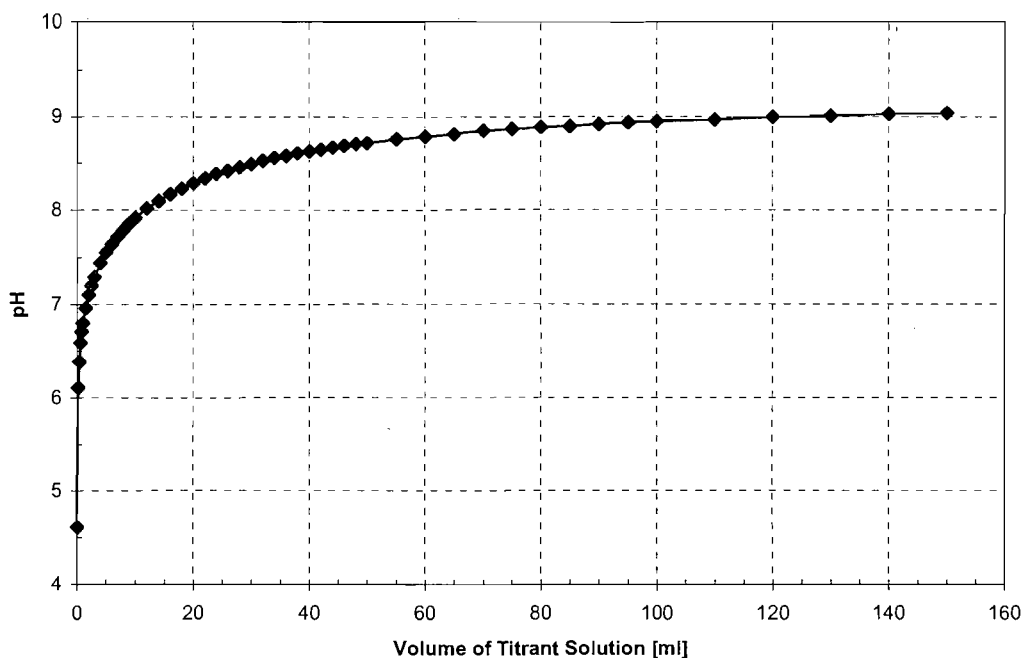
pH of solution: 4.55

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 4.2892 g

Mass of $\text{NaBO}_2 \cdot 4\text{H}_2\text{O}$: 10.1987 g

pH of solution: 9.29



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	11.6	0.473
8.5	30.5	1.24
9.0	120.0	4.895

Test Chemical: Sodium tripolyphosphate

Boron Concentration: 3000 ppm

Preparation of 1 liter of stock 3000 ppm boron solution:

Mass of H_3BO_3 : 17.1472 g

pH of solution: 4.55

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 4.2865 g

Mass of $\text{Na}_5\text{P}_3\text{O}_{10}$: 12.5086 g

pH of solution: 7.80 *

* - not high enough to achieve pH of 8

Test Chemical: Sodium tripolyphosphate

Boron Concentration: 2500 ppm

Preparation of 1 liter of stock 2500 ppm boron solution:

Mass of H_3BO_3 : 14.3040 g

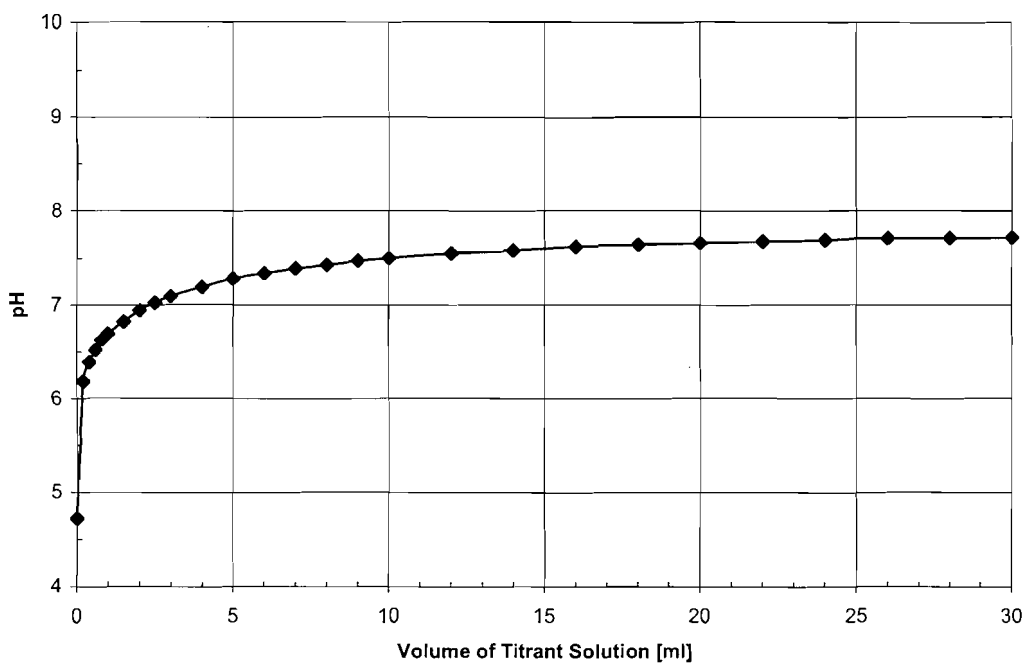
pH of solution: 4.67

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 3.5739 g

Mass of $\text{Na}_5\text{P}_3\text{O}_{10}$: 12.8961 g

pH of solution: 7.88



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
7.0	2.4	0.12
7.5	10	0.52

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium hydroxide

Boron Concentration: 2000 ppm

Preparation of 1 liter of stock 2000 ppm boron solution:

Mass of H_3BO_3 : 11.4178 g

pH of solution: 4.90

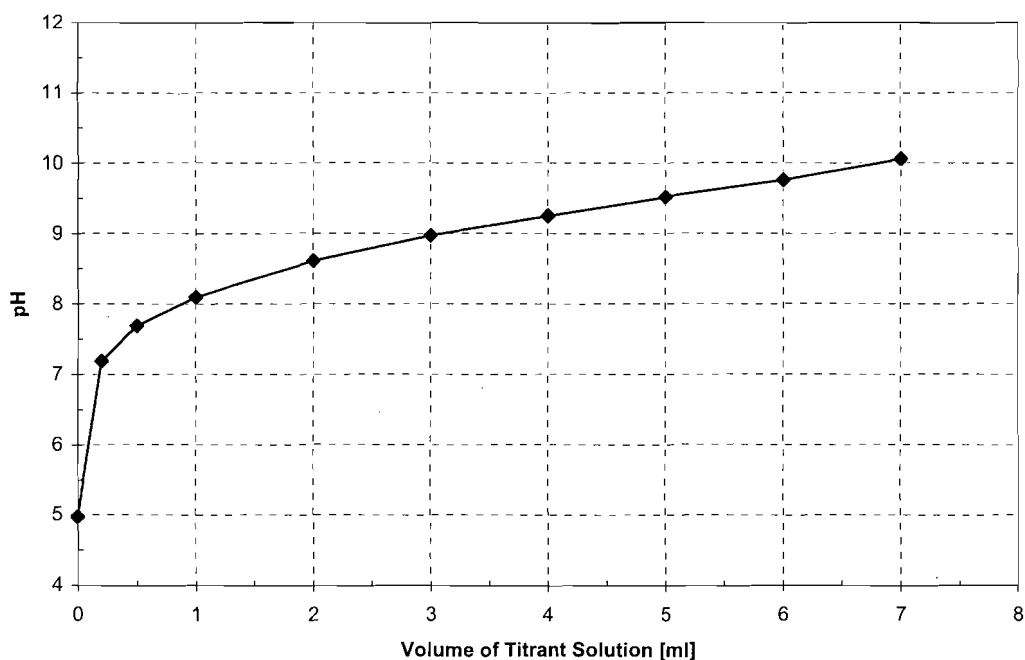
Preparation of 250 ml of titrant solution:

Volume of H_2O : 234 ml

Mass of H_3BO_3 : 2.8646 g

Mass of 50% NaOH: 25.0414 g

pH of solution: 12.77



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	0.94	0.047
8.5	1.8	0.090
9.0	3.1	0.16
9.5	4.9	0.25
10.0	6.8	0.34

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium hydroxide

Boron Concentration: 2500 ppm

Preparation of 1 liter of stock 2500 ppm boron solution:

Mass of H_3BO_3 : 14.3040 g

pH of solution: 4.67

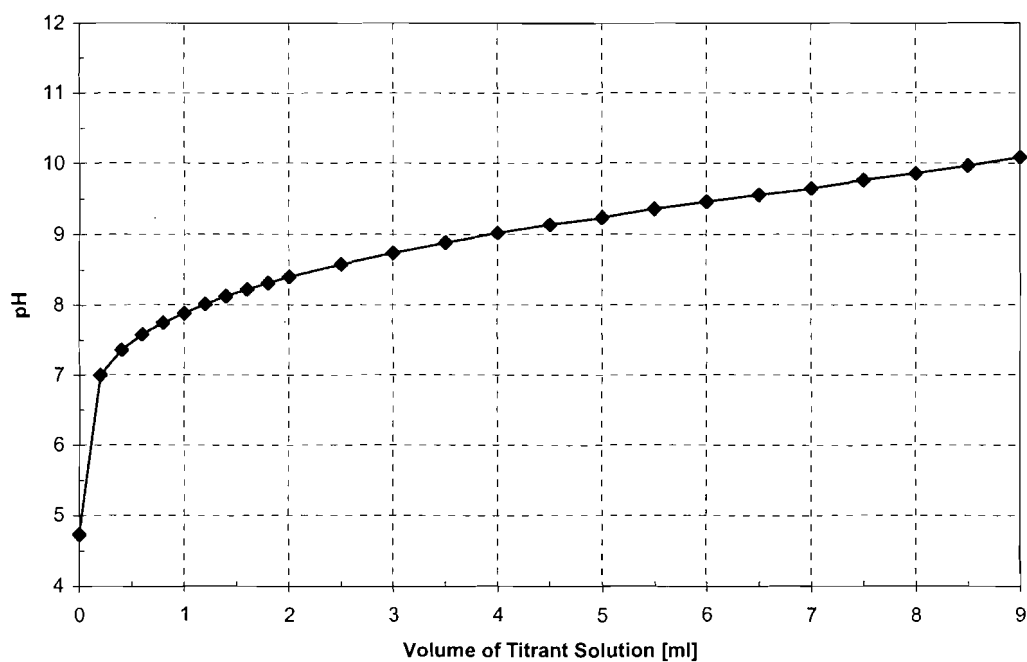
Preparation of 250 ml of titrant solution:

Volume of H_2O : 234 ml

Mass of H_3BO_3 : 3.5694 g

Mass of 50% NaOH: 24.9742 g

pH of solution: 12.80



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	1.2	0.060
8.5	2.3	0.11
9.0	3.9	0.19
9.5	6.3	0.31
10.0	8.6	0.43

Test Chemical: Sodium hydroxide

Boron Concentration: 3000 ppm

Preparation of 1 liter of stock 3000 ppm boron solution:

Mass of H_3BO_3 : 17.1472 g

pH of solution: 4.55

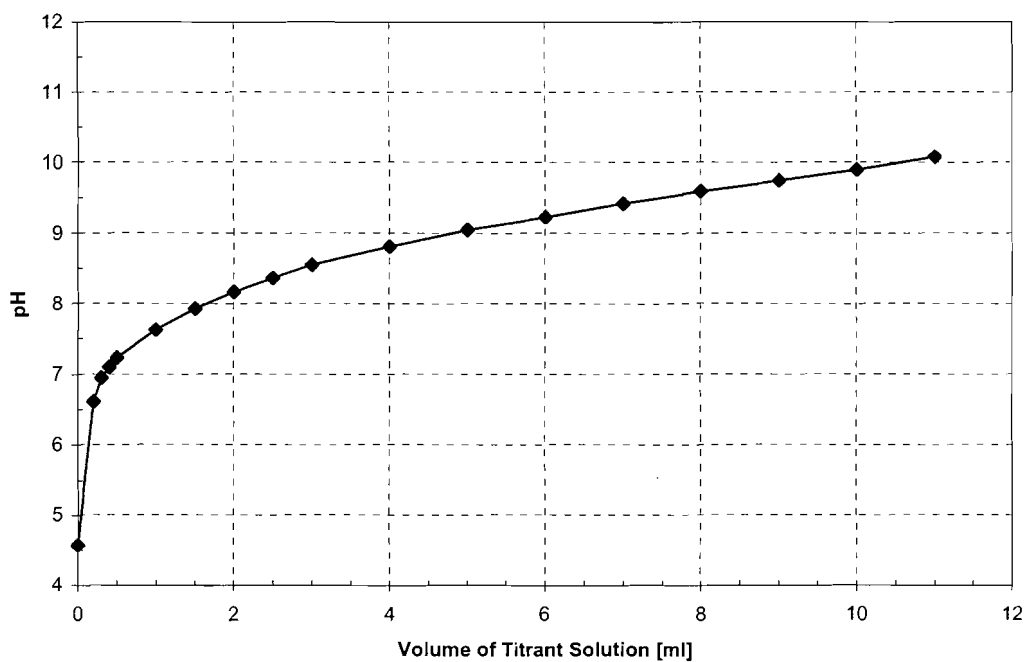
Preparation of 250 ml of titrant solution:

Volume of H_2O : 234 ml

Mass of H_3BO_3 : 4.2848 g

Mass of 50% NaOH: 25.0188 g

pH of solution: 12.66



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	1.6	0.080
8.5	2.9	0.15
9.0	4.8	0.24
9.5	7.5	0.38
10.0	10.6	0.530

Test Chemical: Trisodium phosphate dodecahydrate

Boron Concentration: 2000 ppm

Preparation of 1 liter of stock 2000 ppm boron solution:

Mass of H_3BO_3 : 11.4178 g

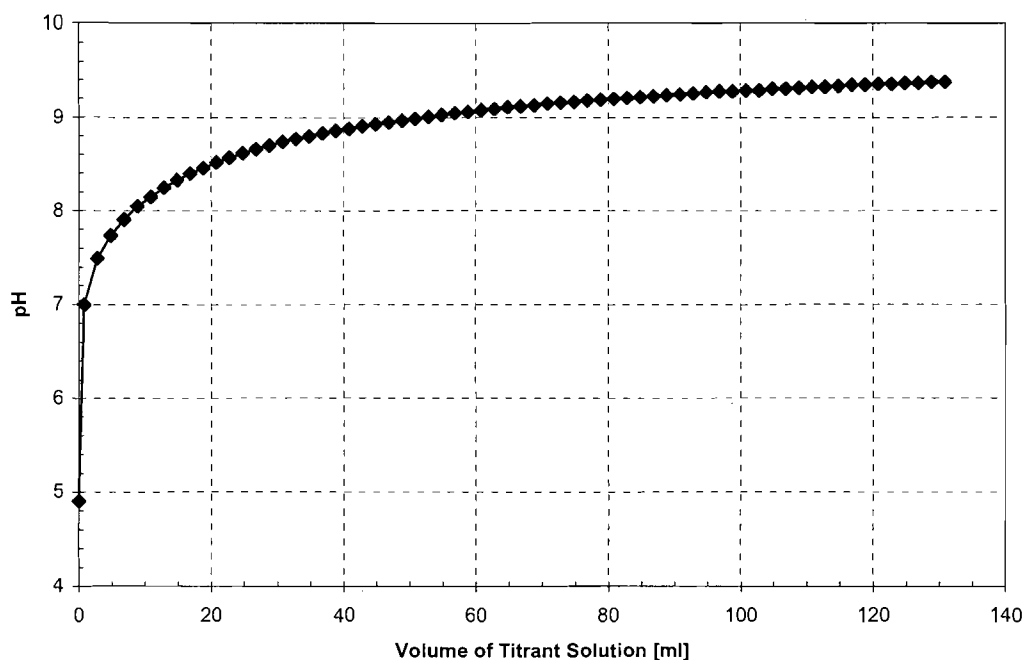
pH of solution: 4.90

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 2.8553 g

Mass of $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$: 12.5106 g

pH of solution: 9.89



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	8.1	0.41
8.5	20.1	1.01
9.0	51.8	2.59

Test Chemical: Trisodium phosphate dodecahydrate

Boron Concentration: 2500 ppm

Preparation of 1 liter of stock 2500 ppm boron solution:

Mass of H_3BO_3 : 14.3040 g

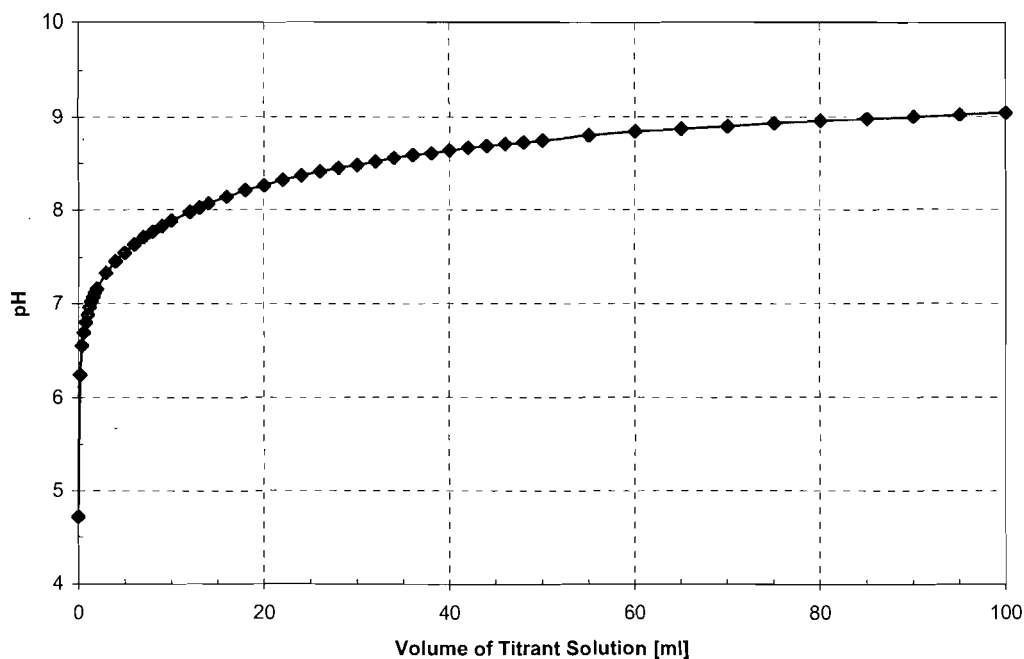
pH of solution: 4.67

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 3.5775 g

Mass of $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$: 12.5044 g

pH of solution: 9.56



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	12.4	0.620
8.5	31	1.6
9.0	90	4.5

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Trisodium phosphate dodecahydrate

Boron Concentration: 3000 ppm

Preparation of 1 liter of stock 3000 ppm boron solution:

Mass of H_3BO_3 : 17.1472 g

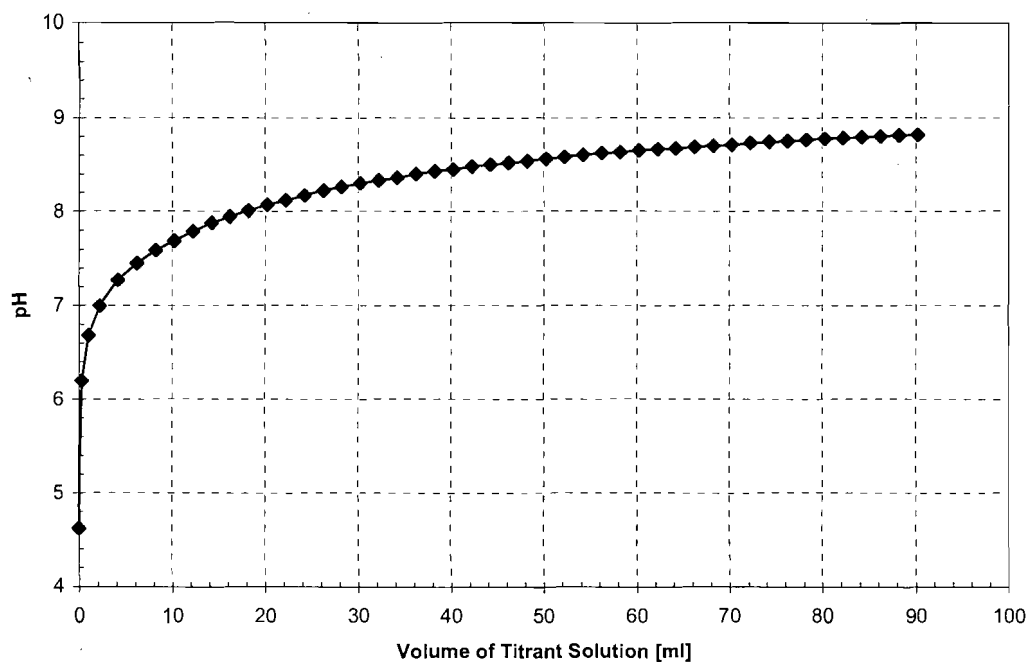
pH of solution: 4.55

Preparation of 250 ml of titrant solution:

Mass of H_3BO_3 : 4.2725 g

Mass of $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$: 12.5107 g

pH of solution: 9.38



pH Value	Volume of Titrant [ml]	Mass of Test Chemical [g]
8.0	17.9	0.896
8.5	44.2	2.21

Appendix B: Detailed Results of Phase 3 Testing

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tetraborate decahydrate

Boron Concentration: 2500 ppm

Desired pH: 8.0

Temperature [°C]	Mass Required [g]	Mass Used [g]	Time for Dissolution [min]	pH After Cooldown
38.6	1.089	1.0910	1:40	7.98
65.3	1.089	1.0902	0:44	7.97
94.2	1.089	1.0888	0:29	7.96

Test Chemical: Sodium metaborate tetrahydrate

Boron Concentration: 2500 ppm

Desired pH: 8.0

Temperature [°C]	Mass Required [g]	Mass Used [g]	Time for Dissolution [min]	pH After Cooldown
39.0	0.5543	0.5584	1:37	8.01
67.0	0.5543	0.5563	1:06	8.01
92.0	0.5543	0.5588	0:37	7.99

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tripolyphosphate

Boron Concentration: 2500 ppm

Desired pH: 7.5

Temperature [°C]	Mass Required [g]	Mass Used [g]	Time for Dissolution [min]	pH After Cooldown
39.8	0.8597	0.8619	2:24	7.49
66.2	0.8597	0.8645	1:42	7.49
94.2	0.8597	0.8521	0:49	7.47

Test Chemical: Trisodium phosphate dodecahydrate

Boron Concentration: 2500 ppm

Desired pH: 8.0

Temperature [°C]	Mass Required [g]	Mass Used [g]	Time for Dissolution [min]	pH After Cooldown
39.0	0.9939	0.9943	2:27	7.99
66.3	0.9939	0.9901	1:55	7.98
91.5	0.9939	0.9934	0:42	7.98

Appendix C: Detailed Results of Phase 4 Testing

C.1 PHASE 4 DETAILED TEST RESULTS

Test Chemical: Sodium tetraborate decahydrate

Boron concentration: 2500 ppm

Desired pH: 8.5

Preparation of 100 ml of stock 10,000 ppm Ca solution:

Mass of CaCl_2 : 2.9004 g

Actual Concentration: 10,475 ppm

pH of solution: 7.37

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1938 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 65.3 °C

Temperature after addition of Ca solution: 63.0 °C

Observations:

- Precipitate was formed immediately upon addition of Ca solution.
- After 60 minutes, there was 29 ml of precipitate at the bottom of the 100 ml graduated cylinder.
- After 120 minutes, the temperature was 22.0 °C and there was 29 ml of precipitate at the bottom of the 100 ml graduated cylinder.
- After 24 hours, there was 24 ml of precipitate at the bottom of the 100 ml graduated cylinder. Also, some precipitate was still suspended in the liquid.
- pH after addition of Ca: 8.44

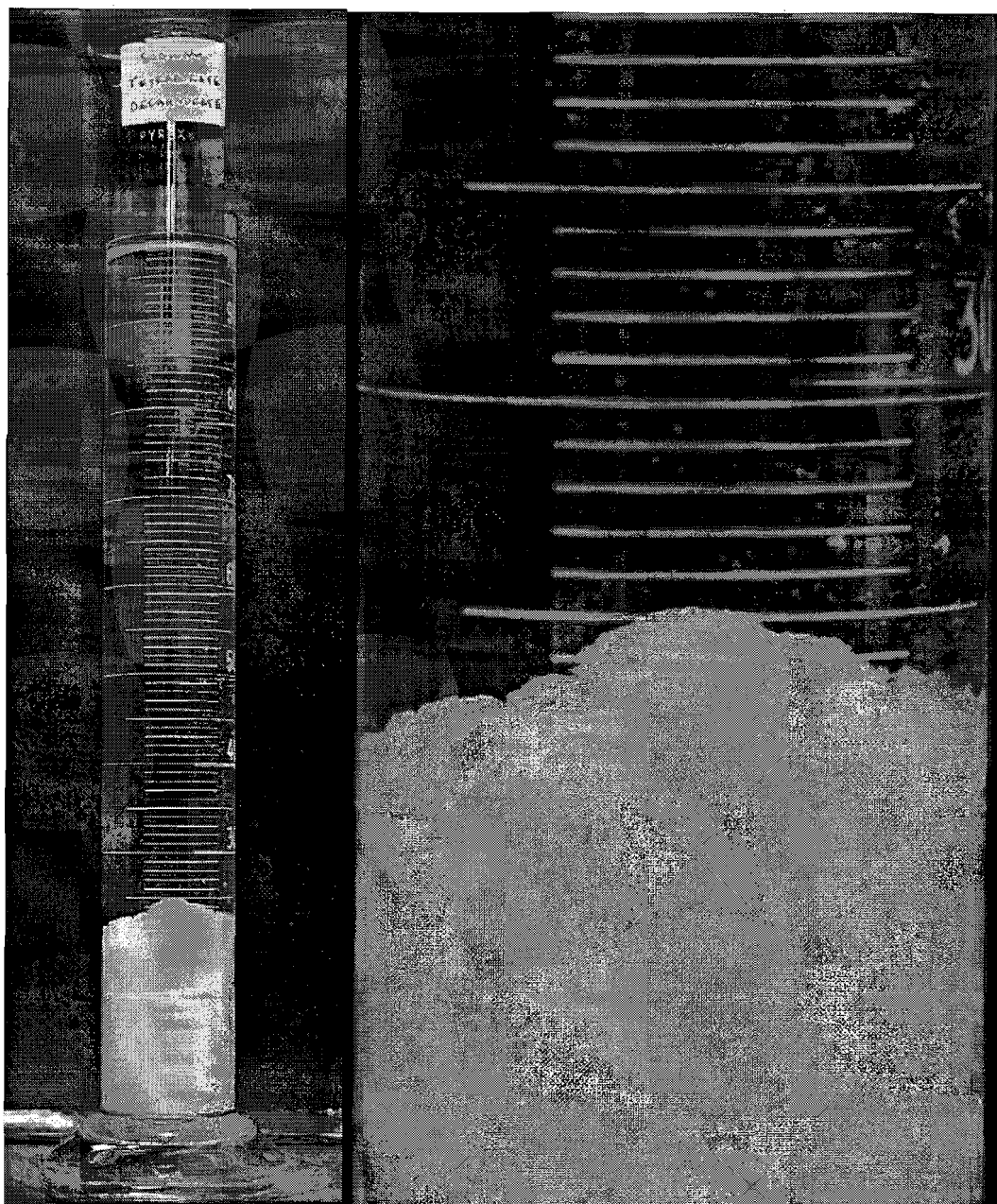


Figure C-1: Overall and close-up views of precipitate formed by sodium tetraborate decahydrate and calcium.

Test Chemical: Sodium metaborate tetrahydrate

Boron concentration: 2500 ppm

Desired pH: 8.5

Preparation of 100 ml of stock 10,000 ppm Ca solution:

Mass of CaCl_2 : 2.9004 g

Actual Concentration: 10,475 ppm

pH of solution: 7.37

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2165 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 67.3 °C

Temperature after addition of Ca solution: 64.8 °C

Observations:

- Precipitate was formed immediately upon addition of Ca solution.
- After 60 minutes, the temperature was 26.5 °C and there was 13 ml of precipitate at the bottom of the 100 ml graduated cylinder.
- After 120 minutes, the temperature was 21.8 °C and there was 13 ml of precipitate at the bottom of the 100 ml graduated cylinder.
- After 24 hours, there was 12 ml of precipitate at the bottom of the 100 ml graduated cylinder. Also, some precipitate was still suspended in the liquid.
- pH after addition of Ca: 8.44

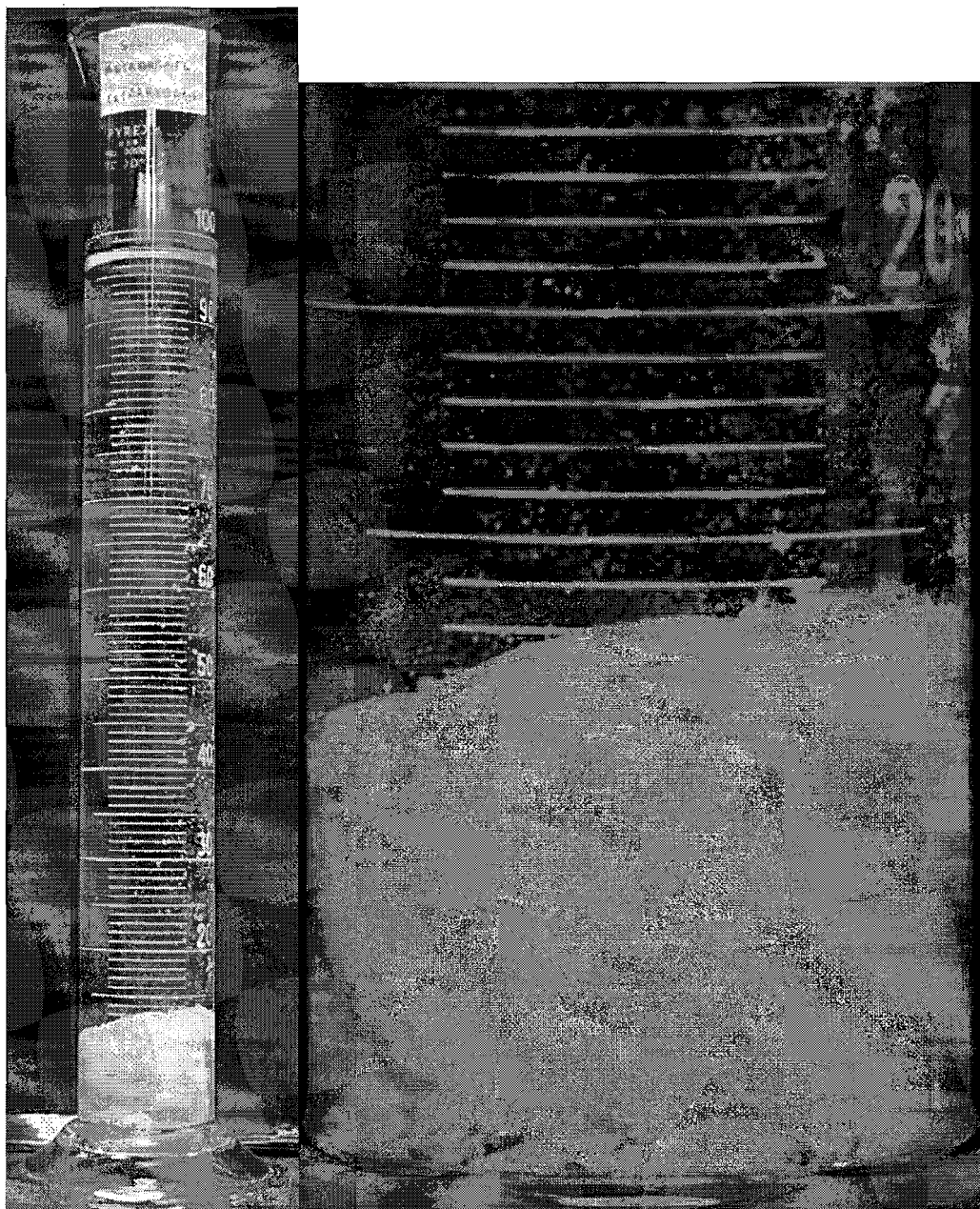


Figure C-2: Overall and close-up views of precipitate formed by sodium metaborate tetrahydrate and calcium.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tripolyphosphate

Boron concentration: 2500 ppm

Desired pH: 7.5

Preparation of 100 ml of stock 10,000 ppm Ca solution:

Mass of CaCl_2 : 2.9004 g

Actual Concentration: 10,475 ppm

pH of solution: 7.37

Mass of buffering candidate:

Mass required: 0.8597 g

Mass used: 0.8651 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 68.6 °C

Temperature after addition of Ca solution: 65.8 °C

Observations:

- A very small amount of precipitate was formed immediately upon addition of Ca solution.
- After 60 minutes, the temperature was 29.2 °C. The liquid was slightly cloudier than immediately after the addition of the Ca solution. No precipitate layer on the bottom of the graduated cylinder yet.
- After 120 minutes, the temperature was 22.8 °C and there was a very thin layer of precipitate at the bottom of the 100 ml graduated cylinder. The liquid is still cloudy.
- After 24 hours, there was about 1 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid was slightly cloudy.
- pH after addition of Ca: 7.32

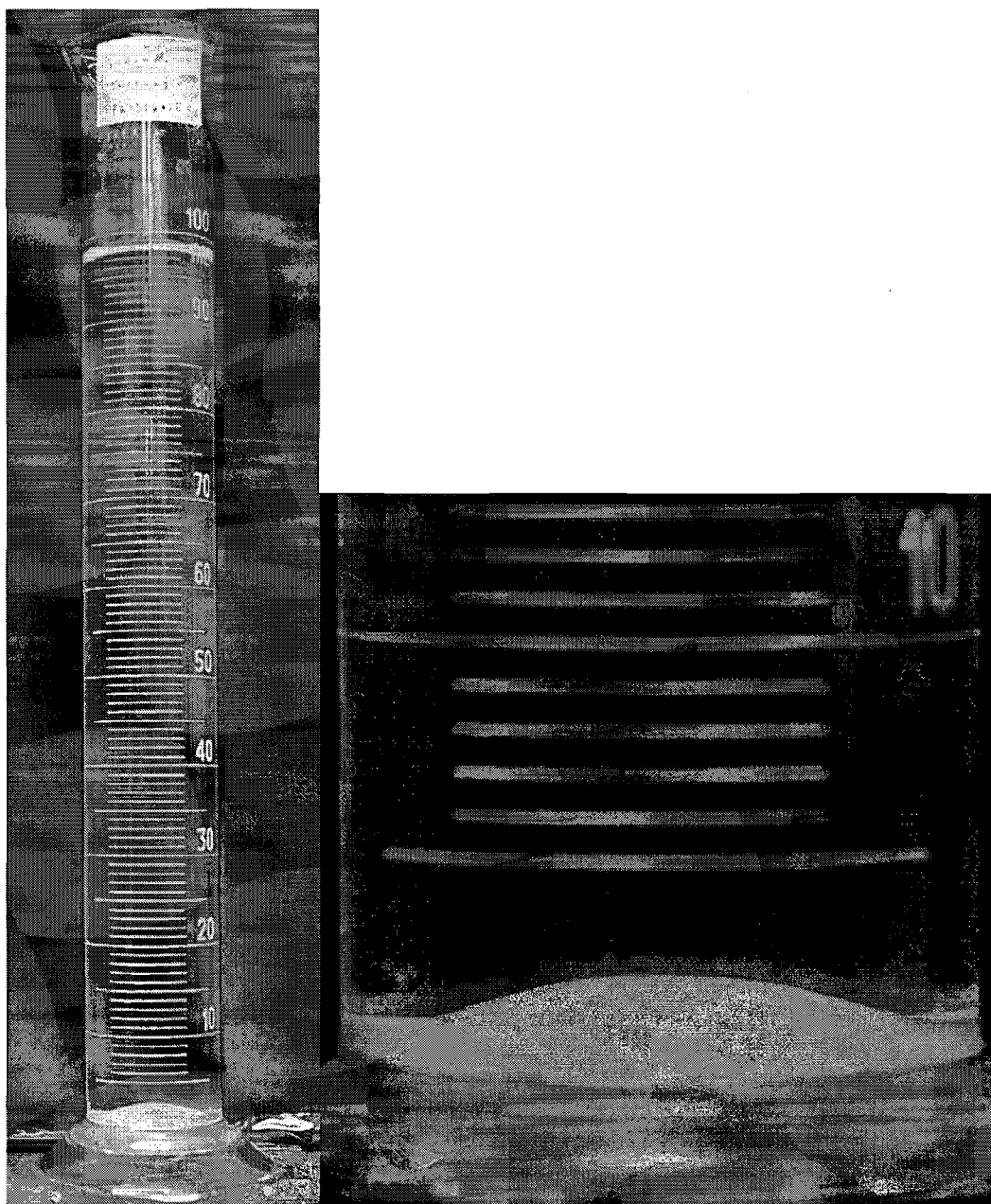


Figure C-3: Overall and close-up views of precipitate formed by sodium tripolyphosphate and calcium.

Test Chemical: Sodium hydroxide

Boron concentration: 2500 ppm

Desired pH: 8.5

Preparation of 100 ml of stock 10,000 ppm Ca solution:

Mass of CaCl_2 : 2.9004 g

Actual Concentration: 10,475 ppm

pH of solution: 7.37

Mass of buffering candidate:

Mass required: 0.2176 g

Mass used: 0.2354 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 68.7 °C

Temperature after addition of Ca solution: 66.5 °C

Observations:

- Precipitate was formed immediately upon addition of Ca solution.
- After 60 minutes, the temperature was 27.2 °C and there was no visible precipitate in the liquid.
- After 120 minutes, the temperature was 22.4 °C and there was no visible precipitate in the liquid.
- After 24 hours, the liquid appeared slightly hazy and a very thin layer of precipitate was at the bottom of the graduated cylinder.
- pH after addition of Ca: 8.47

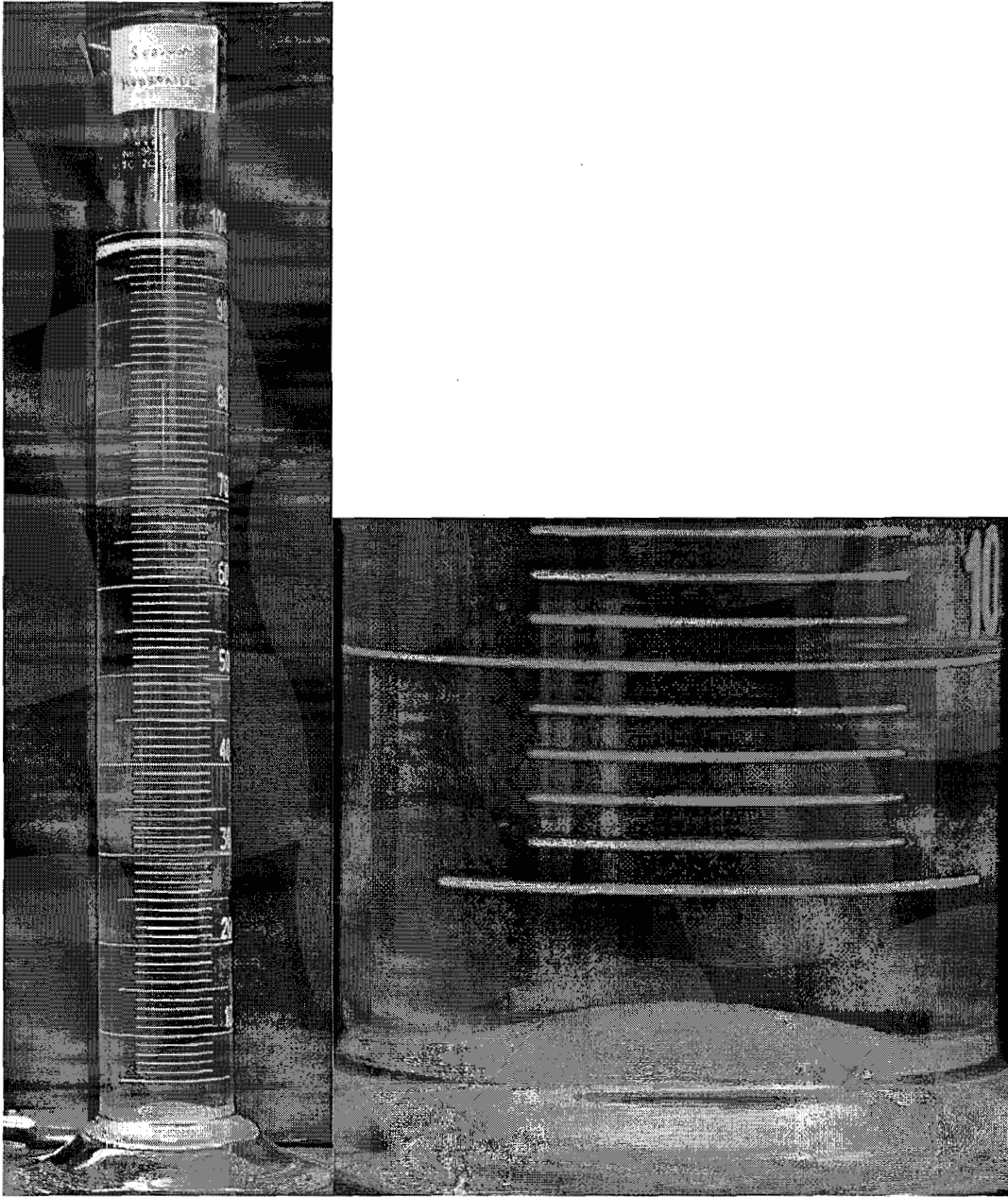


Figure C-4: Overall and close-up views of the precipitate formed by sodium hydroxide and calcium.

Test Chemical: Trisodium phosphate dodecahydrate

Boron concentration: 2500 ppm

Desired pH: 8.5

Preparation of 100 ml of stock 10,000 ppm Ca solution:

Mass of CaCl_2 : 2.9004 g

Actual Concentration: 10,475 ppm

pH of solution: 7.37

Mass of buffering candidate:

Mass required: 1.914 g

Mass used: 1.9787 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 67.8 °C

Temperature at addition of Ca solution: 64.0 °C

Temperature after addition of Ca solution: 61.8 °C

Observations:

- Precipitate was formed immediately upon addition of Ca solution.
- After 60 minutes, the temperature was 27.3 °C and there was 43 ml of precipitate at the bottom of the 100 ml graduated cylinder.
- After 120 minutes, the temperature was 23.6 °C and there was 39 ml of precipitate at the bottom of the 100 ml graduated cylinder.
- After 24 hours, there was 31 ml of precipitate at the bottom of the 100 ml graduated cylinder.
- pH after addition of Ca: 8.44

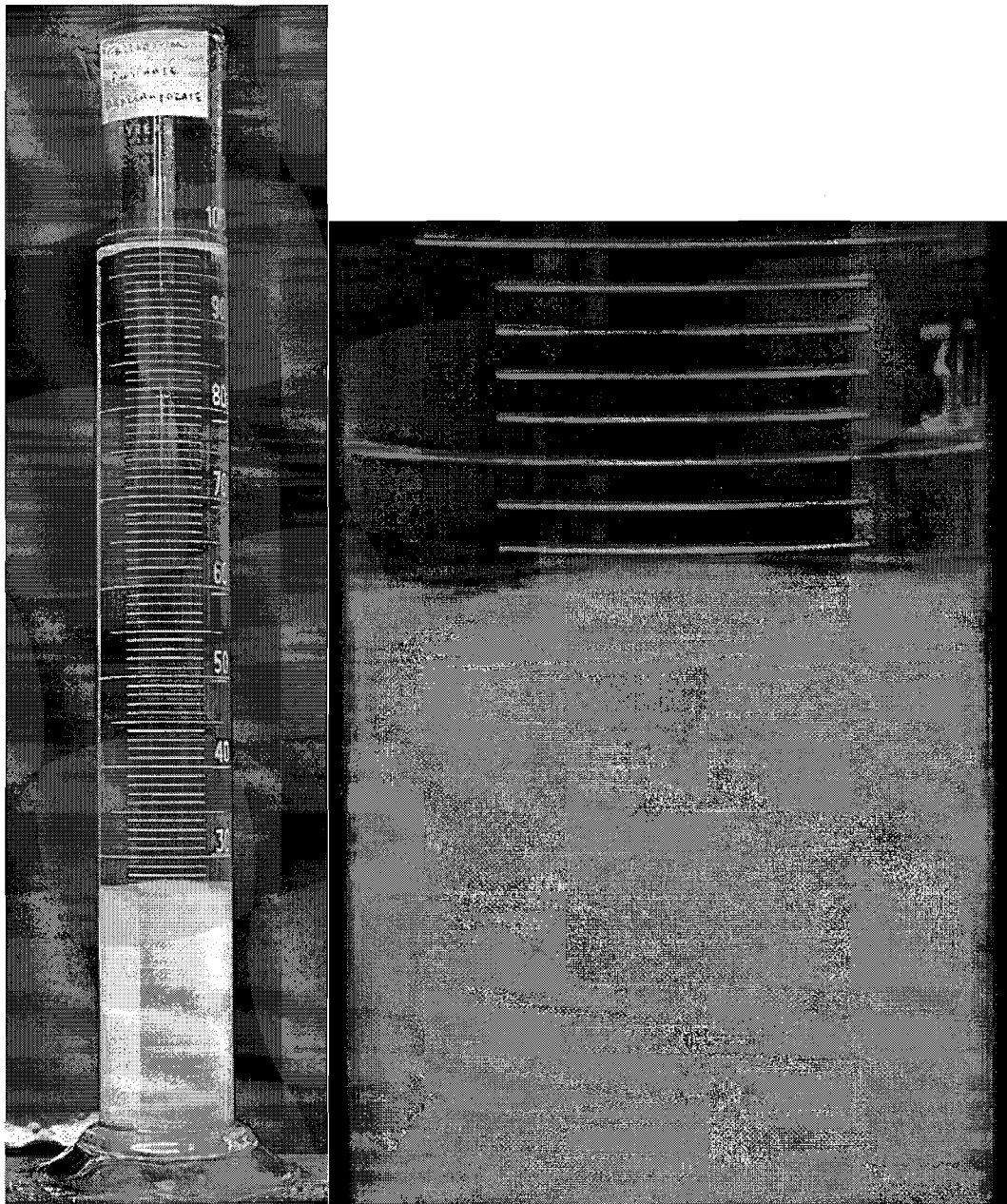


Figure C-5: Overall and close-up views of the precipitate formed by trisodium phosphate dodecahydrate and calcium.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tetraborate decahydrate

Boron concentration: 2500 ppm

Desired pH: 8.5

Preparation of 100 ml of stock 10,000 ppm Al solution:

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$: 13.8960 g

Actual Concentration: 9,994 ppm

pH of solution: 2.63

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1622 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 64.5 °C

Temperature after addition of Al solution: 61.8 °C

Observations:

- Precipitate was formed immediately upon addition of Al solution.
- After 60 minutes, the temperature was 27.4 °C and there was 10 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid above the precipitate was cloudy.
- After 120 minutes, there was 9 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid above the precipitate was still cloudy.
- After 24 hours, there was 7 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid above the precipitate was cloudy.
- pH after addition of Al: 7.98

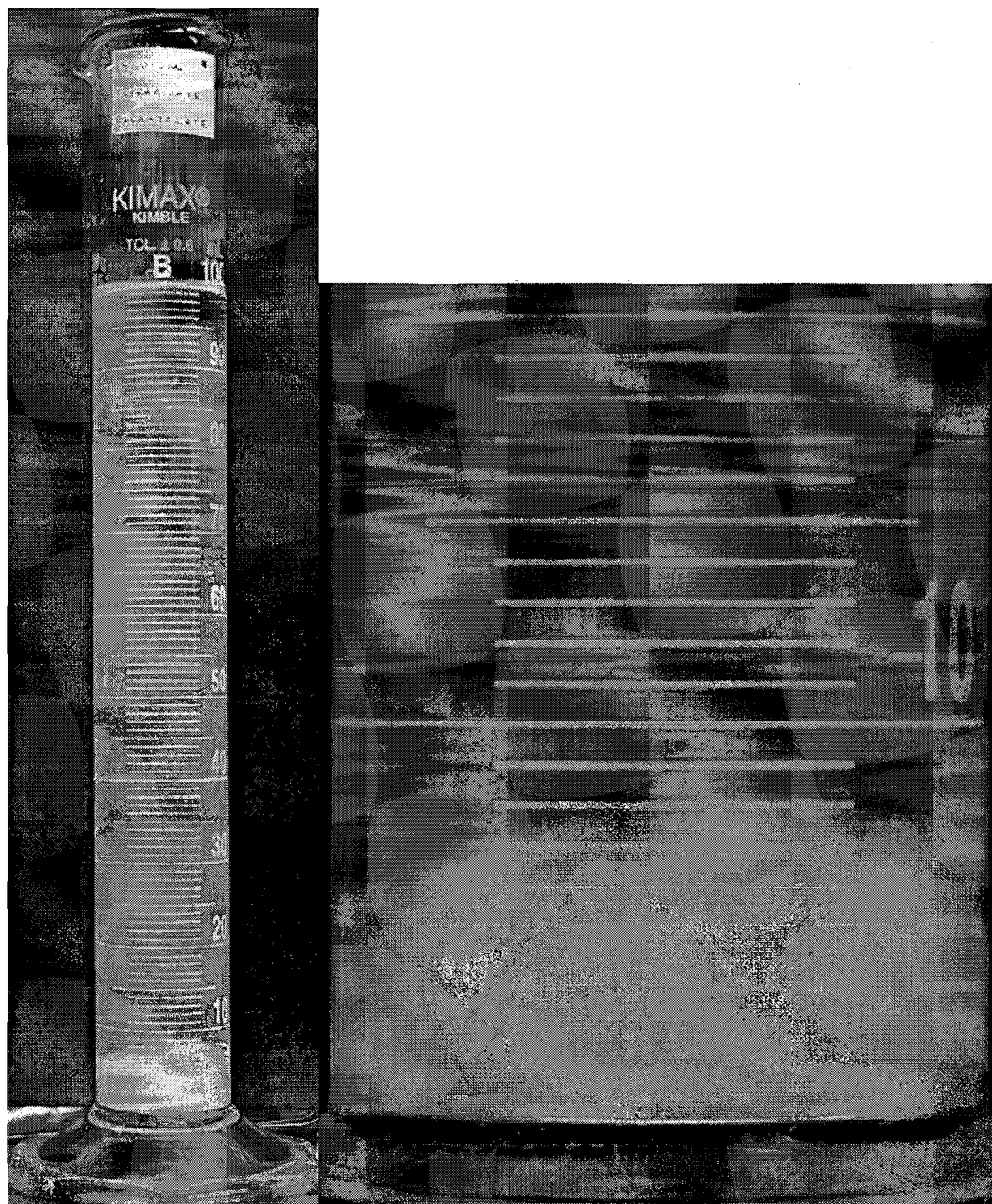


Figure C-6: Overall and close-up views of the precipitate formed by sodium tetraborate decahydrate and aluminum.

Test Chemical: Sodium metaborate tetrahydrate

Boron concentration: 2500 ppm

Desired pH: 8.5

Preparation of 100 ml of stock 10,000 ppm Al solution:

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$: 13.8960 g

Actual Concentration: 9,994 ppm

pH of solution: 2.63

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2100 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 68.8 °C

Temperature at addition of Al solution: 68.0 °C

Temperature after addition of Al solution: 65.4 °C

Observations:

- Precipitate was formed immediately upon addition of Al solution.
- After 60 minutes, the temperature was 28.6 °C and there was 21 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid is still cloudy.
- After 120 minutes, the temperature was 23.8 °C and there was 19 ml of precipitate at the bottom of the 100 ml graduated cylinder.
- After 24 hours, there was 15 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid above the precipitate was cloudy.
- pH after addition of Al: 7.70



Figure C-7: Overall and close-up views of the precipitate formed by sodium metaborate tetrahydrate and aluminum.

Test Chemical: Sodium tripolyphosphate

Boron concentration: 2500 ppm

Desired pH: 7.5

Preparation of 100 ml of stock 10,000 ppm Al solution:

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$: 13.8960 g

Actual Concentration: 9,994 ppm

pH of solution: 2.63

Mass of buffering candidate:

Mass required: 0.8597 g

Mass used: 0.8690 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 66.7 °C

Temperature after addition of Al solution: 64.3 °C

Observations:

- Upon addition of the Al solution, the liquid was momentarily cloudy but then quickly went clear with stirring.
- After 60 minutes, the temperature was 26.1 °C and the solution was clear and free of precipitate.
- After 120 minutes, the solution was clear and free of precipitate.
- After 24 hours, the solution was clear and free of precipitate.
- pH after addition of Al: 6.02

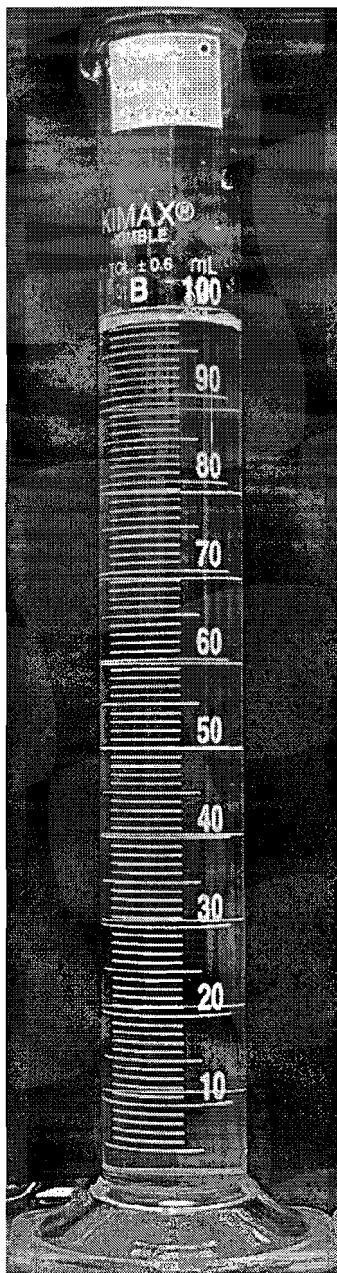


Figure C-8: Overall view of sodium tripolyphosphate and aluminum solution - no precipitate formed.

Test Chemical: Sodium hydroxide

Boron concentration: 2500 ppm

Desired pH: 8.5

Preparation of 100 ml of stock 10,000 ppm Al solution:

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$: 13.8960 g

Actual Concentration: 9,994 ppm

pH of solution: 2.63

Mass of buffering candidate:

Mass required: 0.2176 g

Mass used: 0.2309 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 67.9 °C

Temperature after addition of Al solution: 65.3 °C

Observations:

- Precipitate was formed immediately upon addition of Al solution.
- After 60 minutes, the temperature was 30.2 °C and there was 48 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid above the precipitate is clear.
- After 120 minutes, the temperature was 25.4 °C and there was 42 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid is clear.
- After 24 hours, there was 34 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid was clear.
- pH after addition of Al: 6.93



Figure C-9: Overall and close-up views of the precipitate formed by sodium hydroxide and aluminum.

Test Chemical: Trisodium phosphate dodecahydrate

Boron concentration: 2500 ppm

Desired pH: 8.5

Preparation of 100 ml of stock 10,000 ppm Al solution:

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$: 13.8960 g

Actual Concentration: 9,994 ppm

pH of solution: 2.63

Mass of buffering candidate:

Mass required: 1.914 g

Mass used: 1.9830 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 68.3 °C

Temperature after addition of Ca solution: 65.9 °C

Observations:

- Precipitate was formed immediately upon addition of Ca solution.
- After 60 minutes, the temperature was 29.5 °C and there was 32 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid was slightly cloudy.
- After 120 minutes, the temperature was 23.1 °C and there was 28 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid was slightly cloudy.
- After 24 hours, there was 23 ml of precipitate at the bottom of the 100 ml graduated cylinder. The liquid above the precipitate was slightly cloudy.
- pH after addition of Al: 7.97



Figure C-10: Overall and close-up views of precipitate formed by trisodium phosphate dodecahydrate and aluminum.

C.2 PHASE 4A AND 4B TEST REPORT

PHASE 4A: LOW CONCENTRATION ALUMINUM AND CALCIUM ADDITION

1.1 Procedure

During this phase, calcium and aluminum salts were added to solutions containing the candidate buffers to determine whether precipitates would form.

1. 1 liter of stock 2500 ppm boron solution was prepared using boric acid (see step 1 of Phase 2); the pH of the stock solution was measured and recorded.
2. 100 milliliter stock solutions containing 1,000 ppm aluminum (Al) and 1,000 ppm calcium (Ca) were prepared. Note, 10,000 ppm solutions were used for initial Phase 4 testing. Aluminum nitrate was used for aluminum and calcium chloride was used for calcium. The addition quantities were determined as:

$$\text{Salt (g)} = (1/\text{fraction Al or Ca in salt}) * 0.1 \text{ L} * 1 \text{ g/L}$$

The following steps were performed for sodium tetraborate and sodium metaborate:

3. 95 milliliters of the stock boric acid solution were heated to $150 \pm 5^\circ\text{F}$ ($66 \pm 3^\circ\text{C}$) using a hotplate with stirring.
4. The quantity of the test chemical required to achieve a pH of 8.5 was added, as determined in Phase 2 testing.
5. 7.5 milliliters of stock Al or Ca solution was added; the formation of any precipitates was noted. This was the equivalent to 75 ppm Ca or Al in the test solution.
6. The solution was transferred to a 100 mL graduated cylinder.
7. The solution was allowed to cool to 25°C ; the formation of any precipitates during cooling was noted.
8. After 1, 2 and 24 hours, the volume of any precipitates formed was recorded.
9. Steps 3 to 8 were performed using both Al and Ca.

1.2 Preparation of Stock Solutions

Preparation of 1 L of stock 2,500 ppm boron solution:

Mass of H_3BO_3 required: 14.2993 g

Mass of H_3BO_3 used: 14.2917 g

Actual Concentration: 2,499 ppm

Preparation of 100 mL of stock 1,000 ppm Al solution:

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ required: 1.3904 g

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ used: 1.3936 g

Actual Concentration: 1,002 ppm

Preparation of 100 mL of stock 1,000 ppm Ca solution:

Mass of CaCl_2 required: 0.2769 g

Mass of CaCl_2 used: 0.2983 g

Actual Concentration: 1,077 ppm

1.3 Sodium Tetraborate and Sodium Metaborate with 75 ppm Aluminum and Calcium

1.3.1 Summary of Results

At aluminum and calcium concentrations of 75 ppm, neither sodium tetraborate nor sodium metaborate formed precipitates that remained in the solution longer than approximately one second. The results of testing at this metal concentration can be seen in Figure 1.1 below which was taken approximately 72 hours after the additions of the metal solutions.

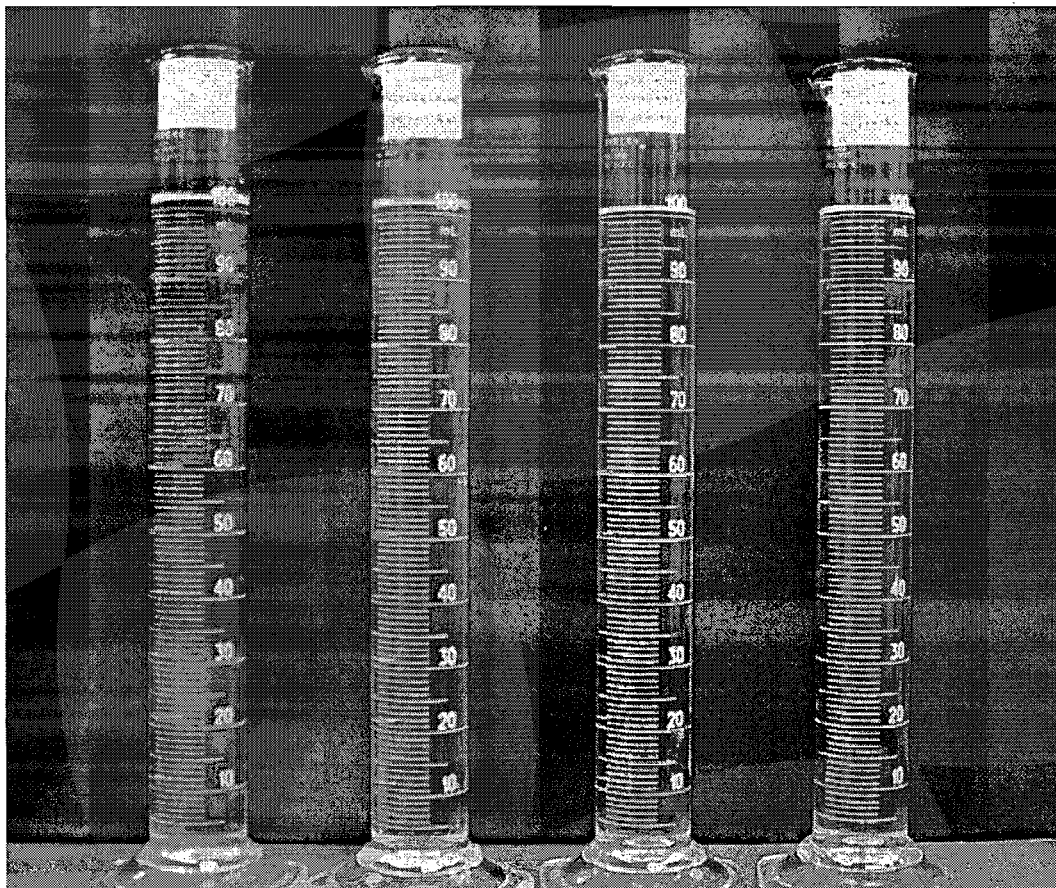


Figure 1.1 Results of precipitate testing at metal concentrations of 75 ppm. (pictured above from left to right: sodium tetraborate decahydrate with 75 ppm Al, sodium tetraborate decahydrate with 75 ppm Ca, sodium metaborate tetrahydrate with 75 ppm Al, and sodium metaborate tetrahydrate with 75 ppm Ca)

1.3.2 Detailed Results

Test Chemical: Sodium tetraborate decahydrate

Metal Concentration: 73.3 ppm Al (7.5 mL of stock 1,002 ppm Al solution)

Initial Boron Concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1671 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 65.8 °C

Temperature after addition of Al solution: 61.1 °C

Observations:

- A very small amount of precipitate formed at the location of aluminum solution addition. The precipitate quickly dissolved (<1 s).
- After 60 minutes, the temperature of the solution was 29.7°C and no precipitate was present.
- After 120 minutes, no precipitate was present.
- After 72 hours, no precipitate was present.

Test Chemical: Sodium tetraborate decahydrate

Metal Concentration: 78.8 ppm Ca (7.5 mL of stock 1,077 ppm Ca solution)

Initial Boron Concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1599 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 68.3 °C

Temperature at addition of Ca solution: 64.5 °C

Temperature after addition of Ca solution: 60.1 °C

Observations:

- A very small amount of precipitate formed at the location of calcium solution addition. The precipitate quickly dissolved (<1 s).
- After 60 minutes, the temperature of the solution was 30.3°C and no precipitate was present.
- After 120 minutes, no precipitate was present.
- After 72 hours, no precipitate was present.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium metaborate tetrahydrate
Metal Concentration: 73.3 ppm Al (7.5 mL of stock 1,002 ppm Al solution)
Initial Boron Concentration: 2,499 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2201 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 65.7 °C

Temperature after addition of Al solution: 61.5 °C

Observations:

- A very small amount of precipitate formed at the location of aluminum solution addition. The precipitate quickly dissolved (<1 s).
- After 60 minutes, the temperature of the solution was 30.2°C and no precipitate was present.
- After 120 minutes, no precipitate was present.
- After 72 hours, no precipitate was present.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium metaborate tetrahydrate

Metal Concentration: 78.8 ppm Ca (7.5 mL of stock 1,077 ppm Ca solution)

Initial Boron Concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.1915 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 68.3 °C

Temperature at addition of Ca solution: 65.5 °C

Temperature after addition of Ca solution: 61.5 °C

Observations:

- A very small amount of precipitate formed at the location of calcium solution addition. The precipitate quickly dissolved (<1 s).
- After 60 minutes, the temperature of the solution was 30.7°C and no precipitate was present.
- After 120 minutes, no precipitate was present.
- After 72 hours, no precipitate was present.

**PHASE 4B: HIGH CONCENTRATION ALUMINUM AND CALCIUM
ADDITION**

2.1 Procedure

During this phase, calcium and aluminum salts were added to solutions containing the candidate buffers to determine whether precipitates would form.

1. 1 liter of stock 2,500 ppm boron solution was prepared using boric acid (see step 1 of Phase 2); the pH of the stock solution was measured and recorded. Additional stock boron solution was prepared as needed.
2. 50 milliliter stock solutions containing 5,000 ppm aluminum, 7,500 ppm aluminum, 5,000 ppm calcium, and 7,500 ppm calcium were prepared. Note, 10,000 ppm solutions were used for initial Phase 4 testing. Aluminum nitrate was used for aluminum and calcium chloride was used for calcium. The addition quantities were determined as:

$$\text{Salt (g)} = (1/\text{fraction Al or Ca in salt}) * 0.05 \text{ L} * 5 \text{ g/L [for 5,000 ppm]}$$

$$\text{Salt (g)} = (1/\text{fraction Al or Ca in salt}) * 0.05 \text{ L} * 7.5 \text{ g/L [for 7,500 ppm]}$$

The following steps were performed for sodium tetraborate and sodium metaborate:

3. 95 milliliters of the stock boric acid solution were heated to 150±5°F (66±3°C) using a hotplate with stirring.
4. The quantity of the test chemical required to achieve a pH of 8.5 was added, as determined in Phase 2 testing.
5. 5.0 milliliters of stock 5,000 ppm Al or Ca solution was added; the formation of any precipitates was noted. This was the equivalent to approximately 250 ppm Ca or Al in the test solution.
6. The solution was transferred to a 100 mL graduated cylinder.
7. The solution was allowed to cool to 25°C; the formation of any precipitates during cooling was noted.
8. After 1, 2 and 24 hours, the volume of any precipitates formed was recorded.
9. Steps 3 to 8 were performed using both Al and Ca.
10. Steps 3 to 9 were repeated, adjusting the metal concentration as required to find the precipitate threshold concentration.

2.2 Preparation of Stock Solutions

Preparation of 1 L of stock 2,500 ppm boron solution:

Mass of H_3BO_3 required: 14.2993 g

Mass of H_3BO_3 used: 14.2917 g

Actual Concentration: 2,499 ppm

Preparation of 1 L of stock 2,500 ppm boron solution:

Mass of H_3BO_3 required: 14.2993 g

Mass of H_3BO_3 used: 14.2932 g

Actual Concentration: 2,499 ppm

Preparation of 1 L of stock 2,500 ppm boron solution:

Mass of H_3BO_3 required: 14.2993 g

Mass of H_3BO_3 used: 14.3052 g

Actual Concentration: 2,501 ppm

Preparation of 50 mL of stock 5,000 ppm Al solution:

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ required: 3.4760 g

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ used: 3.4727 g

Actual Concentration: 4,995 ppm

Preparation of 50 mL of stock 5,000 ppm Ca solution:

Mass of CaCl_2 required: 0.6922 g

Mass of CaCl_2 used: 0.7027 g

Actual Concentration: 5,076 ppm

Preparation of 50 mL of stock 7,500 ppm Al solution:

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ required: 5.2140 g

Mass of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ used: 5.2319 g

Actual Concentration: 7,526 ppm

Preparation of 50 mL of stock 7,500 ppm Ca solution:

Mass of CaCl_2 required: 1.0384 g

Mass of CaCl_2 used: 1.0522 g

Actual Concentration: 7,600 ppm

2.3 Sodium Tetraborate and Aluminum

2.3.1 Summary of Results

Sodium tetraborate precipitate testing was performed using various concentrations of aluminum solutions in order to determine a concentration threshold, below which no precipitate would form. The results of this testing can be seen in Figure 1.1 which was taken approximately 48 hours after the addition of the aluminum solutions.

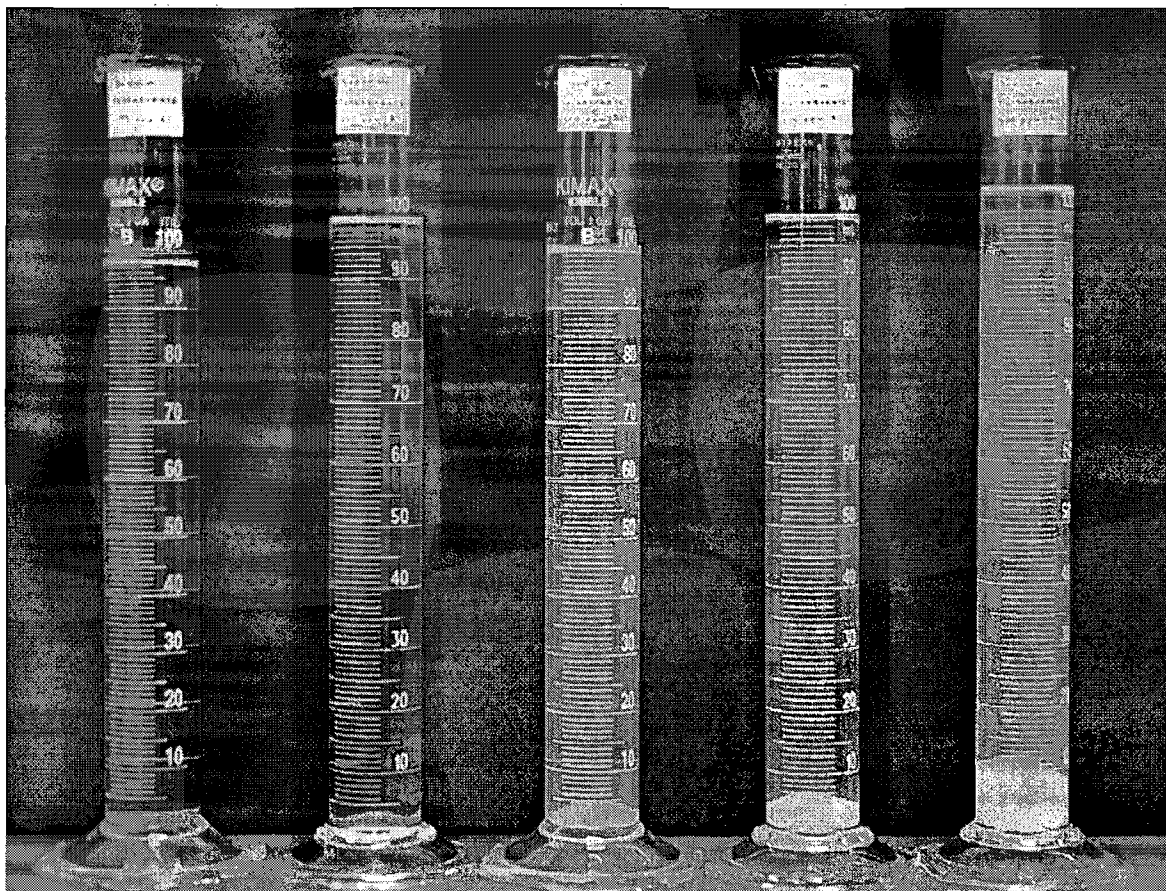


Figure 2.1 Sodium tetraborate precipitate testing with aluminum solutions at various concentrations. (pictured above from left to right: 177 ppm Al, 250 ppm Al, 304 ppm Al, 376 ppm Al, and 476 ppm Al)

While investigating the effects of aluminum concentration on the formation of precipitates, two experiments were performed at room temperature. These tests show that the formation of precipitates is dependent on the temperature of the solution at the time of the addition of the metal solution.^(*) The results of these tests can be seen in Figures 1.2 and 1.3 which were taken approximately 48 hours after the addition of the aluminum solution. When testing at room temperature less precipitates were formed than when tested at the elevated temperature.

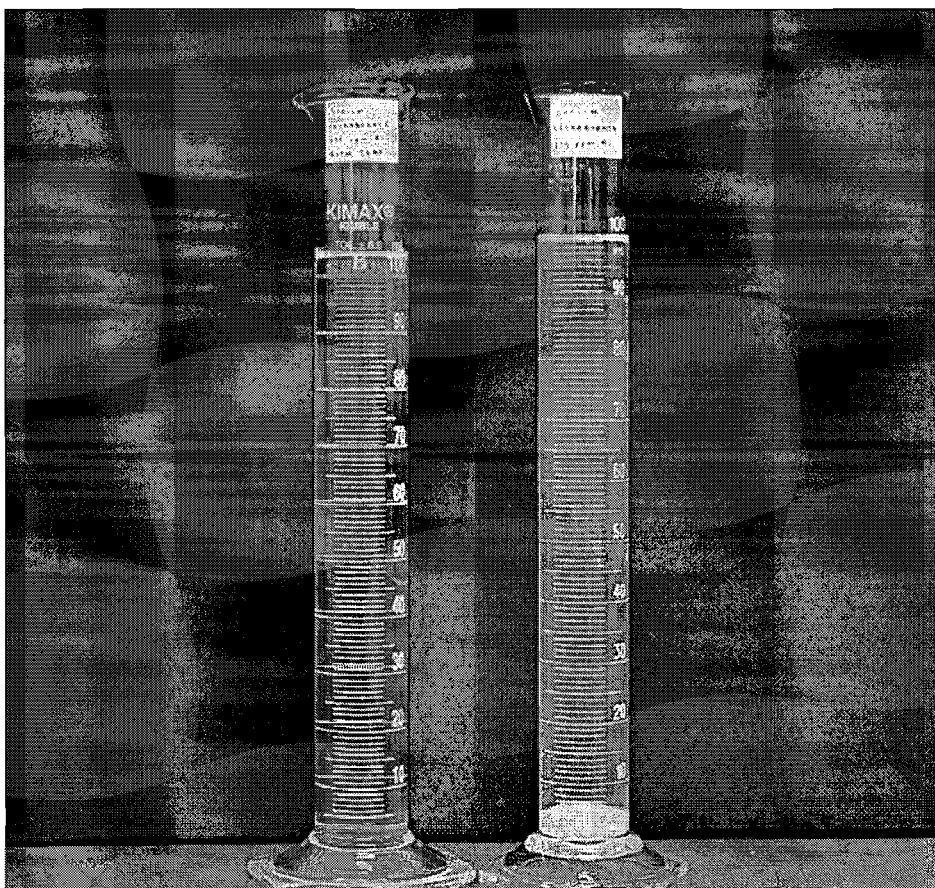


Figure 2.2 Sodium tetraborate precipitate testing at an aluminum concentration of approximately 370 ppm. (pictured above from left to right: testing performed at room temperature, test performed at 66°C)

^(*) These tests were out of the scope of the test plan.

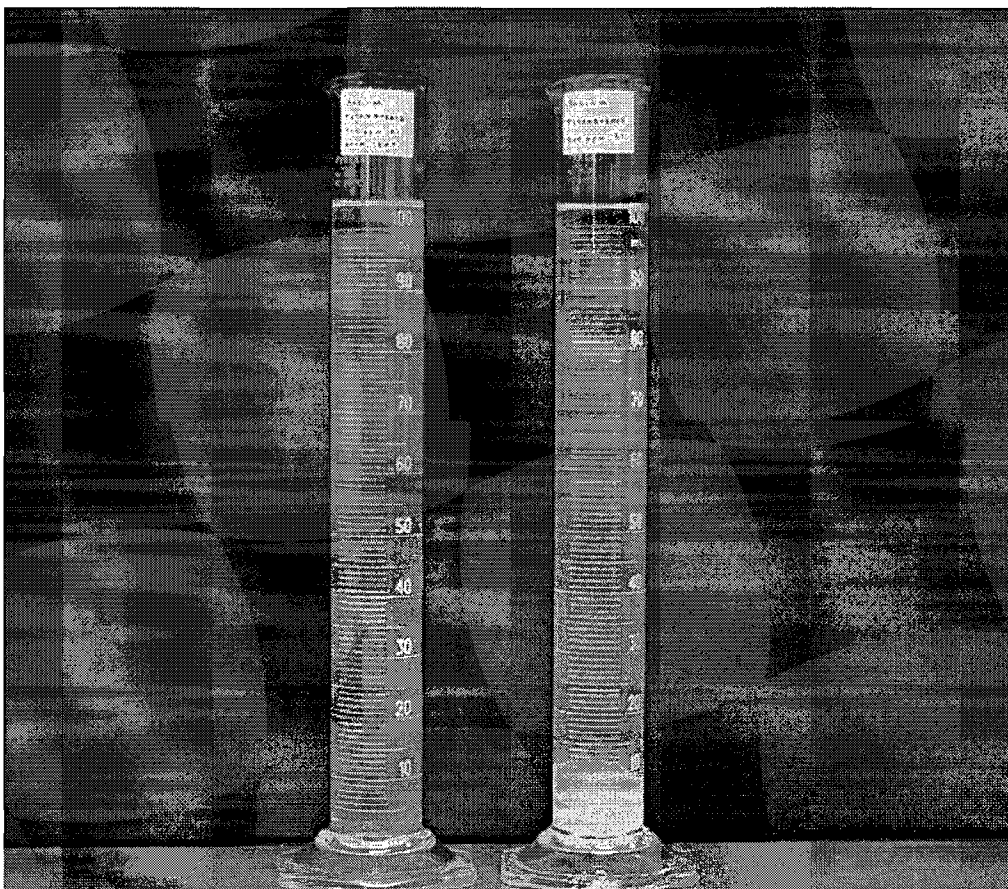


Figure 2.3 Sodium tetraborate precipitate testing at an aluminum concentration of 476 ppm. (pictured above from left to right: testing performed at room temperature, test performed at 66°C)

2.3.2 Detailed Results

Test Chemical: Sodium tetraborate decahydrate

Metal Concentration: 177 ppm Al (3.5 mL of stock 4,995 ppm Al solution)

Initial Boron concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1567 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 66.2 °C

Temperature after addition of Al solution: 63.7 °C

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution. Began clearing up.
- After 60 minutes, the solution was slightly cloudy and no accumulation of precipitate was present.
- After 120 minutes, the solution was slightly cloudy to clear and no accumulation of precipitate was present.
- After 24 hours, the solution was clear to slightly cloudy and no accumulation of precipitate was present.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tetraborate decahydrate
Metal Concentration: 250 ppm Al (5.0 mL of stock 4,995 ppm Al solution)
Initial Boron concentration: 2,499 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1752 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 64.5 °C

Temperature after addition of Al solution: 61.8 °C

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution. Began clearing up.
- After 60 minutes, the solution was slightly cloudy and no accumulation of precipitate was present.
- After 120 minutes, the solution was slightly cloudy to clear and no accumulation of precipitate was present.
- After 24 hours, the solution was slightly cloudy to clear and a very thin layer of precipitate was present at the bottom of the graduated cylinder.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tetraborate decahydrate

Metal Concentration: 304 ppm Al (4.0 mL of stock 7,526 ppm Al solution)

Initial Boron concentration: 2,501 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1682 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 64.8 °C

Temperature after addition of Al solution: -----

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution.
- After 60 minutes, the solution was cloudy and approximately 3 mL of precipitate had accumulated at the bottom of the graduated cylinder.
- After 120 minutes, the solution was cloudy and approximately 3 mL of precipitate was at the bottom of the graduated cylinder.
- After 24 hours, the solution was cloudy and approximately 2 mL of precipitate was at the bottom of the graduated cylinder.

Test Chemical: Sodium tetraborate decahydrate
Metal Concentration: 376 ppm Al (5.0 mL of stock 7,526 ppm Al solution)
Initial Boron concentration: 2,499 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1664 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 64.5 °C

Temperature after addition of Al solution: 61.9 °C

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution.
- After 60 minutes, the solution was cloudy and 7 mL of precipitate had accumulated on the bottom of the graduated cylinder.
- After 120 minutes, the solution was cloudy and there was 7 mL of precipitate on the bottom of the graduated cylinder.
- After 24 hours, the solution was cloudy and there was 6 mL of precipitate on the bottom of the graduated cylinder.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tetraborate decahydrate
Metal Concentration: 365 ppm Al (7.5 mL of stock 4,995 ppm Al solution)
Initial Boron concentration: 2,499 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1672 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 22.7 °C

Temperature at addition of Al solution: -----

Temperature after addition of Al solution: -----

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution.
- After 60 minutes, the solution was slightly cloudy and there was no accumulation of precipitate at the bottom of the graduated cylinder.
- After 120 minutes, the solution was slightly cloudy.
- After 24 hours, the solution was slightly cloudy.

Test Chemical: Sodium tetraborate decahydrate
Metal Concentration: 476 ppm Al (10.0 mL of stock 4,995 ppm Al solution)
Initial Boron concentration: 2,499 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1667 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 65.2 °C

Temperature after addition of Al solution: 61.0 °C

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution. There were also particles suspended in the solution.
- After 60 minutes, the solution was cloudy and 13 mL of precipitate had accumulated on the bottom of the graduated cylinder.
- After 120 minutes, the solution was cloudy and there was 13 mL of precipitate on the bottom of the graduated cylinder.
- After 24 hours, the solution was cloudy and there was 11 mL of precipitate on the bottom of the graduated cylinder.

Test Chemical: Sodium tetraborate decahydrate

Metal Concentration: 476 ppm Al (10.0 mL of stock 4,995 ppm Al solution)

Initial Boron concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1770 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 22.8 °C

Temperature at addition of Al solution: -----

Temperature after addition of Al solution: -----

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution.
- After 60 minutes, the solution was cloudy and there was no accumulation of precipitate at the bottom of the graduated cylinder.
- After 120 minutes, the solution was cloudy.
- After 24 hours, the solution was cloudy.

2.4 Sodium Tetraborate and Calcium

2.4.1 Summary of Results

Sodium tetraborate precipitate testing was performed using various concentrations of calcium solutions in order to determine a concentration threshold, below which no precipitate would form. The results of this testing can be seen in Figure 1.4 below which was taken approximately 48 hours after the addition of the calcium solutions.

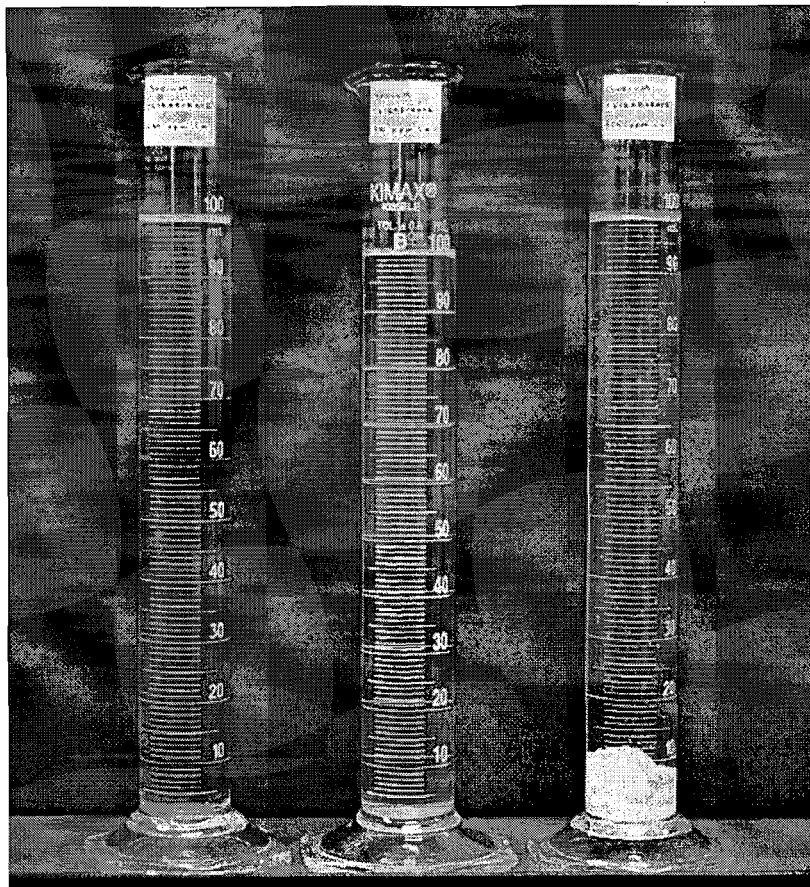


Figure 2.4 Sodium tetraborate precipitate testing with calcium solutions at various concentrations. (pictured above from left to right: 254 ppm Ca, 307 ppm Ca, and 380 ppm Ca)

While investigating the effects of calcium concentration on the formation of precipitates, one experiment was performed at room temperature to determine whether the solution would behave in a similar fashion to that seen with the addition of aluminum. The result of this test can be seen in Figure 1.5 which was taken approximately 48 hours after the addition of the aluminum solution. When testing at room temperature, precipitates were still formed. (*)

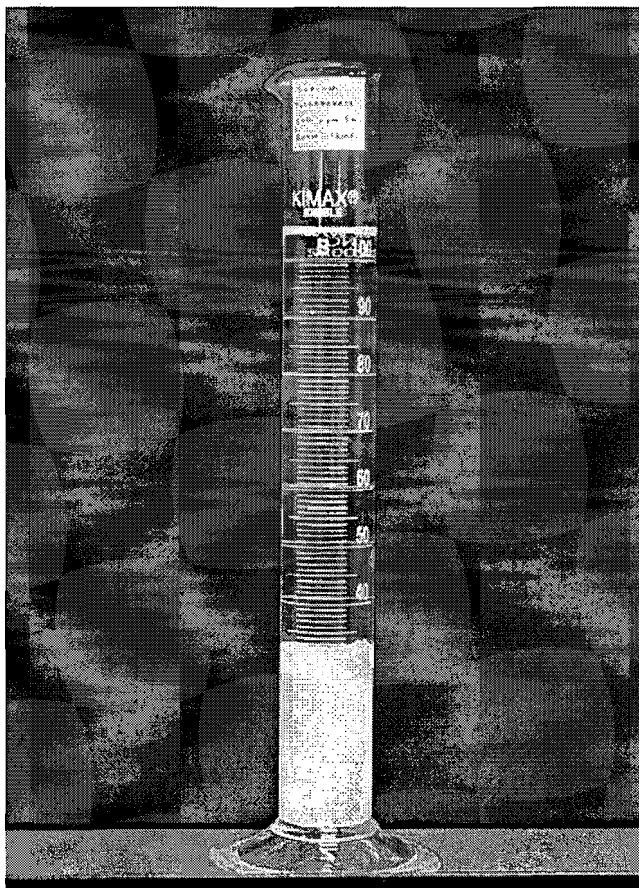


Figure 2.5 Sodium tetraborate precipitate testing at a calcium concentration of 483 ppm.

(*) This test was out of the scope of the test plan.

2.4.2 Detailed Results

Test Chemical: Sodium tetraborate decahydrate

Metal Concentration: 254 ppm Ca (5.0 mL of stock 5,076 ppm Ca solution)

Initial Boron concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1605 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 64.5 °C

Temperature after addition of Ca solution: 61.6 °C

Observations:

- Localized precipitate was seen at the location of addition of the calcium solution. These solids quickly dissolved and the solution was clear.
- After 60 minutes, the solution remained clear.
- After 120 minutes, the solution was clear
- After 24 hours, the solution was clear.

Test Chemical: Sodium tetraborate decahydrate
Metal Concentration: 307 ppm Ca (4.0 mL of stock 7,600 ppm Ca solution)
Initial Boron concentration: 2,501 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1736 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 68.9 °C

Temperature at addition of Ca solution: 64.4 °C

Temperature after addition of Ca solution: -----

Observations:

- The solution was uniformly cloudy upon addition of calcium solution. The solution appeared to become clearer.
- After 60 minutes, the solution was clear with very small particles suspended in it. Also, the bottom of the graduated cylinder was scattered with precipitate particles.
- After 120 minutes, the solution was clear with very small particles suspended in it. Also, more precipitate had accumulated on the bottom of the graduated cylinder.
- After 24 hours, there was approximately 1 mL of precipitate at the bottom of the graduated cylinder and some particles remained suspended in the solution.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tetraborate decahydrate

Metal Concentration: 380 ppm Ca (5.0 mL of stock 7,600 ppm Ca solution)

Initial Boron concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1736 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 68.9 °C

Temperature at addition of Ca solution: 64.4 °C

Temperature after addition of Ca solution: -----

Observations:

- The solution was uniformly cloudy upon addition of calcium solution.
- After 60 minutes, the solution was slightly cloudy and 9 mL of precipitate had accumulated at the bottom of the graduated cylinder.
- After 120 minutes, the solution was slightly cloudy and 9 mL of precipitate was at the bottom of the graduated cylinder.
- After 24 hours, the solution was slightly cloudy and 9 mL of precipitate was at the bottom of the graduated cylinder. There were also suspended particles in the solution.

Test Chemical: Sodium tetraborate decahydrate
Metal Concentration: 483 ppm Ca (10.0 mL of stock 5,076 ppm Ca solution)
Initial Boron concentration: 2,499 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 3.163 g

Mass used: 3.1633 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 22.9 °C

Temperature at addition of Ca solution: -----

Temperature after addition of Ca solution: -----

Observations:

- The solution was uniformly cloudy with suspended particles upon addition of calcium solution.
- After 60 minutes, 44 mL of precipitate had accumulated at the bottom of the graduated cylinder. There were suspended particles in the solution.
- After 120 minutes, there was 40 mL of precipitate at the bottom of the graduated cylinder. There were suspended particles in the solution.
- After 24 hours, there was 37 mL of precipitate at the bottom of the graduated cylinder. There were suspended particles in the solution.

2.5 Sodium Metaborate and Aluminum

2.5.1 Summary of Results

Sodium metaborate precipitate testing was performed using various concentrations of aluminum solutions in order to determine a concentration threshold, below which no precipitate would form. The results of this testing can be seen in Figure 1.6 which was taken approximately 24 hours after the addition of the aluminum solutions.

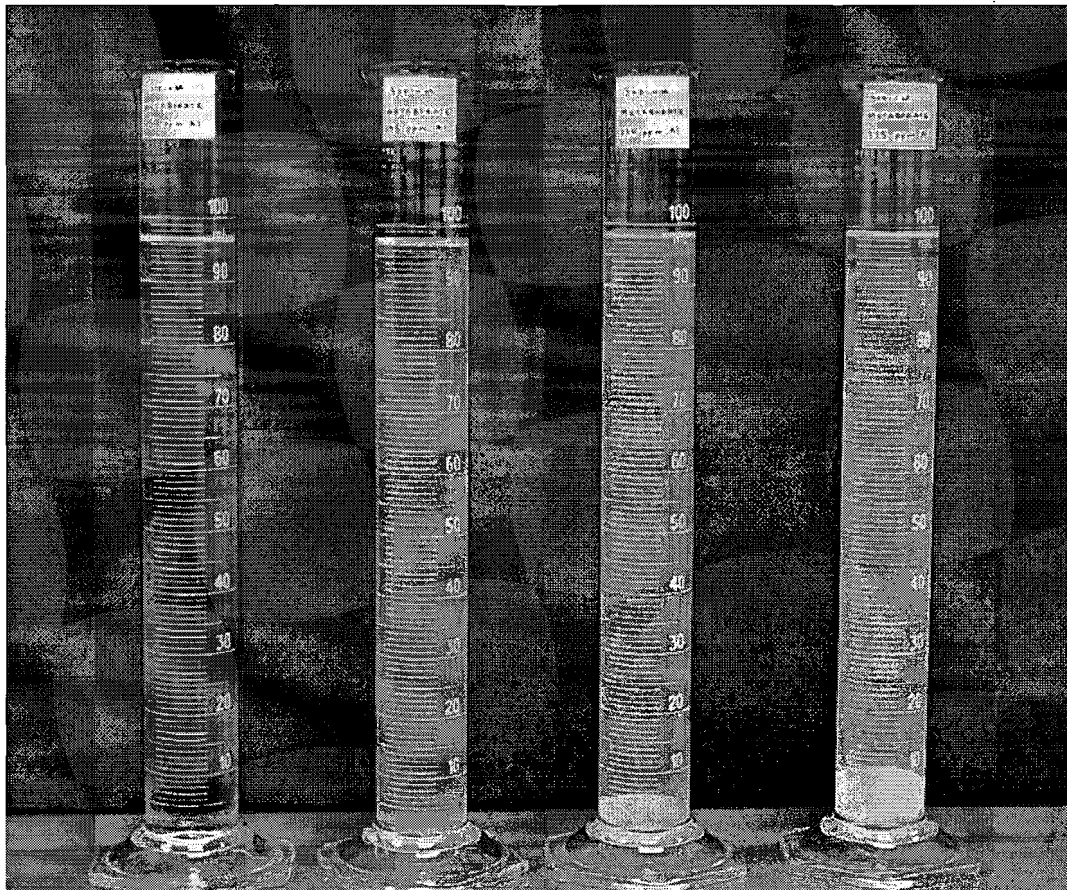


Figure 2.6 Sodium metaborate precipitate testing with aluminum solutions at various concentrations. (pictured above from left to right: 155 ppm Al, 200 ppm Al, 250 ppm Al, and 376 ppm Al)

2.5.2 Detailed Results

Test Chemical: Sodium metaborate tetrahydrate

Metal Concentration: 155 ppm Al (2.0 mL of stock 7,526 ppm Al solution)

Initial Boron concentration: 2,501 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2097 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 68.9 °C

Temperature at addition of Al solution: 65.8 °C

Temperature after addition of Al solution: 63.4 °C

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution. Began clearing up.
- After 60 minutes, the solution was slightly cloudy to clear and no accumulation of precipitate was present.
- After 120 minutes, the solution was slightly cloudy to clear.
- After 24 hours, the solution was slightly cloudy to clear.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium metaborate tetrahydrate
Metal Concentration: 200 ppm Al (2.6 mL of stock 7,526 ppm Al solution)
Initial Boron concentration: 2,501 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2037 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 65.9 °C

Temperature after addition of Al solution: 63.6 °C

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution. Began clearing up.
- After 60 minutes, the solution was slightly cloudy and no accumulation of precipitate was present.
- After 120 minutes, the solution was slightly cloudy.
- After 24 hours, the solution was slightly cloudy.

Test Chemical: Sodium metaborate tetrahydrate
Metal Concentration: 250 ppm Al (5.0 mL of stock 4,995 ppm Al solution)
Initial Boron concentration: 2,499 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2136 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 66.2 °C

Temperature after addition of Al solution: 63.7 °C

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution.
- After 60 minutes, the solution was cloudy and no accumulation of precipitate was present.
- After 120 minutes, the solution was cloudy and there was approximately 3 mL of precipitate at the bottom of the graduated cylinder.
- After 24 hours, the solution was cloudy and there was approximately 3 mL of precipitate at the bottom of the graduated cylinder.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium metaborate tetrahydrate
Metal Concentration: 376 ppm Al (5.0 mL of stock 7,526 ppm Al solution)
Initial Boron concentration: 2,499 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2007 g

Addition of buffering candidate and Al solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Al solution: 65.8 °C

Temperature after addition of Al solution: 63.0 °C

Observations:

- Solution was uniformly cloudy upon addition of aluminum solution.
- After 60 minutes, the solution was cloudy and 10 mL of precipitate had accumulated at the bottom of the graduated cylinder.
- After 120 minutes, the solution was cloudy and there was 9 mL of precipitate at the bottom of the graduated cylinder.
- After 24 hours, the solution was cloudy and there was 8 mL of precipitate at the bottom of the graduated cylinder.

2.6 Sodium Metaborate and Calcium

2.6.1 Summary of Results

Sodium metaborate precipitate testing was performed using various concentrations of calcium solutions in order to determine a concentration threshold, below which no precipitate would form. The results of this testing can be seen in Figure 1.7 below which was taken approximately 24 hours after the addition of the aluminum solutions.

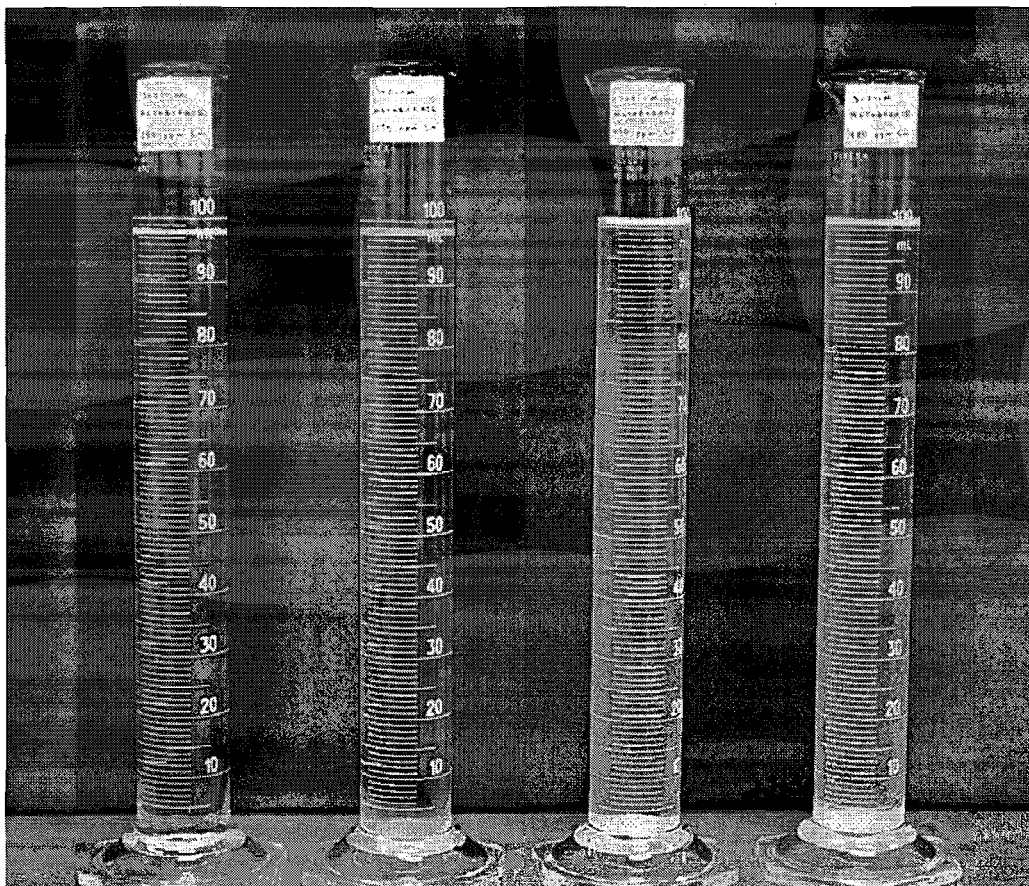


Figure 2.7 Sodium metaborate precipitate testing with calcium solutions at various concentrations. (pictured above from left to right: 254 ppm Ca, 380 ppm Ca, 451 ppm Ca, and 480 ppm Ca)

2.6.2 Detailed Results

Test Chemical: Sodium metaborate tetrahydrate

Metal Concentration: 254 ppm Ca (5.0 mL of stock 5,076 ppm Ca solution)

Initial Boron concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2155 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 66.0 °C

Temperature after addition of Ca solution: 63.6 °C

Observations:

- Solution was clear upon addition of calcium solution.
- After 60 minutes, the solution was clear.
- After 120 minutes, the solution was clear.
- After 24 hours, the solution was clear.

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium metaborate tetrahydrate

Metal Concentration: 380 ppm Ca (5.0 mL of stock 7,600 ppm Ca solution)

Initial Boron concentration: 2,499 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2038 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 65.7 °C

Temperature after addition of Ca solution: 63.2 °C

Observations:

- Solution was clear upon addition of calcium solution.
- After 60 minutes, the solution was clear.
- After 120 minutes, the solution was clear.
- After 24 hours, the solution was clear. A very thin layer of precipitate had accumulated at the bottom of the graduated cylinder.

Test Chemical: Sodium metaborate tetrahydrate

Metal Concentration: 451 ppm Ca (6.0 mL of stock 7,600 ppm Ca solution)

Initial Boron concentration: 2,501 ppm

Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2051 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 69.0 °C

Temperature at addition of Ca solution: 65.8 °C

Temperature after addition of Ca solution: -----

Observations:

- Upon addition of calcium solution, localized cloudiness at the location of addition was observed. The solution was clear to slightly cloudy.
- After 60 minutes, the solution was slightly cloudy.
- After 120 minutes, the solution was slightly cloudy.
- After 24 hours, the solution was clear with suspended particles and there was approximately 1 mL of precipitate at the bottom of the graduated cylinder.

Test Chemical: Sodium metaborate tetrahydrate
Metal Concentration: 480 ppm Ca (6.4 mL of stock 7,600 ppm Ca solution)
Initial Boron concentration: 2,501 ppm
Desired pH: 8.5

Mass of buffering candidate:

Mass required: 1.199 g

Mass used: 1.2149 g

Addition of buffering candidate and Ca solution:

Temperature at addition of buffering candidate: 68.7 °C

Temperature at addition of Ca solution: 66.1 °C

Temperature after addition of Ca solution: -----

Observations:

- Upon addition of calcium solution, localized cloudiness at the location of addition was observed. The solution was clear to slightly cloudy.
- After 60 minutes, the solution was slightly cloudy.
- After 120 minutes, the solution was cloudy.
- After 24 hours, the solution was clear with suspended particles. Approximately 2 mL of precipitate had accumulated at the bottom of the graduated cylinder.

Appendix D: Detailed Results of Phase 5 Testing

Summary of Corrosion Results

Aluminum Corrosion:

Buffer Candidate	Number	Surface Area [mm ²]	Initial Mass [g]	Scaled Mass [g]	Scaled Mass Loss [g]	Scaled Mass Loss [g/m ²]	Descaled Mass [g]	Descaled Mass Loss [g]	Descaled Mass Loss [g/m ²]
Sodium tetraborate decahydrate	1	2286.7	7.2713	7.2352	0.0361	15.8	7.1917	0.0796	34.8
Sodium tetraborate decahydrate	2	2291.1	7.2636	7.2185	0.0451	19.7	7.1827	0.0809	35.3
Sodium metaborate tetrahydrate	3	2286.7	7.2493	7.2387	0.0106	4.64	7.2280	0.0213	9.31
Sodium metaborate tetrahydrate	4	2286.7	7.2231	7.2109	0.0122	5.34	7.2010	0.0221	9.66
Sodium tripolyphosphate	5	2286.7	7.2427	7.1908	0.0519	22.7	7.1876	0.0551	24.1
Sodium tripolyphosphate	6	2286.7	7.2404	7.1876	0.0528	23.1	7.1836	0.0568	24.8
Trisodium phosphate dodecahydrate	7	2301.9	7.2897	7.2886	0.0011	0.478	7.2821	0.0076	3.30
Trisodium phosphate dodecahydrate	8	2288.7	7.2174	7.2160	0.0014	0.612	7.2116	0.0058	2.53
Sodium hydroxide	9	2286.7	7.1894	7.1845	0.0049	2.14	7.1567	0.0327	14.3
Sodium hydroxide	10	2297.5	7.2414	7.2361	0.0053	2.31	7.2062	0.0352	15.3

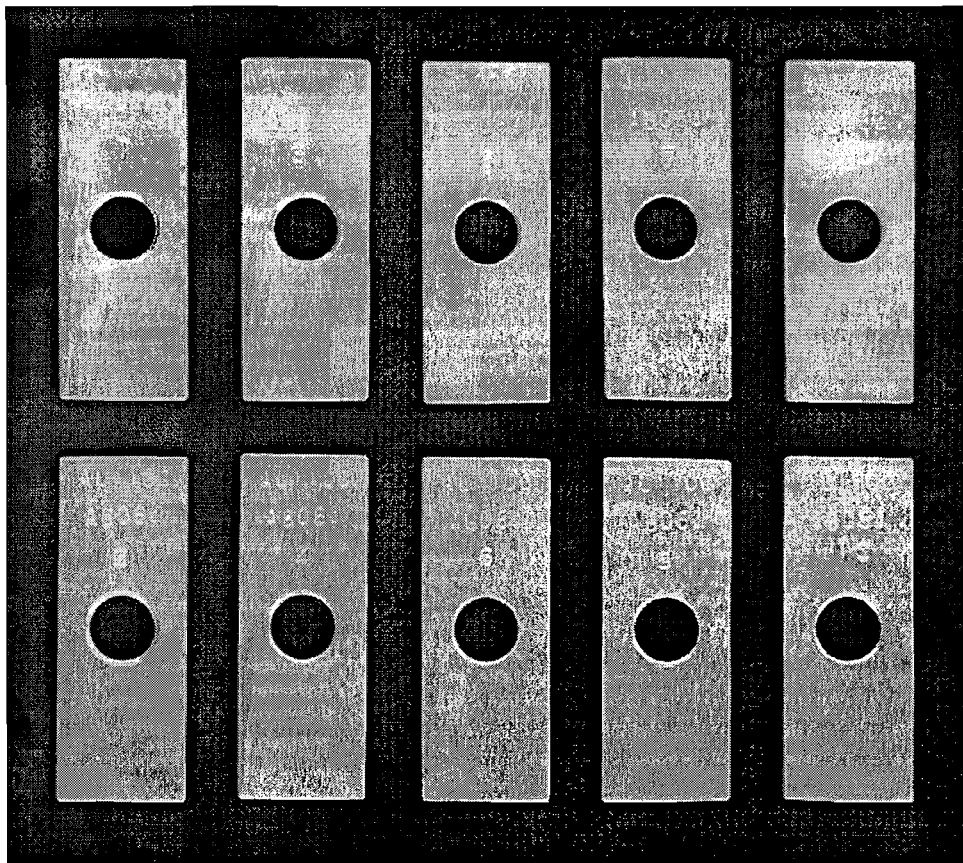
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Steel Corrosion:

Buffer Candidate	Number	Surface Area [mm ²]	Initial Mass [g]	Scaled Mass [g]	Scaled Mass Loss [g]	Scaled Mass Loss [g/m ²]	Descaled Mass [g]	Descaled Mass Loss [g]	Descaled Mass Loss [g/m ²]
Sodium tetraborate decahydrate	1	2331.5	23.0856	23.0835	0.0021	0.901	23.0831	0.0025	1.07
Sodium tetraborate decahydrate	2	2342.4	22.9719	22.9663	0.0056	2.39	22.9696	0.0023	0.982
Sodium metaborate tetrahydrate	3	2336.0	22.7750	22.7729	0.0021	0.899	22.7729	0.0021	0.899
Sodium metaborate tetrahydrate	4	2336.0	22.8077	22.8067	0.0010	0.428	22.8069	0.0008	0.342
Sodium tripolyphosphate	5	2342.4	22.6574	22.6111	0.0463	19.8	22.5304	0.1270	54.2
Sodium tripolyphosphate	6	2337.9	22.4625	22.4076	0.0549	23.5	22.3330	0.1295	55.4
Trisodium phosphate dodecahydrate	7	2342.4	22.0541	22.0520	0.0021	0.897	22.0519	0.0022	0.939
Trisodium phosphate dodecahydrate	8	2331.5	22.8778	22.8760	0.0018	0.772	22.8753	0.0025	1.07
Sodium hydroxide	9	2359.4	22.8053	22.8028	0.0025	1.06	22.8027	0.0026	1.10
Sodium hydroxide	10	2346.8	22.9240	22.9219	0.0021	0.895	22.9217	0.0023	0.980

Initial Aluminum Coupon Masses and Dimensions

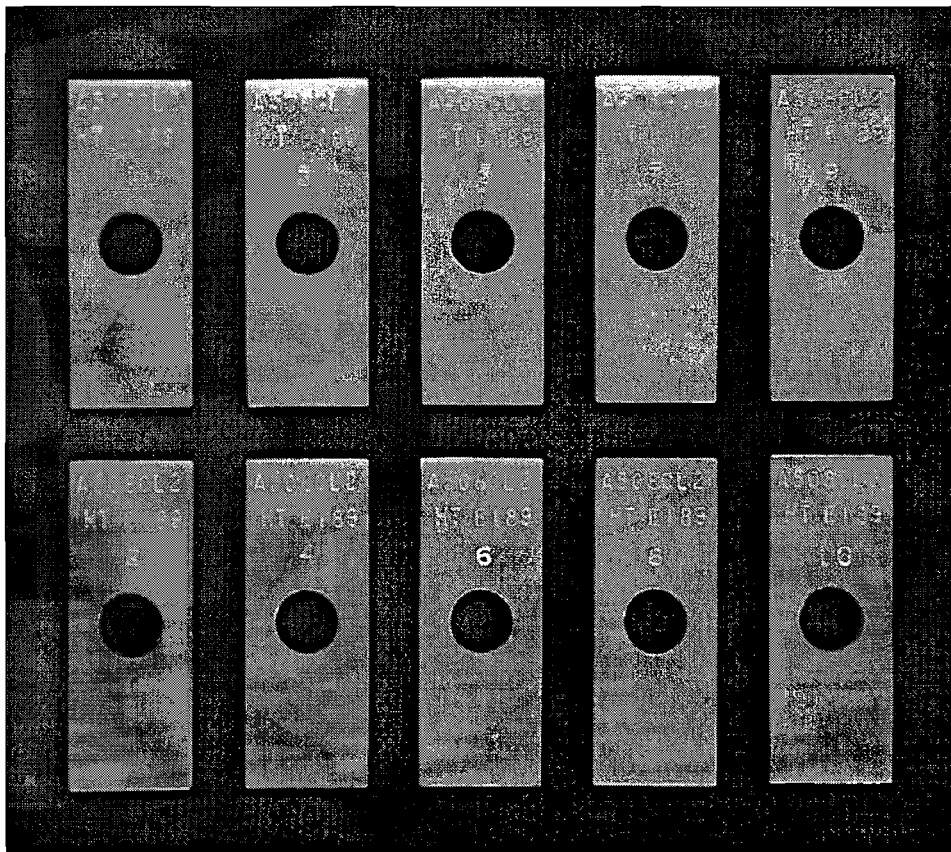
Type	Number	Mass [g]	Length [mm]	Width [mm]	Thickness [mm]	Hole Diameter [mm]
AL1100	1	7.2713	51.0	19.0	2.9	9.6
AL1100	2	7.2636	51.1	19.0	2.9	9.6
AL1100	3	7.2493	51.0	19.0	2.9	9.6
AL1100	4	7.2231	51.0	19.0	2.9	9.6
AL1100	5	7.2427	51.0	19.0	2.9	9.6
AL1100	6	7.2404	51.0	19.0	2.9	9.6
AL1100	7	7.2897	51.1	19.1	2.9	9.6
AL1100	8	7.2174	50.8	19.1	2.9	9.6
AL1100	9	7.1894	51.0	19.0	2.9	9.6
AL1100	10	7.2414	51.0	19.1	2.9	9.6

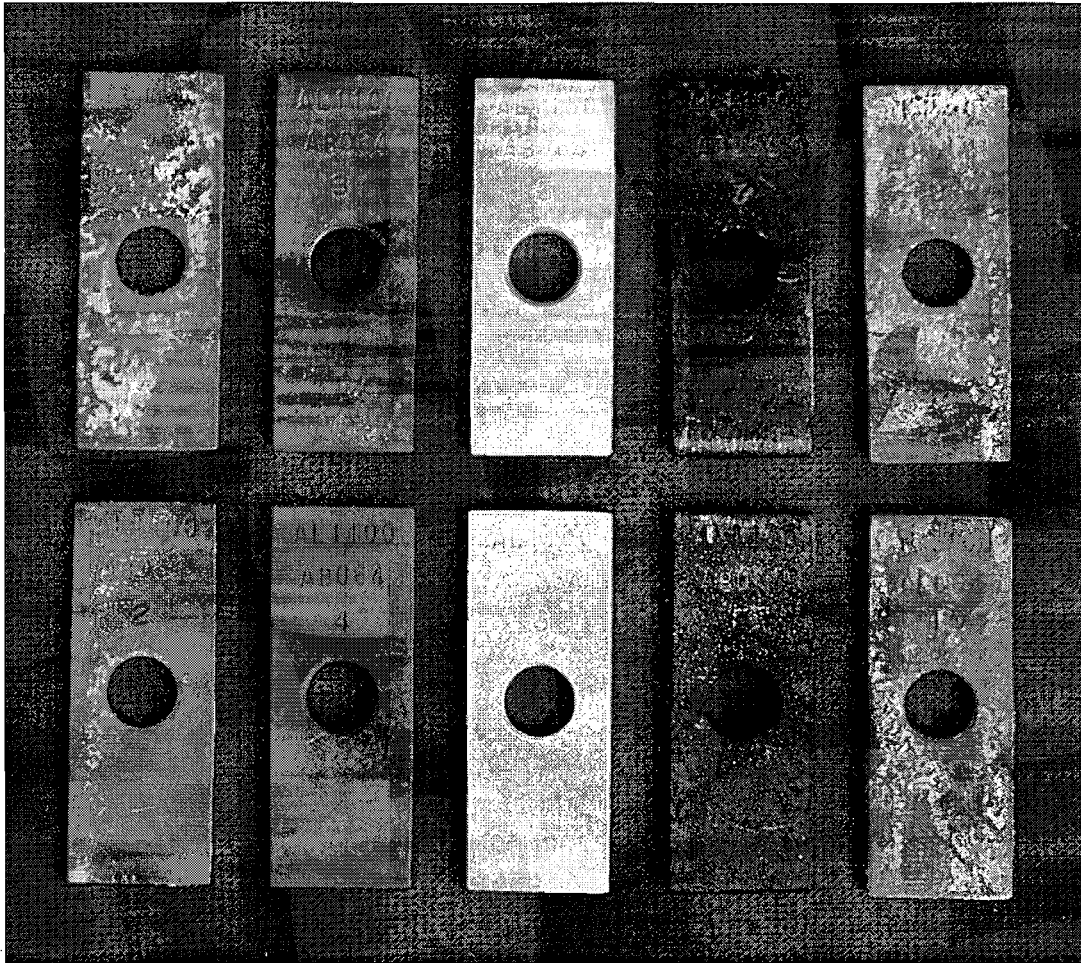


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Initial Carbon Steel Coupon Masses and Dimensions

Type	Number	Mass [g]	Length [mm]	Width [mm]	Thickness [mm]	Hole Diameter [mm]
A508CL2	1	23.0856	51.0	19.1	3.1	9.6
A508CL2	2	22.9719	51.0	19.2	3.1	9.6
A508CL2	3	22.7750	51.1	19.1	3.1	9.6
A508CL2	4	22.8077	51.1	19.1	3.1	9.6
A508CL2	5	22.6574	51.0	19.2	3.1	9.6
A508CL2	6	22.4625	50.9	19.2	3.1	9.6
A508CL2	7	22.0541	51.0	19.2	3.1	9.6
A508CL2	8	22.8778	51.0	19.1	3.1	9.6
A508CL2	9	22.8053	51.0	19.2	3.2	9.6
A508CL2	10	22.9240	51.1	19.2	3.1	9.6

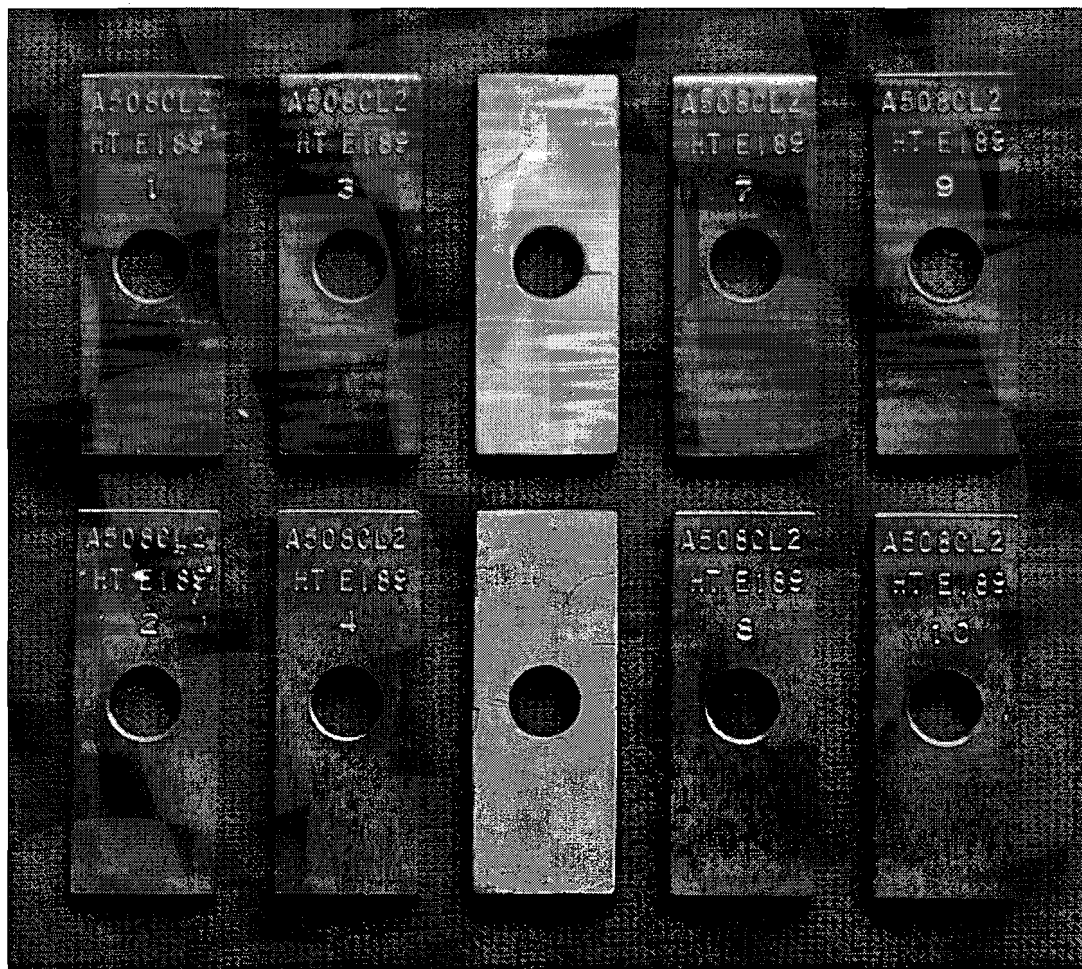


Aluminum Coupons After 2 Weeks of Corrosion

Pictured above from left to right: Sodium tetraborate decahydrate, Sodium metaborate tetrahydrate, Sodium tripolyphosphate, Trisodium phosphate dodecahydrate, Sodium hydroxide.

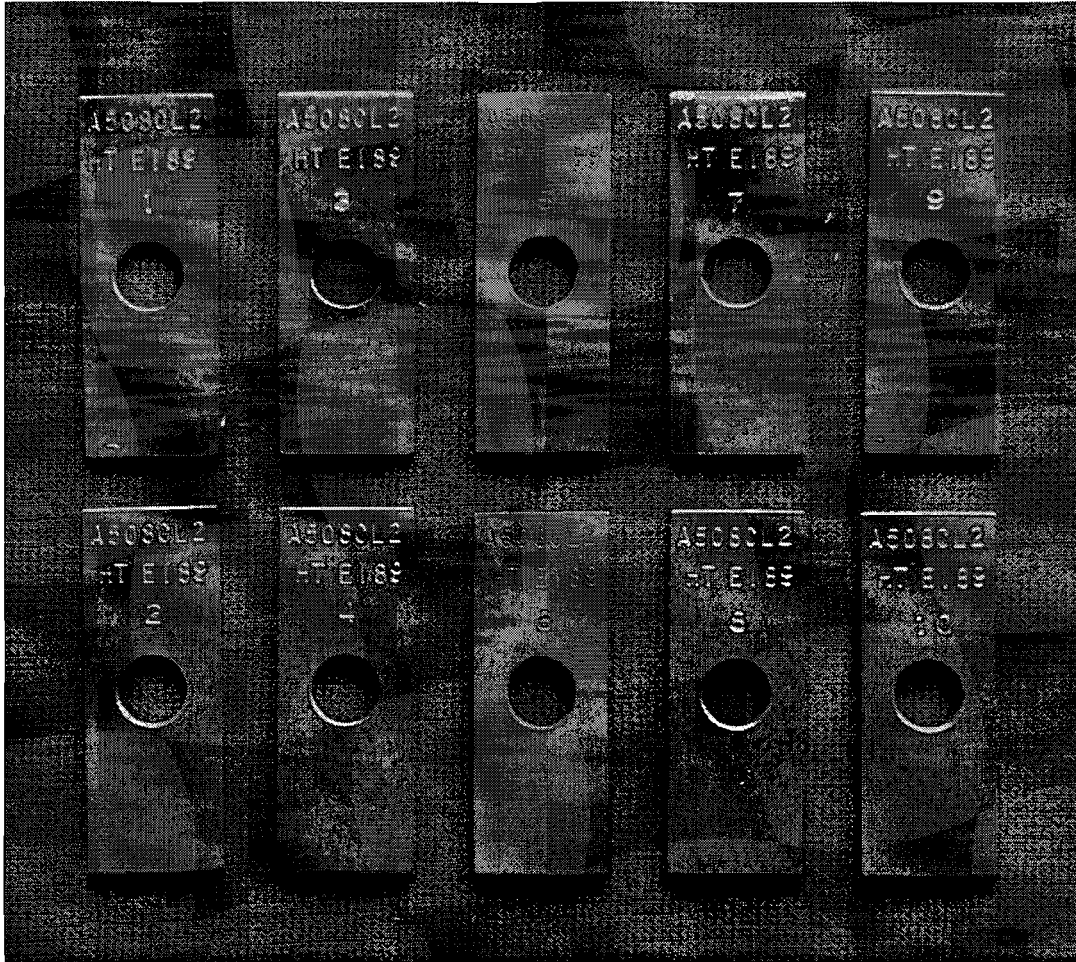
Aluminum Coupons After De-scaling

Pictured above from left to right: Sodium tetraborate decahydrate, Sodium metaborate tetrahydrate, Sodium tripolyphosphate, Trisodium phosphate dodecahydrate, Sodium hydroxide.

Carbon Steel Coupons After 2 Weeks of Corrosion

Pictured above from left to right: Sodium tetraborate decahydrate, Sodium metaborate tetrahydrate, Sodium tripolyphosphate, Trisodium phosphate dodecahydrate, Sodium hydroxide.

Note: Coupons 5 and 6 are pictured face down because they were oriented face down in the corrosion flask and did not corrode uniformly.

Carbon Steel Coupons After De-scaling

Pictured above from left to right: Sodium tetraborate decahydrate, Sodium metaborate tetrahydrate, Sodium tripolyphosphate, Trisodium phosphate dodecahydrate, Sodium hydroxide.

Detailed Results

Test Chemical: Sodium tetraborate decahydrate

Preparation of 500 ml of 2500 ppm boron solution:

Boric acid required: 7.1496 g

Boric acid used: 7.1379 g

pH of solution: 4.69

Addition of sodium tetraborate decahydrate:

Mass for pH of 8.0: 5.4450 g

Mass used: 5.4383 g

pH of solution: 8.01

Coupons used:

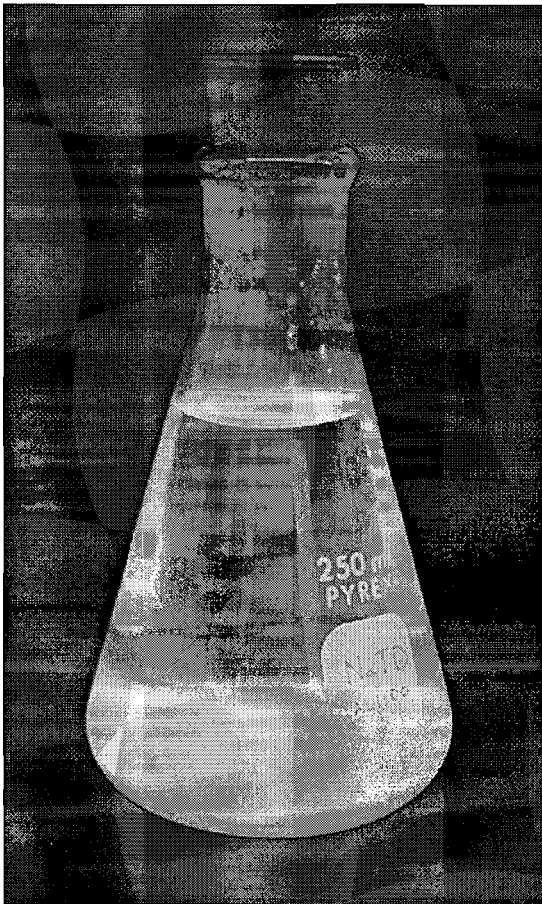
AL1100: 1 & 2

A508CL2: 1 & 2

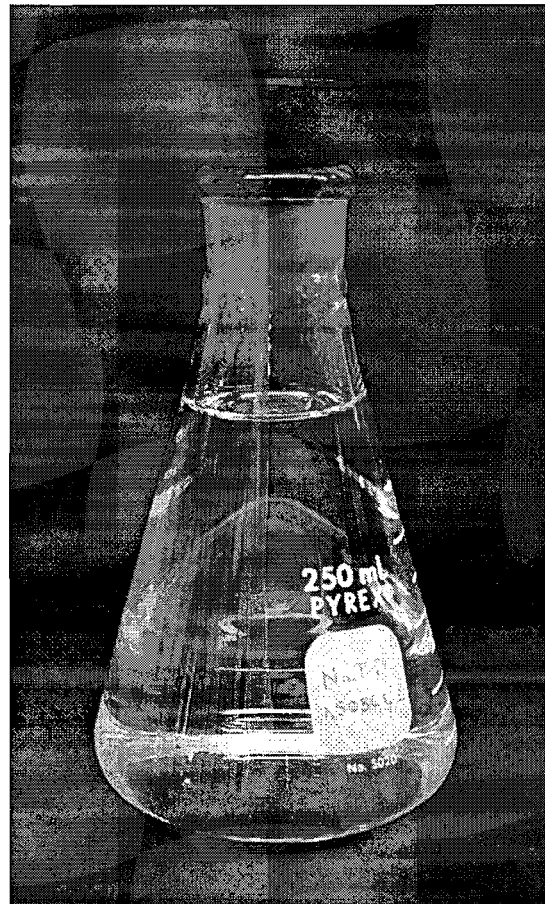
Observations after 2 weeks:

Coupon Type	Coupon ID	Precipitate Formed?	pH of Solution	Final Mass [g]	Final Mass After Descaling [g]
AL1100	1	Yes	8.03	7.2352	7.1917
AL1100	2	Yes	8.03	7.2185	7.1827
A508CL2	1	No	8.03	23.0835	23.0831
A508CL2	2	No	8.03	22.9663	22.9696

Sodium tetraborate decahydrate test solutions after 2 weeks:



Solution that contained aluminum coupons



Solution that contained steel coupons

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Test Chemical: Sodium metaborate tetrahydrate

Preparation of 500 ml of 2500 ppm boron solution:

Boric acid required: 7.1496 g

Boric acid used: 7.1760 g

pH of solution: 4.65

Addition of sodium metaborate tetrahydrate:

Mass for pH of 8.0: 2.7715 g

Mass used: 2.7518 g

pH of solution: 8.00

Coupons used:

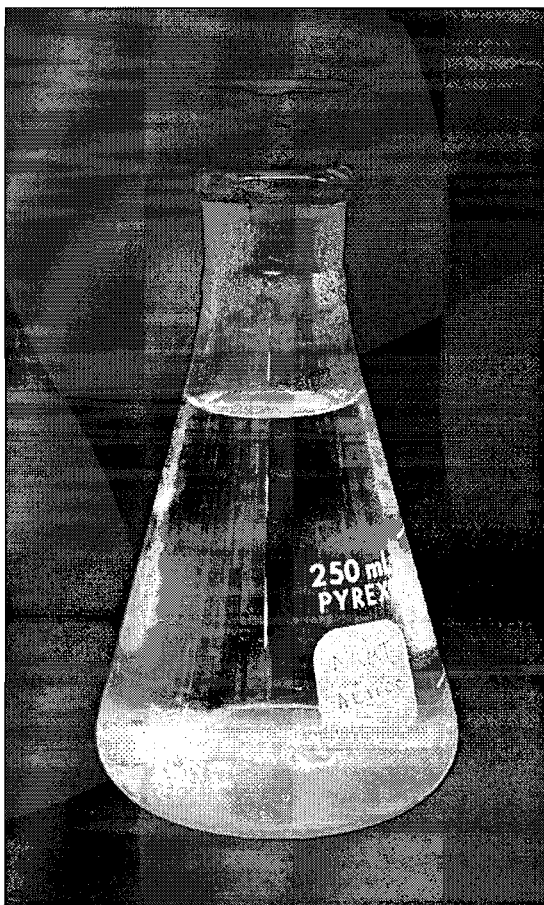
AL1100: 3 & 4

A508CL2: 3 & 4

Observations after 2 weeks:

Coupon Type	Coupon ID	Precipitate Formed?	pH of Solution	Final Mass [g]	Final Mass After Descaling [g]
AL1100	3	Yes	8.04	7.2387	7.2280
AL1100	4	Yes	8.04	7.2109	7.2010
A508CL2	3	No	8.02	22.7729	22.7729
A508CL2	4	No	8.02	22.8067	22.8069

Sodium metaborate tetrahydrate test solution after 2 weeks:



Solution that contained aluminum coupons



Solution that contained steel coupons

Test Chemical: Sodium tripolyphosphate

Preparation of 500 ml of 2500 ppm boron solution:

Boric acid required: 7.1496 g

Boric acid used: 7.1770 g

pH of solution: 4.66

Addition of sodium tripolyphosphate:

Mass for pH of 7.5: 4.2985 g

Mass used: 4.2828 g

pH of solution: 7.49

Coupons used:

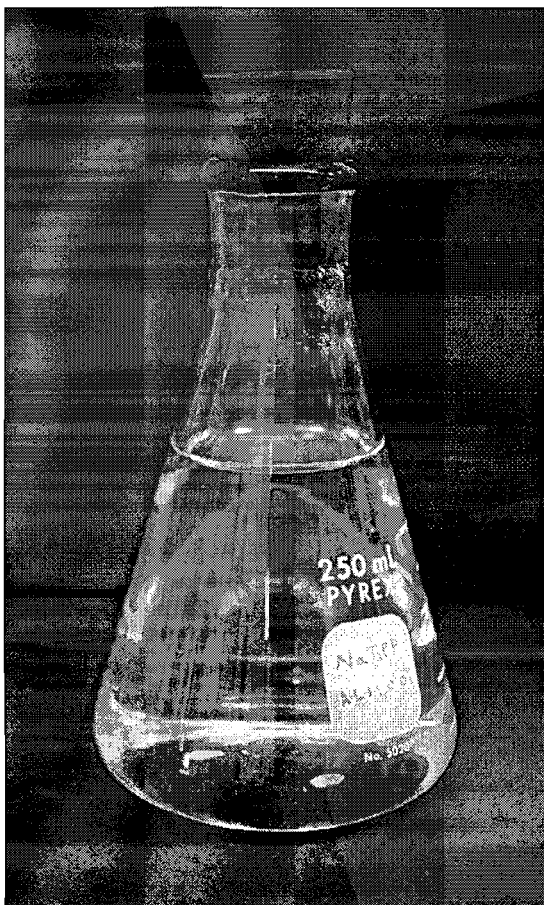
AL1100: 5 & 6

A508CL2: 5 & 6

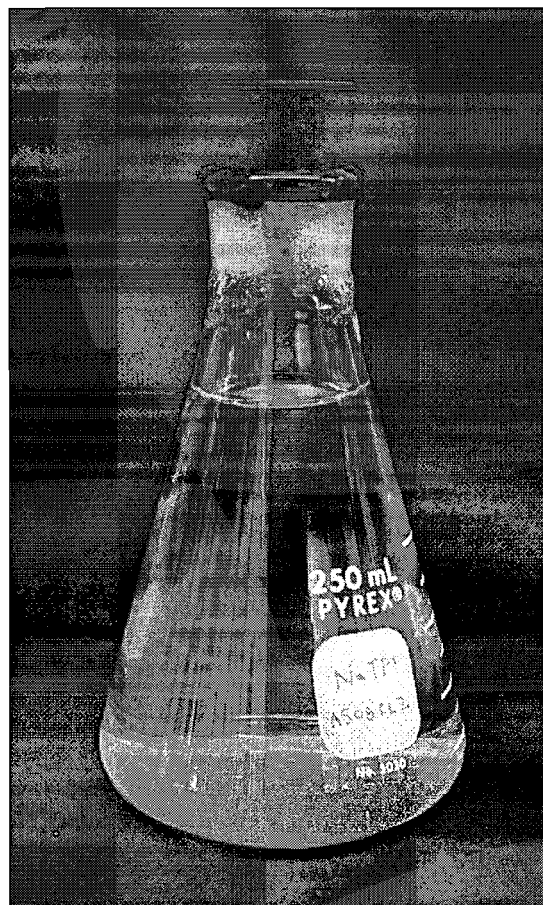
Observations after 2 weeks:

Coupon Type	Coupon ID	Precipitate Formed?	pH of Solution	Final Mass [g]	Final Mass After Descaling [g]
AL1100	5	No	7.56	7.1908	7.1876
AL1100	6	No	7.56	7.1876	7.1836
A508CL2	5	No	7.77	22.6111	22.5304
A508CL2	6	No	7.77	22.4076	22.3330

Sodium tripolyphosphate test solution after 2 weeks:



Solution that contained aluminum coupons



Solution that contained steel coupons

Test Chemical: Trisodium phosphate dodecahydrate

Preparation of 500 ml of 2500 ppm boron solution:

Boric acid required: 7.1496 g

Boric acid used: 7.1342 g

pH of solution: 4.65

Addition of trisodium phosphate dodecahydrate:

Mass for pH of 8.0: 4.9695 g

Mass used: 4.8955 g

pH of solution: 7.99

Coupons used:

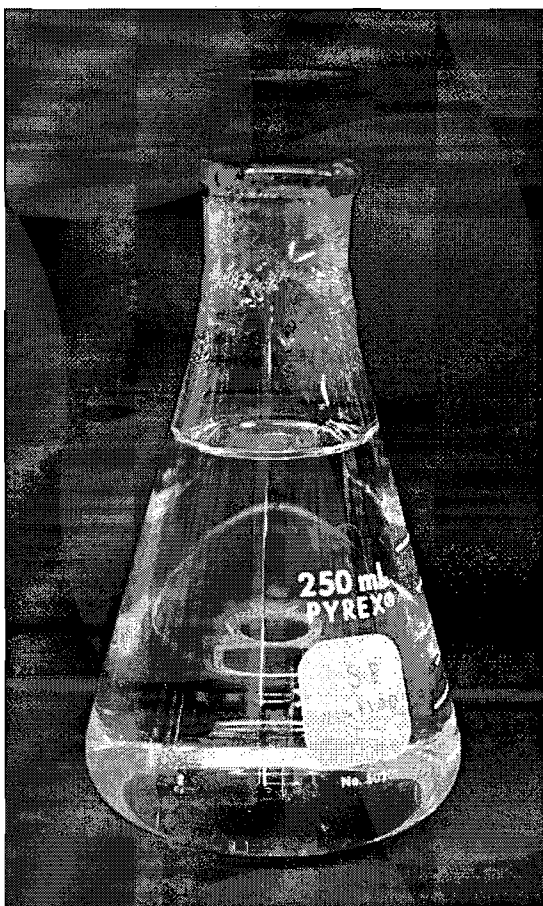
AL1100: 7 & 8

A508CL2: 7 & 8

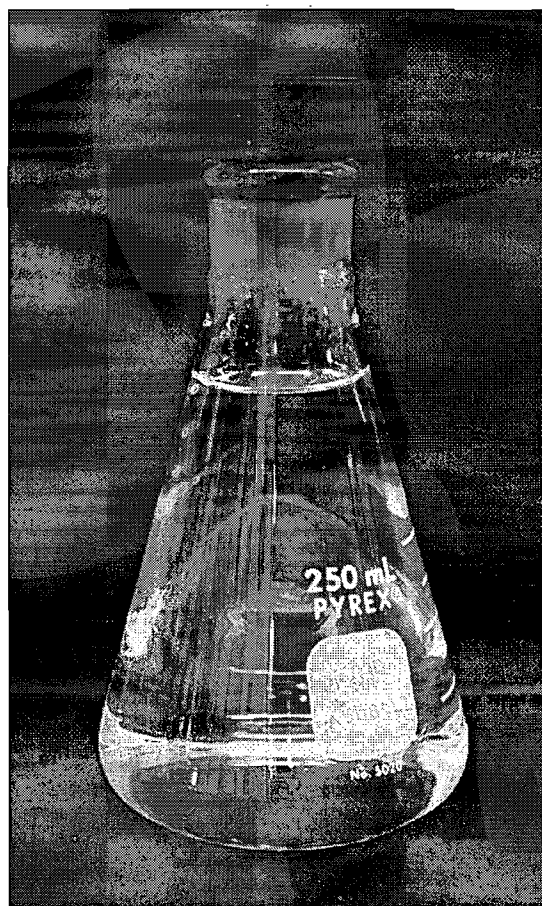
Observations after 2 weeks:

Coupon Type	Coupon ID	Precipitate Formed?	pH of Solution	Final Mass [g]	Final Mass After Descaling [g]
AL1100	7	No	8.01	7.2886	7.2821
AL1100	8	No	8.01	7.2160	7.2116
A508CL2	7	No	8.01	22.0520	22.0519
A508CL2	8	No	8.01	22.8760	22.8753

Trisodium phosphate dodecahydrate test solution after 2 weeks:



Solution that contained aluminum coupons



Solution that contained steel coupons

Test Chemical: Sodium hydroxide

Preparation of 500 ml of 2500 ppm boron solution:

Boric acid required: 7.1496 g

Boric acid used: 7.1367 g

pH of solution: 4.66

Addition of sodium hydroxide:

Mass for pH of 8.0: 0.5780 g

Mass used: 1.2259 g of 50% NaOH

pH of solution: 8.00

Coupons used:

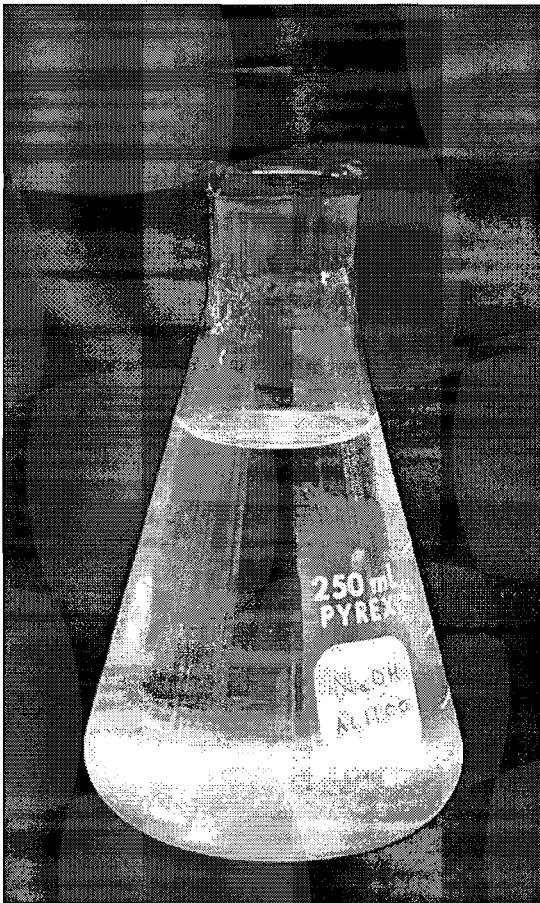
AL1100: 9 & 10

A508CL2: 9 & 10

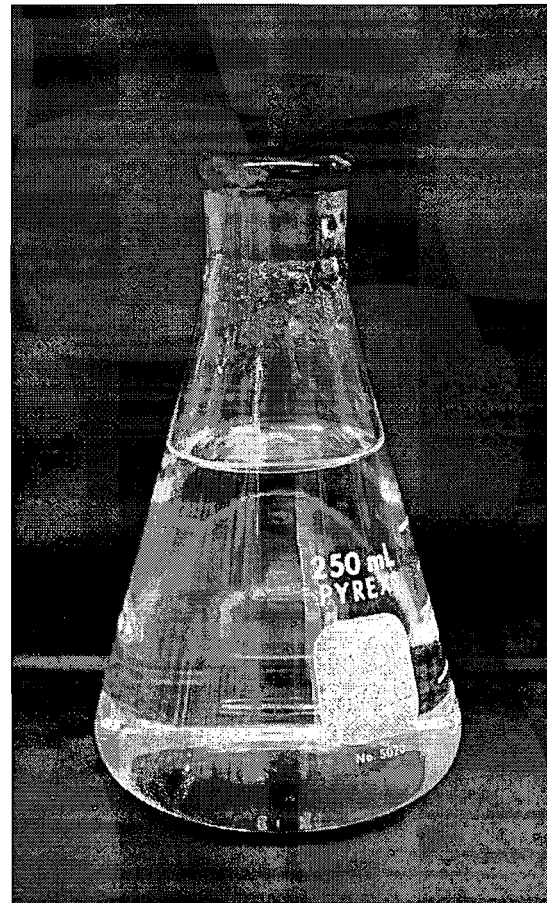
Observations after 2 weeks:

Coupon Type	Coupon ID	Precipitate Formed?	pH of Solution	Final Mass [g]	Final Mass After Descaling [g]
AL1100	9	Yes	8.02	7.1845	7.1567
AL1100	10	Yes	8.02	7.2361	7.2062
A508CL2	9	No	7.99	22.8028	22.8027
A508CL2	10	No	7.99	22.9219	22.9217

Sodium hydroxide test solution after 2 weeks:



Solution that contained aluminum coupons



Solution that contained steel coupons

Analysis of Dissolved Iron and Aluminum**Industrial Analytical Services Inc.**

A Subsidiary of ETR Labs

Customer: Fauske and Associates, LLC
16W070 West 83rd Street
Burr Ridge, IL 60527

Date: 5/17/2006

Report#: 51606368

P.O. #: credit card

Sample ID: see below

Iron & Aluminum Analysis

<u>Sample ID</u>	<u>Iron Results</u>	<u>Aluminum Results</u>
Sodium tetraborate decahydrate w/AL1100	NT	184.00 ppm
Sodium tetraborate decahydrate w/A508CL2	0.03 ppm	NT
Sodium metaborate tetrahydrate w/AL1100	NT	100.00 ppm
Sodium metaborate tetrahydrate w/A508CL2	< 0.01 mg/l	NT
Sodium tripolyphosphate w/AL1100	NT	478.80 ppm
Sodium tripolyphosphate w/A508CL2	1,100.00 ppm	NT
Trisodium phosphate dodecahydrate w/AL1100	NT	12.00 ppm
Trisodium phosphate dodecahydrate w/A508CL2	0.09 ppm	NT
Sodium Hydroxide w/AL1100	NT	42.90 ppm
Sodium Hydroxide w/A508CL2	0.03 ppm	NT

Analysis Method - Inductively Coupled Plasma Mass Spectroscopy

NT = Not Tested

Units = Parts Per Million (ppm) = mg/L

MDL = Minimum Detection Limit

MDL for Iron = 0.01 mg/l

MDL for Aluminum = 0.01 mg/l

The integrity of the sample and results are dependent on the quality of sampling. The results apply only to the actual sample tested. IAS Inc. shall be held harmless from any liability arising out of the use of such results.

29 Fuller Street Leominster, MA 01453 Phone 978-466-3422 Fax 978-466-3492

Appendix E: Detailed Results of Phase 6 Testing

It is important to note that two separate environmental effects tests were performed. Section E.1 documents the results of environmental testing performed while simulating 100% relative humidity. In order to do this, a beaker of water was placed within the oven. Section E.2 documents the results of environmental testing performed without the beaker of water. The relative humidity for the test was approximately 30%.

E.1 ENVIRONMENTAL EFFECTS TESTING WITH 100% HUMIDITY

Start Date: 3-31-06

End Date: 4-13-06

Loading of Samples:

Mass of sodium tetraborate decahydrate used: 10.0934 g

Mass of sodium metaborate tetrahydrate used: 10.0917 g

Mass of trisodium phosphate dodecahydrate used: 10.1012 g

100 ml of deionized water

Observations after 2 weeks:

- Sodium tetraborate decahydrate was still a solid, but both sodium metaborate tetrahydrate and trisodium phosphate dodecahydrate were dissolved.
- Upon cooling, the trisodium phosphate dodecahydrate solidified.
- The pH of the sodium metaborate tetrahydrate solution was 11.78.

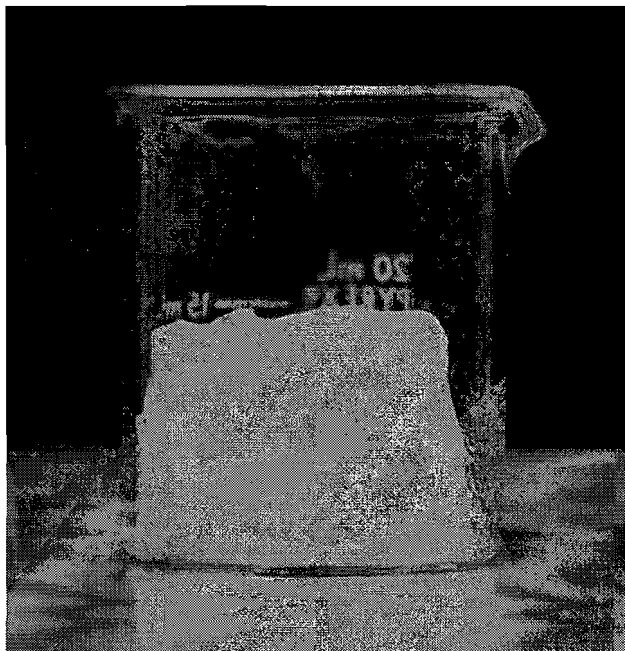


Figure E-1: Sodium tetraborate decahydrate remained a solid but was a clump as opposed to a fine powder.

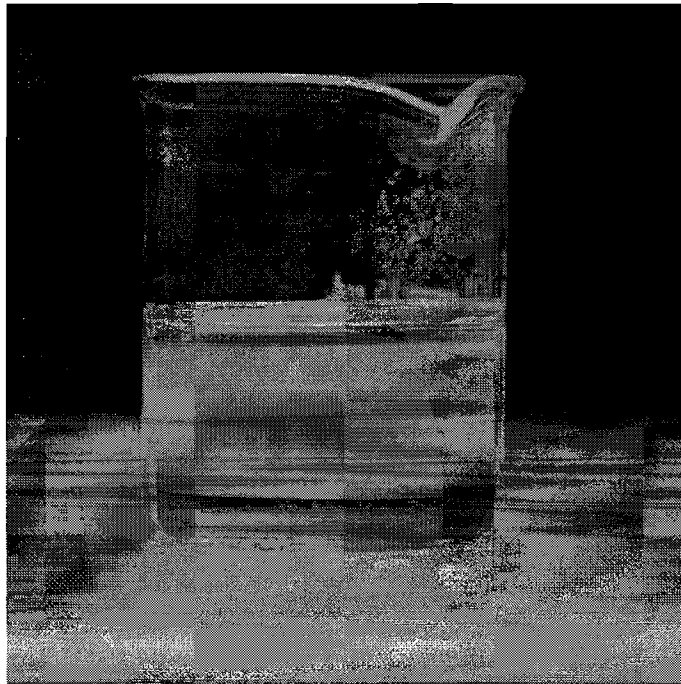


Figure E-2: Sodium metaborate tetrahydrate was completely dissolved.

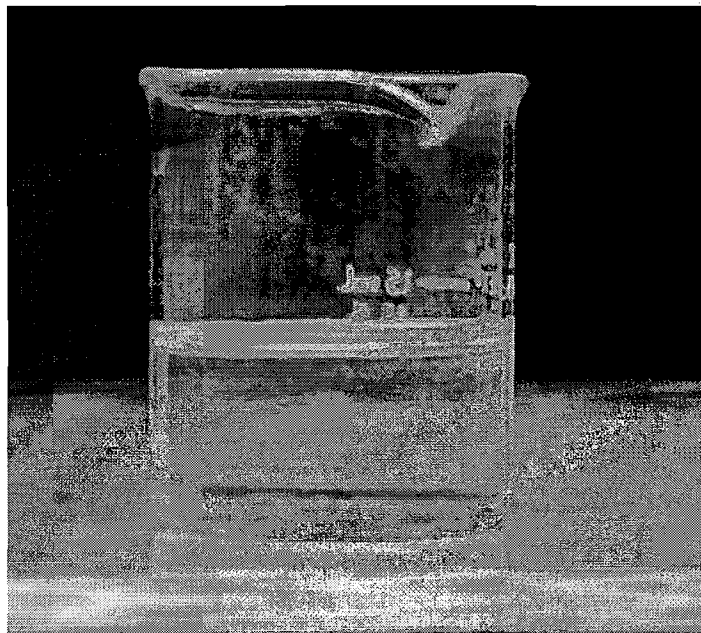


Figure E-3: The trisodium phosphate dodecahydrate sample was a liquid when it was removed from the oven.

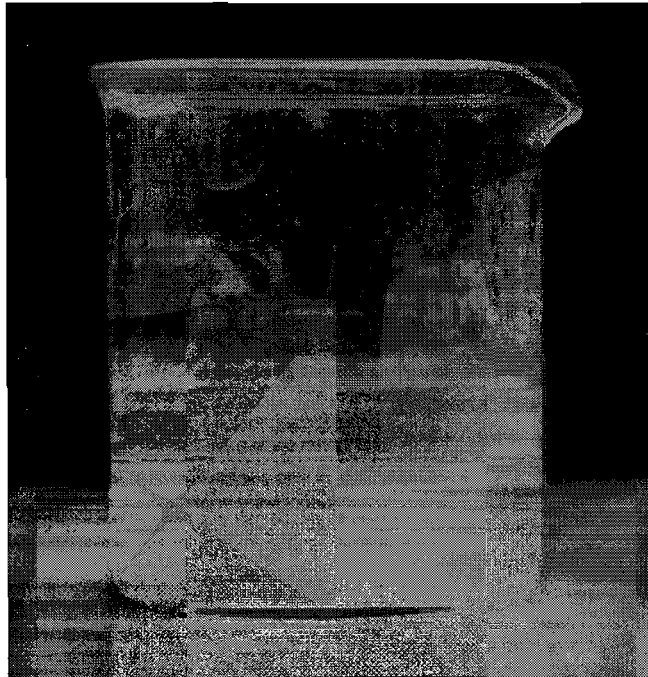


Figure E-4: The trisodium phosphate dodecahydrate sample was a solid after it was cooled to room temperature.



Figure E-5: The top-view of the trisodium phosphate dodecahydrate sample shows the surface after cooling.

E.2 ENVIRONMENTAL EFFECTS TESTING WITH 30% HUMIDITY

Start Date: 4-13-06

End Date: 5-12-06

Loading of Samples:

Mass of sodium tetraborate decahydrate used: 10.0443 g

Mass of sodium metaborate tetrahydrate used: 10.0522 g

Mass of trisodium phosphate dodecahydrate used: 10.0464 g

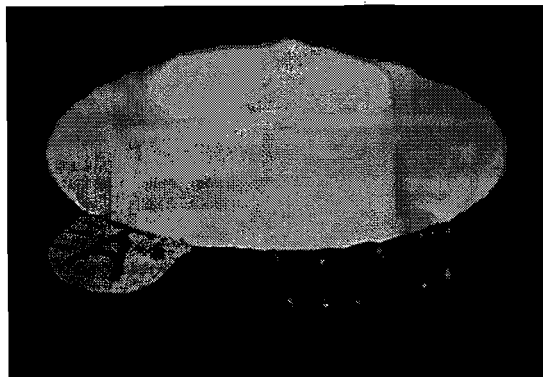
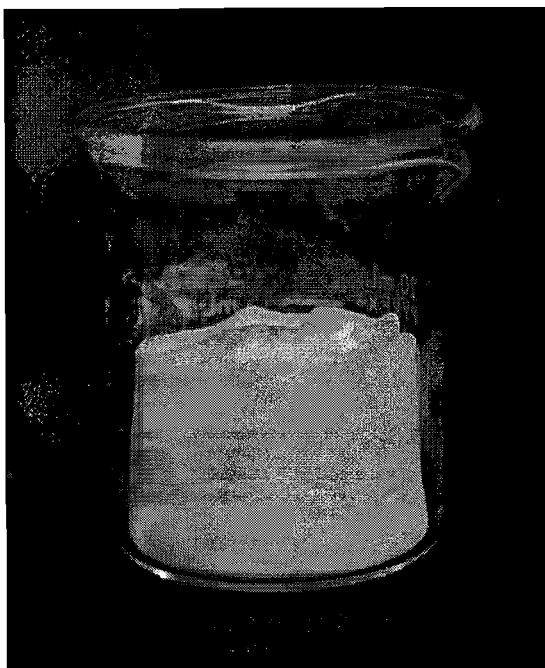
Mass of sodium tripolyphosphate used: 10.0703 g

Relative humidity in oven: ~ 30%

Observations after 30 days:

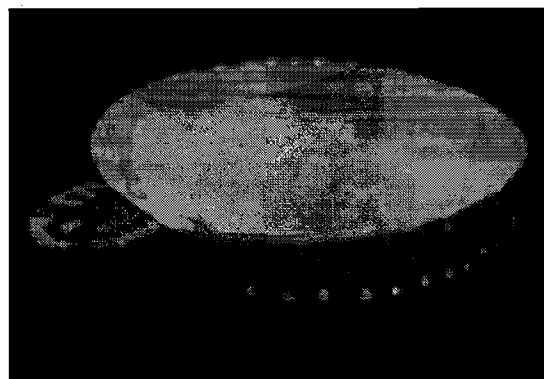
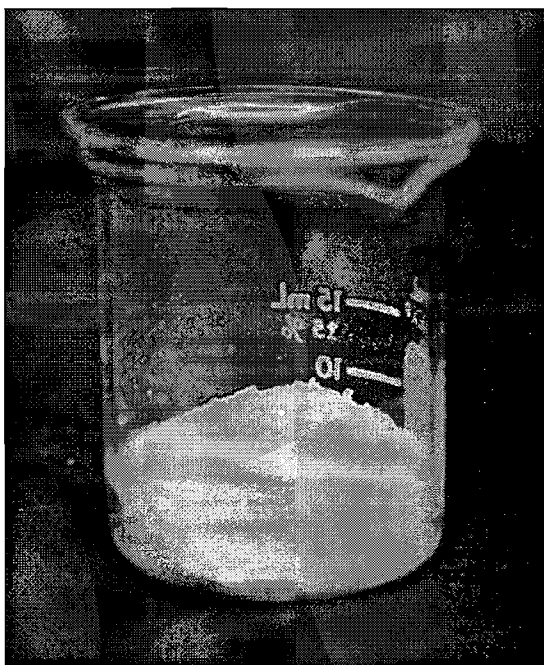
Sodium tetraborate decahydrate:

- Solid clump
- Sample separated from side of beaker
- Clump of sample easily slid out of the beaker



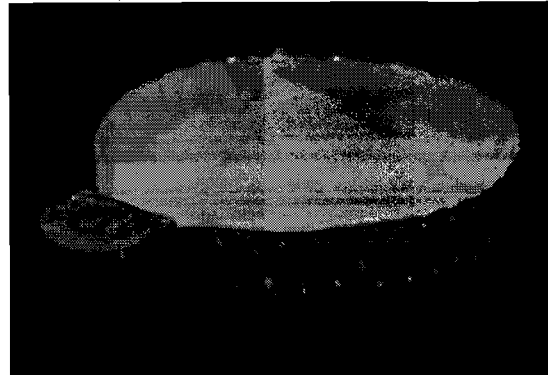
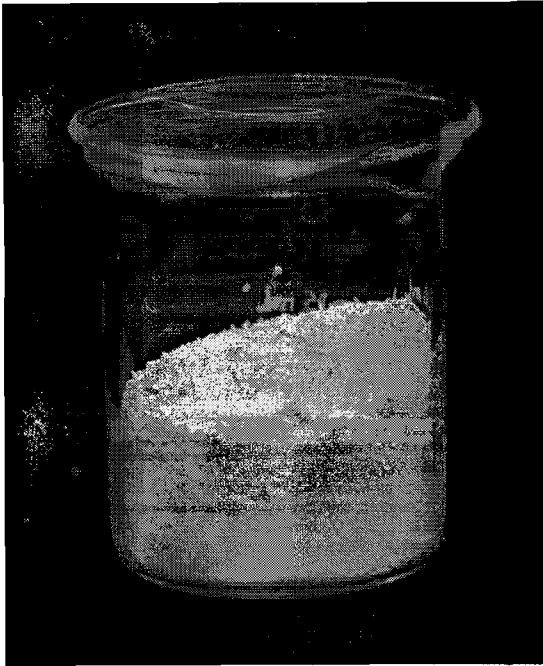
Sodium metaborate tetrahydrate

- Solid crystal-like clump
- Stuck to side of beaker
- Large decrease in volume
- Sample beaker had to be broken so that most of test sample could be dissolved (this resulted in having to add the test sample with a few pieces of glass stuck to sample)



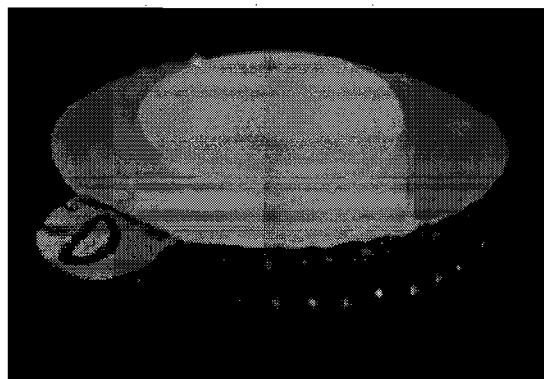
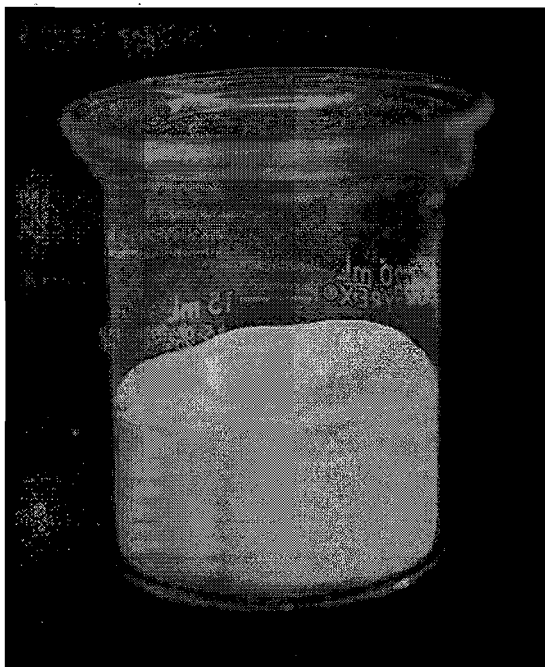
Trisodium phosphate dodecahydrate

- Still looks granular but is clumped together
- Stuck to sides
- When removed, sample broke up into a powder again



Sodium tripolyphosphate

- Solid clump
- Slight separation from side of beaker
- Clump of sample easily slid out of the beaker



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Dissolution results:

Buffer Candidate	Mass of Dissolution Sample [g]	Temperature of Water at Addition [°C]	Dissolution Time [min]	pH of Resulting Solution	Observations
Sodium tetraborate decahydrate	7.6877	67.9	3:29	9.21	Most of clump broke up when added to the water
Sodium metaborate tetrahydrate	7.4978	67.1	4:39	10.07	All of the loose sample was dissolved in 2:59 while sample that was still stuck to glass from the beaker took 4:39
Trisodium phosphate dodecahydrate	4.8797	66.9	2:37	11.39	Dissolved in the same fashion as in phase 1
Sodium tripolyphosphate	10.119	66.6	2:47	8.91	Most of clump broke up when added to the water

Appendix F: Detailed Results of Phase 7 Testing

Test Chemical: Sodium tetraborate decahydrate

Mass req'd for pH of 7.0: 0.1140 g

Time	Temp. [°C]	Boric Acid Addition [g]	Gross Mass [g]	Net Sample Mass [g]	Actual Wt.% Boric Acid	Observations
-	-	-	113.1152	0.0000	-	Mass of flask, stopper, stir bar
-	-	27.8524	140.9676	27.8524	100.00	Boric acid addition
-	-	-	213.5512	100.4360	27.73	Deionized water addition
10:00	-	-	-	-	-	Heat on at 5
10:15	92.3	-	-	-	-	
10:20	99.7	-	-	-	-	Almost all dissolved, heat at 3
10:23	105.1	-	-	-	-	All dissolved
10:25	105.0	-	214.1773	101.0621	27.56	2.1907 g of NaTD, pH = 2.36
10:35	102.8	0.6093	214.3915	101.2763	28.10	Dissolved
10:40	104.0	0.5526	214.6965	101.5813	28.56	Dissolved
10:45	105.1	0.6043	214.7176	101.6024	29.15	Dissolved
10:50	105.1	0.5501	214.1674	101.0522	29.85	Dissolved
10:58	105.2	0.5350	214.2956	101.1804	30.35	Not dissolved
11:09	105.2	-	213.8475	100.7323	30.48	End test

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Test Chemical: Sodium metaborate tetrahydrate

Mass req'd for pH of 7.0: 0.08003 g

Time	Temp. [°C]	Boric Acid Addition [g]	Gross Mass [g]	Net Sample Mass [g]	Actual Wt.% Boric Acid	Observations
-	-	-	115.2390	0.0000	-	Mass of flask, stopper, stir bar
-	-	27.5694	142.8084	27.5694	100.00	Boric acid addition
-	-	-	215.3186	100.0796	27.55	Deionized water addition
2:15	-	-	-	-	-	Heat on @ 5
2:30	53.2	-	-	-	-	
2:40	73.5	-	-	-	-	
2:50	96.1	-	-	-	-	Almost all dissolved
2:55	104.5	-	-	-	-	Dissolved, pH = 1.08, heat @ 3
3:00	104.1	-	215.8754	100.6364	27.40	1.5415 g of NaMT, pH = 2.55
3:04	102.9	0.5447	216.1186	100.8796	27.87	Dissolved
3:08	103.5	0.6323	216.4969	101.2579	28.39	Dissolved
3:11	103.5	0.6768	216.9331	101.6941	28.93	Dissolved
3:14	103.4	0.5704	217.2731	102.0341	29.40	Dissolved
3:19	104.5	0.5901	217.5116	102.2726	29.90	Dissolved
3:24	104.5	0.5270	217.6627	102.4237	30.37	Dissolved
3:30	104.8	0.5021	217.4863	102.2473	30.92	Dissolving very slowly
3:45	104.9	-	215.5525	100.3135	31.51	Not dissolved, End test

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium tripolyphosphate

Mass req'd for pH of 7.0: 0.2339 g

Time	Temp. [°C]	Boric Acid Addition [g]	Gross Mass [g]	Net Sample Mass [g]	Actual Wt.% Boric Acid	Observations
-	-	-	114.3566	0.0000	-	Mass of flask, stopper, stir bar
-	-	27.4753	141.8319	27.4753	100.00	Boric acid addition
-	-	-	214.3594	100.0028	27.47	Deionized water addition
9:50	-	-	-	-	-	Heat on @ 5
10:15	97.5	-	-	-	-	Almost dissolved, heat @ 3
10:18	105.0	-	213.8010	-	-	Dissolved, pH = 0.90
10:23	105.0	-	217.2623	-	-	4.5097 g of NaTPP, pH = 3.05
10:27	1045.0	0.5042	217.2162	-	-	Dissolved
10:30	104.8	0.5104	217.4626	103.1060	27.63	Dissolved
10:33	104.5	0.5431	217.7258	103.3692	28.09	Dissolved
10:37	104.7	0.5031	218.0464	103.6898	28.49	Dissolved
10:40	104.8	0.5394	218.3206	103.9640	28.93	Dissolved
10:44	104.7	0.5108	218.5911	104.2345	29.34	Dissolved
10:47	104.8	0.4957	218.8398	104.4832	29.75	Dissolved
10:52	104.9	0.5535	219.0929	104.7363	30.20	Dissolved
10:56	104.9	0.5791	219.4528	105.0962	30.65	Dissolved
10:58	104.7	0.5103	219.7037	105.3471	31.06	Dissolved
11:01	104.6	0.5105	219.9923	105.6357	31.46	Dissolved
11:05	104.8	0.5154	220.2465	105.8899	31.87	Dissolved
11:08	104.8	0.6051	220.6142	106.2576	32.33	Dissolved
11:12	104.3	0.5239	220.9093	106.5527	32.73	Dissolved
11:16	104.6	0.5359	221.2749	106.9183	33.12	Dissolved
11:22	105.0	0.4924	221.4383	107.0817	33.53	Dissolved
11:28	105.2	0.5310	221.3597	107.0031	34.05	Cloudy, dissolving very slowly
11:45	-	-	-	-	-	Not dissolved, End test

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Sodium hydroxide

Mass req'd for pH of 7.0 (50% NaOH): 0.0398 g

Time	Temp. [°C]	Boric Acid Addition [g]	Gross Mass [g]	Net Sample Mass [g]	Actual Wt.% Boric Acid	Observations
-	-	-	114.3005	0.0000	-	Mass of flask, stopper, stir bar
-	-	27.6164	141.9169	27.6164	100.00	Boric acid addition
-	-	-	214.4199	100.1194	27.58	Deionized water addition
2:13	-	-	-	-	-	Heat on @ 5
2:35	102.3	-	-	-	-	Almost dissolved, heat @ 3
2:37	104.6	-	-	-	-	Dissolved, pH = 1.05
2:40	104.0	-	213.7087	-	-	0.7988 g of 50% NaOH
2:43	104.3	0.5432	213.8901	-	-	Dissolved
2:45	104.7	0.5072	214.1214	99.8209	28.72	Dissolved
2:49	104.0	0.5205	214.0001	99.6996	29.28	Dissolved
2:52	104.3	0.5301	214.2200	99.9195	29.74	Dissolved
2:55	104.9	0.4907	214.3824	100.0819	30.18	Dissolved
2:58	104.4	0.5442	214.6847	100.3842	30.63	Dissolved
3:03	105.1	0.5555	214.6574	100.3569	31.20	Dissolving slowly
3:20	-	-	-	-	-	Not completely dissolved, End test

WESTINGHOUSE NON-PROPRIETARY CLASS 3

Test Chemical: Trisodium phosphate dodecahydrate

Mass req'd for pH of 7.0: 0.1299 g

Time	Temp. [°C]	Boric Acid Addition [g]	Gross Mass [g]	Net Sample Mass [g]	Actual Wt.% Boric Acid	Observations
-	-	-	112.9743	0.0000	-	Mass of flask, stopper, stir bar
-	-	27.5912	140.5655	27.5912	100.00	Boric acid addition
-	-	-	213.1027	100.1284	27.56	Deionized water addition
9:35	-	-	-	-	-	Heat on @ 5
9:55	99.8	-	-	-	-	Almost dissolved, heat @ 3
10:00	104.0	-	212.7788	-	-	Dissolved, pH = 0.98
10:05	103.9	-	215.0117	-	-	2.5050 g of TSP
10:10	104.5	0.5691	214.9390	-	-	Dissolved
10:14	104.5	0.5832	215.1999	102.2256	28.12	Dissolved
10:16	104.8	0.5763	215.4920	102.5177	28.60	Dissolved
10:20	104.6	0.5062	215.8097	102.8354	29.00	Dissolved
10:23	104.1	0.5348	216.1888	103.2145	29.42	Dissolved
10:28	104.4	0.5053	216.4903	103.5160	29.82	Dissolved
10:33	104.7	0.5623	216.8697	103.8954	30.25	Dissolved
10:36	104.5	0.5539	217.2058	104.2315	30.68	Dissolved
10:40	104.6	0.5608	217.6180	104.6437	31.10	Dissolved
10:46	104.6	0.5002	217.8312	104.8569	31.51	Dissolved
10:51	104.6	0.5006	218.0324	105.0581	31.93	Dissolving slowly
10:56	-	-	-	-	-	Almost all dissolved
10:59	-	-	-	-	-	Dissolved
11:02	104.8	-	217.5687	104.5944	32.07	
11:05	104.9	0.5575	217.8885	104.9142	32.50	Cloudy
11:15	105.0	-	-	-	-	Cloudy - not dissolving, End test