QUANTIFICATION OF BORIC ACID CONCENTRATION AND LOSSES DUE

TO VAPORIZATION IN THE PASTA FACILITY

A Thesis

by

MASON PAUL CHILDS

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Chair of Committee,	Yassin Hassan
Committee Members,	Maria King
	Pavel Tsvetkov
	Rodolfo Vaghetto
Head of Department,	Yassin Hassan

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ABSTRACT

During a loss of coolant accident in a pressurized water reactor, borated water is injected into the core through emergency core cooling system to reduce the decay heat, remove excess reactivity, and maintain an adequate core cooling throughout the full range of accident phases. Concentration of boric acid in the core is expected to increase over time due to the continuous vaporization of water from the core. Under certain conditions, the concentration may reach the solubility limit and precipitation of boric acid may occur. Hot leg switchover is a manual emergency procedure involving simultaneous injection through both the hot and cold legs to ensure adequate core flushing and prevent or mitigate precipitation of solid boric acid in the core.

The nuclear research community, industry, and regulatory commission are currently collaborating to understand the possible effects of the precipitation of boric acid on adequate core cooling during the long–term phase of a loss of coolant scenario, particularly in understanding whether modifications of the current procedures are required. An experimental apparatus was constructed to conduct experiments with de–ionized water and boric acid, to observe and study the flow behavior, and to measure the boric acid concentration in boiling water environments in geometry similar to a pressurized water reactor. Three methods for quantifying boric acid content at very high concentrations, even exceeding saturation, were explored. A gravimetric method for concentration determination proved to be the most effective for high values as were observed in the test facility. Concentration of solution in the test section was approximately solved using an analytic approach for comparison to experimentally determined values.

During experimentation, concentrations of boric acid were found to increase lin-

early with time. Rapid boiling in the test section induced uniform mixing in the test section, causing no appreciable difference in the concentration trends between various solution injection locations and other operational parameters of the facility. The rate of increase of boric acid concentration in the solution was observed to be proportional to the applied power to the heating rods in the facility test section. Comparison of boric acid concentration with the analytical solution confirmed that a fraction of the boric acid was transported within the vapor phase outside the test facility. Fractional boric acid loss from the test section was found to be lower than estimated from the observed losses during the calibration procedure.

The constructed facility is used in the study of effects of the precipitate on coolant flow conditions related to cooling capabilities during the long-term cooling phase in a loss of coolant accident. Acquiring this understanding could help the resolution of General Safety Issue 191 as imposed by the United States Nuclear Regulatory Commission.

NOMENCLATURE

10 CFR	Title 10, Code of Federal Regulations
A_{test}	total heat transfer surface area in facility
BAP	boric acid precipitation
C_0	facility initial concentration
C_a	concentration of added solution
C_s	solubility limit of boric acid in water at 100°C
CFD	computational fluid dynamics
D_{core}	inner diameter of typical PWR core
DI	de-ionized
D_{rod}	diameter of heating rods installed in facility
D_{test}	inner diameter of facility test section
EC	electrical conductivity
ECCS	emergency core cooling system
E_l	loss of boric acid due to evaporation
GSI	generic safety issue
H_{core}	height of typical PWR core
HPSI	high pressure safety injection
HLSO	hot leg switchover
H_{test}	height of facility test section
LBLOCA	large-break loss of coolant accident
LOCA	loss of coolant accident
LPSI	low pressure safety injection
M_B	mass of boric acid

$\dot{m}_{in}$	mass injection rate
$\dot{m}_{out}$	mass loss rate
PASTA	precipitation and stratification test apparatus
P_{core}	typical reactor power at time of SSO for a PWR
P_d^{ref}	typical reactor power density at time of SSO for a PWR
P_{test}^{inst}	installed electric power in facility
P_{test}^{ref}	reference power for facility
PWR	pressurized water reactor
Q_{in}	experimental inlet volumetric flow rate
Re	Reynolds number
RPV	reactor pressure vessel
RWST	refueling water storage tank
Ri	Richardson number
SBLOCA	small-break loss of coolant accident
SSO	sump switchover
SG	steam generator
$q^{"\,ref}$	typical reactor heat flux during SSO for a PWR
USNRC	United States Nuclear Regulatory Commission
V_{test}	facility test section free volume

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1. INTRODUCTION

Water circulates as the coolant and moderator in the primary loop of a pressurized water reactor (PWR). Boron is added to the water in the form of dissolved boric acid in order to control reactivity in the core during normal operation; boron also provides the required negative reactivity to maintain a subcritical core configuration during routine maintenance including refueling. In the event of a loss of coolant accident (LOCA), the emergency core cooling system (ECCS) in a PWR is designed to provide sufficient coolant throughput to the core to remove the decay heat, while the voiding of the core provides sufficient reactivity for shutdown. In the first stages of a LOCA, the ECCS injects cold water through the cold legs of the reactor pressure vessel (RPV). In these first stages, the refueling water storage tank (RWST) serves as the source reservoir for ECCS. In certain circumstances, additional borated water may be injected from the cold leg accumulators in order to aid cooldown of the core via the low pressure safety injection (LPSI) or high pressure safety injection (HPSI). Excess water injected into the core overflows through the break, and is collected in the containment sump. Following the depletion of RWST, or at sump switchover (SSO), ECCS begins to draw water from the containment sump and recirculate it back into the core. This stage of a LOCA can last up to several days, in which water is continually recirculated from the sump until a cold shutdown condition can be reached.

During this long term cool down phase, if enough water is evaporated, the coolant may reach a saturated concentration causing boric acid to precipitate out of solution[1]. As a method to prevent buildup of boron precipitates in the core, simultaneous injection through the cold and hot leg is initiated as a manual action by the

operators at a time determined by plant specific analysis. This manual operation, hot leg switchover (HLSO), is an important operational maneuver in the facilitation of long term cooling of the core during LOCA events. Understanding the local behavior of the borated water under boiling conditions and the possible effects of the presence of precipitated boric acid in the solution is necessary for verifying the manual operation procedures currently in place for LOCA scenarios of different break sizes and locations. Of particular interest would be scenarios where precipitation of boric acid occurs, to study the effects of the precipitate on the water cooling capabilities and the overall core coolability during the long-term cooling phase. Acquiring this understanding could help the resolution of generic safety issue (GSI) 191[2], where the combined effect of debris accumulation and boric acid precipitation can affect core coolability.

1.1   Literature Review

With Title 10, Code of Federal Regulations (10 CFR) 50.46, the United States Nuclear Regulatory Commission (USNRC) has mandated operational criteria for ECCS during a variety of LOCA events in which could potentially cause chemical precipitation along with debris to be accumulated on sump screen, negatively affecting ECCS functionality. Resolution of GSI 191 should sufficiently demonstrate that transport and accumulation of debris in containment following a LOCA will not impede ECCS during operation in PWRs [2], and also that the core is maintained at a sufficiently low temperature and power level during long term cooling stages following a LOCA. Since the inception of GSI 191, numerous studies have been conducted on various aspects of the debris and chemical precipitation accumulation in PWR containment.

Buoyancy effects of dilution gradients of boric acid were studied by Cotton *et al.*[3]

to determine loop concentrations following small-break LOCA (SBLOCA) events. With 3D modeling in CFX-5, they were able to determine a strong correlation between the Richardson number (Ri) and the course of the outcome of a concentration transient as in a SBLOCA. Even in low Ri regimes, similar to those seen in natural circulation scenarios, mixing of the boron solution during transient was sufficient to consider concentration stratification of the borated water minimal. In addition, an independence of boron concentration stratification from Reynolds number (Re) was seen for all calculations completed. Also studying the mixing of borated solutions during transient scenarios, da Silva *et al.*[4] observed the effects of buoyancy on the mixibility of two solutions. In PWR boron dilution transients, when borated water is injected through the ECCS, the large differences in density between the injected water and the coolant already contained within the core, due to both temperature gradients and boron concentration, could pose a problem for the safety injection system capability. In their experimental facility, a simple vertical loop equipped with an advanced conductivity probe which provides high spatial and temporal detail, mixing scenarios with water and a glucose solution were compared to results generated from CFX-11 computational modeling software. Their analysis showed good agreement between the experimental results and mixing phenomena seen in the computational fluid dynamics (CFD) simulations, and again highlighted the correlation between Ri and overall effectiveness of mixing. Their analysis, however, seems not to explore extreme concentrations of solution in which densities can be vastly different, and precipitate could impede the mixing flow between analytes.

Recent analysis of the ECCS during LOCA scenarios, especially those of the long term cooling phase as done by Lee *et al.*[5], has made use of the limit that *no* precipitation occurs in the core during the end stages of a LOCA event. In their RELAP5 analysis, the simultaneous injection through both cold and hot legs

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in HLSO, along with containment spray to maintain cooling was seen to be sufficient additive to prevent the high concentrations of boric acid in which precipitation would occur[6]. This was seen to be the case even with a time-variant mixing volume as would be seen in the decreasing in-core coolant level after the beginning stages of a LOCA, as a faster observed increase of boric acid concentration leads only to an earlier initiation of HLSO which provides sufficient core flushing.

Bucalossi *et al.*[7] performed experimental analysis in order to better understand in-vessel conditions during boron dilution transients such as a LOCA event. Their experimental results were also used as a bnechmark for the validation of various numerical studies in regards to boron dilution transients. Their experimental facility consisted of a four loop scaled model of the primary side of a PWR with a simplified structure to simulate core geometry. The facility was used in the observation of two types of experiments:

1. pump start-up scenarios;

2. steady operation of one to four loops

in which tracer slugs of salinated water mixing with tap water was used to measure the mixing of coolant from loops through the RPV via electrical conductivity (EC) measurements. With their facility they were able to see the inverse of flow of loops which had pumps at rest during the startup of the test loop, as well as the propagation of the a concentration gradient through the RPV post injection. They were able to conclude that, with minimal changes in density from the circulating water and the tracer slug, the overall mixing of the two solutions occurred relatively fast, and that perturbations of the concentration of either solutions were small. This was true for all flow rates they studied, with any number of loops isolated from the tested loop.

Of significant concern during the potential precipitation of boric acid is the plate–out of boric acid on the fuel rod cladding surface[8], which could compromise core cooling and lead to fuel damage. With the PKL facility, Umminger *et al.* have sought out to determine the amount of plate–out which may occur at saturation conditions, and its effect on the heat transfer and flow characteristics of coolant during the long term cooling phase. They also study any integral effects including loop pressure drop, increase of pressure drop though the steam generator (SG) due to boric acid accumulation and combined effect on the water level in the core. The PKL facility is a scaled 1:1 elevation facility with diameters reduced by a factor of 12 (volume and power scaling factor 1:145). It is equipped with EC probes to measure boric acid concentration at points located at the core inlet, the core outlet, and the reflector gap, as well as any in the four loops as detailed in [9, 10, 11, 12]. In their most recent analysis with the PKL facility, Umminger *et al.* concluded that even with the loss of a full loop during large-break LOCA (LBLOCA) scenarios, the mixing volume for the coolant and injected borated water expands into the remaining loops' SG, causing minimal plate–out in the RPV and also the SG tubes; overall core cooling is not compromised enough to prevent a decrease of the core power to 1%, low enough to allow for a HLSO and initiate core flushing. Due to the size of the PKL facility, however, it is assumed that the researchers may not want to reach the saturation limit of the boric acid in water and that precipitation of boric acid in the core may be difficult to handle and time consuming. The research conducted at PKL and described in references [9, 10, 11, 12] seems to be limited to concentrations of boric acid in the core lower than the saturation.

Another facility constructed to perform experiments on the in–vessel mixing phenomena, as well as validation benchmarking for CFD simulations has been well documented. The Rossendorf Coolant Mixing Model (ROCOM) facility has been used

alongside the PKL to study characteristics of in–vessel mixing and upper plenum flow[13, 14]. Kliem *et al.* further confirmed the overall mixing quality in the RPV as well as SG loops. Höhne *et al.*[15] also used the ROCOM facility to exhibit the strong dependence on buoyancy in propagating mixing behavior throughout the RPV.

Lastly, Tuunanen *et al.* have presented a large body of work highlighting results of an experimental activity conducted on the REWET-II, VEERA, and modified VEERA facilities[1, 16]. While these facilities include different regions of the vessel and SG, similarities can be found in the two sets of results. A comparison can be summarized as follows:

1. Concentration of boric acid is proven to be uniform in the core. The uniformity is allowed by the mixing due to boiling in the core simulator. The reference states that mixing seems to be more effecting in larger bundle due to the trend observed between REWET–II (19 rods) and VEERA (126 rods). This uniformity due to mixing is confirmed by concentration measurements.

2. Crystallization occurs at certain time during the experiments. While it is unclear how the crystallization was confirmed and observed in the REWET–II and VEERA facilities, it is suspected that this was done by concentration measurements combined by post-test observations of the core simulator.

3. Blockages due to crystallization were possible, and in fact occurred, in the REWET–II facility, and seemed more likely to occur in smaller bundles (due to ineffective mixing) and at the top of the core. Blockage seems to be affected by several parameters and in particular the volume in the core. In the reference it is mentioned that if the liquid level is maintained above the core, no blockage is observed since crystallization takes place outside the core.

Table 1.1: Main features of the experimental facilities used in boron dilution transient testing. Not included in this table is the planar sensors in the VeMix and ROCOM facilities for EC measurements.

Facility and included references	Heating capability	Visualization capability
1:5 Scaled four-loop[7]	None	None
VeMix[4]	None	Full visualization
PKL[8, 9, 10, 12]	1:145 scaled (approx. 650 kW)	None
ROCOM[13, 14, 15]	None	Full visualization
REWET–II[1, 16]	19 heating rods (approx. 10 kW)	Post experiment
VEERA[1, 16]	126 heating rods (approx. 60 kW)	Post experiment

For all the thermal-hydraulic responses of PWR systems to LOCA conditions which have been studied, few of the experimental apparatus provide a visual confirmation of the data recorded with EC measurements to determine boric acid concentration. Further, none of the facilities which provide visualization also include a heating structure to impart thermal energy to the test fluid. An opportunity exists to explore the visual appearance of flow, as well as any plate–out of boric acid, during the extended boiling as seen during the long term cool down phase. Full visualization of a precipitate particle distribution would aid in the understanding of plate–out characteristics. The transition and two–phase flow which would exist within the core during this phase should be accurately captured with full visualization wherever possible. This additional heating which takes place in the core may define the plate–out phenomenon, or alter mixing flow during the HLSO phase, when injected water is counter current with the escaping steam. As such, a facility was

originally designed to study the combined effect of the boron precipitation with a hypothetical core blockage at the bottom of the core which prevents any mixing of the solution in the core with other RPV regions, including the lower plenum.

2. THE PASTA FACILITY

The precipitation and stratification test apparatus (PASTA) facility was designed to provide novel observations on the characteristics of boiling flow for concentrated solutions of boric acid in de-ionized (DI) water. The facility was created to reproduce key geometry from that of a typical PWR core. While rigorous scaling was not implemented in the design, the facility can provide qualitative visualization of solutions in various boiling scenarios and precipitation effects at concentrations beyond saturation. Figure 2.1 shows the initial construction of the facility prior to shakedown testing.

As shown, the PASTA facility was constructed of a 6" diameter polycarbonate pipe with 0.25" polycarbonate flanges welded at either end to form a test section. 45 heating rods internal of the test section are held in place with stainless steel plate flanges with holes drilled to allow the ends of each heating rod to protrude slightly into the upper plenum as seen in Figure 2.2. Following initial shakedown tests of the PASTA facility, four of the shown corner rods were removed to allow for test section sampling, as well as temperature measurement instrumentation to be added.

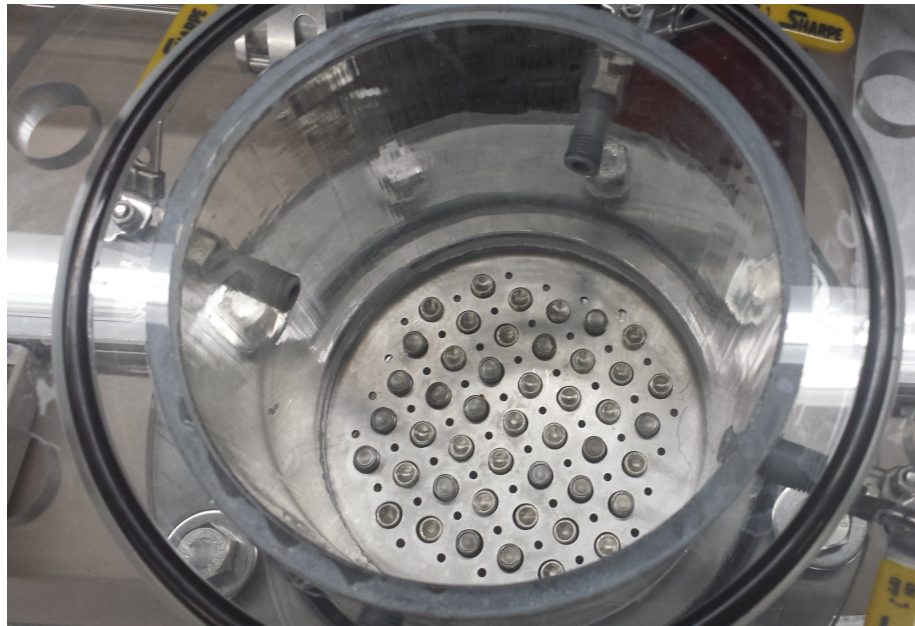Figure 2.1: The fully assembled PASTA facility test section.

Figure 2.2: Ends of the heating rods held in place with the top plate flange.

The smaller vent holes in the top plate flange are included to allow the flow of liquid water into, and steam out of, the test section during experimentation. At the bottom of the test section, the heating rods are fastened into a 0.25" stainless steel bottom plate flange with 45 threaded holes as shown in Figure 2.3. Two corner holes are left void of heating rods in order to allow for instrumentation to be inserted into the test section. In each of these positions, a k–type thermocouple was inserted to measure the temperature of the test section fluid near the periphery of the heating assembly bundle. The other two empty corner holes are fitted with valved ports which serve as injection points, or sample collection ports (Figure 2.4) depending on the experiment at hand. These flange plates at each end of the test section hold the heating rods parallel during experiments, without interfering with the mixing volume. The bottom flange plate also served as a seal for the lower end of the test section. Drawings of the top and bottom plate flanges used for production of the the PASTA facility can be found in Appendix A.1.

At the top of the test section, an extension piece of 6" polycarbonate pipe with welded flange was fitted to serve as an upper plenum. The upper plenum pipe was fitted with four valved ports as seen in Figure 2.5, which primarily served as the injection points for the borated solution during experimentation. Although the injection lines were not scaled, injection from the top of the facility, as through the upper plenum ports is representative of injection through the top of a PWR core, as in HLSO. This design was conceived to observe core characteristics during a full core blockage at the bottom of the core; no lower plenum mixing is simulated with the PASTA facility.

Also visible in Figure 2.5 is the exhaust port flange, which is fixed to the top of the upper plenum pipe section. A threaded elbow is connected with an 1" insulative hose to vent the exhausted steam away from the facility without affecting pressure

Figure 2.3: Bottom plate flange with shown threaded holes to secure heating rods at bottom of test section.

in the test section.

As shown in Figure 2.3, each heating element was wired separately, which were each connected to an individual power supply. As such, these heating elements are all able to be controlled independently. Each power supply was equipped with a gauge to monitor its power output. This allows the facility to be used with various power profiles, both radially and azimuthally to test the effect of different radial power distributions.

Figure 2.4: Photo of the valved ports at the bottommost flange of the PASTA facility test section.

2.1 Scaling and Facility Dimensions

Although rigorous scaling laws were not followed in the assembly of the PASTA facility, dimensions were defined with proportions calculated to represent PWR core geometries as close as necessary for qualitative analysis. The test section enclosure dimensions were driven by the core aspect ratio for a typical PWR, defined as D_{core}/H_{core}. For a typical PWR this was assumed to equal 0.78. Based on availability of polycarbonate pipes and flanges, as well as the total electric power to be installed, a test section with an inner diameter of 15.24 cm (6" standard) was selected. The height of the test section was then selected in order to preserve the aspect ratio as close as possible to a typical PWR:

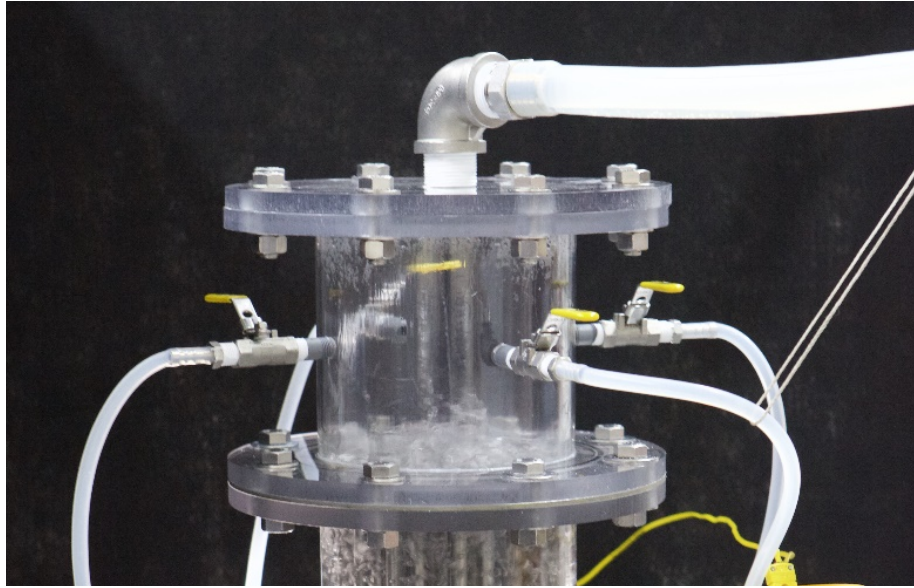$$H_{test} = D_{test} \frac{H_{core}}{D_{core}} \tag{2.1}$$

Figure 2.5: Photo of the upper plenum of the PASTA facility. The four injection ports near the top, and exhaust line from the topmost flange can be seen.

Using the polycarbonate pipe available, Eq. 2.1 fixes the facility height to $H_{test} = 19.38$ cm. The electrical heating rods were selected to be as close as possible to the diameter seen in a typical PWR. Based on manufacturing standards available, rods of diameter $D_{rod} = 0.95$ cm were selected. To preserve the rod bundle pitch to diameter ratio, 1.33 for a typical PWR, the center–to–center distance for the test section heating rods was determined as 1.26 cm. Due to the size and shape of the installed rods' 1.27 cm threaded heads(Figure 2.3), however, the pitch to diameter ratio of the test section necessarily differed from the reference value. A ratio of 1.75 was selected as closest to the reference while still allowing for the installation of the rods into the bottom plate flange. The selected pitch to diameter ratio resulted in a center–to–center distance of 1.66 cm for the heating rods. The total heat transfer surface area of the rods is $A_{test} = 0.24$ m^2. The enclosure and heating rod bundle defined the facility free volume, which the coolant–like solution will occupy. This

facility free volume was calculated as $V_{test} = 2.97$ l.

As described in [17], the total power to be installed in the experimental facility was estimated based on the assumption that observed reactor power in the PASTA facility is comparable to scaled decay power at the time of SSO in a typical PWR. For a LBLOCA, the SSO can be assumed to be approximately 30 minutes after the event of the break. Reactor power at this time is estimated as $P_{core} = 75$ MW. Two scaling parameters were then considered:

1. reactor power density P_d^{ref} (W/m^3), defined as the ratio of reactor power to the total core free volume (volume occupied by the coolant)

2. heat flux $q^{"ref}$ (W/m^2), defined as the ratio of reactor power to the total core heat transfer surface area

The reactor reference values used for this evaluation were $P_d^{ref}=3.7$ MW/m^3 and $q^{"ref} = 11.5$ kW/m^2, both estimated assuming a reactor power equal to decay power described above. This permitted a scaled total facility power to be calculated using the reference values as

$$P_{test}^{ref} = \max \left(q^{"ref} A_{test}, P_d^{ref} V_{test} \right). \tag{2.2}$$

Using Eq. 2.2 the facility reference power, P_{test}^{ref}, is calculated as 11 kW. With 41 rods in the test section the individual rod power is fixed to a minimum of 270 W. Based on electrically heated rods which were readily available, rods of 500 W nominal power were selected. With selected rods, the total maximum power installed in the facility was $P_{test}^{inst} = 20.5$ kW$_e$, allowing for a wide range of experimental power densities to be supported by the PASTA facility. The dimensional features of the PASTA facility are summarized in Table 2.1. Additional description and photos of

the facility can be found in [17, 18].

Table 2.1: Main features of the PASTA experimental facility.

Test Section Parameter	Value	Unit
Inner Diameter	15.24	cm
Height	19.38	cm
Diameter to Height Ratio	0.785	-
Free Volume	2.97	l
Number of Heating Rods	41	-
Heating Rod Diameter	0.95	cm
Heating Rod Pitch to Diameter Ratio	1.75	-
Total Power Installed	20.5	kW_e

Due to the loose scaling methodology used for the construction of the PASTA facility, a number of characteristics necessarily varied from the reference PWR value. For example, the pitch to diameter ratio was altered to allow for the physical construction of the bottom plate flange to allow for the heating rods to pass through the threaded holes and fit side by side. This small distortion may cause differences in the accumulation of boric acid between the heating elements as compared to a PWR. Also, due to the small size of the facility test section, spacer grids for the heating rods, similar to those seen in PWR geometry, were not included. Similarly, the power levels attainable in the facility are not exactly equal to power densities as might be seen approximately during SSO in an LBLOCA. This difference in installed power allows for flexibility in experimentation; varying power levels as well

as different power profiles can be studied. Additional descriptions and photos of the PASTA facility can be found in [17, 18].

2.2 Operation of PASTA Facility

Each test done with the PASTA facility was conducted according to written procedures which were defined and improved during shakedown of said facility. Operation of the PASTA facility is centered around two functions: the evaporative heating of solution in the test section, and the continual injection of a solution of borated DI water to replenish the evaporated solution. Heating of the solution in the test section is accomplished with the 41 inserted 0.25" heating rods which protrude through the bottom plate flange and extend the length of the test section. Addition of solution to the facility is accomplished with injection via a MityFlex 913 metering pump which is located under the facility as can be seen in Figure 2.6. Suction of the pump is submerged in a reservoir of solution of boric acid, which is heated during experimentation to maintain an approximately constant temperature. The pump injects water into the facility through the valved ports described above. With the valves and a small manifold, the pump can inject water through any combination of the four upper plenum and two lower plate flange injection points.
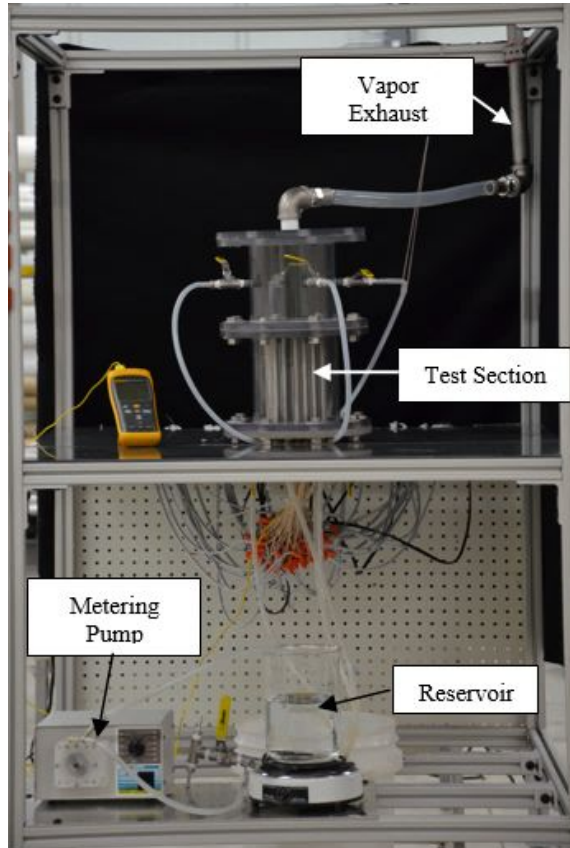
Figure 2.6: Fully assembled PASTA facility used for experimentation. The metering pump and reservoir can be seen below the test section.

2.2.1 Test Section Measurement and Sampling

Measurements within the PASTA facility are necessary to correlate the observed flow characteristics to the boric acid concentration. The facility provides a challenge for measurement, however, due to the high concentration of solution in the test section during experimentation. At these high concentrations, boric acid molecules are in a constant flux between dissolved and precipitated states. EC probe measurements within the facility, as used in other facilities[3, 9, 11, 14], are impossible because of the impingement of these particles with proposed instrumentation causing interfer-

ence. An alternative method for determining concentration of the solution within the facility was therefore developed. Description of the solution analysis method is contained in the following sections.

The PASTA facility was constructed with 4 valved ports which open to the top of the test section, Figure 2.5. These ports are used for the injection of addition solution which maintains a constant volume of boric acid solution during routine experimentation. These ports are above the top of the solution during boiling and do not present a chance for collection from the test solution as seen in Figure 2.5. There are 2 additional valved ports which open to the bottom of the test section as seen in Figure 2.4 which provide direct access to the heated solution. While these bottom ports can be used to inject solution to the test section, as might be seen prior to HLSO, they provide most useful for the collection of samples from the solution during the boiling phase of experimentation. Methods for determining the concentration of the solution via the samples collected from the PASTA facility are necessary for the analysis of solution within the test section.

2.2.2   Fixed Volume Sample Collection

To measure the boric acid concentration of solution in the test section during experimentation, samples were collected via the valved ports at the bottom of the facility as seen in Figure 2.4 . Collection of samples is described in this section.

During experimentation, the heating of the solution causes a volatile boil throughout the test section volume. The boiling ensures a well mixed solution in the test section, and the dissolution of boric acid within the DI water. Due to the well mixed status of the test section volume caused by volatile boiling, samples collected from these ports well represent the average solution throughout the test section. While power is applied to the heating elements, and the solution is well mixed, one of the

bottom valved ports was be opened. An amount of solution was then allowed to flow through the port into a waste bucket to ensure a clean flushing of the port length of any boric acid precipitate. A small vial was then used to collect a known volume of solution as it streams from the facility. Collected samples, which well represents the average solution throughout the test section, were used to determine the concentration of boric acid in the test solution.

3. SAMPLE ANALYSIS

Once a sample was collected from the facility, it was used to determine the average concentration of the boric acid solution within the test section at the time of collection. Three methods for determining concentration are described in this section.

Prior to measurement of samples collected from the facility during experimentation, a calibration of measurements for known concentrations was generated. The calibrations in this section were accomplished with the same DI water, and boric acid used in the PASTA facility during experimentation. Calibrations were performed at concentrations of boric acid in DI water which were characteristic of the facility, and as similar as possible to those expected, even up to precipitation.

3.1   Gravimetric Method

Gravimetric analysis methods provide a simple and effective method for determining the content of a sample solution when the analyte is known. For a constant volume of boric acid solution, a higher concentration correlates to a greater mass of boron in the sample. Samples collected from the PASTA facility were pure DI water mixed only with boric acid. As such, uncertainty of mass measurements specifically due to impurities were minimal. A sample collected from the facility was able to be dried directly, and the mass of boric acid measured. The mass of boric acid was be used to determine concentration of the known volume which was collected, and correlate to the average concentration of the facility at time of sampling.

3.1.1 Calibration Procedure and Error Estimation

A calibration curve was generated of the measurable mass of boric acid from various known–concentration solutions. The calibration was generated to befitting concentrations of boric acid in DI water which are seen in the PASTA facility. Drying trays were prepared with absorbent paper sheets and then weighed as shown in Figure 3.1a. Calibration solution was then created, after which a liquid sample containing boric acid was collected in a tray and dried until only boric acid remained. The mass of boric acid was determined by weighing the tray after drying and subtracting the predetermined weight of the tray with the paper sheets.

(b) Tray after drying process.
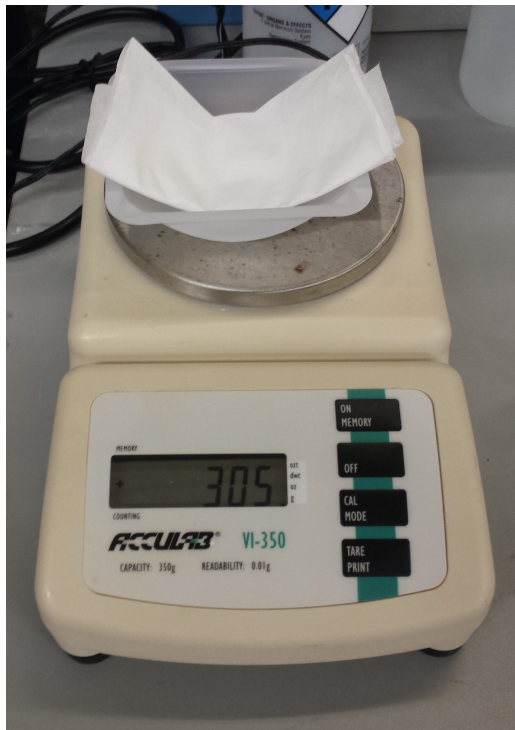
(a) Tared tray before sample collection.

Figure 3.1: Example drying trays used for gravimetric calibration of samples from the PASTA facility.

To perform the calibration, five solutions of known boric acid concentrations were prepared. A solution of 100 g/l boric acid in DI water was prepared at 100°C. From this calibration solution, a 16 ml sample was collected and immediately transferred to a prepared drying tray. The tray was placed on a heated surface to dry at a constant temperature of 50°C. This process was repeated to produce 10 samples for error estimation. The same procedure was applied for calibration solutions of 250 g/l, 275 g/l, 280 g/l, and 300 g/l of boric acid in DI water at 100°C. Control samples were also prepared using pure DI water to verify the calibration method, by confirming that the original mass was recovered after the drying procedure, and to estimate the drying time. After the samples were dried, as shown in Figure 3.1b, the trays for each calibration solution were again weighed using the Acculab® VI-350 (0.01 g accuracy) scale. The average mass of boric acid from the 10 samples was calculated for each calibration solution, along with the uncertainty, reported as 2σ, and is plotted in Figure 3.2.
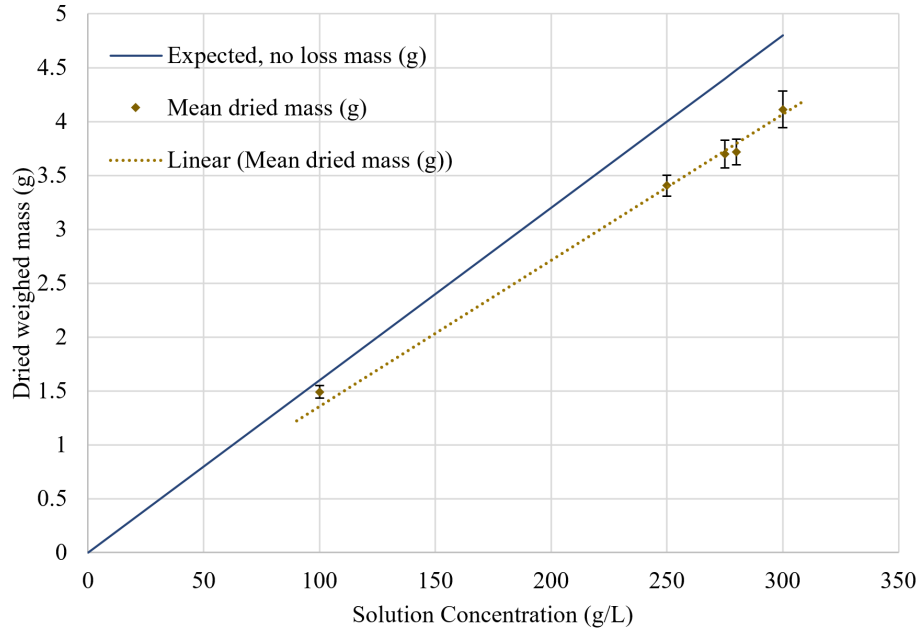
Figure 3.2: Boric acid dried mass as a function of calibration solution concentration for a 16 ml sample. A linear fit was made for the calibration solutions. Data contained in Table B.1.

From the dry mass of the gravimetric calibration samples, it was determined a fraction of the boric acid in solution was lost from each sample during the drying process. Losses were found to follow a linear relationship which results in a multiplication coefficient of

$$\frac{\text{boric acid in solution (g)}}{\text{measurable boric acid after drying (g)}} = 1.18 \qquad (3.1)$$

to be used to determine the concentration of boric acid in DI water from the 16 ml samples taken from the PASTA facility during experimentation.

3.2   pH Measurements Method

Another proposed method for determining the concentration of boric acid in a solution was measurements of pH. Because the concentration of boric acid in the PASTA facility can reach the solubility limit, however, samples needed to be diluted such that the sample is below saturation. Calibrations of pH measurements of diluted samples collected from known solution concentrations were used to generate a curve that can be used to determine concentrations of samples collected from the PASTA facility.

3.2.1   Calibration Procedure and Error Estimation

Dilution beakers and graduated cylinders were cleaned and dried in preparation of sample collection. A solution of boric acid with known concentration (50 g/l) was prepared in DI water heated near to 100°C. A 16 ml sample was collected from the calibration solution and transferred to a graduated cylinder. The sample was then diluted to a total volume 100 ml using DI water and set aside to cool to room temperature. Allowing samples to cool to room temperature after dilution ensured minimized uncertainty in pH measurements due to temperature variance. This process was repeated with ten samples for error estimation. After the samples had cooled to room temperature (25-27°C), a Mettler Toledo® SevenCompact pH and ion meter was used to measure the pH of each sample. For the ten diluted samples measured, the average pH was calculated, along with the uncertainty reported as 2σ. Ten similar samples were also collected from the calibration solution and diluted to a total of 200 ml to quantify the effect of dilution on the measurements taken. The process for 100 ml and 200 ml diluted samples was repeated for calibration solutions of concentrations 100 g/l, 150 g/l, 200 g/l, 250 g/l, and 300 g/l, which are characteristic of the PASTA facility. The average pH of the samples for both 100

ml and 200 ml dilutions as well as logarithmic best fits was plotted as a function of calibration solution concentration in Figure 3.3.
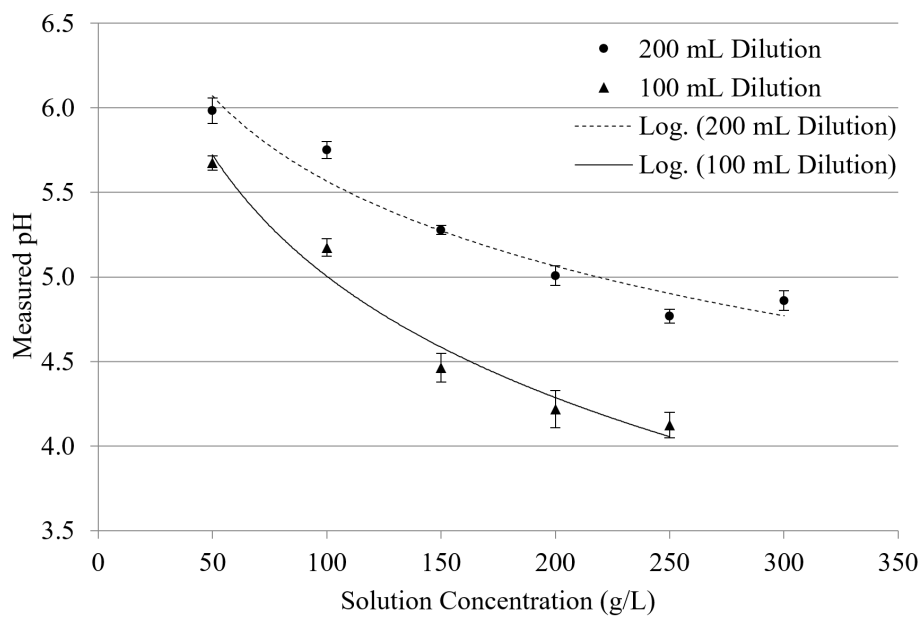

Figure 3.3: Measured pH of diluted 16 ml samples as a function of calibration solution concentration. Logarithmic fits were made for the set of pH vs concentration. Data contained in Table B.2.

Due to the high concentration of solutions required for calibration, it was determined unfeasible to measure the pH of a 100 ml dilution for the 300 g/l calibration solution. A logarithmic fit proved to be the best matching curve for the data. The decrease in slope at the higher concentrations, which are characteristic of the facility, would decrease precision of measurements of concentration using pH, as the same measurement was seen for various calibration solutions, as can be seen in comparison of the 200 g/l, 250 g/l, and 300 g/l data points for 200 ml dilutions. As such, quantitative analysis of concentration using pH measurements was determined to be unrealistic.

3.3   EC Measurements Method

The last method for determining concentration of boric acid in DI water was EC measurement. Similar to the pH measurements, this method also required the use of dilution, as the high concentration of boric acid in samples interfere with the instrumentation used. Calibrated EC measurements of diluted samples collected from known concentrations of calibration solutions were used to generate curves that can be used to determine the concentration of a sample collected from the PASTA facility. The calibration curve was made using known concentrations of boric acid solution in a similar sampling method as for the pH method.

3.3.1   Calibration Procedure and Error Estimation

Dilution beakers and graduated cylinders were cleaned and dried in preparation of sample collection. A calibration solutions were prepared as for the pH measurements, and twenty samples of 16 ml were collected concurrently. For error estimation, ten samples each were diluted with DI water to total volumes of 100 ml and 200 ml. The samples were allowed to cool to room temperature, and a MultiParameter PCSTestr$^{\text{TM}}$ 35 by Eutech Instruments® and Oakton® was used to measure the EC of each sample. As done for the pH calibrations, the process was done for calibration solutions of concentrations 50 g/l, 100 g/l, 150 g/l, 200 g/l, 250 g/l, and 300 g/l in order to represent the full range of concentrations which would be seen in the PASTA facilitiy. The resultant EC measurements are plotted as a function of solution concentration in Figure 3.4.
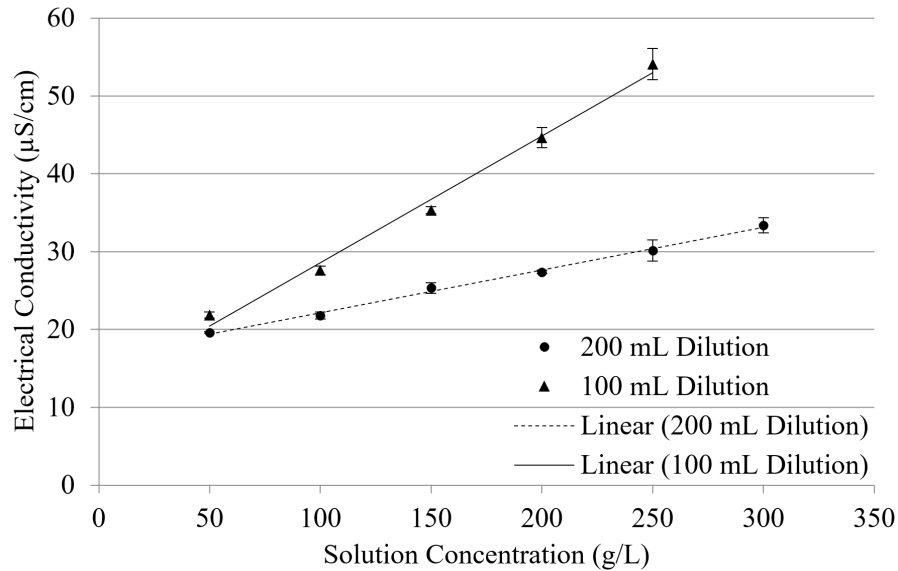
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Figure 3.4: Measured EC of diluted 16 ml samples as a function of calibration solution boric acid concentration. Linear fits were made for the set of EC vs concentration. Data contained in Table B.3.

Again, for the high concentration of solutions required for calibration, it was determined unfeasible to measure the EC of a 100 ml dilution for the 300 g/l calibration solution. Linear fits proved to be the best fitting curves for the data. The 100 ml diluted samples, however, would be unable to be used in conjunction with and experimental run in the PASTA facility, as concentration would be unable to be quantitatively analyzed. As such, the 200 ml dilutions were determined to be the only useful method for measurements to determine concentration of a solution.

3.4 Calibration Results

Comparing the three calibration methods, a number of features stand out. The method of pH measurements appears to have very little application to the determination of boric acid concentration from the PASTA facility. Due to the high concentration expected in the facility test section, the pH measurements, even after dilution of

samples, provided very little quantitative difference between samples. Without any differentiation of the measured samples, it could not be accurately determined what the original solution concentration was. The linear fits of the gravimetric and EC measurements seemed to provide a more appropriate tool for determining concentration of unknown solutions. The 100 ml dilutions for measurements of EC provides the best resolution in differences of measurement when compared to the original solution concentration. At concentrations in the PASTA facility, however, which are in excess of 275 g/l, the dilution to 100 ml does not provide enough DI water to maintain an all-dissolved boric acid solution. Due to this, the EC measurements would only be feasible with the 200 ml diluted samples. In all calibrations, uncertainty of measurements was seen to be proportional to concentration of the sampled solution. This was of particular concern in the EC calibration, in which there was overlapping uncertainty in measurements of the higher concentration regime. Due to the overlapping uncertainty coupled with the visibly smaller slope of the calibration line generated from the samples diluted to 200 ml, the EC measurements were also determined to have little application in determination of solution concentration for high values as were expected to be seen in the PASTA facility.

Even with the losses of boric acid due to the drying process required in the gravimetric measurement method, the uncertainty of measured values provided less overlap than was seen in the EC measurements, while still providing a reasonably sloped line from calibration measurements to resolve differences in concentration of original solution from measured boric acid of dried trays. The gravimetric method for determination of boric acid concentration was therefore used for all experimental trials described in this thesis.

4. EXPERIMENTAL PROCEDURE

All experimental runs were conducted according to written procedures, which were developed and refined during the facility shakedown. This section provides a brief description of the official written procedure, which is contained in Appendix C. The facility was first filled with DI water and heated to a boiling state to prevent any thermal damage or shock to the facility hardware. During the initial preheating, an initial solution was prepared with 660 g of Optibor$^®$ Orthoboric Acid in 2 l of DI water at approximately 50°C. After draining the test section of the preheating water, the initial solution was poured into the test section from the upper plenum which was kept open. An additional 0.5 l of DI water at the same temperature was then used to clean any boric acid which had accumulated at the upper plate flange. This final solution in the test section at the beginning of an experimental runs, which contained 660 g of boric acid in 2.5 l of DI water to make facility initial concentration (C_0) = 264 g/l, was chosen to be slightly below solubility limit of boric acid in water at 100°C (C_s) = 275 g/l[6] so that the saturation could be reached shortly following test solution boiling. The total initial volume corresponded to a liquid level just above the top plate to allow for full coverage of the heating rods. With the initial boric acid solution in the facility, the test section was allowed to come to a quiescent state as shown in Figure 4.1 with the excess of boric acid deposited at the bottom of the test section.
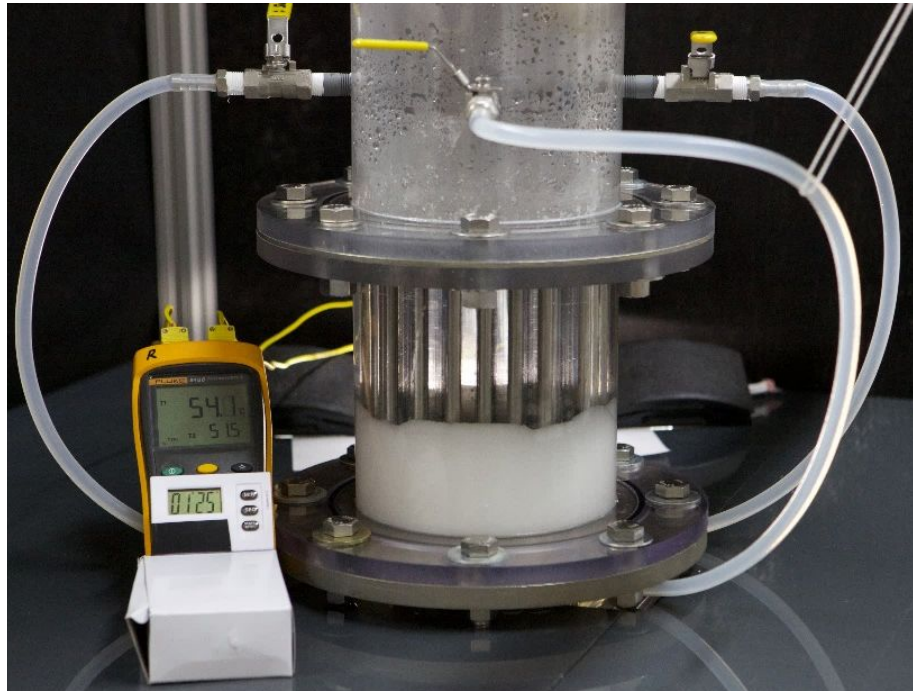
Figure 4.1: Initial loading of test section during experimentation.

A solution was prepared for injection into the facility using similar techniques described above. The concentration of added solution (C_a) selected was equal to 60 g/l, which is not typical for any plant conditions and was selected arbitrarily based on solubility of boric acid at the preparation temperature of 50°C. Addition solution was held at preparation temperature in the reservoir beaker as shown in Figure 2.6. After the addition solution was prepared, the heaters were turned on to the prescribed operational power for the experiment. The temperature of the test section solution was monitored closely during the first heating, as well as the power of the heating rods, which varied slightly due to thermal resistance differences. Applied power to the heating rods was adjusted during the first five minutes of facility operation to maintain a constant power draw.

Once the test section solution was observed to have completely dissolved the

excess boric acid, the injection solution in the reservoir beaker was added to the facility via the metering pump. Time of initiation of injection solution addition was recorded for each experimental run, and was considered the beginning of the change of concentration regime for the experiment. Additional prepared injection solution was added to the reservoir when the remaining solution reached the 300 ml mark on the graduated reservoir beaker. Time of addition of each injection solution batch was also recorded during the experiment. Periodically throughout the experiment, the heaters were turned off for a minimal amount of time for the boiling solution to come to a quiescent state and the level of solution to be determined. Injection rate via the metering pump was adjusted for any differences in solution level from the beginning of experiment and from the previous solution level check. This procedure was used to maintain the constant solution volume in the test section during experimentation.

Throughout the experiment, 16 mL samples were collected from the port at the bottom of the facility test section as per the sample collection procedure described above, while recording the time of collection. In following of the gravimetric method for concentration determination also previously described, each sample collected was transferred to a analysis tray and dried on a heating plate. From the samples dried mass and using Equation 3.1, the concentration of the solution in the test section at the time of collection was determined with associated uncertainty.

The heaters were left on, except for short periods to determine solution level, until the test section solution was observed to exceed the solubility limit as determined by a change in the turbidity of the solution which obscured light passing through the facility test section as can be seen in comparison of Figure 4.1 with Figure 4.2, as well as the appearance of solid precipitates in suspension in the solution.

Figure 4.2: Final state of test section during experimentation.

Once the solubility limit was exceeded, the test was terminated by cutting off power to the heating rods and flushing the test section with DI water repeatedly until it was clean. The samples collected from the facility during experimentation were placed on the heating surface to dry. After the analyte samples were dried completely, they were weighed as described above to determine concentration of the facility at time of collection. For each experimental trial, concentration of the solution in the test section was plotted as a function of elapsed time since the initial injection from the reservoir.

4.1 Test Matrix and Conditions

To determine the facility dependence on physical features and construction pieces, a number of tests were run with varying parameters of operation. Features that were altered included the sealing of the top lid, total power applied to the heating rods,

as well as injection location from either the top or bottom of the facility. Other operational parameters were unaltered during the perturbed experiments, including radial power distribution, concentration of injected solution, and initial concentration of facility solution. Table 4.1 summarizes the conditions of the perturbed shakedown experiments.

Table 4.1: Operational parameters of perturbed shakedown experiments.

Test number	C_0 (g/l)	C_a (g/l)	Total power (kW)	Power shape profile	Lid Status	Injection point
1	264	60.0	4.0	Flat	Closed	Top
2	264	60.0	4.0	Flat	Open	Top
3	264	60.0	4.0	Flat	Closed	Bottom
4	264	60.0	2.0	Flat	Closed	Top

During the experiment described in this thesis the total power used was uniformly distributed among the 41 rods, constituting a flat power profile. This flat power profile is not characteristic of a full PWR core, but within the geometry described, may provide a more appropriate description of a smaller segment as might be seen in a single assembly. Test 1 was used as a baseline, as it presented the easiest setup and operation of the facility. Injection from the top of the facility was hypothesized to create a gradient of boric acid concentration in which the bottom of the test section near the sampling port would be lower than near the top. Injection from the bottom was thought to induce greater mixing in the test section solution, which would create a more uniform concentration throughout the facility. Opening the facility lid for the

duration of an experiment was hypothesized to decrease the overall concentration of the solution in the facility, due to additional losses in rapid vaporization and exhaust. The rapid boiling of the facility solution was also thought to cause splashes of solution out of the facility which could also entrain boric acid out of the facility. Decrease of the total power applied via the heating rods was expected to slow the overall increase of test section solution concentration. Each of the hypotheses was tested.

5. ANALYTICAL SOLUTION

For comparison of the determined concentration of the test section with the an-alytical expected value, the rate of change of mass of the boric acid in solution was analytically calculated by solving the mass conservation equation expressed as

$$\frac{d\mathrm{M_B}}{dt} = \dot{m}_{in} - \dot{m}_{out}.$$
(5.1)

In Equation 5.1, the injected mass rate, $\dot{m}_{in}$ (g/s), was determined by the con-centration, $\mathrm{C_a}$ (g/l), and approximately constant volumetric flow rate, $\mathrm{Q_{in}}$ (l/s), of the injection solution into the facility as

$$\dot{m}_{in} = \mathrm{Q_{in}C_a}.$$
(5.2)

The boric acid loss rate, $\dot{m}_{out}$ (g/s), was calculated from the calibration data. To determine loss of boric acid due to evaporation ($\mathrm{E_l}$) (g/l), the fractional loss was cal-culated for each calibration concentration of boric acid as per the samples collected. As described in the experimental procedure, solution volume in the test section was held constant, which permitted the assumption that volume of solution injected was equal to volume of solution evaporated, ie. $\mathrm{Q_{in}} = \mathrm{Q_{out}}$. These assumptions allowed for a mass loss rate to be determined as

$$\dot{m}_{in} = \mathrm{Q_{in}E_l}.$$
(5.3)

For comparison to an ideal experimental operation, the loss of boric acid due to evaporation was neglected, and the rate of change of boric acid was approximated as

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$$\frac{d\mathrm{M_B}}{dt} = \dot{m}_{in}. \tag{5.4}$$

Equations 5.1 and 5.4 were divided by the facility test section free volume, $\mathrm{V_{test}}$ (l), to express the rate of change in boric acid concentration. The injection rate for both equations was determined using the time of batch injection during experimentation, and analytical solutions of 5.1 and 5.4 were used in comparison to the concentration of the test section as determined from the samples collected.

6. RESULTS

During experimentation, after the electric heaters were turned on, solution temperature in the test section was found to increase until reaching the boiling point. At this point, the solution was observed to undergo a volatile boil–off which caused highly turbulent flow conditions in the test section due to the rapid bubble formation and migration to the upper plenum, as can be seen in Figures 4.2 and 6.1. Elevated temperatures achieved and highly turbulent flow in the test section produced dissolution of the entire quantity of boric acid initially deposited at the bottom of the test section during preparation. As described in the experimental procedure, once all boric acid was observed to have dissolved and no solid traces were visible in the test section, the metering pump was turned on and set to a rate required to compensate for the evaporation of DI water from the test section for the selected test power. Rate of solution injection via the metering pump was defined during shakedown testing and, as described in the experimental procedure, manually fine–tuned during the experiment.

Figure 6.1: Volatile boiling in test section during experimentation.

6.1   Flow Visualization

During shakedown tests, and defined experimentation within the scope as described in Table 4.1, a number of observations were made on the qualitative flow characteristics within the test section. Observations of the test section included the following:

- After the first complete batch of injection solution, which was expected to raise the concentration beyond the solubility limit, the solution in the test section remained as clear as the initial loading of the solution.

- Later, during experimentation, small visible particles started appearing in the solution. These particles were entrained in the turbulent flow or maintained in suspension via the volatile bubbles departing from the surface of the rods

- Due to the particle entrainment or suspension phenomenon, deposition on the heating rods or test section wall of the boric acid precipitate was prevented.

- Also due to the flow turbulence and bubble formation, the test section solution fully represented a well mixed homogeneous volume.

- As the mass of boric acid in the liquid increased, turbidity of the test section solution visibly increased and a layer of boric acid precipitate at the top plate flange formed which continued to grow until termination of the experiment as in Figure 6.2.

- At high concentrations just prior to the termination of experimentation, the appearance of a boric acid precipitate layer was accompanied by the appearance of larger particles, as well as deposition of precipitate near the rod bundle periphery and test section walls.

  - The density of these larger particles was observed to be great enough to prevent the entrainment of the particles in the boiling flow as was previously seen.

- For experiments in which the lid flange was left open, the volatile boiling caused a quantity of boric acid to splash up out of the upper plenum and deposit boric acid precipitate around the uppermost flange and on the PASTA working surfaces, as seen in Figure 6.3

Figure 6.2: Boric acid precipitate at top of test section during experimental runs near termination.

Figure 6.3: Boric acid precipitate deposition during unlidded experimental runs.

6.2   Concentration Measurements

Following the termination of experimentation by shutting off the heating rods, the collected samples were placed on the heated surface to dry. When it was determined the analyte trays had dried completely, the measurable mass of boric acid was used to determine the concentration of the solution in the facility as described in the calibration and sample analysis sections. Concentration of the solution was then plotted as a function of time after injection initiation for each experiment. Equations 5.1 and 5.4 were used to calculate a range of theoretical concentration in the facility and were plotted as a function of time according to the injection rate, Q_{in}, during experimentation as determined by the metering pump. The analytical approximations of facility concentration along with the experimental data from test experiments 1, 2, and 3 is plotted in Figure 6.4.
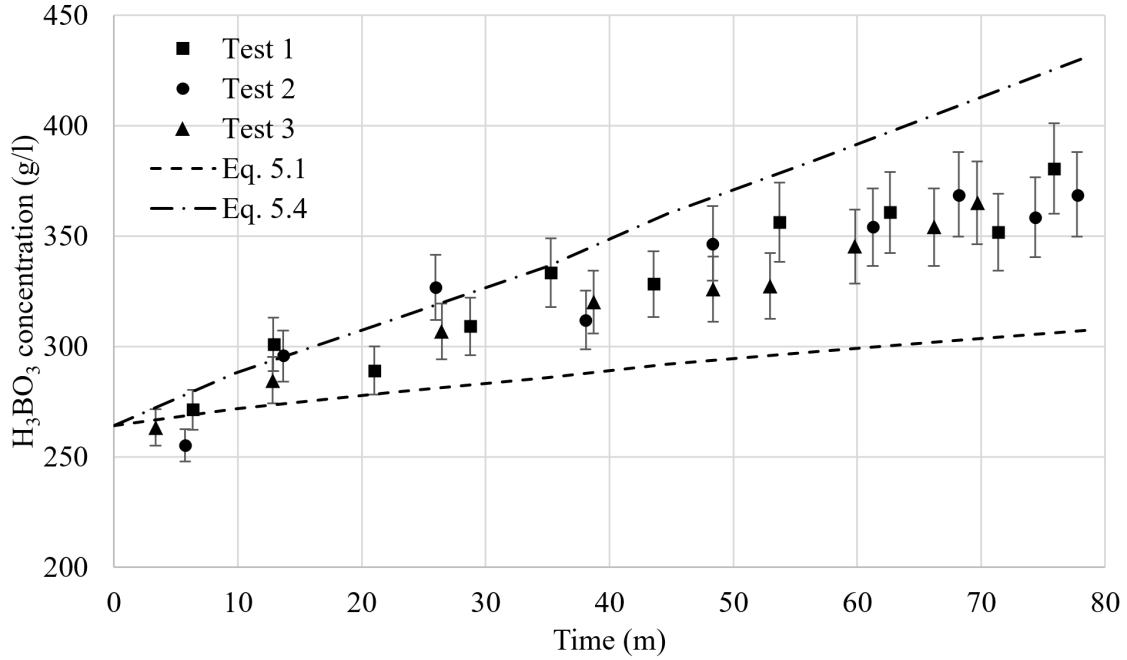
Figure 6.4: Analytically approximated alongside experimentally determined boric acid concentration for Tests 1, 2, and 3.

Experimental data resulted a linear trend for the test section solution concentration which is loosely bounded by the analytical solutions, Equations 5.1 and 5.4. Experimental results deviate from the evaporative loss approximation in Equation 5.1, indicating that losses due to evaporation of the solution are less significant than what was observed during calibration of the gravimetric measurement method. Boric acid, however, is certainly lost from the facility, as the collected data are well below the lossless approximation as described by Equation 5.4. Deviation from the lossless operation of the facility is hypothesized to be due to entrainment of liquid droplets out the exhaust vent in the vapor phase. This exhaust vapor was not collected or analyzed for boric acid content to determine concentration.

To quantitatively determine the dependence on applied power to the rate of in-

crease of boric acid concentration in the PASTA facility during experimentation, Test 4 was executed with half the total applied power through the heating rods. Samples were collected as previously described and were permitted to dry for gravimetric analysis. After weighing the collected samples, the experimentally determined boric acid concentration was plotted as a function of time for both Test 1 and 4. Linear fits were made to both data sets as shown in Figure 6.5.
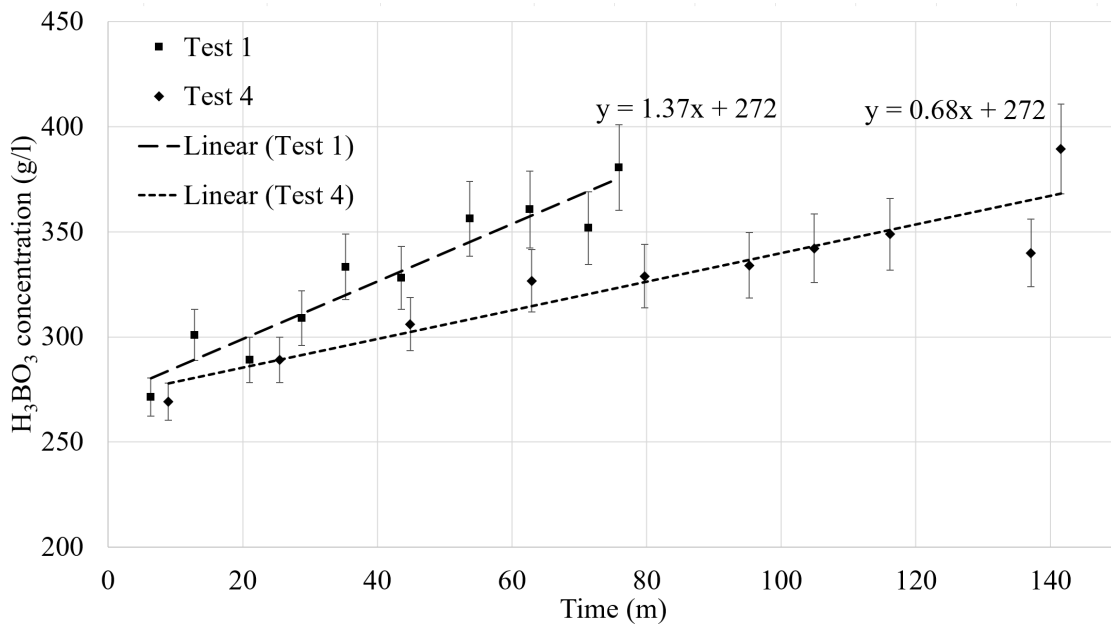

Figure 6.5: Experimentally determined boric acid concentration for Tests 1 and 4.

The experimental data for Test 4 was also seen to follow a linear trend. As seen by the trend line equations, the rate of increase of concentration was half of the baseline Test. Similar losses were observed in all four Tests, which require additional analysis to determine the full mass conservation relationship of boric acid in the PASTA facility during experimentation.

6.3 Discussion

Each of the Tests 1, 2, and 3 were observed to follow an almost identical rate of increase of concentration throughout the duration of the experiment. Even with the variance to the lid, which was left open in Test 2, the experiment reached precipitation at approximately the same time as the other Tests. This disproved the initial hypothesis of dependence of evaporative losses as had been seen in the gravimetric calibration process. It was also observed that due to the highly volatile boiling within the test section, the solution was homogeneous regardless of injection location, as varied from Test 1 to 3. No gradient was observed in any orientation in the test section, and mixing was driven by the boiling flow and bubble formation, rather than the flow disturbance due to solution injection, which was minor. This disproved the hypotheses of improved mixing due as well as concentration gradients due to injection position.

In comparing the data sets from Tests 1 and 4, it can be seen that the total time required to reach boric acid precipitation within the PASTA facility is inversely proportional to the total thermal power applied to the solution via the heating rods. With half the total power applied in Test 4 as Test 1, the rate of increase of concentration was almost exactly halved as shown by the trend line solutions in Figure 6.5. This is in agreement with the hypothesized effect as described.

As shown in all data sets in Figures 6.4 and 6.5, concentration of the solution in the test section was greater than $C_s = 275$ g/l for the majority duration of the experiment. This high concentration of boric acid in the facility, however, did not have a visible effect on the flow characteristics of the boiling, even when particles of boric acid were visible throughout the test section. Turbidity of the test section solution was the best indicator of approximate concentration during each experiment, rather

than any perceptible change in flow characteristics. Further, change in water density caused by increased boric acid concentration did not bring about stratification of the test section soltuion. Instead uniform flow mixing was observed, facilitated by the rapid boiling in the test section.

For all Tests observed, the rapid bubble generation which drove the test section mixing prevented deposition of boric acid precipitate within the rod bundle. Experimentation clearly visualizes the mixing due to boiling and the distribution of the precipitate particles in the solution when they become visible. In particular, no appreciable deposition of solid precipitate was observed between the internal rods of the bundle or on the rods surface. Instead, deposition of the precipitate occurred in regions of lower power density or at colder surfaces, ie. the rod bundle periphery or the test section walls, respectively. This is confirmed in all presented experiments in the PASTA facility. This phenomenon can be observed in real time during the experimentation. Crystallization starts at the top of the mixture level and continues in the liquid in the form of small spherical particles of approximately 0.5–1 mm in diameter. The majority of these particles remains suspended due to the violent boiling. Towards the end of experimentation, when concentration is well beyond the solubility limit, the behavior of the precipitate accumulating at the top of the mixture level seems similar in all experimental activities. This is in agreement with results as reported from experiments at the VEERA facility[16], although it is unclear if the referenced experiments were conducted considering the liquid level (collapsed level) or the mixture level as reference. In the case of the Tests conducted in the PASTA facility, the liquid level, verified at the clear test section before heating the solution via the rods, was maintained right at the core top plate elevation. This may explain the fact that no blockage was observed in the presented Tests due to the larger water inventory.

7. CONCLUSIONS

An experimental apparatus was constructed to conduct experiments with DI water and boric acid, to observe and study the flow behavior, and to measure the boric acid concentration in boiling water. The PASTA facility focuses on studying the effect of the increase in the boric acid concentration exclusively in the core and does not account for other effects such as volume mixing. This facility was originally designed to study the combined effect of the boron precipitation with a hypothetical core blockage at the bottom of the core which prevents any mixing of the solution in the core with other RPV regions, including the lower plenum. Three methods for determining the concentration of boric acid at very high concentrations, even exceeding saturation, were explored. A gravimetric method for concentration determination proved to be the most effective for high values as were characteristic of the PASTA facility. The concentration of solution in the test section was approximately solved using an analytic approach for comparison to experimentally determined values.

During experimentation, concentrations of boric acid were found to increase linearly with time. Due to initial experiment conditions, the saturation limit was reached very early in the experiments, with no major effect on the flow observed. Volatile boiling in the test section induced uniform mixing in the test section, causing no appreciable difference in the concentration trends between the top and bottom solution injection locations. Opening the lid during experimentation did not affect the overall rate of increase of solution concentration in the facility test section. Applied power to the heating rods in the test section was observed to be proportional to the rate of increase of concentration of the test section solution in the facility. Comparison of boric acid concentration with the analytical solution confirmed that

a fraction of the boric acid was transported within the vapor phase outside the test facility. The fraction released contributed to the reduced rate of increase of boric acid concentration in the solution. Fractional boric acid loss from the test section was found to be lower estimated from the observed losses during the calibration procedure. This may be attributed to condensation in the upper plenum and through the test exhaust and the subsequent reflux of the liquid into the test section.

7.1   Future Work

Further work with the PASTA facility allows for many directions. To address issues seen in this thesis, it should first be noted that the concentrations of boric acid from the test section which were determined by the gravimetric method were above the values initially calibrated. This extrapolation of a calibration curve should be improved. Whether a gravimetric method can still be used at the supersaturated conditions observed must be further explored to validate these measurements reported. Also of note, is the conservation of mass equation which describes the boric acid in the test section solution during experimentation. Experimental results lie between a lossless approximation and an overestimated value for evaporative losses. The observed losses are attributed to the entrainment of boric acid precipitate particles in the vapor phase of the water escaping the facility. These losses, however, may be a procedural error in the sample analysis, which is not accurately determining the concentration of samples from the test section compared to the calibration samples. To resolve this, the exhaust vapor should be collected and condensed to perform a similar concentration analysis as on the directly collected samples. Additional construction to the facility would require the the test section maintain ambient pressure for direct comparison with the current analysis.

Held constant in this analysis is the variance of power in a radial and azimuthal

direction. This approximation is used to simplify the construction of the facility as well as qualitatively observe a smaller region of the proposed PWR core, rather than looking at an entire core for boric acid precipitate analysis. The smaller scope of even a single assembly in a PWR, however, has a "radial" power profile that can be quantified. It was observed that boric acid precipitates deposited near the colder sections of the test section. Varying power profiles with pronounced cold spots would provide further insight to the deposition of boric acid solids in regions of minimal decay heat during LOCA events.

To further improve the PASTA facility's likeness with a typical PWR, the test section could be reconstructed to include an annular downcomer at the periphery, with bottom injection from all azimuthal positions simultaneously. This alteration to the facility would even further improve the analysis of cold spots in the RPV for boric acid precipitate accumulation. A spacer grid could also be constructed to fit around the rods in the test section. Accumulation of boric acid in those regions are not captured by the current PASTA construction, and may be of interest for the final resolution of GSI 191.

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APPENDIX A

PASTA PRODUCTION IMAGES

A.1 Drawings

The following are drawings used in the manufacturing of the PASTA facility.

The pattern of holes is centered about the flange and the 6" ø construction line

Standard 6"
Flange

55 threaded holes
The thread is:
NPT Size 1/4--18

52 1/8" ø holes

The pitch is the same for the threaded holes and 1/8"holes in the x,y dimensions.

The pitch is .66"

0.50

D

Ø0.125

DETAIL D
SCALE 1 : 1
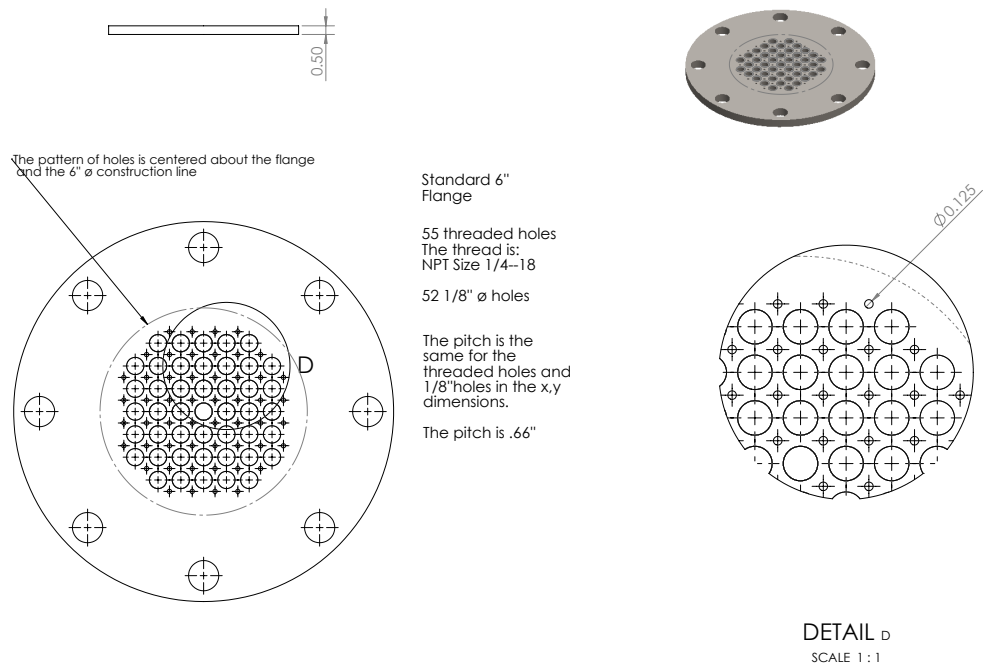
Figure A.1: Manufacturing drawing of the top plate flange.

0.065

The pattern of holes is centered about the flange
and the 6'' construction line

Standard 6''
Flange

55 holes

E

∅0.4375

DETAIL E
SCALE 1 : 2
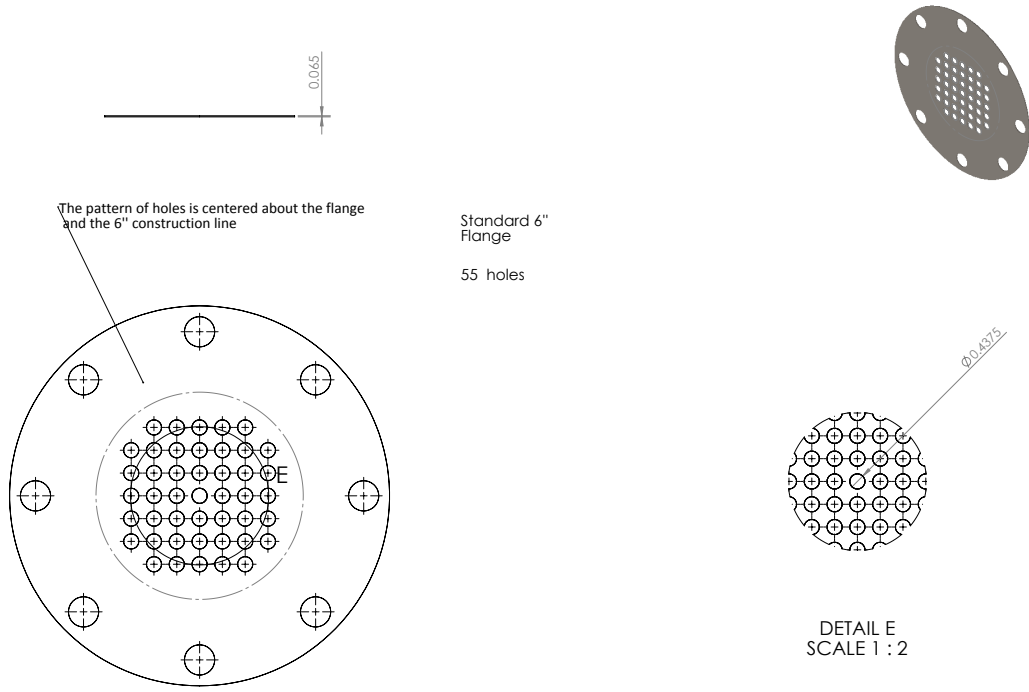
Figure A.2: Manufacturing drawing of the bottom plate flange.

APPENDIX B

CALIBRATION DATA

Following is the collected data used in calibration.

Table B.1: Expected mass and mean dried mass for each ten samples, 16 ml, collected from the various gravimetric calibration solutions.

Solution Concentration (g/l)	Expected mass (g)	Mean dried mass (g)
100	1.60	1.49 ± 0.06
250	4.00	3.41 ± 0.10
275	4.40	3.70 ± 0.13
280	4.48	3.72 ± 0.12
300	4.80	4.11 ± 0.17

Table B.2: Measured pH for each ten samples, 16 ml, diluted to 100 ml and 200 ml for various calibration boric acid solutions. Samples diluted to 100 ml for the calibration solution of 300 g/l were unable to be measured with the Mettler Toledo® SevenCompact pH meter, as boric acid had precipitated out of solution and interfered with the instrumentation.

Solution Concentration (g/l)	100 ml dilutions measured pH	200 ml dilutions measured pH
50	5.67 ± 0.04	5.98 ± 0.08
100	5.17 ± 0.05	5.75 ± 0.05
150	4.46 ± 0.08	5.28 ± 0.03
200	4.22 ± 0.11	5.01 ± 0.06
250	4.12 ± 0.08	4.77 ± 0.04
300	N/A	4.86 ± 0.06

Table B.3: Measured EC for each ten samples, 16 ml, diluted to 100 ml and 200 ml for various calibration boric acid solutions. Samples diluted to 100 ml for the calibration solution of 300 g/l were unable to be measured with the MultiParameter PCSTestr$^{\text{TM}}$, as boric acid had precipitated out of solution and interfered with the instrumentation.

Solution Concentration (g/l)	100 ml dilutions EC (µS/cm)	200 ml dilutions EC (µS/cm)
50	21.9 ± 0.4	19.6 ± 0.1
100	27.6 ± 0.6	21.8 ± 0.5
150	35.3 ± 0.5	25.3 ± 0.7
200	44.6 ± 1.3	27.4 ± 0.3
250	54.1 ± 2.0	30.2 ± 1.3
300	N/A	33.4 ± 1.0

APPENDIX C

EXPERIMENTAL PROCEDURE

The following pages are the procedure for preparation and operation of the PASTA test facility. Each test described in this thesis was accomplished using these instructions after development during construction and shakedown testing of the facility.
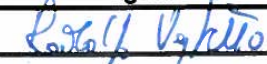
TECHNICAL DOCUMENT COVER PAGE

Document No:	TAMU-PASTA-TPC	Revision: 1.0	Page 1 of 21
		Date: 3/28/15	

Doc Title: TAMU PASTA Test Procedure
Project No: N/A
Project Name: N/A
Client: N/A
Document Purpose/Summary: This procedure provides the instructions for preparation and operation of the PASTA test facility.   Total Page Count: 21 pages.
Continuous Use Procedure

TECHNICAL DOCUMENT COVER PAGE

Document No:	TAMU-PASTA-TPC		Revision: 1.0	Page 2 of 21

Prepared By:	Philip Jones	ʻ6₂	3-28-15
	Printed/Typed Name	Signature	Date
	Mason Childs	*(signature)*	3/28/15
	Printed/Typed Name	Signature	Date
TAMU Reviewed By:	Rodolfo Vaghetto	*(signature)*	03/28/2015
	Printed/Typed Name	Signature	Date

REVISION HISTORY LOG

Document Number: TAMU-PASTA-TPC   Revision: 1.0

Document Title: TAMU PASTA Test Procedure

REVISION	DATE	DESCRIPTION
1.0	3/28/15	First release

TABLE OF CONTENTS

DEFINITIONS AND ACRONYMS

PASTA	Precipitation And Sedimentation Test Apparatus
DEGB	Double Ended Guillotine Break
GSI	Generic Safety Issue
LB	Large Break
LOCA	Loss of Coolant Accident
MB	Medium Break
NRC	Nuclear Regulatory Commission
SB	Small Break
PWR	Pressurized Water Reactor
TAMU	Texas A&M University

1.0   PURPOSE

The purpose of this document is to provide instructions to conduct boron precipitation experiments on the PASTA test facility at Texas A&M University (TAMU).

2.0   EXPERIMENTAL FACILITY

The facility was designed for high temperature tests in presence of chemicals. The facility consists of an acrylic cylindrical section (test section) with 41 heated rods. A variable speed metering pump injects boric acid solution through the injection lines into the test section to compensate the liquid water lost by evaporation, continually increasing the concentration of boric acid until precipitation occurs.

3.0   TEST PROCEDURE

3.1   GENERAL PROCEDURE

This section describes how to use this document. Sections 3.2 through 3.4 must be completed before the actual experiment can begin, outlined in section 3.5.

Section 3.2 describes how to prepare the general materials required, 3.3 describes how to prepare the PASTA facility, and 3.3 describes how to prepare the solution.

When initiating a step of the procedures, the step will be marked with an "O". The "O" will be crossed when the step is completed. Steps that are skipped, or do not apply to an experimental run, will be marked as N/A.

3.2   MATERIALS PREPERATION

The following steps provide instructions for preparing the materials that will be required during the experiment.

Prerequisite Materials

- 11 Plastic closed containers (Figure 1a)
- Minimum of 1260 g of boric acid
- Minimum of 5 gallons of DI water
- 10 glass vials with a 16 mL indicator mark (Figure 1b)
- 10 Trays with each containing 2 lab foils (Figure 1c)
- Safety masks for boric acid handling

Figure 1a. Plastic container used for boric acid preparation. b. Glass vial for sampling with 16 mL indicator. c. Tray and foils used for sample drying.

Date: _____

1. _____ Ensure the 16 mL indicator on the glass vials is clearly visible

2. _____ Label the trays 1-10, and measure with the foil in the tray

3. _____ Record the masses of the trays in Table 3.2.1.

4. _____ Prepare 660 g of boric acid in a closed plastic container

5. _____ Prepare 10 x 60 g of boric acid in closed individual plastic containers

Tray Number	Measured mass (g)
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	

Table 3.2.1. Test sampling tray masses

Signatures:

Test Operator 1

Name	Signature	Date

Test Operator 2

Name	Signature	Date

Quality Assurance Officer or Test Reviewer

Name	Signature	Date

3.3   TEST SECTION PREPARATION

The following steps provide instructions for setting up the facility to begin the experiment.

Prerequisite Materials

- 1 hot plate with stirring capability (Figure 2a)
- Water Heater – a tea kettle works well
- Stainless steel rod
- Syringe, minimum of 20 mL
- 2 Thermocouple readers
- 2 x 2000 mL clean glass beaker
- 1000 mL clean glass beaker
- 1000 mL clean graduated cylinder
- Stainless steel container, minimum of 2500 mL
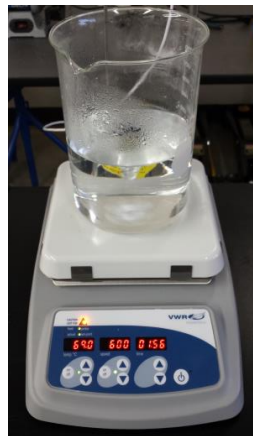
Figure 2a. Stirring hot plate used for boric acid batch solution preparation.

Date: _____

1. _____ Fill the test facility with DI water until the heater rods are completely covered – 2500 mL.

2. _____ Connect the thermocouple reader to the thermocouple inside the test facility.

3. _____ Turn on only a few of the rods, or a maximum of 400 W to slowly increase the water temperature until boiling.

4. _____ Add the 660 g of boric acid to the 2500 mL clean stainless steel container.

5. _____ Using the water heater, heat up an excess of 2800 mL of DI water to boiling.

6. _____ Add 2000 mL of hot DI water to the 660 g of boric acid in the stainless steel container and stir with stainless steel rod until as much of the boric acid is dissolved as possible.

7. _____ Place the stainless steel beaker with the 660 g solution near the PASTA facility and.

8. _____ Turn off the heaters in the test facility and open the valves to drain the DI water from the facility via the lower drain into the catch bucket located below the test section.

9. _____ Prepare a small amount (approximately 300 ml) of hot DI water in a clean glass beaker and connect to the suction of the PASTA metering pump.

10. _____ Open the selected injection port and turn on the metering pump to flush the injection with hot DI water.

11. _____ Turn off the pump and immediately close the injection valve to ensure that water remains in the injection lines.

12. _____ Close the drain valves used to drain the water from the test section.

13. _____ Pour the 2000 ml heated solution (step 7) from the stainless steel container into the test section.

14. _____ Use the remaining hot DI water container to clean any remaining boric acid from the container and add to the test facility. (Note the total volume of DI water injected in steps 14 and 15 must be 500 ml)

15. _____ With a syringe filled with DI hot water from the remaining 500ml, clean the walls of the upper test section with heated DI water.

16. _____ Ensure the liquid level is at the top of the steel plate.

17. _____ Close the top lid of the facility, confirming the O-ring is properly set.

18. _____ Install and hand-tighten the lid hold-down bolt.

19. _____ Connect silicon hose exhaust to the vertical exhaust pipe.

20. _____ Place video camera and adjust settings to view the experimental facility. Turn off audio recording.

21. _____ Take a final picture of the facility after test preparation.

Procedure notes:

Signatures:

Test Operator 1

Name	Signature	Date

Test Operator 2

Name	Signature	Date

Quality Assurance Officer or Test Reviewer

Name	Signature	Date

3.4   Batches Preparation

The following steps provide the instructions for preparing the boric acid solutions that will be injected during the duration of the experiment.

Prerequisite Materials

- DI water from section 3.2
- Boric acid from section 3.2
- Water heater
- Hot plate with temperature control and stirring device

Date: _____

Preparation of Batch 1

1. _____ Prepare 1000 ml of hot DI water.

2. _____ Add 60 g of boric acid in a 2000 mL glass beaker.

3. _____ Add the hot DI water prepared in step 1 to the beaker.

4. _____ Use the stirring hot plate, set at 69, to keep solution agitated and hot

5. _____ Place the beaker of solution on the injection reservoir heater.

6. _____ Place the suction hose from the metering pump into the solution.

7. _____ Place the thermocouple probe into the solution and turn on the thermocouple

   reader.

8. _____ Monitor the temperature of the solution and ensure it remains at approximately

   70°C - 80°C throughout the entirety of the experiment.

Preparation of Subsequent Batches

1. _____ Begin preparing subsequent batch immediately after adding a batch to the injection

   reservoir.

2. _____ Follow steps 1-4 from the batch 1 preparation to make another 1 L solution of 100

   g/L concentration.

Procedure notes:

Signatures:

Test Operator 1

Name	Signature	Date

Test Operator 2

Name	Signature	Date

Quality Assurance Officer or Test Reviewer

Name	Signature	Date

3.5   TEST EXECUTION: TEST #_____

The following steps provide instructions for operating the PASTA facility. The test initiation is outlined below. Throughout the experiment it will be required to check that the level of solution in the test section remains just over the plate. It will also be required to take samples during the experiment to later determine the concentration of the solution.

Prerequisites

- Prepared batches of boric acid solution
- Timer
- Camera
- Backlight
- Vials and trays
- Syringe
- Cold DI water

Date: _____

Test Initiation

1. _____ These steps are to be completed after Test Preparation procedures.

2. _____ Turn on the video camera and backlight.

3. _____ Record the time of test initiation and start timer. _____

4. _____ Turn on all switches for the electrical heaters.

5. _____ Calibrate heaters' power by adjusting the knob on the power supply while reading the output from the power meter. Fine tune the power supplies until each power meter reads 400 W (Each power supply will be approximately 40%).

6. _____ Check power after 5 minutes for stability.

7. _____ Record each reading from the power meters and total to determine total power output from the heaters.

     _____

     _____

     Total: _____ kW.

8. _____ Monitor the water as the temperature increases until complete dissolution of boric acid.

9. _____ Perform first **Solution Level Check** (see next section). If liquid level is below target, turn on pump until the appropriate level is reached.

10. _____ When all boric acid is dissolved and the solution is boiling and clear, begin injection by starting the pump and opening the injection valve (Pump should start at approximately 30-35%).

11. _____ Start pump and record the start time of injection. _____ m:s.

12. _____ When 300 mL solution remains in the injection reservoir, add the prepared solution batch to the injection reservoir beaker.

13. _____ Record the time of solution batch addition in Table 3.5.1.

14. _____ Record time of experiment termination. _____ m:s.

15. _____ Stop camera recording.

16. _____ Turn off experimental facility heaters and injection pump.

Solution Level Check

This is suggested to be repeated at each batch injection

1. _____ Ensure the experimental facility heaters and injection pump are turned off.

2. _____ Check the solution level in the test facility.

3. _____ Adjust pump rate with heaters off until more suitable level is achieved.

4. _____ Turn heaters and pump back on.

5. _____ Check power and fine tune if necessary.

Sampling

1. _____ Record the time of sampling in Table 4.0.1.

2. _____ Fill the vial with solution from the test section to the marked 16 mL level by opening the sampling valve.

3. _____ Immediately pour the solution into the tray without the lab foils in them.

4. _____ Clean boric acid from the vial with small amounts of DI water with the syringe.

5. _____ Pour any remaining DI water in the vial into the tray.

6. _____ Repeat steps 4 and 5 until vial is clean.

7. _____ Replace lab foils in the tray and allow to absorb the solution.

8. _____ Place the tray on the drying plate.

Batch Number	Injection time
1	
2	
3	
4	
5	
6	
7	
8	
9	
10	

Table 3.5.1. Time of batch injections.

Procedure notes:

Signatures:

Test Operator 1

Name	Signature	Date

Test Operator 2

Name	Signature	Date

Quality Assurance Officer or Test Reviewer

Name	Signature	Date

3.6   TEST SECTION CLEANING

The following steps provide instructions for cleaning the precipitated boric acid from the test section

Prerequisites

- DI Water

Date:_____

1. _____ Turn on only a few rods to boil at a much lower rate than during the experiment.

2. _____ Remove the boric acid injection reservoir from the lower hot plate.

3. _____ Replace with a beaker containing hot DI water.

4. _____ Turn on the pump to a fairly high speed and inject until the water level is at the injection nozzles.

5. _____ Open the lower sampling valve and allow the solution to drain into the lower catch bucket.

6. _____ Close the valve when the water level reaches the plate.

7. _____ Repeat steps 4 through 6 until the water is completely clear and there is no longer precipitated boric acid on any of the test section interior surfaces.

8. _____ Turn off the heaters.

9. _____ Allow the facility to reach room temperature.

10. _____ Drain the facility.

Procedure notes:

Signatures:

Test Operator 1

Name	Signature	Date

Test Operator 2

Name	Signature	Date

Quality Assurance Officer or Test Reviewer

Name	Signature	Date

4.0   DATA ACQUISITION

The following steps provide instructions for measuring the samples to determine the concentration of the sample when it was taken during the procedure.

Date: _____

1. _____ Remove the samples from the drying plate and measure their masses with a scale (weight is assumed to be final when the scale reading is stable)

2. _____ Record their masses on table 4.0.1.

Tray Number	Acquisition time	Measured mass (g)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

Table 4.0.1. Test sample acquisition time and masses

Procedure notes:

Signatures:

Test Operator 1

Name	Signature	Date

Test Operator 2

Name	Signature	Date

Quality Assurance Officer or Test Reviewer

Name	Signature	Date