

Summary of Tests to Determine the Physical Properties of Buffered and Un-buffered Boric Acid Solutions

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Summary of Tests to Determine the Physical Properties of Buffered and Un-buffered Boric Acid Solutions

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LIST OF ACRONYMS AND ABBREVIATIONS

ACRS	Advisory Committee on Reactor Safeguards
ASME	American Society of Mechanical Engineers
B&W	Babcock and Wilcox
CE	Combustion Engineering
ECCS	Emergency Core Cooling System
EPU	Extended Power Uprate
LOCA	Loss of Coolant Accident
LTC	Long Term Cooling
MCE	Materials Center of Excellence
NIST	National Institute of Standards and Technology
NRC	Nuclear Regulatory Commission
NUREG	Nuclear Regulatory Commission Regulation
pH	Potential of Hydrogen
PIRT	Phenomena Identification and Ranking Table
PWR	Pressurized Water Reactor
PWROG	Pressurized Water Reactor Owners Group
RCS	Reactor Coolant System
SI	Safety Injection
STC	(Westinghouse) Science and Technology Center
WCAP	Westinghouse Technical Report Number Preface (formerly Westinghouse Commercial Atomic Power)

EXECUTIVE SUMMARY

Following a LOCA in a PWR, the RCS begins to depressurize and, for all but the smallest breaks, significant boiling occurs in the core. The ECCS injects borated water into the reactor vessel so as to keep the core fuel temperatures at acceptably low levels. All three US PWR nuclear plant designs (Westinghouse, CE, and B&W, advanced plant designs excluded) have ECCS features that, with or without operator action, initiate a core dilution mechanism to prevent the core region boric acid concentration from reaching the precipitation point. The three US PWR designs have different ECCS designs, different procedures for preventing boric acid precipitation, and different methodologies for evaluating the potential for boric acid precipitation. [Reference 1]

The NRC staff has cited the disparity of unjustified methods and assumptions used to address long term cooling boric (LTC) acid control across the US PWR fleet. An official NRC suspension of the use of Topical Report CENPD-254-P, "Post-LOCA Long-Term Cooling Model," was issued on August 1, 2005 [Reference 2]. This letter was followed by a clarification on November 23, 2005 [Reference 3].

In response to the suspension, the PWROG funded a program to develop a LOCA boric acid precipitation control analytical approach, applicable to all PWR designs that would be acceptable to the NRC staff. An early objective in this program was to develop a Phenomena Identification and Ranking Table (PIRT) that would provide guidance as to what phenomena the methodology needed to address and how sophisticated of a methodology was needed. While developing the PIRT, it became clear that there is a relatively low state-of-knowledge in the area of the physical properties of buffered boric acid solutions that would be typical of those found in a PWR containment sump after a LOCA [Reference 1]. For these reasons, the PWROG boric acid precipitation control program funded tests to investigate the properties of buffered and un-buffered boric acid solutions under conditions simulating that expected during the LTC phase following a LOCA.

This report is meant to help facilitate the development of an analytical approach for precipitation control by determining key physical properties of buffered and un-buffered boric acid solutions. Section 1 provides an introduction including background, the purpose of the tests, and the test matrix. Section 2, Section 3, and Section 4 outline detailed tests and results pertaining to the physical properties of density, viscosity, and surface tension, respectively. Section 5 presents solubility test data for buffered and un-buffered boric acid solutions at 65.6°C/150°F and 100.0°C/212°F. Section 6 presents observations, conclusions and recommendations concerning use of the data.

Density measurements were within expectations and support density driven convection between the core and lower reactor vessel regions as a mechanism that reduces the risk of bulk precipitation in the core region. Viscosity measurements indicated no highly viscous solutions that might inhibit free flow and mixing in the core. Observed differences in surface tension of the tested chemical solutions relative to water support enhanced nucleation for nucleate boiling heat transfer. As expected, the test data indicate that the solubility of boric acid is increased by the presence of pH buffering agents. Detailed measurement and calibration data are provided in appendices.

1 INTRODUCTION

1.1 BACKGROUND

Following a LOCA in a PWR, the RCS begins to depressurize and, for all but the smallest breaks, significant boiling occurs in the core. The ECCS injects borated water into the reactor vessel so as to keep the core fuel temperatures at acceptably low levels. All three US PWR nuclear plant designs (Westinghouse, CE, B&W) use boron as a core reactivity control method and are subject to concerns regarding potential boric acid precipitation in the core for scenarios that preclude direct safety injection (SI) flow through the core for extended periods following a LOCA. All three plant designs have ECCS features that, with appropriate operator action, initiate a flow through the core to prevent the core region boric acid concentration from reaching the boric acid solubility limit.

The common approach for demonstrating adequate boric acid dilution in a post-LOCA scenario includes simplified methods with conservative boundary conditions and assumptions. These simplified methods are used with limiting scenarios in calculations that determine the time at which appropriate operator action must be taken to initiate an active boron dilution flow path. All three US PWR designs have different ECCS designs, different procedures for preventing boric acid precipitation, and different methodologies for evaluating the potential for boric acid precipitation. There are common approaches, assumptions and simplifications that have been used in virtually all PWR calculations that address the potential for boric acid precipitation. Recent Extended Power Uprate (EPU) Programs have provided the opportunity for the NRC to challenge some of these common approaches, assumptions and simplifications with regards to regulatory compliance and technical justification.

On August 1, 2005, shortly after the Waterford EPU was approved, the NRC staff suspended the use [Reference 2] of Topical Report CENPD-254-P, "Post-LOCA Long-Term Cooling Model" [Reference 4]. This suspension gave an extensive list of NRC questions related to the potential for boric acid precipitation after a LOCA event. This suspension was later clarified by the NRC in a letter on November 23, 2005 [Reference 3]. In response to these questions and to the NRC's concerns regarding the methodologies used by industry to analyze the potential for boric acid precipitation, the PWROG funded a long-term program to develop a LOCA boric acid precipitation control analytical approach that will be applicable to all PWR designs and that would be acceptable to the NRC staff.

Phase 1 of the long term program is to establish evaluation model scenarios, assumptions, and acceptance criteria that would appropriately address the issues that were raised and be acceptable to the NRC. The initial task of Phase 1 was to develop a PIRT that would provide guidance as to what phenomena the methodology needed to address and how sophisticated of a methodology was needed. In the series of PIRT expert panel sessions that followed, it became clear that there is a relatively low state-of-knowledge in the area of the physical properties of buffered sodium borate solutions that would be typical of those found in a PWR containment sump after a LOCA [Reference 1].

A follow-up study of LOCA coolant chemistry and possible post LOCA chemical reactions provided insights on how different chemistries might impact LTC of the reactor core. The study concluded that a variety of different coolant chemistries could exist after a LOCA. For plants which add sodium hydroxide for pH control, the coolant would contain a variety of dissolved species which may behave quite differently than simple boric acid solutions. Such solutions, depending on pH, would tend to precipitate sodium borates rather than boric acid and would do so at different concentration levels. Likewise, plants adding trisodium phosphate or sodium tetraborate would also have different properties than boric acid solutions [Reference 5].

The presence of pH control agents in sump solutions will impact not only precipitation from solution, but also other properties such as density, viscosity and surface tension. Understanding the changes in these properties would increase the state-of-knowledge of medium and high ranked mixing phenomena identified in the PIRT for buffered and un-buffered boric acid solutions. Data on the physical properties of sump solutions could also aid in determining appropriate testing to be done which will ultimately be used to address questions raised by the NRC staff and ACRS members concerning the fluid properties of buffered and un-buffered boric acid solutions. One example of such a question (from Reference 3) is as follows:

The impact of the boric acid on transport properties and other quantities affecting drift velocity and level swells, as concentrations increase, has not been evaluated. The transport properties as well as the drift velocity correlations may need to be justified when computing the void fraction in the mixing volume. The fluid density in the mixing volume needs to include the impact of the boric acid.

For the above reasons, the PWROG boric acid precipitation control program funded tests to investigate the properties of buffered and un-buffered boric acid solutions under conditions simulating that expected during the LTC phase following a LOCA.

1.2 PURPOSE OF THE TESTS

The purpose of these tests was to investigate the properties of buffered and un-buffered boric acid solutions under conditions simulating that expected during the long-term cooling phase following a LOCA. Based upon the first two rounds of the small scale buffered and un-buffered boric acid nucleate boiling heat transfer tests (single rod tests conducted at post-LOCA heat flux levels) performed at the STC [Reference 6], thermodynamic properties in relation to nucleate boiling heat transfer for buffered and un-buffered boric acid solutions are not significantly different from water [Reference 5]. It was identified that the physical properties of buffered and un-buffered boric acid pertaining to surface tension, viscosity, and density need to be further investigated. These properties are expected to be important for post-LOCA mixing/transport phenomena in the reactor vessel [Reference 6].

To increase the state of knowledge in the PIRT [Reference 6] and to address previous questions raised by the NRC staff and ACRS members concerning fluid properties of buffered and un-buffered boric acid solutions, experiments quantifying the density, viscosity, surface tension, and a solubility limit over a range of concentrations of buffered and un-buffered boric acid solutions are desired.

The motivations for investigating solubility limits of buffered and un-buffered boric acid is directly related to requests made by the NRC to justify the solubility limit of boric acid if containment pressures greater than 14.7 psia are assumed or additives are contained in the sump water. This report is meant to address the NRC requirement for justification of solubility limits in the presence of additives.

In summary, the results of these tests will be used for the following.

- To increase the state-of-knowledge of solution properties for medium and high ranked mixing phenomena identified in the PIRT for boric acid and sodium borate solutions.
- To address NRC/ACRS questions on concentrated sump solution physical properties.
- To provide insights for subsequent small scale mixing tests.
- To provide data to determine critical property data for use in analytical mixing models.
- To support the assumption of water-like behavior at low concentrations, thus validating common LOCA clad heat-up evaluation model assumptions.

1.3 TEST MATRIX

The objective of these tests was to further investigate the physical properties of buffered and un-buffered boric acid solutions to simulate conditions expected during the cooling phase following a LOCA. The solutions of interest are as follows:

- Boric Acid (H_3BO_3 , un-buffered)
- Boric Acid buffered with Sodium Hydroxide (NaOH) pH 7
- Boric Acid buffered with Sodium Hydroxide (NaOH) pH 10
- Boric Acid buffered with Trisodium Phosphate (TSP, $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$)
- Boric Acid buffered with Sodium Tetraborate (Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$)

Solution concentrations were selected to cover a broad range of reactor coolant chemical compositions that might be present after a LOCA. Concentrations representing the reactor water composition early in an accident were selected for physical property testing, along with concentrations that would be expected after a 10X – 20X evaporative concentration in the core. A solubility limit was determined by adding additional boric acid and pH buffer beyond the 10X – 20X evaporative concentration level, while maintaining constant pH.

Boric acid is the most concentrated chemical additive in the coolant during normal operation where its concentration may approach 1800 ppm B¹. It is also the main additive contained within the high pressure

-
1. The boric acid concentration in reactor coolant water is usually reported in units of ppm B, which is milligrams of boron per kilogram of solution. To convert this quantity to ppm boric acid, multiply by the molecular weight of boron, 61.83, and then divide by the atomic weight of boron, 10.81 (i.e., 1800 ppm B=10,295 ppm boric acid).

accumulators and the refueling water storage tank, where concentrations near 2500 ppm B are typical. It will not be present in the reactor without an alkaline pH control agent after recirculation from the sump, but it was included in the test matrix as a bounding case and to provide data relevant to the short period of time after a LOCA when significant quantities of coolant have not yet entered the reactor.

Three alkaline pH control agents are used in PWRs: sodium hydroxide, NaOH, trisodium phosphate, (TSP, $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$), and Sodium Tetraborate (Borax, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$).

Sodium hydroxide is introduced into the coolant via the containment spray system in some plants. Within the containment spray system, a concentrated sodium hydroxide solution is added to water from the refueling water storage tank to produce a solution which may have a pH as high as 12 in the spray. However, when the spray solution is mixed with the coolant in the sump, the pH is expected to be near pH 9.3, the design pH for plants with NaOH pH adjustment [Reference 8]. For the purposes of the tests here, pH values of 7 and 10 were selected as representatively low and high values for the NaOH – H_3BO_3 system before evaporative concentration in the core. The bounding lower pH was selected since all plants have designed their ECCS to achieve this minimum pH to meet regulatory requirements for iodine retention. The maximum pH was selected to be consistent with the maximum sump pH used by industry and the NRC for LTC testing [Reference 9].

Trisodium phosphate is used for pH control in the ECCS at some plants. The trisodium phosphate is contained in baskets located within containment. If a break occurs, the trisodium phosphate is dissolved by coolant exiting the break location onto the containment floor. It is then carried into the reactor by recirculation of coolant which has collected in the sump. Four (4) trisodium phosphate solution/boric acid concentrations were selected for physical property testing. The first two (2) were representative of the ECCS concentrations before evaporative concentration, and the other two (2) after concentration by 10X – 20X.

Sodium tetraborate (a form of borax) is also used for ECCS pH control in PWRs. In some plants, it is added to the coolant via baskets on the containment floor. In plants with ice condensers, the sodium tetraborate is contained in an ice reservoir that remains frozen during normal operation, but which melts during an accident. Two sodium tetraborate/boric acid solutions were tested. The first was representative of the ECCS concentrations before evaporative concentration, and the other after concentration by 10X.

It should be noted that extremes in pH were not tested for the sodium tetraborate and trisodium phosphate solutions. Since trisodium phosphate and sodium tetraborate are weak bases, they form good buffers with the weak acid, boric acid, and extremes in pH are not likely for any reasonable scenario within the reactor.

The species present in the aqueous solutions which were selected for testing will reflect chemical reactions which occur both with water and between the solutes. Sodium hydroxide is a strong base, and will totally dissociate in water to yield hydrated sodium cations and hydroxide anions. The other solution components are either weak acids or bases, meaning that they do not dissociate completely into ionic species when they dissolve. Boric acid will remain primarily as free H_3BO_3 molecules at low pH and in dilute solution. As pH is increased by the addition of NaOH or another base, some of the boric acid molecules will associate with the hydroxide anions, forming $\text{B}(\text{OH})_4^-$. At higher concentrations and higher pH values, polyborate species such as $\text{B}_3\text{O}_3(\text{OH})_4^-$ will begin to form, and there will be an equilibrium between undissociated boric acid and the polyborate anions. Sodium tetraborate will dissolve to produce

free sodium and a variety of boron species, including boric acid. Trisodium phosphate will dissociate into a variety of phosphate anions and free sodium. Like the boron species, phosphates tend to form polyphosphates and they may also associate with boron species to form mixed phosphate-borate complexes [Reference 11].

During the nucleate boiling channel tests performed at the STC, thermodynamic properties of buffered and un-buffered boric acid solutions were not significantly different from water. However, there were some differences observed with respect to surface tension, viscosity, and density. These properties are expected to be important for post-LOCA mixing/transport phenomena in the reactor vessel. To increase the state of knowledge in the PIRT and to address concerns raised by the NRC staff and ACRS members concerning fluid properties of buffered and un-buffered boric acid solutions, experiments quantifying density, viscosity, and surface tension over a range of concentrations of buffered and un-buffered boric acid solutions were performed.

Physical properties for the aforementioned solutions were determined at various concentrations and temperatures. The experiments were conducted in four phases:

- Phase 1: Density
- Phase 2: Viscosity
- Phase 3: Surface Tension
- Phase 4: Solubility Limits

The properties were obtained at atmospheric pressure consistent with low pressure, post-LOCA conditions. This covers large break scenarios where the RCS would quickly depressurize to near atmospheric pressure, as well as intermediate and small break scenarios where reactor operators would take actions to cool down and depressurize the RCS. The chemical solution temperature range should cover the bulk conditions in the boiling region and lower plenum of the reactor vessel. Therefore, the temperature range was chosen to be 65.6°C/150°F – 100.0°C/212°F [Reference 7].

2 PHASE 1: DENSITY

During Phase 1, the densities of the boric acid solutions were determined at various temperatures and concentrations. The following steps were taken to determine the density of each solution:

1. The quantities of boric acid and buffer required to achieve the desired concentrations were calculated using equation 2-1 and equation 2-2:

$$m_{\text{H}_3\text{BO}_3} = \frac{M_{\text{H}_3\text{BO}_3}}{M_{\text{B}}} \cdot \frac{m_{\text{total}} \cdot C_{\text{B}}}{1,000,000} \quad (2-1)$$

$$m_{\text{buffer}} = \frac{m_{\text{total}} \cdot C_{\text{buffer}}}{1,000,000} \quad (2-2)$$

where:

C_{B}	=	target concentration of boron [ppm]
C_{buffer}	=	target concentration of buffer [ppm]
m_{total}	=	total mass of solution [g]
$m_{\text{H}_3\text{BO}_3}$	=	mass of boric acid required [g]
m_{buffer}	=	mass of buffer required [g]
$M_{\text{H}_3\text{BO}_3}$	=	molecular weight of boric acid [61.83 g/gmol]
M_{B}	=	molecular weight of boron [10.81 g/gmol]

2. 150 grams of solution were prepared using the required masses of boric acid, buffer, and water. The chemicals were weighed and added to an Erlenmeyer flask.
3. The solution was mixed and heated to the desired temperature using a hot plate magnetic stirrer.
4. The pH of the solution was measured using a pH probe.
5. 100 milliliters of solution were measured using a calibrated graduated cylinder.
6. The mass of the solution was measured.

The mass of the buffer included waters of hydration. The trisodium phosphate (TSP) formula was $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$. The formula for the sodium tetraborate (NaTB) used in this testing was $\text{Na}_2\text{B}_4\text{O}_7 \cdot 12\text{H}_2\text{O}$.

7. The density of the solution was calculated using the measured mass and volume.

$$\rho = \frac{m}{V} \quad (2-3)$$

where:

ρ = density of solution
 m = mass of solution
 V = volume of solution

The results of Phase 1 testing are shown in Table 2-1. The experimental results are compared to the accepted values [Reference 12] for water at 65.6°C/150°F and 100.0°C/212°F. Table 2-1 is depicted graphically in Figure 2-1 through Figure 2-4. Detailed results are shown in Appendix A.

The pH values displayed by the pH meter and tabulated in Table 2-1 were obtained “at temperature.” The pH meter applied a temperature correction factor to account for electrode response variation with temperature. No attempt was made to correct the pH values to a standard reference temperature such as 25°C/77°F.

Table 2-1 Results of Density Testing							
	Concentration of Boron from Boric Acid	Concentration of Buffer	Temperature		Measured pH	Density	
Coolant	(ppm)	(ppm)	°C	°F		kg/m³	lbm/ft³
Pure Water ⁽¹⁾	0	0	65.6	150		980.2	61.2
Pure Water ⁽¹⁾	0	0	100.0	212		958.1	59.8
Boric Acid	2,520	0	65.6	150	4.88	981.4	61.3
Boric Acid	2,520	0	100.0	212	4.74	963.5	60.1
Boric Acid	25,012	0	65.6	150	2.38	1019.4	63.6
Boric Acid	45,002 (2)	0	100.0	212	2.13	1042.4	65.1
Boric Acid, NaOH (pH 10)	2,497	6,190	65.6	150	9.68	983.4	61.4
Boric Acid, NaOH (pH 10)	2,497	6,190	100.0	212	9.22	962.2	60.1

**Table 2-1 Results of Density Testing
(cont.)**

Coolant	Concentration of Boron from Boric Acid (ppm)	Concentration of Buffer (ppm)	Temperature		Measured pH	Density	
			°C	°F		kg/m ³	lbm/ft ³
Boric Acid, NaOH (pH 10)	25,067	63,452	65.6	150	9.64	1098.1	68.6
Boric Acid, NaOH (pH 10)	49,946	126,006	100.0	212	9.66	1237.5	77.3
Boric Acid, NaOH (pH 7)	2,512	955	65.6	150	7.73	982.3	61.3
Boric Acid, NaOH (pH 7)	2,512	955	100.0	212	7.62	964.4	60.2
Boric Acid, NaOH (pH 7)	25,015	9,050	65.6	150	5.73	1026.8	64.1
Boric Acid, NaOH (pH 7)	50,119	17,977	100.0	212	4.28	1072.1	66.9
Boric Acid, TSP	2,466	9,965	65.6	150	8.46	988.2	61.7
Boric Acid, TSP	2,510	10,035	100.0	212	8.26	976.0	60.9
Boric Acid, TSP	24,972	100,004	65.6	150	7.75	1128.3	70.4
Boric Acid, TSP	49,966	199,816	100.0	212	7.39	1304.4	81.4
Boric Acid, NaTB	2,508	12,070	65.6	150	8.03	985.1	61.5
Boric Acid, NaTB	2,508	12,070	100.0	212	7.92	967.7	60.4
Boric Acid, NaTB	25,002	120,071	65.6	150	6.58	1069.3	66.8
Boric Acid, NaTB	50,034	240,283	100.0	212	5.21	1189.8	74.3

Notes:

1. The 1967 ASME steam tables [Reference 12] were utilized for water data. It is noted that water property values obtained from NIST are slightly different [Reference 10].
2. 50,000 ppm resulted in precipitation

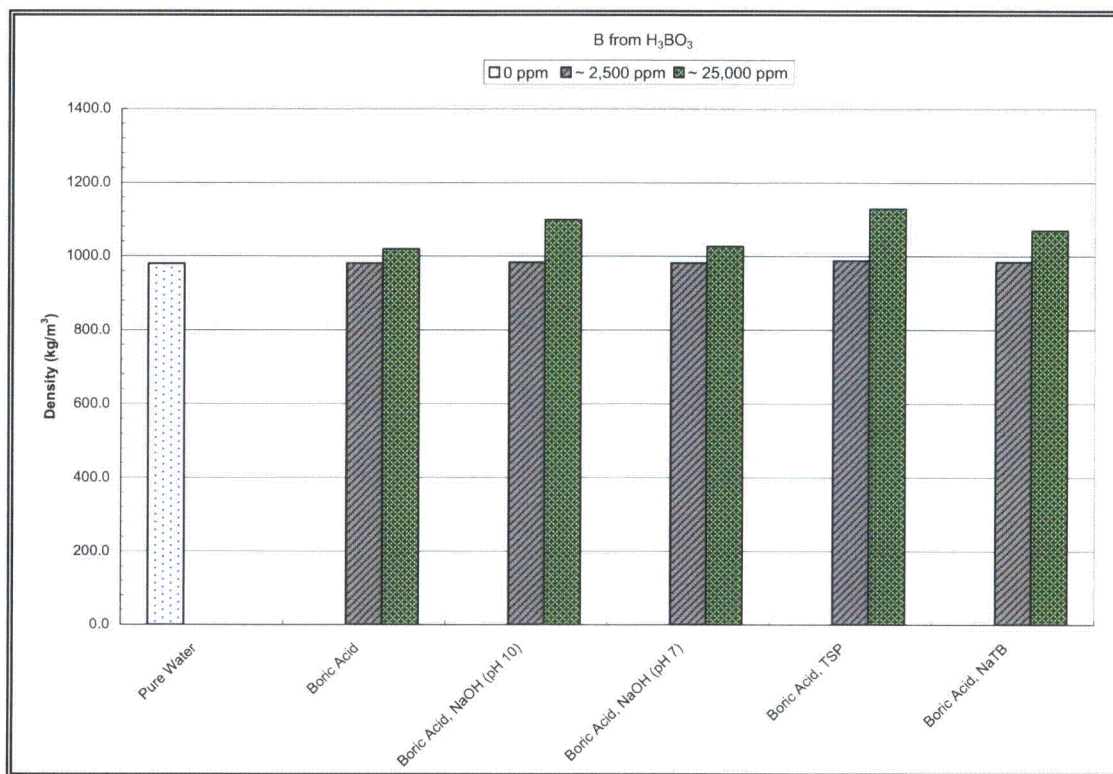


Figure 2-1 Density of Buffered and Un-Buffered Boric Acid at 65.6°C

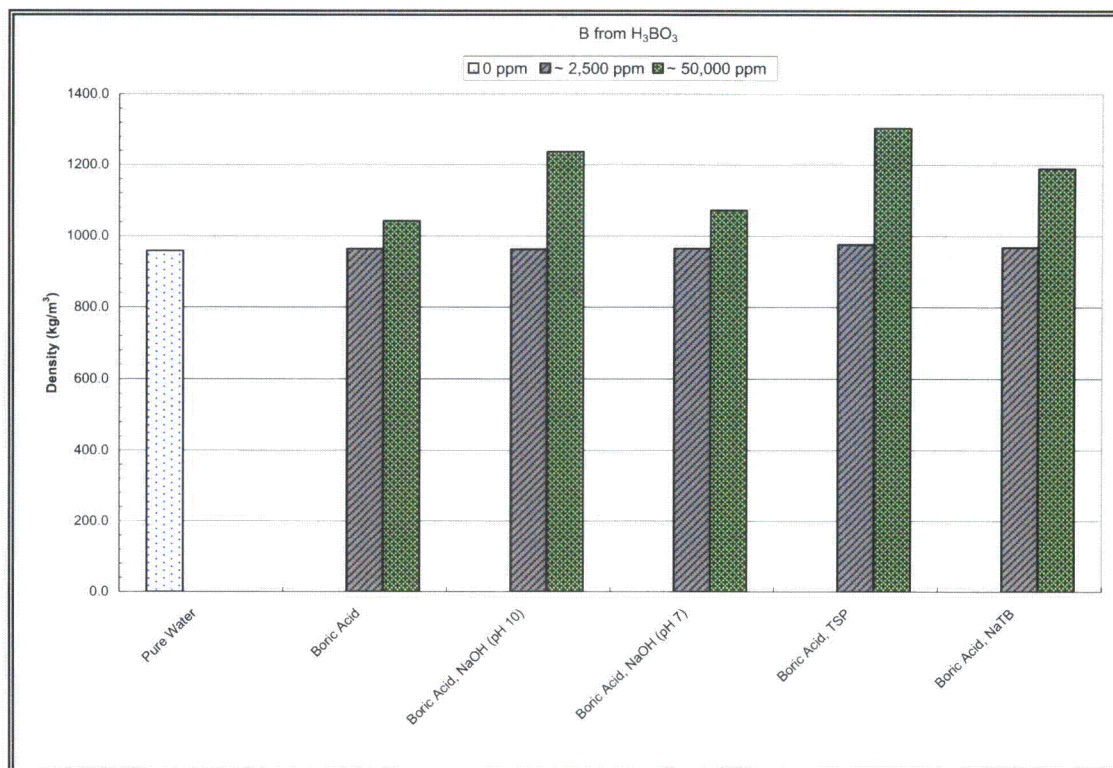


Figure 2-2 Density of Buffered and Un-Buffered Boric Acid at 100.0°C

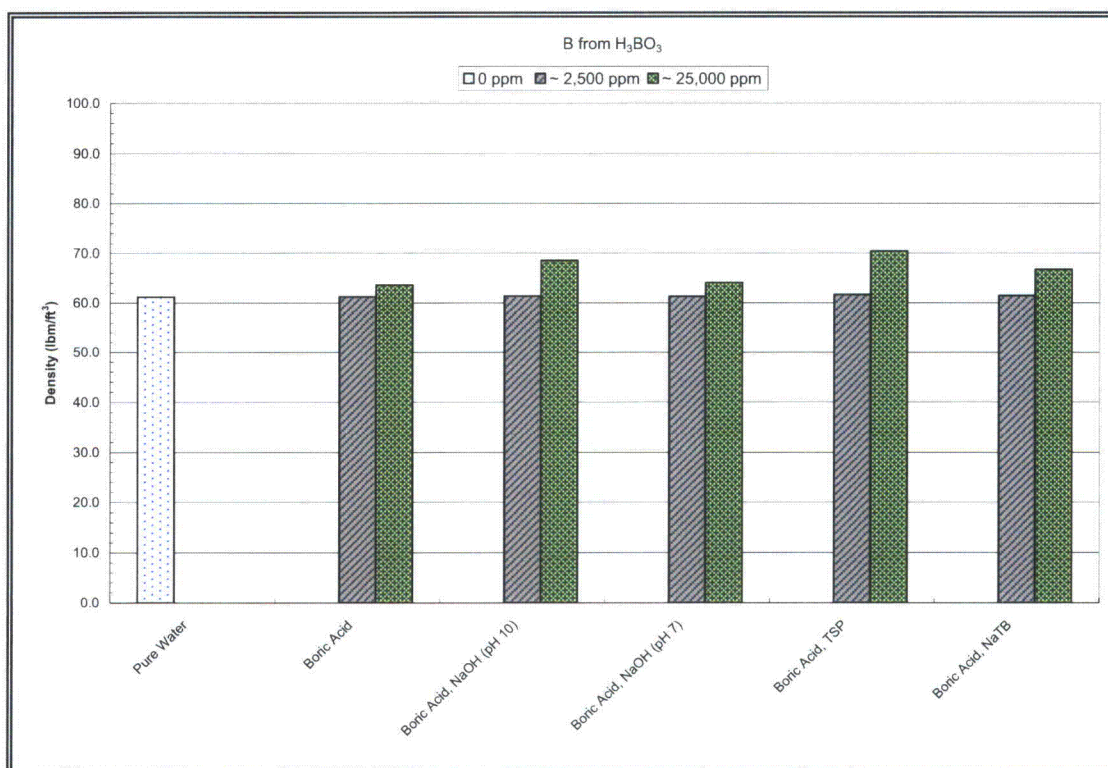


Figure 2-3 Density of Buffered and Un-Buffered Boric Acid at 150°F

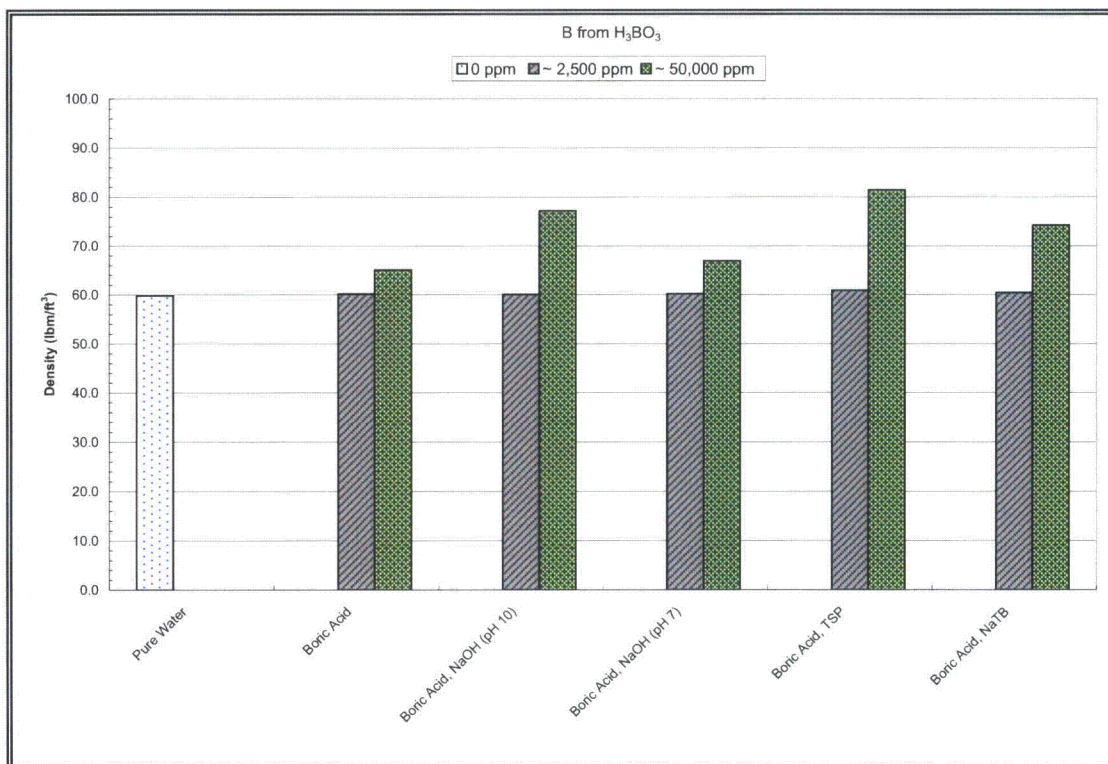


Figure 2-4 Density of Buffered and Un-Buffered Boric Acid at 212°F

3 PHASE 2: VISCOSITY

During Phase 2, the viscosity of each boric acid solution was determined using an Ubbelohde viscometer with a measuring range appropriate for the solution being tested. A circulating temperature controlled bath of ethylene glycol was used in conjunction with a large graduated cylinder so that the viscometer could be maintained at the desired temperature. For this phase, the same solutions prepared in Phase 1 were used for consistency of data. The apparatus constructed for the viscosity measurements can be seen in Figure 3-1.

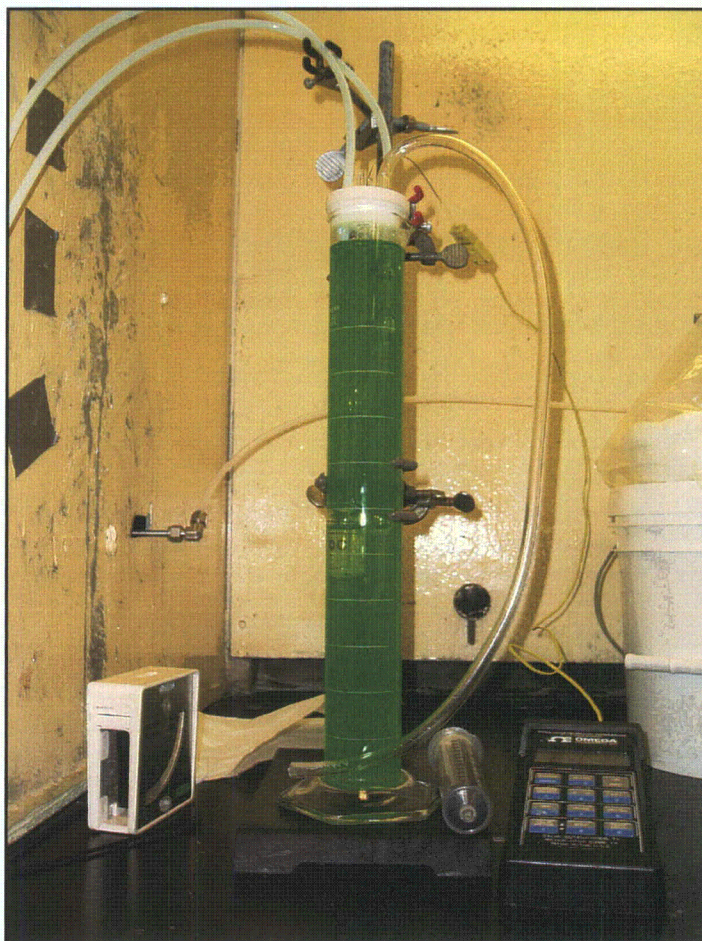


Figure 3-1 Experimental Setup for Viscosity Measurement

The following steps were performed to determine the viscosity of each solution:

1. The ethylene glycol bath was turned on and set to the desired temperature.
2. Approximately 35 milliliters of preheated solution were added to the Ubbelohde viscometer using a syringe.
3. The temperature of the solution was allowed to reach the desired steady state value.

4. The solution was drawn through the capillary by creating a slight vacuum in the capillary tube.
5. The time required for the solution to fall to a predetermined level was measured and recorded.
6. The kinematic viscosity of the solution was determined by multiplying the efflux time by the viscometer constant provided by the viscometer manufacturer.
7. The dynamic viscosity was calculated as follows:

$$\mu = \rho \cdot \nu \quad (3-1)$$

where:

μ = dynamic viscosity
 ρ = density of solution
 ν = kinematic viscosity

8. Steps 4-7 were repeated five times for each solution.
9. The average dynamic viscosity along with a standard deviation of the measurements was calculated.

The results of Phase 2 testing are shown in Table 3-1. Table 3-1 is depicted graphically in Figure 3-2 through Figure 3-5. Detailed results are shown in Appendix A. Calibration data for viscosity measurements are provided in Appendix D.

Table 3-1 Results of Viscosity Testing								
Coolant	Concentration of Boron from Boric Acid	Concentration of Buffer	Temperature		Viscosity		Standard Deviation	
	(ppm)	(ppm)	°C	°F	kg/m-s	lbm/ft-s	kg/m-s	lbm/ft-s
Pure Water ⁽¹⁾	0	0	65.6	150	4.21E-04	2.83E-04	N/A	N/A
Pure Water ⁽¹⁾	0	0	100.0	212	2.77E-04	1.86E-04	N/A	N/A
Boric Acid	2,520	0	65.6	150	4.49E-04	3.02E-04	1.51E-06	1.01E-06
Boric Acid	2,520	0	100.0	212	2.97E-04	2.00E-04	1.58E-06	1.06E-06
Boric Acid	25,012	0	65.6	150	5.42E-04	3.64E-04	2.21E-06	1.48E-06
Boric Acid	45,002	0	100.0	212	4.31E-04	2.90E-04	2.93E-06	1.97E-06
Boric Acid, NaOH (pH 10)	2,497	6,190	65.6	150	4.58E-04	3.08E-04	4.19E-06	2.82E-06

Table 3-1 Results of Viscosity Testing (cont.)

Coolant	Concentration of Boron from Boric Acid	Concentration of Buffer	Temperature		Viscosity		Standard Deviation	
	(ppm)	(ppm)	°C	°F	kg/m-s	lbm/ft-s	kg/m-s	lbm/ft-s
Boric Acid, NaOH (pH 10)	2,497	6,190	100.0	212	3.04E-04	2.04E-04	1.70E-06	1.14E-06
Boric Acid, NaOH (pH 10)	25,067	63,452	65.6	150	8.26E-04	5.55E-04	1.55E-06	1.04E-06
Boric Acid, NaOH (pH 10)	49,946	126,006	100.0	212	1.21E-03	8.10E-04	2.71E-05	1.82E-05
Boric Acid, NaOH (pH 7)	2,512	955	65.6	150	4.53E-04	3.04E-04	4.55E-07	3.06E-07
Boric Acid, NaOH (pH 7)	2,512	955	100.0	212	3.01E-04	2.02E-04	1.73E-06	1.16E-06
Boric Acid, NaOH (pH 7)	25,015	9,050	65.6	150	5.57E-04	3.74E-04	7.10E-07	4.77E-07
Boric Acid, NaOH (pH 7)	50,119	17,977	100.0	212	5.11E-04	3.44E-04	3.37E-06	2.27E-06
Boric Acid, TSP	2,466	9,965	65.6	150	4.54E-04	3.05E-04	6.66E-07	4.47E-07
Boric Acid, TSP	2,510	10,035	100.0	212	3.12E-04	2.09E-04	2.92E-06	1.96E-06
Boric Acid, TSP	24,972	100,004	65.6	150	9.65E-04	6.48E-04	5.64E-06	3.79E-06
Boric Acid, TSP	49,966	199,816	100.0	212	2.08E-03	1.39E-03	2.66E-05	1.79E-05
Boric Acid, NaTB	2,508	12,070	65.6	150	4.62E-04	3.10E-04	1.83E-06	1.23E-06
Boric Acid, NaTB	2,508	12,070	100.0	212	3.02E-04	2.03E-04	1.30E-06	8.72E-07
Boric Acid, NaTB	25,002	120,071	65.6	150	7.25E-04	4.87E-04	6.50E-06	4.37E-06
Boric Acid, NaTB	50,034	240,283	100.0	212	1.04E-03	6.97E-04	5.37E-06	3.61E-06

Note:

1. The 1967 ASME steam tables [Reference 12] were utilized for water data. It is noted that water property values obtained from NIST are slightly different [Reference 10].

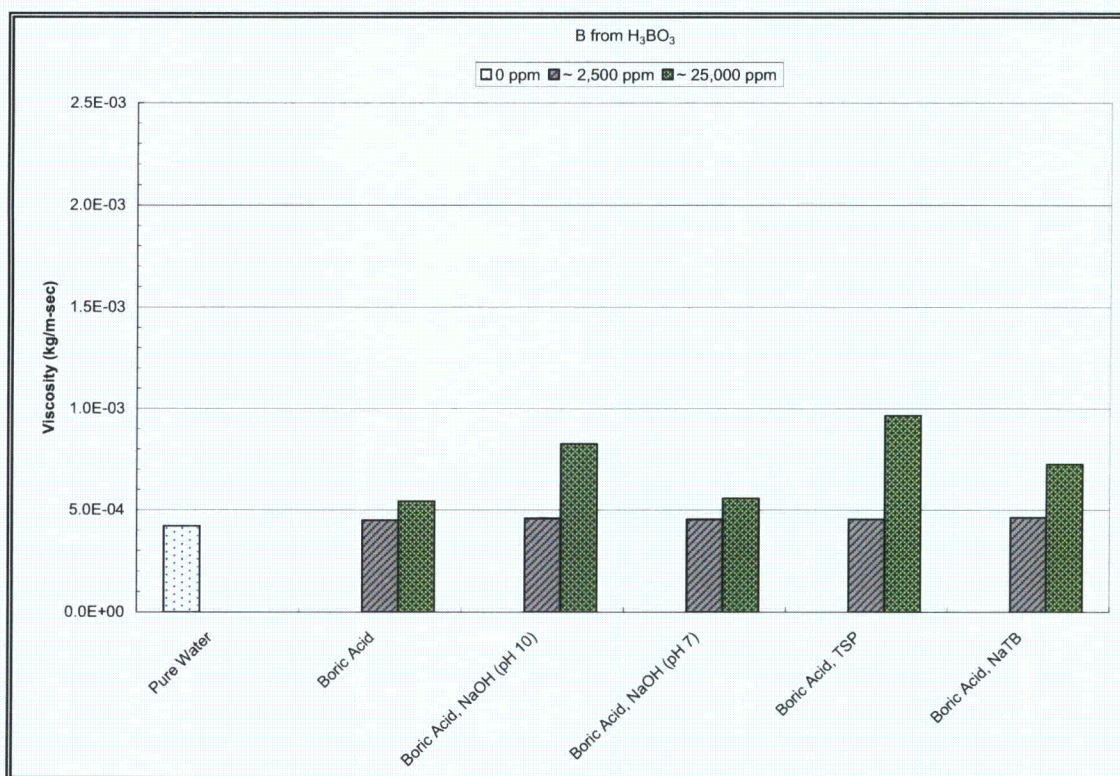


Figure 3-2 Viscosity of Buffered and Un-Buffered Boric Acid at 65.6°C

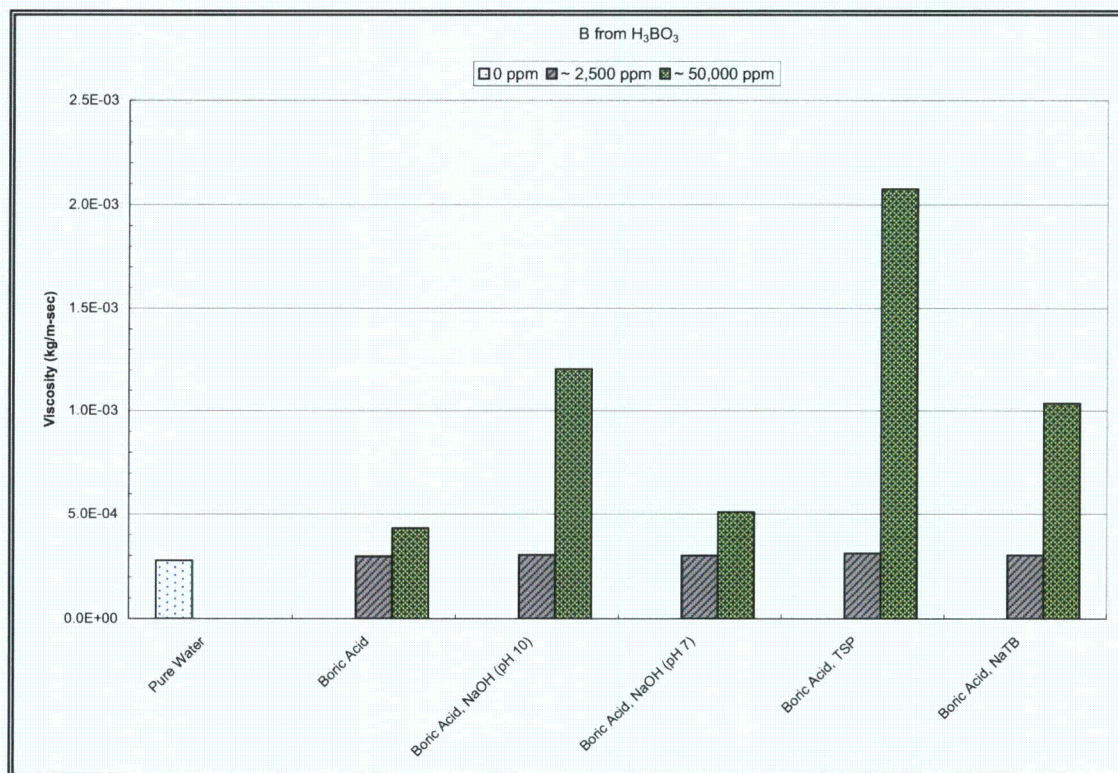


Figure 3-3 Viscosity of Buffered and Un-Buffered Boric Acid at 100.0°C

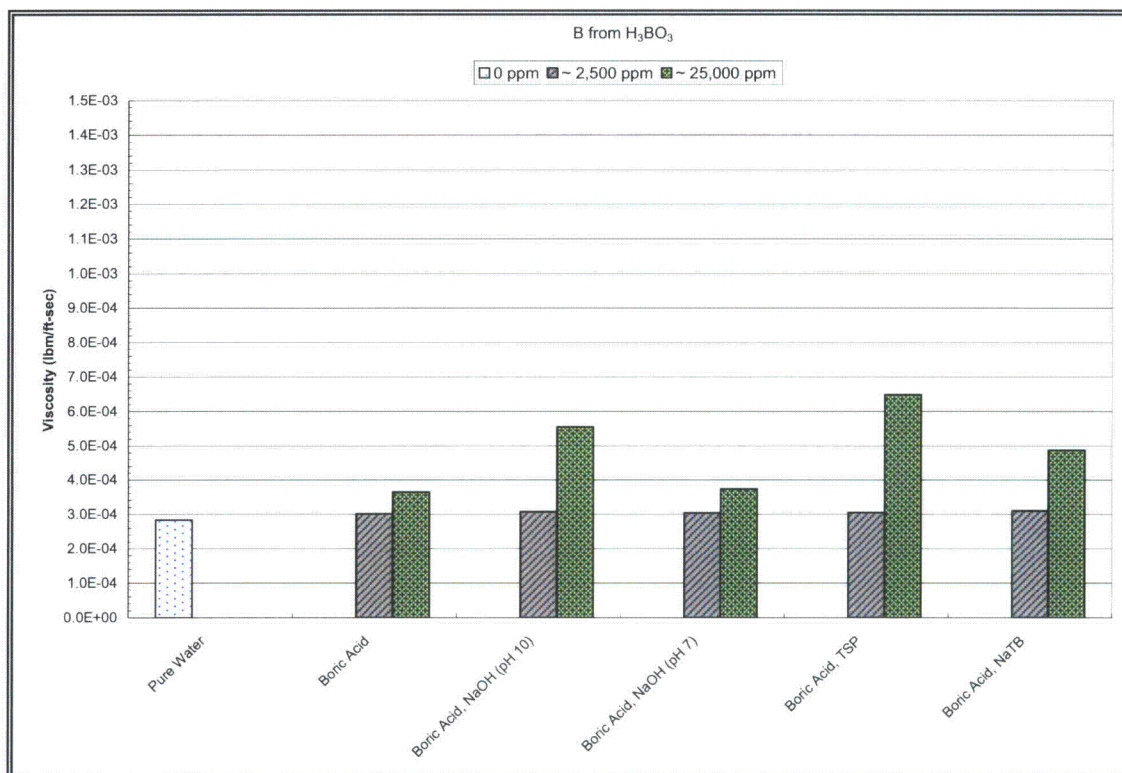


Figure 3-4 Viscosity of Buffered and Un-Buffered Boric Acid at 150°F

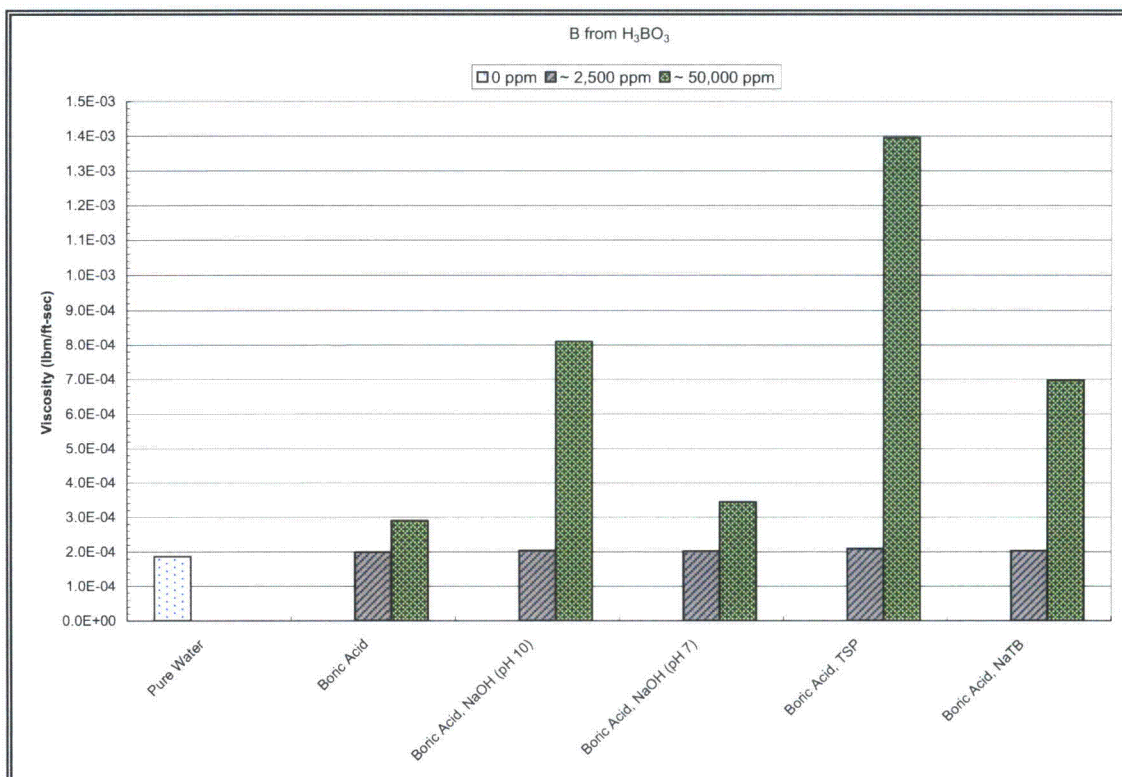


Figure 3-5 Viscosity of Buffered and Un-Buffered Boric Acid at 212°F

4 PHASE 3: SURFACE TENSION

During Phase 3, the surface tension of each boric acid solution was determined. To determine the surface tension, a Surface Electro Optics DST 60 tensiometer was used in conjunction with an ethylene glycol bath as seen in Figure 4-1.

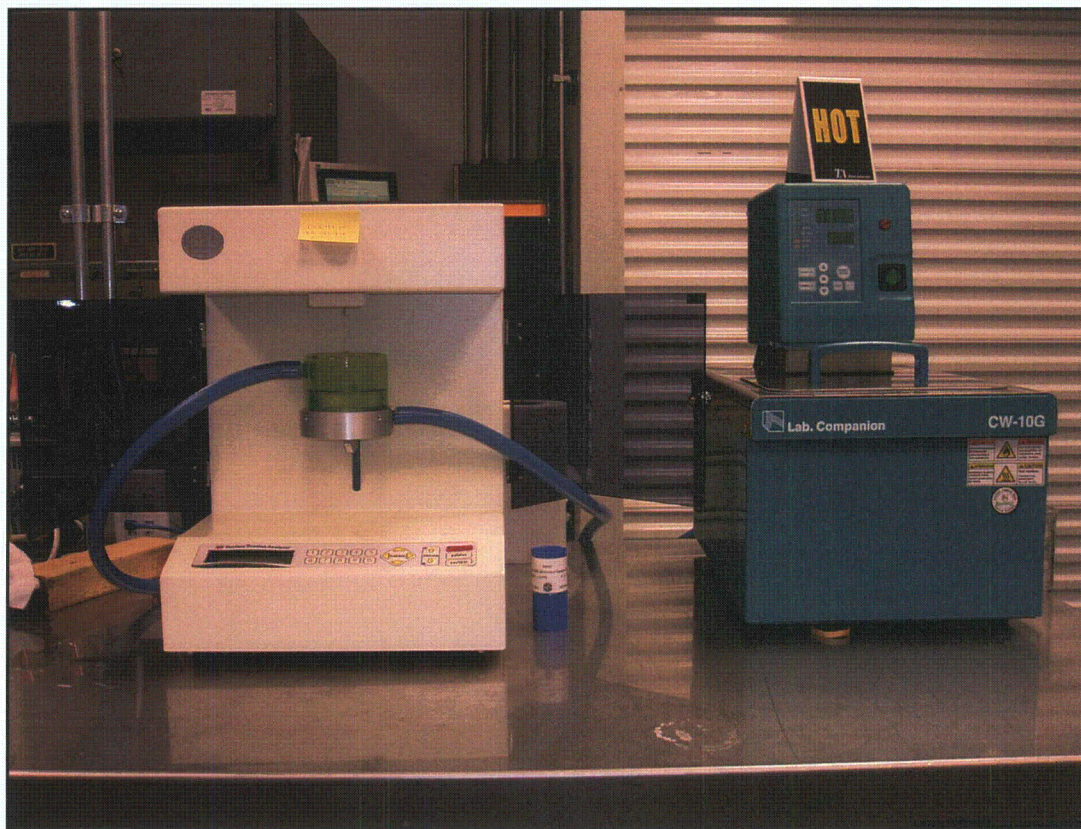


Figure 4-1 Experimental Setup for Surface Tension Measurement

The DST 60 employed the Du Nouy Ring method of surface tension measurement. The device used an internal balance to give an equivalent mass of surface force acting on the ring. Using the simple force balance given in Equation 4-1, the measured mass can be converted to a surface tension.

$$\sigma = \frac{m \cdot g}{4 \pi R} \quad (4-1)$$

where:

σ	=	surface tension
m	=	measured mass acting on ring
g	=	acceleration due to gravity
R	=	radius of Du Nouy Ring

The following steps for measuring surface tension with the DST 60 were used:

1. Solutions with the desired concentrations of boric acid and buffer were prepared as in Phase 1 of testing.
2. The ethylene glycol bath was brought up to a temperature slightly higher than the specified test temperature.
3. The sample was loaded into the jacketed cup and allowed to reach the specified test temperature.
4. The Du Nouy ring was lowered at least 5 mm below the surface of the sample and then was slowly withdrawn.
5. The maximum force exerted on the ring was recorded.
6. Using the measured mass value, the surface tension was calculated using the Equation 4-1.
7. Steps 4-6 were repeated five times for each solution tested.
8. The average surface tension along with a standard deviation of the measurements was calculated.

The results of Phase 3 testing are shown in Table 4-1. Table 4-1 is depicted graphically in Figure 4-2 through Figure 4-5. Detailed results are shown in Appendix B. Calibration data for surface tension measurement is provided in Appendix E.

Table 4-1 Results of Surface Tension Testing								
Coolant	Concentration of Boron from Boric Acid	Concentration of Buffer	Temperature		Surface Tension		Standard Deviation	
	(ppm)	(ppm)	°C	(°F)	kg/s²	(lbf/ft)	kg/s²	(lbf/ft)
Pure Water ⁽¹⁾	0	0	65.6	150	0.0651	0.0045	N/A	N/A
Pure Water ⁽²⁾	0	0	100.0	212	0.0589	0.0040	N/A	N/A
Boric Acid	2,490	0	65.6	150	0.0564	0.0039	3.43E-04	2.35E-05
Boric Acid	2,490	0	100.0	212	0.0494	0.0034	3.08E-04	2.11E-05
Boric Acid	23,968	0	65.6	150	0.0584	0.0040	2.32E-04	1.59E-05
Boric Acid	44,889	0	100.0	212	0.0511	0.0035	1.14E-03	7.81E-05
Boric Acid, NaOH (pH 10)	2,493	6,377	65.6	150	0.0549	0.0038	4.10E-04	2.81E-05
Boric Acid, NaOH (pH 10)	2,493	6,377	100.0	212	0.0463	0.0032	1.04E-04	7.13E-06

Table 4-1 Results of Surface Tension Testing (cont.)

Coolant	Concentration of Boron from Boric Acid	Concentration of Buffer	Temperature		Surface Tension		Standard Deviation	
	(ppm)	(ppm)	°C	(°F)	kg/s ²	(lbf/ft)	kg/s ²	(lbf/ft)
Boric Acid, NaOH (pH 10)	24,930	62,938	65.6	150	0.0511	0.0035	5.33E-04	3.65E-05
Boric Acid, NaOH (pH 10)	49,878	126,894	100.0	212	0.0373	0.0026	5.54E-04	3.80E-05
Boric Acid, NaOH (pH 7)	2,488	950	65.6	150	0.0644	0.0044	3.77E-04	2.58E-05
Boric Acid, NaOH (pH 7)	2,488	950	100.0	212	0.0477	0.0033	4.53E-04	3.10E-05
Boric Acid, NaOH (pH 7)	24,992	9,103	65.6	150	0.0634	0.0043	1.20E-03	8.22E-05
Boric Acid, NaOH (pH 7)	50,119	17,977	100.0	212	0.0489	0.0034	2.54E-04	1.74E-05
Boric Acid, TSP	2,496	9,978	65.6	150	0.0630	0.0043	5.04E-04	3.45E-05
Boric Acid, TSP	2,516	10,040	100.0	212	0.0508	0.0035	3.66E-04	2.51E-05
Boric Acid, TSP	25,028	99,660	65.6	150	0.0524	0.0036	4.08E-04	2.80E-05
Boric Acid, TSP	49,849	200,194	100.0	212	0.0453	0.0031	8.80E-04	6.03E-05
Boric Acid, NaTB	2,498	11,969	65.6	150	0.0581	0.0040	2.00E-04	1.37E-05
Boric Acid, NaTB	2,497	12,004	100.0	212	0.0544	0.0037	3.79E-04	2.60E-05
Boric Acid, NaTB	24,957	120,604	65.6	150	0.0462	0.0032	9.43E-04	6.46E-05
Boric Acid, NaTB	50,055	239,715	100.0	212	0.0481	0.0033	8.93E-04	6.12E-05

Notes:

1. The 1967 ASME steam tables [Reference 12] were utilized for water data. It is noted that water property values obtained from NIST are slightly different [Reference 10].
2. The 1979 ASME steam tables [Reference 13] were utilized for water data. It is noted that water property values obtained from NIST are slightly different [Reference 10].

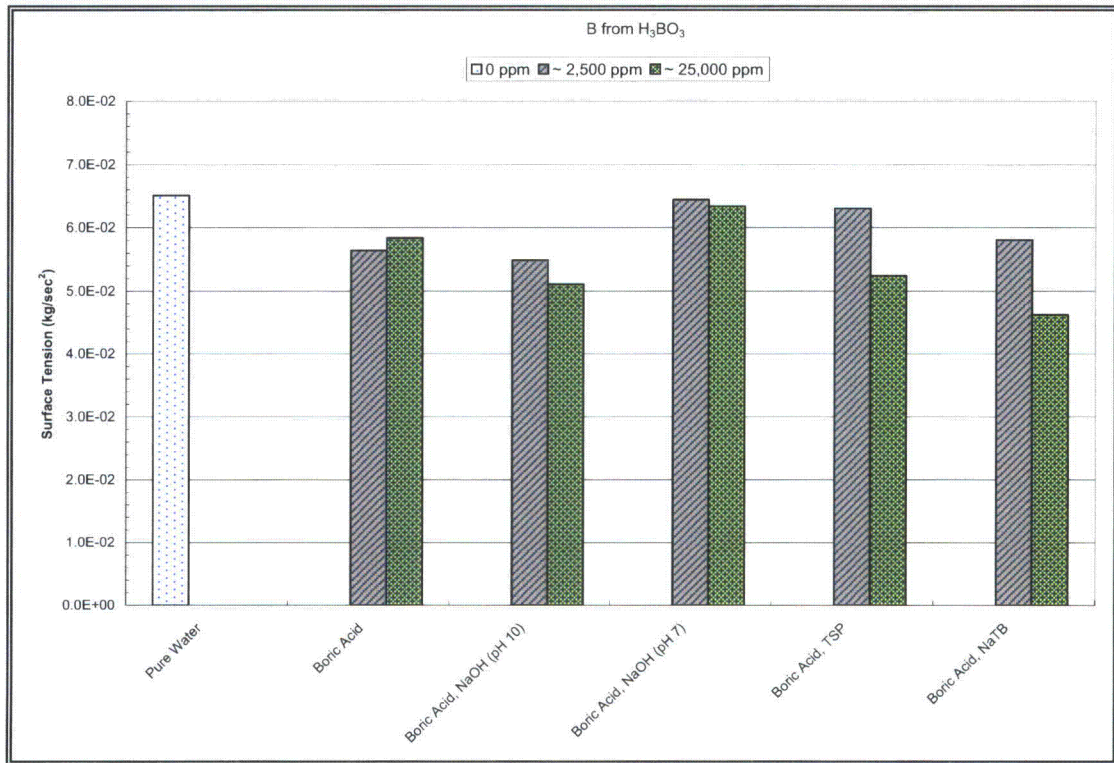


Figure 4-2 Surface Tension of Buffered and Un-Buffered Boric Acid at 65.6°C

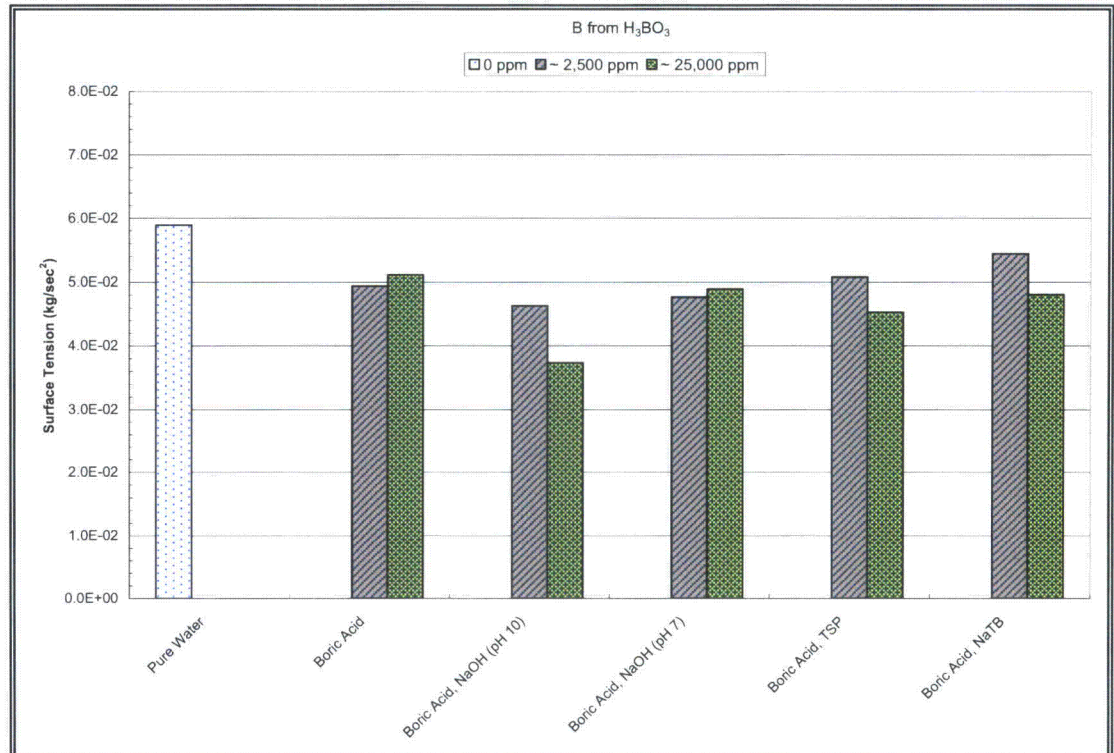


Figure 4-3 Surface Tension of Buffered and Un-Buffered Boric Acid at 100.0°C

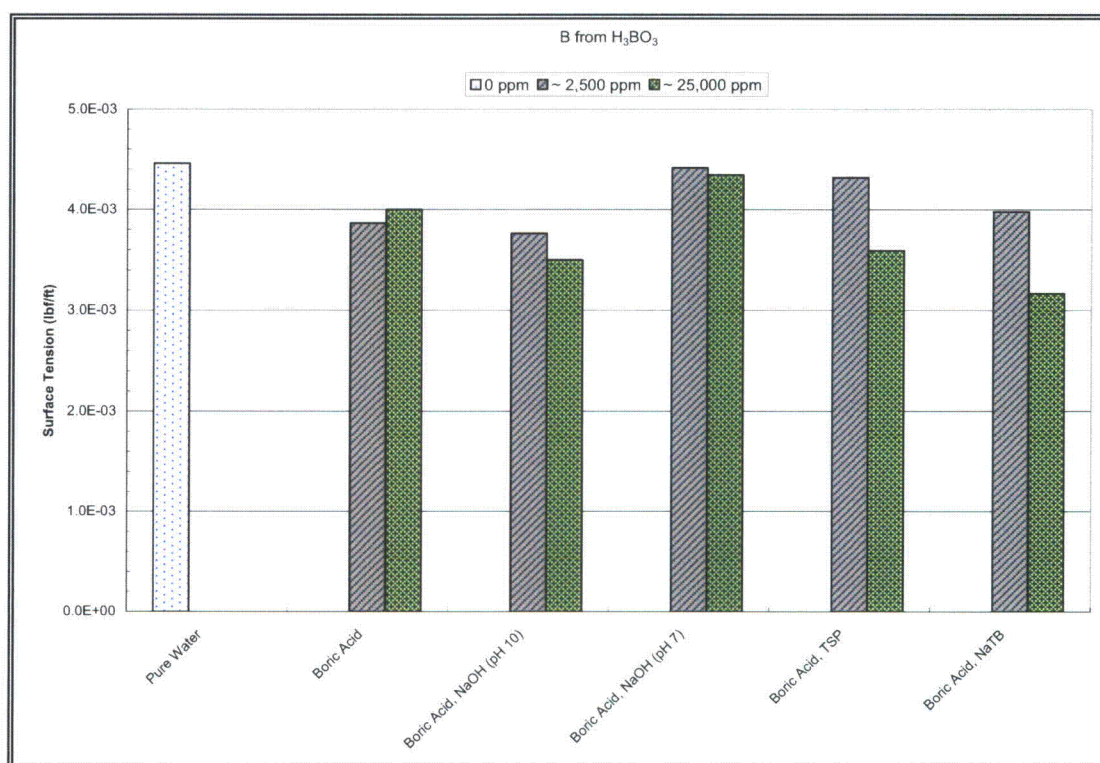


Figure 4-4 Surface Tension of Buffered and Un-Buffered Boric Acid at 150°F

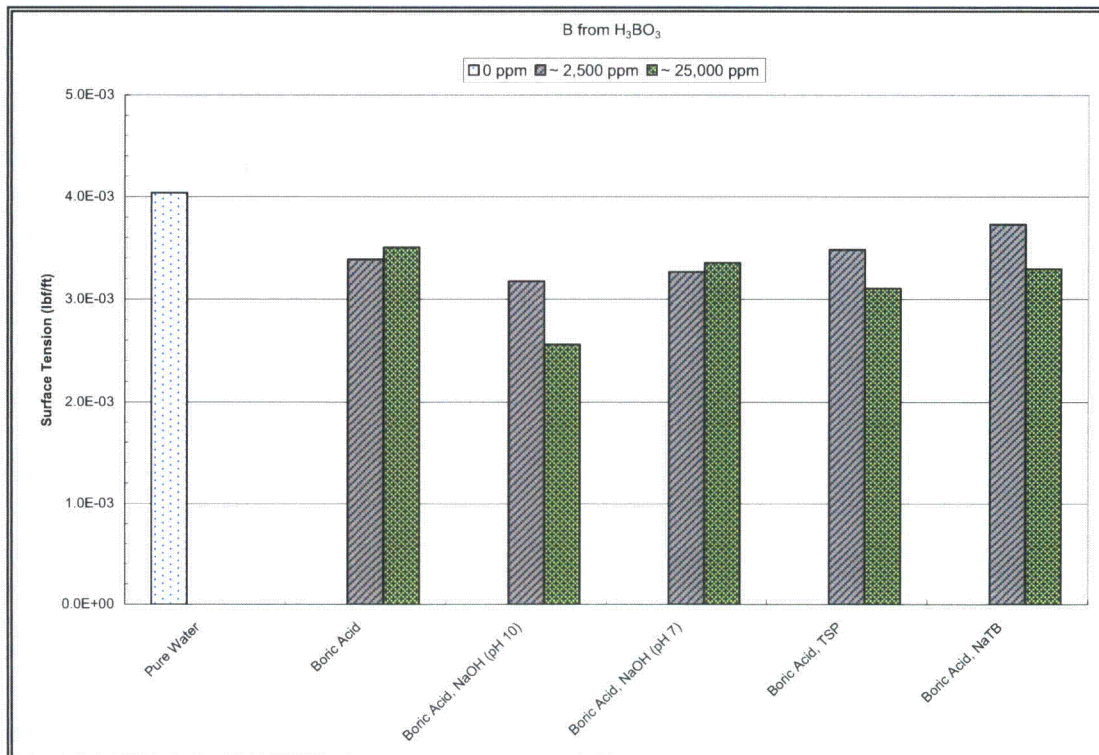


Figure 4-5 Surface Tension of Buffered and Un-Buffered Boric Acid at 212°F

5 PHASE 4: SOLUBILITY LIMITS

The solubility limits of ten different boric acid solutions were determined.

The term solubility ‘limit’ is defined as the concentration of solute at which precipitation is observed to occur and the solid and dissolved species are in equilibrium. It is possible to maintain soluble species in solution at levels above the solubility limit, and this state is referred to as ‘supersaturation.’ Solutions containing borates are prone to supersaturation, and the ability to form supersaturated solutions is controlled by the availability of nucleation sites and other factors such as stirring. Supersaturation was minimized in this testing by using a procedure where a solid was progressively added to the solution while looking at the dissolution behavior. This approach provided a large number of nucleation sites for growth or dissolution of crystalline phases.

A solubility limit was determined by starting at the concentrations from previous phases of testing. These initial concentrations were known to be soluble at the given temperatures. The following procedure was utilized:

1. Solutions with the desired concentrations of boric acid and buffer were prepared as in Phase 1 of testing.
2. The solution was heated (while being stirred) to the required test temperature and a pH reading was made using a pH meter.
3. Additional boric acid was added.
4. The pH was checked to ensure that the pH was at the specified level. If the pH had dropped below this level, additional buffer was added to raise the pH.
5. Steps 3-4 were repeated until a precipitate was formed.
6. The final boron and buffer concentrations were calculated based on the masses added.

The results of Phase 4 testing are shown in Table 5-1. Table 5-1 is depicted graphically in Figure 5-1. Detailed results are shown in Appendix C. Visual observation prior to precipitation suggested a relatively low viscosity.

Table 5-1 Boric Acid Precipitation Limits					
Coolant	Temperature		Boron Concentration		Buffer Concentration
	(°C)	(°F)	(ppm)	wt %	(ppm)
Boric Acid	65.6	150	25,301	14.5	0
Boric Acid	100.0	212	47,121	27.0	0
Boric Acid, NaOH (pH 10)	65.6	150	38,350	21.9	152,390
Boric Acid, NaOH (pH 10)	100.0	212	69,891	40.0	156,235
Boric Acid, NaOH (pH 7)	65.6	150	82,018	46.9	70,743
Boric Acid, NaOH (pH 7)	100.0	212	113,532	64.9	117,867
Boric Acid, TSP	65.6	150	57,585	32.9	363,843
Boric Acid, TSP	100.0	212	67,753	38.8	514,012
Boric Acid, NaTB	65.6	150	47,874	27.4	400,056
Boric Acid, NaTB	100.0	212	51,552	29.5	545,649

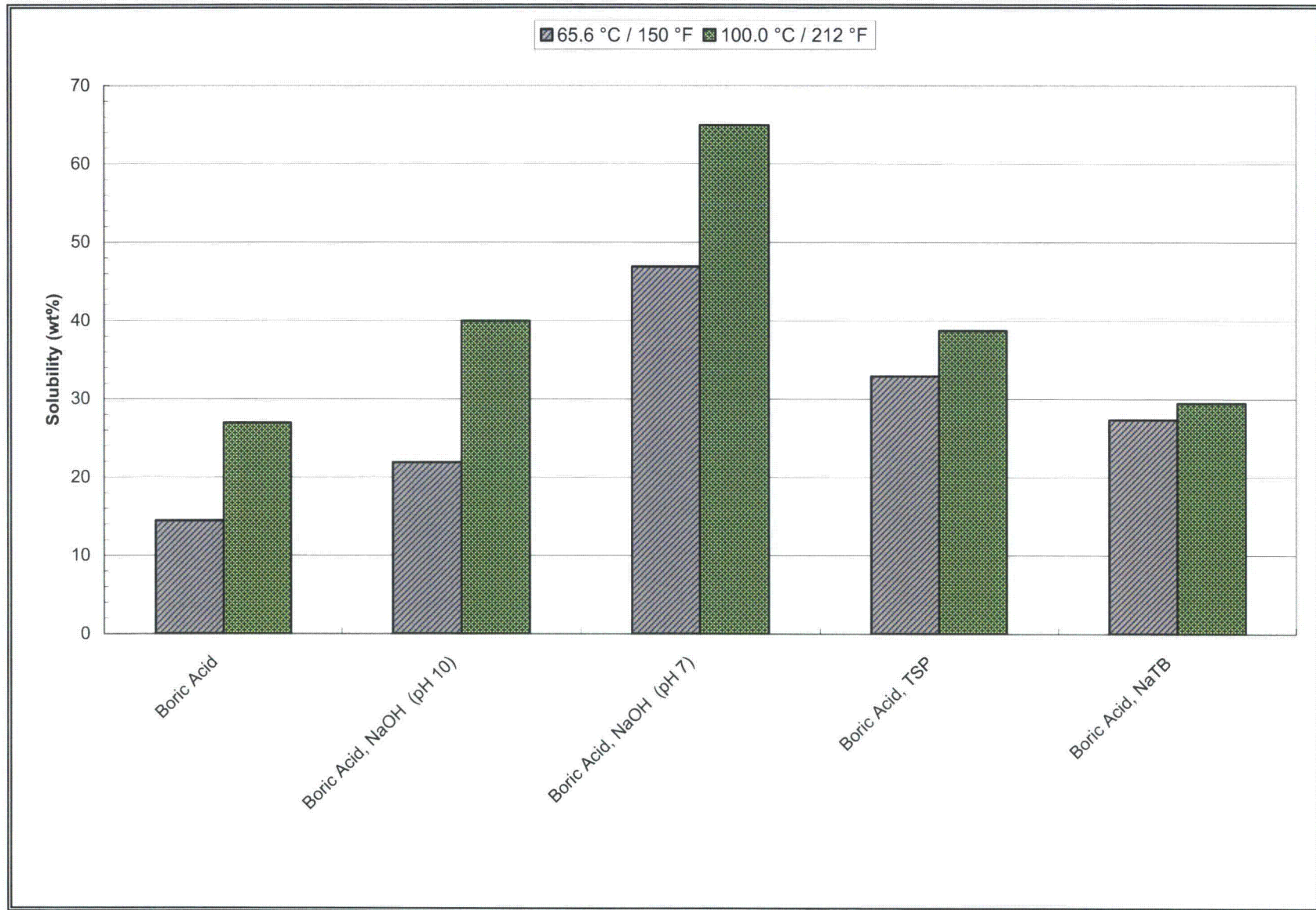


Figure 5-1 Boric Acid Precipitation Limit as a Function of Temperature

6 CONCLUSIONS

Overall, the density measurements were reasonable and were consistent with expectations. The dilute boric acid solutions had densities slightly greater than water. Density increased with increasing concentration, and decreased with increasing temperature.

An important aspect of the data is that the densities of the concentrated pH buffered solutions at 100.0°C/212°F were higher than the corresponding dilute solutions at 65.6°C/150°F. This implies that after a cold leg break LOCA, a driving force for core mixing would develop before precipitation within the core took place (unless post-LOCA core uncover occurs), even if the lower plenum was as cold as 65.6°C/150°F. In some cases, densities of the concentrated buffers were quite high. For instance, the most concentrated boric acid/trisodium phosphate solution had a density that was more than 30 percent higher than the corresponding dilute solution. Such high densities would provide a powerful force for circulation of the core volume with the lower plenum after a cold leg break, which would in turn provide a mechanism for core dilution. This mixing would provide a larger margin to precipitation than would otherwise be predicted if lower plenum mixing was ignored. However, since the solubilities of the buffer solutions tested in this work were high, the density of the core coolant itself could be a limiting factor in core cooling. It is also worth noting that since the height of the coolant in the downcomer is fixed, increases in core coolant density would cause a lowering of the liquid mixture level in the inner vessel since the downcomer and inner vessel are in hydrostatic balance.

There was little difference in viscosity between the different solutions at the 2500 ppm boron concentration level. This was true both for 65.6°C/150°F and 100.0°C/212°F.

At higher concentrations, considerable increases in viscosity were noted and there were large differences between the different chemical systems. Viscosity increased in the order Boric Acid < Boric Acid-NaOH pH 7 < Boric Acid – Sodium Tetraborate < Boric Acid-NaOH pH 10 < Boric Acid – TSP.

While the concentrated solutions showed viscosity increases, no highly viscous solutions that could inhibit free flow and mixing in the core were noted.

The surface tension values for all of the solutions were lower than that of pure water at the same temperature. That was true, regardless of the presence or absence of a pH control agent. Increasing the solute concentration caused, on average, a decrease in surface tension. Decreases in surface tension promote boiling nucleation, so with regard to the surface tension parameter, the presence of boric acid and buffered boric acid would be expected to improve nucleate boiling heat transfer within the core compared to pure water.

The amount of evaporative concentration that can be tolerated before precipitation was greater for all of the buffered solutions than for boric acid alone. In the case of sodium hydroxide added to boric acid producing an initial pH near 7.7 at temperature, the maximum concentration increased more than a factor of 2. This indicates that there is a large amount of conservatism using solubility for un-buffered boric acid solutions when it is justified to use the solubility for buffered boric acid solutions. Applications of solubility data to reactor vessel precipitation should consider precipitation mode and mixing/transport phenomena identified and discussed in WCAP-17047-NP, "PIRTs for Un-Buffered/Buffered Boric Acid Mixing/Transport and Precipitation in Reactor Vessel During Post-LOCA Conditions," [Reference 14].

It should be acknowledged that in the reactor post-LOCA environment, supersaturation could occur, and the amount of supersaturation possible will be dependent on the suspended debris, the system surfaces, and the rate of system cooling. The solubility limits determined in this testing were measured using a procedure which should minimize supersaturation, so from this respect, they should represent conservative limits. However, chemical reactions between the simulated coolants and coolant impurities were not considered in this work, and these interactions could alter solubility limits in either direction.

7 REFERENCES

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APPENDIX A

DETAILED RESULTS OF DENSITY AND VISCOSITY TESTING

pH Buffer	Temp [°C]	Preparation of Solution					Density Measurement					Viscosity Measurement					
		Mass of H ₃ BO ₃ [g]	Mass of Buffer [g]	Mass of Water [g]	Total Solution Mass [g]	Conc. of Boron [ppm]	Conc. of Buffer [ppm]	pH	Mass of 100 ml of Solution [g]	Density [kg/m ³]	Visc. Type	Viscometer Constant [cSt/s]	Efflux Time [s]	Kinematic Viscosity [cSt]	Dynamic Viscosity [kg/m·s]	Average Dynamic Viscosity [kg/m·s]	Standard Deviation [kg/m·s]
None	65.6	4.3240	0.0000	295.6732	299.9972	2520	0	4.88	98.1448	981.4	0	0.001093	416.53	0.4553	4.4682E-04	4.4898E-04	1.5056E-06
													417.62	0.4565	4.4799E-04		
													419.28	0.4583	4.4977E-04		
													419.60	0.4586	4.5011E-04		
													419.69	0.4587	4.5021E-04		
None	100.0	4.3240	0.0000	295.6732	299.9972	2520	0	4.74	96.3520	963.5	0	0.001093	281.41	0.3076	2.9636E-04	2.9745E-04	1.5816E-06
													281.34	0.3075	2.9629E-04		
													284.44	0.3109	2.9955E-04		
													281.35	0.3075	2.9630E-04		
													283.69	0.3101	2.9876E-04		
None	65.6	21.4617	0.0000	128.5548	150.0165	25012	0	2.38	101.9380	1019.4	0	0.001093	488.87	0.5343	5.4469E-04	5.4219E-04	2.2052E-06
													488.44	0.5339	5.4421E-04		
													484.81	0.5299	5.4017E-04		
													486.38	0.5316	5.4192E-04		
													484.63	0.5297	5.3997E-04		
None	100.0	38.6100	0.0000	111.3913	150.0013	45002	0	2.13	104.2394	1042.4	0	0.001093	374.84	0.4097	4.2707E-04	4.3149E-04	2.9277E-06
													378.09	0.4133	4.3077E-04		
													381.19	0.4166	4.3430E-04		
													378.58	0.4138	4.3133E-04		
													380.92	0.4163	4.3400E-04		
NaOH	65.6	4.2842	1.8570	293.8313	299.9724	2497	6190	9.68	98.3385	983.4	0	0.001093	425.46	0.4650	4.5730E-04	4.5826E-04	4.1937E-06
													430.75	0.4708	4.6299E-04		
													427.09	0.4668	4.5905E-04		
													420.28	0.4594	4.5173E-04		
													428.18	0.4680	4.6022E-04		
NaOH	100.0	4.2842	1.8570	293.8313	299.9724	2497	6190	9.22	96.2201	962.2	0	0.001093	290.06	0.3170	3.0505E-04	3.0378E-04	1.6988E-06
													289.66	0.3166	3.0463E-04		
													290.34	0.3173	3.0535E-04		
													287.05	0.3137	3.0189E-04		
													287.16	0.3139	3.0200E-04		
NaOH	65.6	21.5100	9.5195	118.9966	150.0261	25067	63452	9.64	109.8085	1098.1	OC	0.002861	262.40	0.7507	8.2436E-04	8.2593E-04	1.5504E-06
													263.31	0.7533	8.2722E-04		
													263.35	0.7534	8.2735E-04		
													263.10	0.7527	8.2656E-04		
													262.34	0.7506	8.2417E-04		
NaOH	100.0	42.8519	18.9010	88.2476	150.0004	49946	126006	9.66	123.7469	1237.5	OC	0.002861	329.31	0.9422	1.1659E-03	1.2059E-03	2.7124E-05
													350.41	1.0025	1.2406E-03		
													339.81	0.9722	1.2031E-03		
													343.72	0.9834	1.2169E-03		
													339.75	0.9720	1.2029E-03		

pH Buffer	Temp [°C]	Preparation of Solution				Density Measurement				Viscosity Measurement							Standard Deviation [kg/m·s]
		Mass of H ₃ BO ₃ [g]	Mass of Buffer [g]	Mass of Water [g]	Total Solution Mass [g]	Conc. of Boron [ppm]	Conc. of Buffer [ppm]	pH	Mass of 100 ml of Solution [g]	Density [kg/m ³]	Visc. Type	Viscometer Constant [cSt/s]	Efflux Time [s]	Kinematic Viscosity [cSt]	Dynamic Viscosity [kg/m·s]	Average Dynamic Viscosity [kg/m·s]	
NaOH	65.6	4.3112	0.2866	295.4378	300.0356	2512	955	7.73	98.2337	982.3	0	0.001093	421.47	0.4607	4.5253E-04	4.5285E-04	4.5524E-07
													421.25	0.4604	4.5229E-04		
													421.94	0.4612	4.5303E-04		
													421.84	0.4611	4.5293E-04		
													422.34	0.4616	4.5346E-04		
NaOH	100.0	4.3112	0.2866	295.4378	300.0356	2512	955	7.62	96.4434	964.4	0	0.001093	287.66	0.3144	3.0323E-04	3.0124E-04	1.7262E-06
													284.54	0.3110	2.9994E-04		
													283.71	0.3101	2.9907E-04		
													286.93	0.3136	3.0246E-04		
													286.00	0.3126	3.0148E-04		
NaOH	65.6	21.4650	1.3576	127.1978	150.0204	25015	9050	5.73	102.6797	1026.8	0	0.001093	496.44	0.5426	5.5715E-04	5.5662E-04	7.1013E-07
													495.82	0.5419	5.5645E-04		
													494.97	0.5410	5.5550E-04		
													496.56	0.5427	5.5728E-04		
													496.07	0.5422	5.5673E-04		
NaOH	100.0	42.9999	2.6966	104.3027	149.9992	50119	17977	4.28	107.2102	1072.1	0	0.001093	432.62	0.4729	5.0695E-04	5.1133E-04	3.3727E-06
													436.07	0.4766	5.1099E-04		
													437.72	0.4784	5.1292E-04		
													435.09	0.4756	5.0984E-04		
													440.31	0.4813	5.1596E-04		
TSP	65.6	2.1203	1.4979	146.6943	150.3125	2466	9965	8.46	98.8165	988.2	0	0.001100	418.28	0.4601	4.5466E-04	4.5418E-04	6.6566E-07
													416.91	0.4586	4.5317E-04		
													417.87	0.4597	4.5422E-04		
													417.63	0.4594	4.5396E-04		
													418.47	0.4603	4.5487E-04		
TSP	100.0	2.1531	1.5053	146.3458	150.0042	2510	10035	8.26	97.5950	976.0	0	0.001093	289.28	0.3162	3.0858E-04	3.1154E-04	2.9235E-06
													296.13	0.3237	3.1589E-04		
													292.19	0.3194	3.1168E-04		
													289.81	0.3168	3.0914E-04		
													292.88	0.3201	3.1242E-04		
TSP	65.6	21.4252	15.0007	113.5750	150.0009	24972	100004	7.75	112.8252	1128.3	0C	0.002861	297.81	0.8520	9.6131E-04	9.6469E-04	5.6425E-06
													298.47	0.8539	9.6344E-04		
													298.28	0.8534	9.6283E-04		
													297.79	0.8520	9.6124E-04		
													301.94	0.8639	9.7464E-04		
TSP	100.0	42.8677	29.9721	77.1584	149.9982	49966	199816	7.39	130.4372	1304.4	0C	0.002861	544.16	1.5568	2.0307E-03	2.0756E-03	2.6610E-05
													555.56	1.5895	2.0732E-03		
													561.12	1.6054	2.0940E-03		
													558.72	1.5985	2.0850E-03		
													561.44	1.6063	2.0952E-03		

pH Buffer	Temp	Preparation of Solution						Density Measurement			Viscosity Measurement						
		Mass of H ₃ BO ₃	Mass of Buffer	Mass of Water	Total Solution Mass	Conc. of Boron	Conc. of Buffer	pH	Mass of 100 ml of Solution	Density	Visc. Type	Viscometer Constant	Efflux Time	Kinematic Viscosity	Dynamic Viscosity	Average Dynamic Viscosity	Standard Deviation
		[g]	[g]	[g]	[g]	[ppm]	[ppm]		[g]	[kg/m ³]		[cSt/s]	[s]	[cSt]	[kg/m·s]	[kg/m·s]	[kg/m·s]
NaTB	65.6	4.3028	3.6211	292.0813	300.0052	2508	12070	8.03	98.5115	985.1	0	0.001093	427.72	0.4675	4.6054E-04	4.6179E-04	1.8325E-06
													428.44	0.4683	4.6131E-04		
													427.82	0.4676	4.6065E-04		
													431.85	0.4720	4.6499E-04		
													428.56	0.4684	4.6144E-04		
NaTB	100.0	4.3028	3.6211	292.0813	300.0052	2508	12070	7.92	96.7684	967.7	0	0.001093	284.81	0.3113	3.0124E-04	3.0226E-04	1.2973E-06
													285.62	0.3122	3.0209E-04		
													284.91	0.3114	3.0134E-04		
													287.85	0.3146	3.0445E-04		
													285.68	0.3122	3.0216E-04		
NaTB	65.6	21.4502	18.0102	110.5354	149.9958	25002	120071	6.58	106.9284	1069.3	OC	0.002861	238.41	0.6821	7.2935E-04	7.2518E-04	6.5047E-06
													240.13	0.6870	7.3461E-04		
													235.41	0.6735	7.2017E-04		
													235.88	0.6749	7.2161E-04		
													235.41	0.6735	7.2017E-04		
NaTB	100.0	42.9280	36.0430	71.0314	150.0024	50034	240283	5.21	118.9766	1189.8	OC	0.002861	304.00	0.8697	1.0348E-03	1.0366E-03	5.3692E-06
													302.18	0.8645	1.0286E-03		
													304.84	0.8721	1.0377E-03		
													305.25	0.8733	1.0390E-03		
													306.41	0.8766	1.0430E-03		

APPENDIX B

DETAILED RESULTS OF SURFACE TENSION TESTING

pH Buffer	Temp	Preparation of Solution						Measurement of Surface Tension				
		Mass of H ₃ BO ₃	Mass of Buffer	Mass of Water	Total Solution Mass	Conc. of Boron	Conc. of Buffer	Equiv. Surface Tension Mass	Surface Tension	Surface Tension	Average Surface Tension	Standard Deviation
	[°C]	[g]	[g]	[g]	[g]	[ppm]	[ppm]	[g]	[dyne/cm]	[kg/s ²]	[kg/s ²]	[kg/s ²]
None	65.6	2.8492	0.0000	197.2098	200.0590	2490	0	0.6879	56.34	0.05634	0.05642	3.4261E-04
								0.6839	56.01	0.05601		
								0.6869	56.26	0.05626		
								0.6909	56.58	0.05658		
								0.6949	56.91	0.05691		
None	100.0	2.8492	0.0000	197.2098	200.0590	2490	0	0.5969	48.89	0.04889	0.04942	3.0842E-04
								0.6049	49.54	0.04954		
								0.6059	49.62	0.04962		
								0.6058	49.61	0.04961		
								0.6039	49.46	0.04946		
None	65.6	27.4267	0.0000	172.6380	200.0647	23968	0	0.7104	58.18	0.05818	0.05838	2.3189E-04
								0.7099	58.14	0.05814		
								0.7138	58.46	0.05846		
								0.7131	58.40	0.05840		
								0.7169	58.71	0.05871		
None	100.0	51.3522	0.0000	148.6542	200.0064	44889	0	0.6387	52.31	0.05231	0.05114	1.1437E-03
								0.6298	51.58	0.05158		
								0.6058	49.61	0.04961		
								0.6139	50.28	0.05028		
								0.6339	51.92	0.05192		
NaOH	65.6	2.8512	1.2753	195.8681	199.9946	2493	6377	0.6692	54.81	0.05481	0.05492	4.1013E-04
								0.6789	55.60	0.05560		
								0.6709	54.95	0.05495		
								0.6659	54.54	0.05454		
								0.6679	54.70	0.05470		
NaOH	100.0	2.8512	1.2753	195.8681	199.9946	2493	6377	0.5647	46.25	0.04625	0.04634	1.0385E-04
								0.5659	46.35	0.04635		
								0.5659	46.35	0.04635		
								0.5679	46.51	0.04651		
								0.5649	46.26	0.04626		
NaOH	65.6	28.5656	12.6085	159.1593	200.3334	24930	62938	0.6326	51.81	0.05181	0.05110	5.3270E-04
								0.6278	51.42	0.05142		
								0.6232	51.04	0.05104		
								0.6204	50.81	0.05081		
								0.6158	50.43	0.05043		
NaOH	100.0	57.0992	25.3975	117.6507	200.1474	49878	126894	0.4546	37.23	0.03723	0.03734	5.5411E-04
								0.4678	38.31	0.03831		
								0.4530	37.10	0.03710		
								0.4527	37.08	0.03708		
								0.4513	36.96	0.03696		

pH Buffer	Temp [°C]	Preparation of Solution					Measurement of Surface Tension					
		Mass of H ₃ BO ₃ [g]	Mass of Buffer [g]	Mass of Water [g]	Total Solution Mass [g]	Conc. of Boron [ppm]	Conc. of Buffer [ppm]	Equiv. Surface Tension Mass [g]	Surface Tension [dyne/cm]	Surface Tension [kg/s ²]	Average Surface Tension [kg/s ²]	Standard Deviation [kg/s ²]
NaOH	65.6	2.8458	0.1900	196.9548	199.9905	2488	950	0.7869	64.45	0.06445	0.06442	3.7689E-04
								0.7879	64.53	0.06453		
								0.7919	64.86	0.06486		
								0.7869	64.45	0.06445		
								0.7792	63.82	0.06382		
NaOH	100.0	2.8458	0.1900	196.9548	199.9905	2488	950	0.5891	48.25	0.04825	0.04766	4.5331E-04
								0.5859	47.98	0.04798		
								0.5759	47.17	0.04717		
								0.5814	47.62	0.04762		
								0.5776	47.30	0.04730		
NaOH	65.6	28.5892	1.8207	169.5913	200.0012	24992	9103	0.7612	62.34	0.06234	0.06340	1.1973E-03
								0.7563	61.94	0.06194		
								0.7799	63.87	0.06387		
								0.7899	64.69	0.06469		
								0.7835	64.17	0.06417		
NaOH	100.0	57.3332	3.5954	139.0703	199.9989	50119	17977	0.5948	48.71	0.04871	0.04894	2.5358E-04
								0.6009	49.21	0.04921		
								0.5949	48.72	0.04872		
								0.5964	48.84	0.04884		
								0.6009	49.21	0.04921		
TSP	65.6	2.8550	1.9955	195.1360	199.9865	2496	9978	0.7624	62.44	0.06244	0.06303	5.0397E-04
								0.7656	62.70	0.06270		
								0.7769	63.63	0.06363		
								0.7749	63.46	0.06346		
								0.7681	62.91	0.06291		
TSP	100.0	2.8779	2.0080	195.1044	199.9903	2516	10040	0.6189	50.69	0.05069	0.05080	3.6551E-04
								0.6269	51.34	0.05134		
								0.6148	50.35	0.05035		
								0.6189	50.69	0.05069		
								0.6217	50.92	0.05092		
TSP	65.6	28.6334	19.9341	151.4532	200.0208	25028	99660	0.6339	51.92	0.05192	0.05244	4.0824E-04
								0.6447	52.80	0.05280		
								0.6389	52.32	0.05232		
								0.6459	52.90	0.05290		
								0.6379	52.24	0.05224		
TSP	100.0	57.0380	40.0483	102.9612	200.0475	49849	200194	0.5369	43.97	0.04397	0.04531	8.8035E-04
								0.5489	44.95	0.04495		
								0.5559	45.53	0.04553		
								0.5607	45.92	0.04592		
								0.5639	46.18	0.04618		

Preparation of Solution								Measurement of Surface Tension				
pH Buffer	Temp	Mass of H ₃ BO ₃	Mass of Buffer	Mass of Water	Total Solution Mass	Conc. of Boron	Conc. of Buffer	Equiv. Surface Tension Mass	Surface Tension	Surface Tension	Average Surface Tension	Standard Deviation
	[°C]	[g]	[g]	[g]	[g]	[ppm]	[ppm]	[g]	[dyne/cm]	[kg/s ²]	[kg/s ²]	[kg/s ²]
NaTB	65.6	2.8580	2.3940	194.7640	200.0160	2498	11969	0.7119	58.30	0.05830	0.05806	2.0027E-04
								0.7085	58.02	0.05802		
								0.7109	58.22	0.05822		
								0.7063	57.84	0.05784		
								0.7069	57.89	0.05789		
NaTB	100.0	2.8563	2.4007	194.7434	200.0004	2497	12004	0.6600	54.05	0.05405	0.05443	3.7892E-04
								0.6659	54.54	0.05454		
								0.6720	55.04	0.05504		
								0.6627	54.27	0.05427		
								0.6625	54.26	0.05426		
NaTB	65.6	28.5486	24.1202	147.3258	199.9946	24957	120604	0.5489	44.95	0.04495	0.04623	9.4339E-04
								0.5569	45.61	0.04561		
								0.5669	46.43	0.04643		
								0.5737	46.99	0.04699		
								0.5763	47.20	0.04720		
NaTB	100.0	57.2640	47.9464	94.8040	200.0144	50055	239715	0.5879	48.15	0.04815	0.04807	8.9269E-04
								0.5739	47.00	0.04700		
								0.5787	47.39	0.04739		
								0.5938	48.63	0.04863		
								0.6007	49.20	0.04920		

APPENDIX C

DETAILED RESULTS OF SOLUBILITY TESTING

pH Buffer	Temp	Preparation of Solution					Solubility Determination						
		Initial Mass of H ₃ BO ₃	Initial Mass of Buffer	Initial Mass of Water	Initial Solution Mass	Initial Conc. of Boron	Initial Conc. of Buffer	Additional H ₃ BO ₃ Added	Additional Buffer Added	Final Solution Mass	Final Conc. of Boron	Final Conc. of Buffer	Final pH
		[g]	[g]	[g]	[g]	[ppm]	[ppm]	[g]	[g]	[g]	[ppm]	[ppm]	[-]
None	65.6	21.47	0.00	128.37	149.84	25051	0	0.25	0.00	150.09	25301	0	-
None	100.0	34.37	0.00	116.03	150.40	39954	0	8.44	0.00	158.84	47121	0	-
NaOH	65.6	5.19	2.10	22.71	30.00	30246	70000	4.31	4.50	43.31	38350	152390	10.30
NaOH	100.0	10.09	3.73	15.50	29.32	60166	127217	6.26	2.66	40.90	69891	156235	10.05
NaOH	65.6	21.61	1.32	127.04	149.97	25193	8802	129.85	21.52	322.86	82018	70743	6.91
NaOH	100.0	5.74	0.38	13.91	20.03	50102	18972	70.73	13.50	117.76	113532	117867	6.98
TSP	65.6	4.29	3.02	22.96	30.27	24778	99769	20.36	24.21	74.84	57585	363843	7.48
TSP	100.0	8.61	6.03	15.74	30.38	49550	198486	53.34	76.14	159.86	67753	514012	7.45
NaTB	65.6	4.34	3.07	23.20	30.61	24789	100294	15.14	25.39	71.14	47874	400056	6.90
NaTB	100.0	8.58	7.28	14.15	30.01	49986	242586	17.58	41.13	88.72	51552	545649	7.06

APPENDIX D

CALIBRATION DATA FOR VISCOSITY MEASUREMENT

Sample	Temperature [°C]	Trial	Viscometer Type	Viscometer Constant [cSt/s]	Efflux Time [s]	Kinematic Viscosity [cSt]	Average Kinematic Viscosity [cSt]	Standard Deviation [cSt]	Literature Kinematic Viscosity ⁽¹⁾ [cSt]	Error [%]
Water	23.3	1	OC	0.002861	320.28	0.9163	0.9406	1.6940E-02	0.9521	1.22
		2			328.87	0.9409				
		3			334.22	0.9562				
		4			334.38	0.9567				
		5			326.13	0.9331				
Water	44.5	1	OC	0.002861	214.06	0.6124	0.6140	5.3737E-03	0.6253	1.85
		2			217.56	0.6224				
		3			215.16	0.6156				
		4			212.72	0.6086				
		5			213.5	0.6108				

⁽¹⁾ - Literature data obtained from Design Institute for Physical Property Data

APPENDIX E

CALIBRATION DATA FOR SURFACE TENSION MEASUREMENT

Substance	Temperature	Trial	Equivalent Surface Tension Mass	Surface Tension	Average Surface Tension	Literature Surface Tension ⁽¹⁾	Error
	[°C]		[g]	[dyne/cm]	[dyne/cm]	[dyne/cm]	[%]
Water	23.0	1	0.8989	73.62	73.60	73.22	0.528
		2	0.8989	73.62			
		3	0.8990	73.63			
		4	0.8999	73.70			
		5	0.8969	73.45			
Water	91.0	1	0.7067	57.88	57.46	59.96	-4.162
		2	0.7117	58.29			
		3	0.7079	57.98			
		4	0.7079	57.98			
		5	0.6739	55.19			
Glycerol	23.0	1	0.8202	67.17	67.36	63.18	6.610
		2	0.8220	67.32			
		3	0.8252	67.58			
		4	0.8229	67.39			
		5	0.8220	67.32			
Glycerol	91.0	1	0.7399	60.60	59.52	57.85	2.875
		2	0.7189	58.88			
		3	0.7219	59.12			
		4	0.7249	59.37			
		5	0.7279	59.61			
Pentadecane	23.0	1	0.3469	28.41	27.97	26.89	4.002
		2	0.3389	27.76			
		3	0.3389	27.76			
		4	0.3489	28.57			
		5	0.3339	27.35			
Pentadecane	93.0	1	0.2739	22.43	22.41	20.84	7.568
		2	0.2719	22.27			
		3	0.2740	22.44			
		4	0.2749	22.51			
		5	0.2737	22.42			

(1) - Literature data obtained from Design Institute for Physical Property Data