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HYDRAGARD® SAMPLING OF MELTER FEED SLURRY CONTAINING CST: A NONPROPRIETARY SUMMARY (U)

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1 Executive Summary

This report is a modified version of WSRC-TR-2000-00378, Revision 0. All proprietary data related to Crystalline Silicotitanate (CST) have been removed for this version. Two proprietary components of CST are identified only as PM_1 and PM_2 in this report, and their ranges along with the ranges of SiO_2 and TiO_2 also CST components are not revealed in accordance with the nondisclosure agreement between WSRC and UOP, Inc.

For a 26 wt% oxide sludge and 74 wt% oxide frit (this represents Phase 3 and is defined as the baseline case), an ~9.5% enrichment of sludge and a 3.4% depletion of frit were seen in Hydragard® samples relative to grab samples. Two campaigns were carried out with CST present in the feed. The Phase 1 campaign had the same solids wt% in the slurry (~46%) as the baseline case, with a sludge loading of 26 wt%, CST loading of 10 wt%, and a frit loading of 64 wt%, all in wt% oxide in the glass. Phase 2 used the same feed concentrations as Phase 1, but reduced the solids wt% in the slurry to 42%. The 10 wt% CST oxide loading is assumed to be conservative (i.e., the actual flowsheet will most likely use less than 10 wt% CST).

The results of this study indicate that introduction of size-reduced CST into a melter feed slurry of frit and sludge does not enhance the enrichment of sludge or the depletion of frit observed in the baseline case for the Hydragard® vs. the grab samples. In fact the results suggest that the differentiation of sludge and frit observed with the Hydragard® sampler may be mitigated slightly in the presence of CST.

The sludge enrichment was reduced to 8.1% in Phase 1 and 5.5% in Phase 2 relative to the 9.5% observed in the baseline case. Frit depletion decreased from 3.4% in the baseline case to 1.5% in Phase 1 and 2.2% in Phase 2. However, the mass balance was not as good for Phases 1 and 2 as it was for the baseline case. If a mass balance based on the enrichments observed for sludge and CST is assumed, then Phase 1 would have a frit depletion of 4.5% and Phase 2 a frit depletion of 3.2 wt%.

CST behaves much like the sludge does in terms of differentiation in the Hydragard® sampler. That is, CST is enriched in the Hydragard® samples relative to the grab samples in roughly the same percentage as the sludge. For Phase 1 the CST was enriched by 7.7% while for Phase 2, it was enriched by 6.7%. These enrichments for CST are close to the enrichments observed for the sludge (8.1% for Phase 1 and 5.5% for Phase 2).

In order to make a valid comparison between the Hydragard® samples and the grab samples, it was also necessary to prove that:

(1) The tank was homogeneously mixed,

(2) The recirculation flowrate did not significantly influence the sample results from the Hydragard® sampler, and

(3) The sampling time did not affect the Hydragard® results.

Tank homogeneity was proven by taking grab samples at the top and the bottom of the tank, analyzing the resulting samples, and statistically comparing the results. This analysis demonstrated that the system used in this task produced a well-mixed tank.

The recirculation flow rate was set at 4, 7 and 10 gallons per minute (gpm), and samples were taken at each flow rate. These flow rates cover the anticipated range for DWPF operations. Statistical analysis of the compositional results demonstrated that any effect of flow rate was minimal and did not alter the conclusions of this study.

During an additional set of tests, the sampling time was randomly varied between 5, 10, 20, 40 and 60 seconds. Analysis of results from samples taken at these times demonstrated no significant dependence of the sample composition on sampling time.

Rheological properties of the sludge-frit melter feed and the sludge-frit with size-reduced CST melter feed were measured. Adding CST to the slurry significantly increased the yield stress. However, the consistencies of both types of slurry were comparable implying that once the slurries were flowing they behaved similarly.

2 Introduction

Ion exchange using Crystalline Silicotitanate (CST) is one of the alternative processes being evaluated for removal of cesium from the aqueous fraction of high level waste currently stored at the Savannah River Site (SRS). The proposed process using CST ion exchange would pass filtered salt solution through a fixed bed of CST that would sorb the cesium. The spent resin, loaded with cesium and highly radioactive, would then be slurried out of the column and ultimately blended with the sludge waste stream in the Defense Waste Processing Facility (DWPF) Sludge Receipt and Adjustment Tank (SRAT) for incorporation into waste glass for permanent disposal. The product of the SRAT process is blended with glass formers (frit) and concentrated in the Slurry Mix Evaporator (SME). The SME represents the final control point in the glass making process. This means that the slurry in the tank must be homogeneously mixed and representatively sampled to determine the composition. Significant errors in the composition measurement may result in producing unacceptable glass product.

The contents of the SME are sampled using a Hydragard® sampling valve to direct a stream of slurry into a 15-ml "peanut" vial. The slurry is pumped out of the tank and returned to the tank through a recirculation loop. DWPF maintains this recirculation flow between 4 and 10 gpm. Although 7 gpm is nominally the average recirculation flow, the flow can vary between 4 and 10 gpm. The Hydragard® valve draws a side stream from this recirculation flow and directs the stream through the sampling valve. The stream flows through the sample vial and overflows into the Recycle Collection Tank (RCT). After a preset time, usually 40 seconds, the Hydragard® valve is closed, and a sample of the slurry is trapped in the vial. Chemical analyses of these samples form the basis for glass quality assurance.

To obtain a workable pressure drop across the ion exchange column, the CST has been engineered into a particle approximately 500 – 700 μm in diameter. Preliminary considerations indicated that several questions needed to be addressed to evaluate the impact of adding the CST particles to the DWPF slurry mixture:

1. Can the CST be mixed homogeneously into the sludge-frit slurry using the current DWPF agitation system? Frit particles are in the size range of 80 to 200 mesh (177 – 74 μm) so as-received CST is considerably larger. Sludge particles are typically in the range of 1 – 5 microns or smaller.

2. Can the Hydragard® valve obtain representative samples from a melter feed slurry that contains CST? Initial testing in 1999 [1] using a mock-up of the DWPF Hydragard® sampling system with a melter feed of sludge simulant and frit that also contained approximately 10 wt% as-received CST revealed that the Hydragard® sampler rapidly plugged because of the large particle size [1]. As a result of these tests, it was evident that the particle size of spent CST resin would have to be reduced before blending with the DWPF sludge stream. The testing was repeated with CST particles that were size-reduced to a maximum size less than 177 μm. In these

experiments, some bias toward frit depletion in the Hydragard® samples was observed.

3. Does the recirculation flow rate have an influence on the Hydragard® sample composition? The opening of the Hydragard® valve is positioned at a 90° angle to the recirculation flow stream. It has been speculated that the larger and heavier frit and CST particles will have a more difficult time making this abrupt change in flow direction than the sludge and water. This effect could lead to non-representative sampling of the slurry and the effect could possibly be more pronounced at higher recirculation flow rates.

4. Does the time that the Hydragard® valve is left opened have an influence on the composition of the sample? In previous testing and in DWPF operations a decrease in the flow through the sample valve over the time that the valve is opened has been observed. It is possible that the rapidly settling CST may preferentially segregate in the sample vial over time changing the composition of the sample.

As an initial estimate of the required particle size, it was decided that CST size reduced such that the maximum particle size is less than the largest frit particle would likely have both mixing and sampling properties adequate for processing in the DWPF. A Task Technical and QA Plan was written and approved to address these and other issues on interfacing CST ion exchange with the DWPF flowsheet [2]. As a part of this plan, the Savannah River Technology Center (SRTC) was tasked with the responsibility of testing Hydragard® sampling of melter feed slurries containing CST.

This report is a modified version of WSRC-TR-2000-00378, Revision 0. All proprietary data related to Crystalline Silicotitanate (CST), an ion sorbent produced by UOP, Inc., have been removed for this version. Two proprietary components of CST are identified only as PM_1 and PM_2 in this report, and their ranges along with the ranges of SiO_2 and TiO_2, also CST components, are not revealed in accordance with the nondisclosure agreement between WSRC and UOP, Inc.

2.1 Review of 1994 Hydragard® Test Results

During the DWPF startup program, Steimke [3] tested the efficiency of the Hydragard® system for sampling sludge-frit slurries. In the original Hydragard® design the valve stem protruded into the recirculation flow stream when the valve was opened. This configuration was found to create a bias toward frit enrichment in samples collected at low flow rates through the vial. That is, when compared to grab samples collected from the test tank, samples collected by the Hydragard® valve were higher in frit and lower in sludge. The bias was significant when the flow rate through the vial was less than 1 gpm. Based on this finding the Hydragard® valve was redesigned so that the valve stem would be flush with the side of the recirculation line when the valve was opened. This modification significantly reduced the sampling bias. However, a slight bias toward frit depletion in the Hydragard® samples was observed with the modified design. This was interpreted as a tendency for the larger and heavier frit particles to bypass the valve opening and not be sampled with the same efficiency as the sludge components. Because the sampling bias was reduced, Hydragard® valves with the modified design were installed on the DWPF SME.

To facilitate the testing, Steimke devised a method of assessing the frit content of the samples through the sample density. This simple method was used to evaluate the sampling behavior for much of the testing. To confirm that the density method gave a reliable indication of sampling behavior, some samples were also analyzed for a limited elemental composition. Aluminum and iron were chosen as markers for the sludge and lithium and silicon for the frit. Two different melter feed slurries (designated as TNX and DWPF) representing slightly different simulants of DWPF melter feed were tested during the 1994 program. At a density of 1.35 g/ml and a temperature of 25 C, the TNX slurry had a yield stress of 105 dyne/cm^2 and a consistency of 15.7 cp. At a density of 1.38 g/ml and a temperature of 25 C, the DWPF slurry had a yield stress of 83 dyne/cm^2 and a consistency of 17.9 cp. The testing was performed over a range of total solids in the slurry between 35% and 52%.

In Table 2.1, some of the compositional data reported by Steimke are reevaluated to estimate the bias toward frit depletion in the Hydragard® system observed during the 1994 testing. The evaluation is based on the ratio of lithium (Li) to iron (Fe) observed in grab samples compared to the ratio measured in the Hydragard® samples. If the composition of the Hydragard® samples is identical to that of the grab samples, both elemental ratios will be the same and the ratio of the ratios will be unity. In fact, as shown in Table 2.1, the Li/Fe ratio in the Hydragard® samples is about 6% less than the ratio in the grab samples indicating that the samples are somewhat lower in frit.

Table 2.1 1994 Hydragard® Test Results

Test	n	Li/Fe Grab	Li/Fe Hydragard	Hydragard/ Grab	% Difference
DWPF Slurry	8	0.221	0.208	0.944	5.61
TNX Slurry	14	0.228	0.214	0.938	6.23

It should be noted that the percent difference in Table 2.1, based on the ratio of the elemental ratios, is not the same as a bias toward frit depletion or sludge enrichment. This is demonstrated as follows. First define the following terms (on a dried basis):

F ....frit mass fraction in slurry
$S = 1 - F$ ....sludge mass fraction in slurry
L ....lithium mass fraction in frit
I ....iron mass fraction in sludge
W ....sample mass

Then the lithium to iron ratio measured in either the Hydragard® or grab sample is

$$\left.\frac{Li}{Fe}\right|_{H,G} = \frac{L\,F\,W}{I\,S\,W} = \left.\frac{L\,F}{I\,S}\right|_{H,G} \qquad (2.1)$$

Taking the ratio of the two measurements and canceling the common lithium and iron fractions, since the same material is sampled in both cases, gives

$$\frac{(Li/Fe)_H}{(Li/Fe)_G} = \frac{(F/S)_H}{(F/S)_G} = \left(\frac{F}{1-F}\right)_H \left(\frac{1-F}{F}\right)_G \tag{2.2}$$

We will assume that the grab sample is measuring the true amount of frit and sludge in the slurry. Then, taking the frit in the Hydragard® sample to be some fraction x of the frit in the grab sample, Eq. (2.2) becomes

$$\frac{(Li/Fe)_H}{(Li/Fe)_G} \equiv R = x\left(\frac{1-F}{1-xF}\right) \tag{2.3}$$

Solving Eq. (2.3) for x gives the relationship

$$x = \frac{R}{1-F(1-R)} \tag{2.4}$$

Similarly, taking the sludge in the Hydragard® sample to be some fraction y of the sludge in the grab sample, and performing the same analysis leads to the relationship

$$y = \frac{1}{R+(1-F)(1-R)} \tag{2.5}$$

From Table 2.1, the ratio R is, on average, approximately 0.941. Frit is typically 66% to 75% of the total slurry mass. Assuming $F = 0.7$, Eq. (2.4) gives $x = 0.982$ and Eq. (2.5) gives $y = 1.043$ or about 1.8% less frit and 4.3% more sludge in the Hydragard® samples than in the grab. DWPF has analyzed this effect and concluded that sampling biases on the order of 5% are acceptable.

These results and the more extensive density measurements reported by Steimke indicate that samples collected using the modified Hydragard® valve would be expected to show some small bias toward frit depletion and, correspondingly, some sludge enrichment over simultaneous grab samples.

2.2 Review of 1999 Hydragard® Test Results

As a part of the evaluation of the CST ion-exchange process it was determined that Hydragard® sampling should be retested with a melter feed slurry that contained CST. A mockup of the DWPF sampling system was built in the SRTC Thermal Fluids Laboratory (TFL) as described by Qureshi [1]. The mockup used a full scale sampling valve but a small (1/240th scale) feed tank and reduced length recirculation and sampling lines. These tests had as their objectives:

- Determine if the Hydragard® sampling system is capable of sampling sludge-frit slurry without any CST. This test was intended to verify the experimental setup.

- Determine if the Hydragard® sampling system is capable of sampling as-received CST, sludge and frit slurry. As mentioned previously, the as-received CST particles were too large to pass through the sampling valve and sufficient size reduction did not occur during the DWPF process to allow use of as-received CST in DWPF feed material. Therefore, representative sampling of the melter feed slurry containing as-received CST was not achieved and these tests will not be discussed further in this report. In the following discussion, "melter feed slurry containing CST" will be understood to refer to size-reduced CST.

- Determine if the Hydragard® sampling system is capable of sampling a size-reduced CST, sludge and frit slurry. Anticipating that as-received CST could not be sampled, melter feed with CST size-reduced to have a maximum particle size less than the largest frit particle was also prepared and tested.

To accomplish this test program, three batches of melter feed material were prepared in the Glass Feed Preparation System (GFPS) in 1999 as described by Koopman and Lambert [4]. The sludge simulant was based on the composition of Tank 42 sludge with 110% of the expected noble metals. Frit addition for the sludge-only run targeted 65 wt% frit and 35 wt% sludge as oxides in the glass product. Frit addition for the CST-sludge runs targeted 64 wt% frit, 10 wt% CST and 26 wt% sludge as oxides in the glass. Frit 200 was used for the sludge only run since this is the frit normally used in DWPF operations. Frit 202 was added to the CST-sludge slurry since this is similar to the frit that would be used during coupled DWPF operation. (The particle sizes for Frit 200 and 202 are essentially equivalent.)

The CST used was IONSIV® IE-911 (UOP, LLC, Molecular Sieves Division, Des Planes, IL), Lot Number 999098810006. The CST was pretreated by washing with water to remove fines and washing with a caustic solution to condition the resin. The CST was also loaded with cesium and noble metals to simulate the condition of the material following ion-exchange. The size-reduced CST was prepared as described by Baich [5] to have a maximum particle size smaller than 177 μm so that the CST particles in the slurry would be no larger than the frit particles. Figure 2.1 shows the size distribution of the size-reduced CST (volumetric mean diameter of 15 μm).

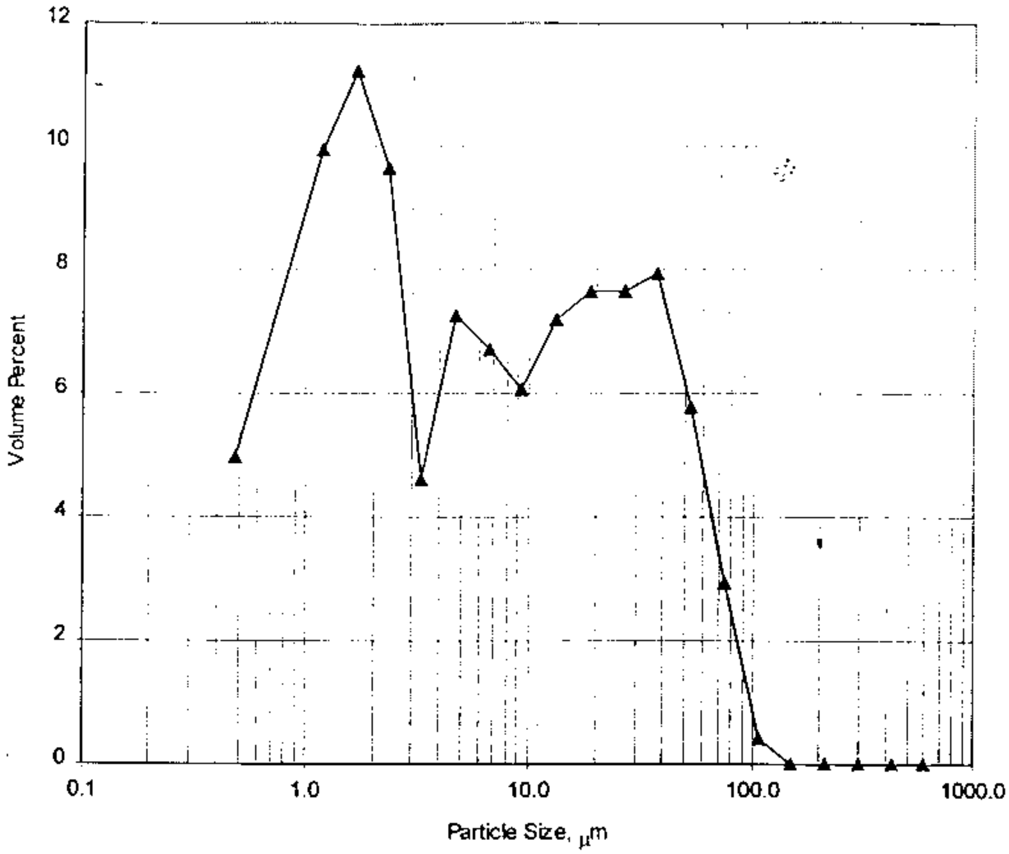

Figure 2.1 CST particle size distribution.

The test slurries were diluted to a total solids content of approximately 40 wt% for the 1999 tests. This somewhat low solids content was dictated by slurry pump limitations during this series of tests. Recirculation flow rates of 4, 6, 8 and 10 gpm were tested. A limited number of Hydragard® and grab samples were collected during the testing. As in 1994, a complete chemical analysis was not performed. The samples were analyzed for aluminum and iron to determine the sludge content, lithium and silicon to determine the frit content and titanium for the CST. Grab samples were collected from both high and low positions in the mixing tank to provide information on slurry homogeneity and to compare to Hydragard® samples. The Hydragard® valve was left opened 40 seconds during each sample collection to replicated DWPF operating procedures.

A summary of results from the 1999 testing is presented in Table 2.2. These results are presented in terms of the lithium to iron ratios for direct comparison to the 1994 data. The values in Table 2.2 are presented to more significant digits than those originally reported in [1]. The measurements were reported with more precision after the final report on that test program was issued.

Table 2.2 1999 Hydragard® Test Results

Test	Samples	n	Li/Fe Grab	Li/Fe Hydragard	Hydragard/ Grab	% Difference
Without CST	**Grab**	**4**	**0.121**			
	4 gpm Hydragard	5		0.121	1.006	-0.62
	6 gpm Hydragard	5		0.121	1.002	-0.17
	8 gpm Hydragard	5		0.123	1.020	-2.01
	10 gpm Hydragard	5		0.117	0.973	2.74
	Overall Average	**20**		**0.120**	**1.000**	**-0.02**
With CST	**Grab**	**4**	**0.239**			
	4 gpm Hydragard	5		0.208	0.869	13.11
	6 gpm Hydragard	5		0.218	0.912	8.82
	8 gpm Hydragard	5		0.216	0.904	9.58
	10 gpm Hydragard	5		0.225	0.939	6.08
	Overall Average	**20**		**0.217**	**0.906**	**9.40**

The results in Table 2.2 indicate that without CST in the slurry the Hydragard® and grab samples are in almost perfect agreement. This result is contrary to what was observed during the earlier testing in 1994 (see Table 2.1). Table 2.2 also indicates that with CST in the melter feed slurry the Hydragard® samples are significantly lower in frit than the grab samples. On average, the observed bias toward frit depletion in the 1999 tests is somewhat greater than that observed in 1994. Using Eqs. (2.4) and (2.5) with $F = 0.64$ would indicate about 3.6% less frit and 6.4% more sludge in the Hydragard® samples than in the grab samples. The test results with CST summarized in Table 2.2 also appear to show a trend toward poorer frit sampling at lower recirculation flows and a lower bias at higher flows. This trend is again contrary to the assumption that the bias is caused by the inability of the larger and heavier frit particles to change flow direction and enter the sampler. If this is correct, it would be expected that the bias toward frit depletion would increase as the flow rate increased and the frit particles had greater momentum in the direction of the recirculating flow. The testing in 1999 also showed that, as indicated by the titanium, CST was sampled in the same ratio as the sludge components and did not follow the pattern of frit depletion in the Hydragard®. These data suggest, but do not conclusively prove, that the presence of CST in the slurry is influencing the sampling behavior and perhaps exacerbating the tendency toward frit depletion in the Hydragard® samples.

By comparing high and low grab samples from the feed tank it was concluded that the slurry was well mixed during the sampling experiments. It was also observed that the sample loop flow decreased by as much as 60% during the 40 seconds that the sample valve was opened. This and the observation that the flowrate could be temporarily recovered by tapping on the valve suggested that the sample line was plugging. Table 2.3 shows a comparison of the rheological properties measured on the sludge-frit melter feed and the sludge-frit with size-reduced CST melter feed. Adding CST to the slurry significantly increases the yield stress. However, the consistencies of both types of slurry are comparable implying that once the slurries are flowing they should behave similarly.

Table 2.3 Rheological Properties of Melter Feed Slurries

Slurry	Total Solids (wt%)	Yield Stress (dynes/cm^2)	Consistency (cp)
Sludge-frit-CST	50.5	390	34
	47.0	190	24
	42.0	90	17
Sludge-frit	50.0	290	39
	46.0	110	22
	39.1	60	9

2.3 Basis for 2000 Testing

Based on a technical review of the 1999 test results it was concluded that additional testing was required to determine the effect of size-reduced CST on Hydragard® performance. A statistical analysis of the 1999 results indicated that insufficient data was obtained to draw conclusions at a 95% confidence level. Therefore, plans were made to obtain better sampling data using statistically designed experiments comparing Hydragard® and grab samples from both sludge-frit-CST and sludge-frit melter feed slurry. The samples would be analyzed for a full set of constituent elements to be certain that no analytical problems obscured the results. Since the chemical analysis would be both expensive and time consuming, it was decided to reduce the recirculation flows tested to 4, 7 and 10 gpm. This test matrix still covers the expected range of DWPF operations and includes the nominal flow.

In addition to comparing Hydragard® and grab samples from both slurries, the following tests were planned:

- With both feed slurries, obtain a set of profiles of sample flowrate as a function of elapsed time after valving in the sample loop for each recirculation flowrate. This test was designed to determine if the presence of CST is the cause of the apparent sampler plugging.

- At the nominal slurry recirculation flowrate of 7 gpm, obtain Hydragard® samples at various times (5, 15, 30, 40 and 60 seconds) after valving in the sample loop to determine if the sample composition changes over time.

The same melter feed slurries prepared during the 1999 testing were used in the extended test program with the exception that Frit 200 was added to the sludge-frit melter feed to adjust the waste and frit oxide contents to 26 wt% and 74 wt% respectively. This adjustment made this material consistent with the sludge-frit-CST melter feed. Calculations indicated this frit adjustment would produce a melter feed with a total solids content of 45 to 46 wt%. Initial plans were to test the CST melter feed at 52, 47 and 42 wt% solids to span the range of normal DWPF operating conditions.

3 Experimental Methods

3.1 Experimental Apparatus

The Hydragard® sampling tests were conducted in a 1/240th scale mockup of the Slurry Mix Evaporator (SME) of DWPF. The SME tank dimensions were reduced by a factor of 6.213. Additionally, the tank internals (coils and agitator) were also scaled down by this factor. The agitator speed was determined by preserving power per unit volume. This yielded an agitator speed of 440 rpm to represent full scale mixing conditions. Note that the full-scale agitator speed is 130 rpm. The 1/240th scale was chosen to match the Glass Feed Prep System batch size, which produced the sludge-frit-CST batches utilized in the tests reported here.

Figure 3.1 shows the 1/240th scale facility. The slurry tank is 23.2" in diameter and 31" high. The tank is equipped with an agitator system consisting of a variable speed motor drive (0-700 rpm) and two prototypic impellers (an upper axial impeller and a lower radial impeller of 5.8" diameter). For Hydragard® sampling, the critical slurry flow paths were replicated to a full scale. A 1.5" and 1" diameter PVC circulation loop containing a 5 HP centrifugal pump and a flow meter provided slurry circulation ability. A Hydragard® sampling loop (0.75" transfer line) draws slurry from the 1" loop and returns it to the tank as shown in Figure 3.1. The Hydragard® overflow is also returned to the tank via a flow meter. The Hydragard sampler could be isolated using three ball valves for water/air flushing as needed during the test program. All of these ball valves are fitted with injection ports for air/water flushing.

A grab-sampling device was fabricated to collect ~ 15 ml of slurry samples from the tank. It consisted of a peanut vial, a rubber stopper and support hardware to open or close the stopper as needed. The grab sampler could obtain samples at any desired depth and circumferential location.

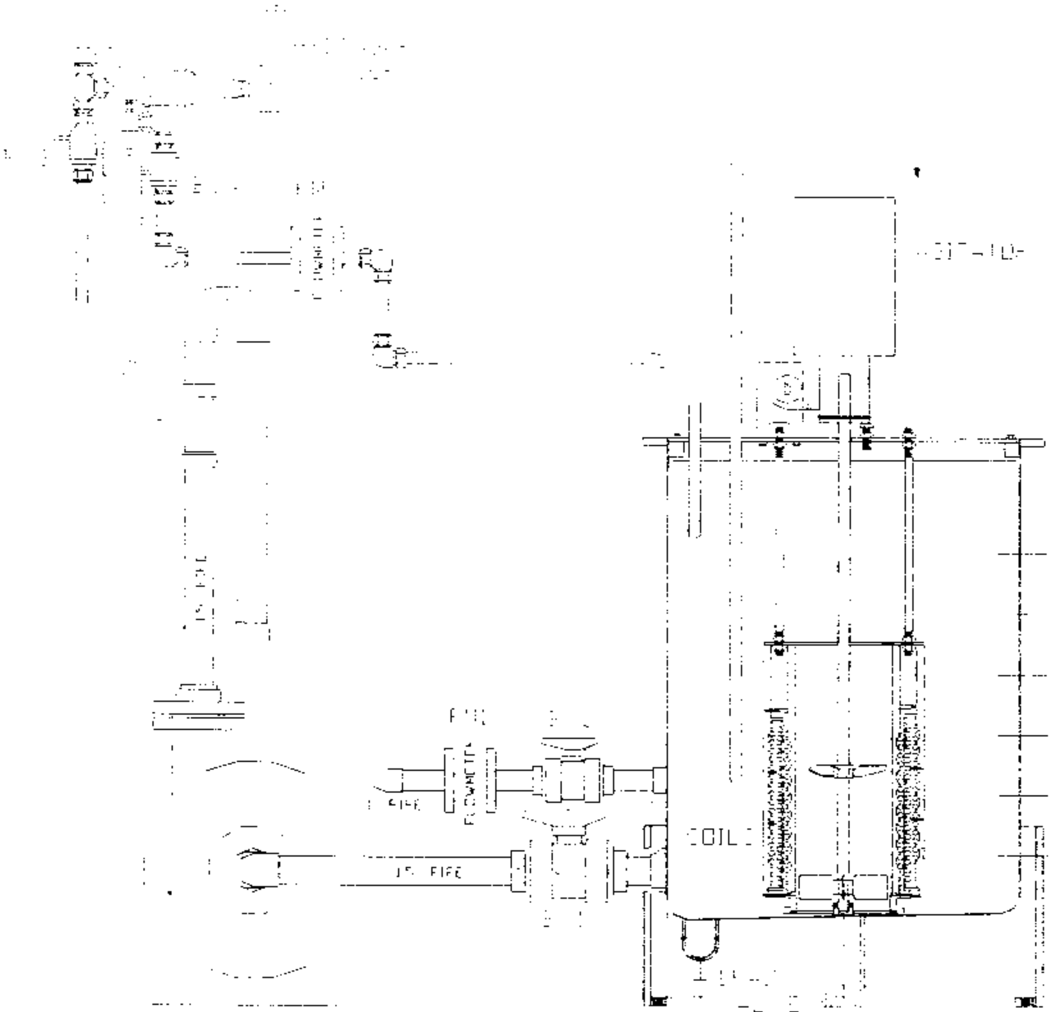

Figure 3.1 CST Mixing and Sampling Test Facility

3.2 Experimental Procedures

Initially, it was planned to test slurries with three different wt% solids – 51%, 47% and 42%, starting with 51 wt% solids and then diluting it for subsequent tests. It was observed that the 51 wt% solids slurry could not be adequately mixed in the test facility. This was mainly attributed to the close spacing of the cooling coils. Note that the 1/240th test facility was designed by scaling down the SME tank dimensions by a factor of 6.213. The cooling coil diameter and spacing were also scaled down by this factor. The high yield stress of the slurry and narrow coil spacing prevented adequate mixing even at high agitation speeds. Another factor that contributed to poor mixing of this batch was the total mass. Only about 254 lbs of 51 wt% solids slurry were available. This was insufficient to cover the cooling coils completely. Typical batch size for good mixing is around 320 lbs of slurry. As a result of these observations and considerations, it was decided to test only 47 wt% and 42 wt% solids slurries containing CST. After testing these two batches of CST based slurries, a batch of sludge-frit slurry (at ~46 wt%) was also tested to provide baseline performance of the Hydragard® sampler without any CST present in the system.

For each of the two slurry batches, it was decided to run two sets of experiments. In the first set the Hydragard® valve opening time was fixed at 40 seconds (to match the plant conditions). A total of 72 slurry samples were obtained in this set. This included 18 grab samples and 54 Hydragard® samples at the Hydragard® transfer line flow rates of 4, 7 and 10 gpm. The agitator speed was set at 600 rpm for all tests. This agitator speed (which is above the equivalent full-scale speed of 440 rpm) was selected to ensure a well-mixed tank. The test conditions were randomized to preclude any systematic error due to any drift in the system. The system pressure downstream of the Hydragard® valve (see location of pressure tap in Fig. 3.1) was set at 14 psi +/- 0.5 psi for the entire test program to match DWPF operating conditions. Table 3.1 provides the sample sequence for 47 wt% solids slurry.

Table 3.1 Pull Sequence for Samples At a Targeted 47 wt% Solids

Test #	Grab Sample Location/ FM2 Flow Rate	Number of Samples	Sample ID
1	High Grab	5	GRS-47-01 to GRS-47-05
2	4 gpm	6	H04-47-01 to H04-47-06
3	10 gpm	6	H10-47-01 to H10-47-06
4	7 gpm	6	H07-47-01 to H07-47-06
5	Low Grab	4	GRS-47-06 to GRS-47-09
6	7 gpm	6	H07-47-07 to H07-47-12
7	4 gpm	6	H04-47-07 to H04-47-12
8	10 gpm	6	H10-47-07 to H10-47-12
9	High Grab	4	GRS-47-10 to GRS-47-13
10	10 gpm	6	H10-47-13 to H10-47-18
11	7 gpm	6	H07-47-13 to H07-47-18
12	4 gpm	6	H04-47-13 to H04-47-18
13	Low Grab	5	GRS-47-14 to GRS-47-18

The second set of tests was conducted to determine the effect of Hydragard® valve opening time on slurry composition. It was observed that the flow through the Hydragard® sampler decreased with time. It starts out at around 1.2 – 1.4 gpm and within few seconds it drops to a value below 0.5 gpm. The nominal sampler valve opening time is 40 seconds as described above. Figures 3.2 and 3.3 provide plots of the flow rate versus time for two of the test phases (there were limited data for the Phase 2 slurry, but the behavior seen for that phase was similar to that seen for Phase 1).

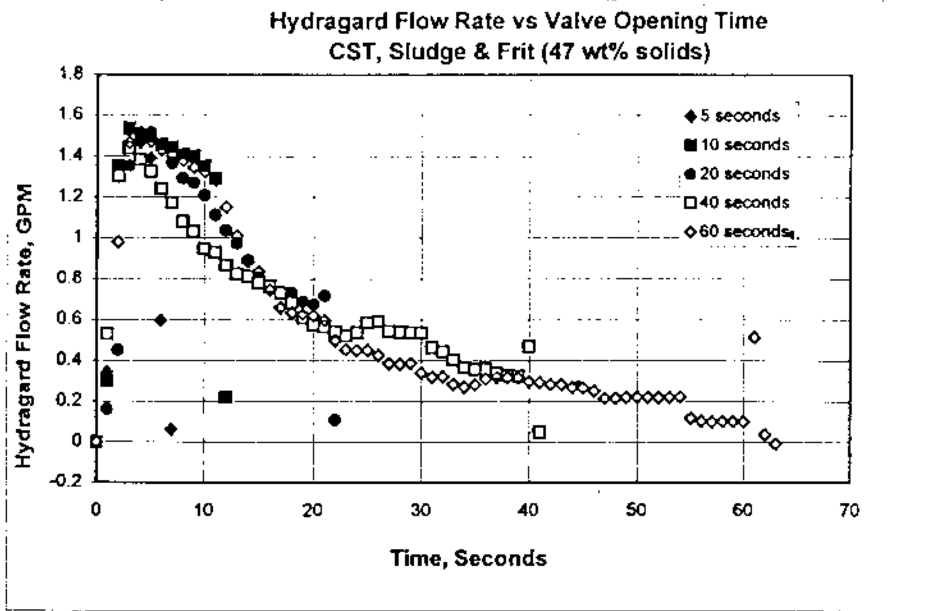

Figure 3.2 Hydragard® Flow Rate versus Valve Opening Time for Phase 1 (Sludge-Frit-CST at ~ 47 wt% Total Solids)

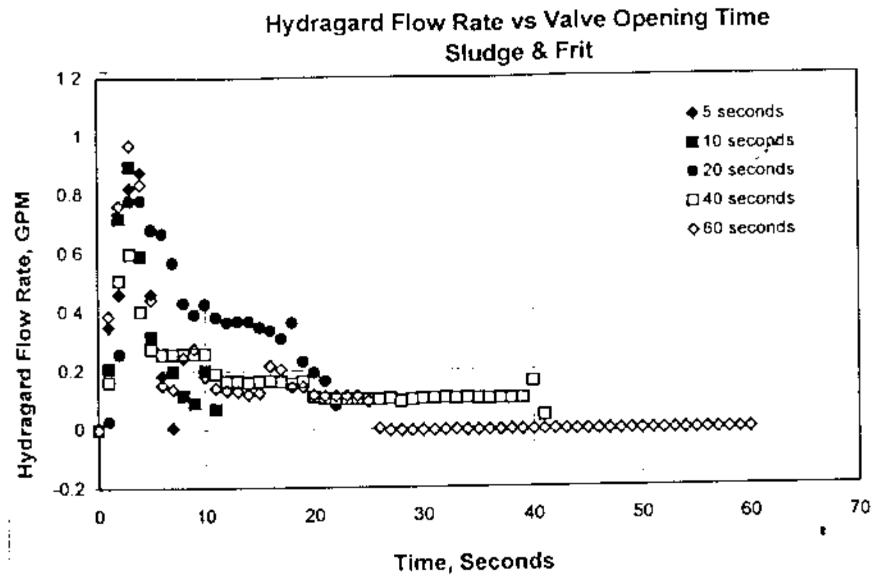

Figure 3.3 Hydragard® Flow Rate versus Valve Opening Time for Phase 3 (Sludge-Frit Only at ~ 47 wt% Total Solids)

Note that in the presence of CST (Figure 3.2), the maximum flow through the Hydragard® is quite high (~ 1.5 gpm) and that the flow drops gradually to a lower value. While for the sludge-frit only slurry (Figure 3.3), the maximum flow is less than 1 gpm and the flow through the value drops drastically after about 10 to 20 seconds. Thus, the valve plugging is more pronounced for the sludge-frit only slurry (i.e., for Phase 3).

In order to determine the effect of valve opening time on sample composition, it was decided to set the valve opening time at 5, 10, 20, 40 and 60 seconds. The flow rate to the transfer line (as measured by flow meter, FM2) was set at 7 gpm. At each of these conditions, four samples were obtained, thus yielding a total of 20 samples. These 20 samples were also pulled in a randomized manner as given below in Table 3.2.

Table 3.2 Pull Sequence for Samples At a Targeted 47 wt% Solids for Various Valve Opening Times

Pull Order	Valve Opening Time, Seconds	Sample ID
1	5	slt05-1
2	10	slt10-1
3	20	slt20-1
4	40	slt40-1
5	10	slt10-2
6	5	slt05-2
7	60	slt60-1
8	20	slt20-2
9	40	slt40-2
10	60	slt60-2
11	10	slt10-3
12	60	slt60-3
13	5	slt05-3
14	10	slt10-4
15	20	slt20-3
16	40	slt40-3
17	5	slt05-4
18	60	slt60-4
19	40	slt40-4
20	20	slt20-4

After the two test series with the 47 wt% solids slurry were completed, the slurry was diluted with 29 lbs of water, yielding 42 wt% solids. After complete agitation and mixing, 38 lbs of slurry were removed to maintain the slurry level in the tank in order to preserve the mixing behavior. Grab samples were taken to confirm the concentration of solids in the slurry after the dilution. Similar to the 47 wt% slurry, two test series were conducted with 42 wt% slurry. In the first series, 72 samples were pulled according to the sequence given in Table 3.3.

Table 3.3 Pull Sequence for Samples At 42 wt% Solids

Test #	Grab Sample Location/ FM2 Flow Rate	Number of Samples	Sample ID
1	High Grab	5	GRS-42-01 to GRS-42-05
2	4 gpm	6	H04-42-01 to H04-42-06
3	10 gpm	6	H10-42-01 to H10-42-06
4	7 gpm	6	H07-42-01 to H07-42-06
5	Low Grab	4	GRS-42-06 to GRS-42-09
6	7 gpm	6	H07-42-07 to H07-42-12
7	4 gpm	6	H04-42-07 to H04-42-12
8	10 gpm	6	H10-42-07 to H10-42-12
9	High Grab	4	GRS-42-10 to GRS-42-13
10	10 gpm	6	H10-42-13 to H10-42-18
11	7 gpm	6	H07-42-13 to H07-42-18
12	4 gpm	6	H04-42-13 to H04-42-18
13	Low Grab	5	GRS-42-14 to GRS-42-18

The second series of test consisted of 20 Hydragard® samples pulled at various valve opening times. The flow rate in the Hydragard® loop transfer line was set at 7 gpm. The sample pull sequence is given in Table 3.4.

Table 3.4 Pull Sequence for Samples At 42 wt% Solids for Various Valve Opening Times

Pull Order	Valve Opening Time, Seconds	Sample ID
1	5	42-slt05-1
2	10	42-slt10-1
3	20	42-slt20-1
4	40	42-slt40-1
5	10	42-slt10-2
6	5	42-slt05-2
7	60	42-slt60-1
8	20	42-slt20-2
9	40	42-slt40-2
10	60	42-slt60-2
11	10	42-slt10-3
12	60	42-slt60-3
13	5	42-slt05-3
14	10	42-slt10-4
15	20	42-slt20-3
16	40	42-slt40-3
17	5	42-slt05-4
18	60	42-slt60-4
19	40	42-slt40-4
20	20	42-slt20-4

The third batch of slurry consisted of sludge and frit only. No CST was present in this batch. Similar to the above slurry batches, 18 grab and 54 Hydragard® samples were pulled in the sequence given in Table 3.5 below.

Table 3.5 Pull Sequence for Samples of Sludge-Frit Slurry

Test #	Grab Sample Location/ FM2 Flow Rate	Number of Samples	Sample ID
1	High Grab	5	GRS-sf-01 to GRS-sf-05
2	4 gpm	6	H04-sf-01 to H04-sf-06
3	10 gpm	6	H10-sf-01 to H10-sf-06
4	7 gpm	6	H07-sf-01 to H07-sf-06
5	Low Grab	4	GRS-sf-06 to GRS-sf-09
6	7 gpm	6	H07-sf-07 to H07-sf-12
7	4 gpm	6	H04-sf-07 to H04-sf-12
8	10 gpm	6	H10-sf-07 to H10-sf-12
9	High Grab	4	GRS-sf-10 to GRS-sf-13
10	10 gpm	6	H10-sf-13 to H10-sf-18
11	7 gpm	6	H07-sf-13 to H07-sf-18
12	4 gpm	6	H04-sf-13 to H04-sf-18
13	Low Grab	5	GRS-sf-14 to GRS-sf-18

The 20 samples of sludge-frit slurry at different valve opening times were pulled in the random order given in Table 3.6.

Table 3.6 Pull Sequence for Samples For Sludge-Frit Slurry for Various Valve Opening Times

Pull Order	Valve Opening Time, Seconds	Sample ID
1	20	spf20-1
2	10	spf10-1
3	40	spf40-1
4	5	spf05-1
5	60	spf60-1
6	5	spf05-2
7	40	spf40-2
8	20	spf20-2
9	60	spf60-2
10	10	spf10-2
11	10	spf10-3
12	60	spf60-3
13	5	spf05-3
14	20	spf20-3
15	40	spf40-3
16	10	spf10-4
17	40	spf40-4
18	60	spf60-4
19	20	spf20-4
20	05	spf05-4

4 Data Analysis

As described above, samples were taken over 3 primary phases of testing: a high-solids slurry containing CST (Phase 1), a low-solids slurry containing CST (Phase 2), and a sludge-frit-only slurry (Phase 3). For each of these primary phases, two groups of samples were acquired. The first group consisted of samples from the Hydragard® and grab samplers, while the second group consisted of samples only from the Hydragard. The first group of samples was used to investigate the main objectives of the study, while the second group was used to determine if the Hydragard® sample composition changes over time after the sample loop is valved in.

Samples drawn at the TFL during the various phases of this testing were submitted to the SRTC-Mobile Laboratory (ML) for vitrification and chemical analysis. A statistical review of each set of data generated by the SRTC-ML in support of the CST sampling study was conducted using JMP® Version 3.2.2 [6]. Samples of standard glasses were submitted to the SRTC-ML along with test samples for chemical analysis. This approach provided the opportunity to adjust (or correct) results from the test glasses for biases seen in the results from the standards. In addition, the SRTC-ML reported the concentrations of some of the oxides of interest by two different dissolution (or preparation) methods. Subsequently, there were different ways available to represent the glass compositions. The statistical review investigated the objectives of the study against the various compositional views. The analytical plans utilized by the SRTC-ML and the details of the statistical review for each phase were organized and reported by Edwards [7, 8, & 9].

Summaries of the information from those reports are provided in the sections that follow. Specifically, two compositional views are provided: original measurements (derived by averaging all available measurements for each oxide of interest) and bias-corrected measurements (derived by averaging the bias-corrected measurements for each dissolution for each oxide).

4.1 Tank Homogeneity

Tank homogeneity was determined for each phase by taking grab samples at the top and the bottom of the tank, analyzing the samples for chemical composition, and comparing the results. The tank contents were considered to be well-mixed if the composition of samples taken from the top of the tank were within 5% of those taken from the bottom of the tank for all oxides present in the glass at concentrations greater than 0.5 wt%. Exhibit A1 in Appendix A provides plots for each phase of the average oxide concentrations for samples taken at the bottom (00) and top (15) tank positions using the Grab sampler[1]. The statistical graphics, utilized in these plots, are described in Appendix B.

The comparisons between high and low grab samples were made for both measured and bias corrected (bc) measured compositions. The only statistically significant differences noted were for Cu and Cr, two components in the sludge that were present at very low concentrations. These variations are a reflection of the uncertainty in the analytical methods at the region near the detection limit and are therefore not meaningful. For the major sludge, frit and CST components, there are relatively small differences between the high and low grab samples.

The results (for the original and bias-corrected measurements) are summarized in Table 4.1, and they provide good evidence that the contents of the tank are homogeneously distributed (i.e., the tank was well mixed). Shading is used in Table 4.1 to help identify the primary source (sludge, frit, or CST) of each of the oxides. Note that Na_2O (bolded in the table) is present in each of the sources. The weight percent solids (wt% solids) results are also provided. The mean (or average) and standard deviation (Std Dev) of all samples taken by the Grab sampler at each of the two (low and high) tank locations are provided for each test phase. In addition, the differences between the high and low averages and % differences (relative to the low averages) are given.

The % differences provide the key information in assessing the homogeneity of the tank. An oxide whose average concentration (from the Low Grab samples) is greater than 0.5 wt% and whose % difference value is no more than 5% would be considered well mixed. The results in Table 4.1 indicate that the contents of the TFL vessel were well mixed for every major oxide for each test phase.

1 The bottom and top designations as "00" and "15" respectively, were selected so that they might be easily distinguished from the Hydragard® flowrate values of 4, 7, and 10. Thus, there is no physical interpretation of these numbers for the Grab sampler.

Table 4.1 Summary of High Versus Low Grab Sample Results

	Phase 1: Sludge-Frit-CST (High Solids)						Phase 2: Sludge-Frit-CST (Low Solids)						Phase 3: Sludge-Frit Only					
	Low Grab		High Grab		Differences		Low Grab		High Grab		Differences		Low Grab		High Grab		Differences	
Oxide	Mean	Std Dev	Mean	Std Dev	(H-L)	(H-L)/L	Mean	Std Dev	Mean	Std Dev	(H-L)	(H-L)/L	Mean	Std Dev	Mean	Std Dev	(H-L)	(H-L)/L
Al_2O_3 (wt%)	5.820	0.102	5.780	0.202	-0.040	-0.7%	5.930	0.128	6.015	0.092	0.085	1.4%	5.710	0.030	5.637	0.055	-0.073	-1.3%
B_2O_3 (wt%)	5.526	0.072	5.398	0.228	-0.128	-2.3%	5.576	0.157	5.719	0.086	0.142	2.5%	8.061	0.176	7.999	0.059	-0.062	-0.8%
CaO (wt%)	1.276	0.057	1.256	0.030	-0.020	-1.6%	1.246	0.027	1.263	0.037	0.017	1.4%	1.338	0.034	1.342	0.016	0.004	0.3%
Cr_2O_3 (wt%)	0.099	0.007	0.102	0.004	0.004	3.6%	0.098	0.006	0.101	0.006	0.003	3.1%	0.099	0.023	0.086	0.009	-0.013	-13.0%
CuO (wt%)	0.009	0.003	0.012	0.006	0.003	28.7%	0.019	0.006	0.022	0.007	0.003	15.3%	0.011	0.007	0.010	0.007	-0.001	-9.9%
Fe_2O_3 (wt%)	12.797	0.153	12.534	0.417	-0.263	-2.1%	12.466	0.343	12.631	0.194	0.165	1.3%	11.721	0.234	11.668	0.097	-0.053	-0.4%
K_2O (wt%)	0.130	0.005	0.129	0.008	-0.002	-1.3%	0.097	0.003	0.102	0.010	0.004	4.5%	0.123	0.005	0.121	0.004	-0.002	-1.5%
Li_2O (wt%)	3.992	0.063	3.938	0.161	-0.055	-1.4%	3.901	0.139	4.009	0.059	0.108	2.8%	3.413	0.050	3.384	0.018	-0.029	-0.9%
MgO (wt%)	2.010	0.051	2.013	0.040	0.002	0.1%	1.949	0.025	1.968	0.028	0.019	1.0%	2.248	0.034	2.228	0.014	-0.021	-0.9%
MnO (wt%)	1.519	0.022	1.495	0.044	-0.024	-1.6%	1.465	0.035	1.485	0.011	0.020	1.4%	1.483	0.021	1.478	0.020	-0.005	-0.4%
Na_2O (wt%)	8.573	0.087	8.647	0.289	0.074	0.9%	8.711	0.164	8.651	0.103	-0.060	-0.7%	10.555	0.148	10.471	0.260	-0.084	-0.8%
NiO (wt%)	0.166	0.003	0.163	0.005	-0.003	-2.0%	0.149	0.005	0.152	0.006	0.003	1.8%	0.148	0.002	0.145	0.005	-0.003	-2.1%
SiO_2 (wt%)						-1.4%						0.3%						-0.3%
TiO_2 (wt%)						-1.8%						1.2%						-0.9%
PM_1 (wt%)						-3.0%						0.1%						3.0%
PM_2 (wt%)						-4.0%						-0.1%						1.6%
wt% Solids	46.000	0.082	45.740	0.114	-0.260	-0.6%	42.525	0.050	42.240	0.055	-0.285	-0.7%	45.522	0.286	45.549	0.487	0.027	0.1%
Al_2O_3 bc (wt%)	6.176	0.054	6.011	0.162	-0.165	-2.7%	6.173	0.142	6.267	0.092	0.094	1.5%	5.912	0.060	5.862	0.069	-0.050	-0.8%
B_2O_3 bc (wt%)	5.503	0.080	5.370	0.170	-0.133	-2.4%	5.515	0.166	5.663	0.107	0.148	2.7%	7.982	0.184	7.923	0.059	-0.059	-0.7%
CaO bc (wt%)	1.205	0.052	1.180	0.035	-0.025	-2.0%	1.210	0.033	1.231	0.025	0.021	1.7%	1.316	0.045	1.328	0.024	0.012	0.9%
Cr_2O_3 bc (wt%)	0.111	0.001	0.109	0.004	-0.002	-1.8%	0.116	0.002	0.119	0.002	0.004	3.1%	0.108	0.024	0.093	0.003	-0.015	-14.1%
CuO bc (wt%)	0.010	0.003	0.012	0.006	0.003	27.5%	0.019	0.006	0.022	0.007	0.003	15.5%	0.011	0.006	0.010	0.007	-0.001	-5.8%
Fe_2O_3 bc (wt%)	12.614	0.109	12.502	0.437	-0.112	-0.9%	12.900	0.304	13.088	0.119	0.188	1.5%	11.896	0.289	11.765	0.146	-0.132	-1.1%
K_2O bc (wt%)	0.128	0.007	0.127	0.006	-0.001	-0.5%	0.091	0.003	0.095	0.009	0.004	4.3%	0.124	0.004	0.126	0.006	0.002	1.3%
Li_2O bc (wt%)	4.119	0.030	4.072	0.117	-0.047	-1.1%	4.125	0.140	4.247	0.042	0.121	2.9%	3.628	0.061	3.584	0.040	-0.043	-1.2%
MgO bc (wt%)	1.955	0.032	1.920	0.047	-0.035	-1.8%	1.986	0.025	2.008	0.026	0.021	1.1%	2.229	0.026	2.208	0.018	-0.021	-1.0%
MnO bc (wt%)	1.477	0.021	1.463	0.046	-0.013	-0.9%	1.494	0.030	1.515	0.009	0.021	1.4%	1.481	0.021	1.478	0.019	-0.002	-0.1%
Na_2O bc (wt%)	8.978	0.155	8.899	0.228	-0.079	-0.9%	8.628	0.104	8.546	0.154	-0.082	-0.9%	10.829	0.299	10.850	0.215	0.021	0.2%
NiO bc (wt%)	0.167	0.003	0.164	0.005	-0.003	-1.6%	0.156	0.005	0.159	0.005	0.003	2.0%	0.154	0.002	0.152	0.004	-0.003	-1.8%
SiO_2 bc (wt%)						-0.9%						0.4%						0.2%
TiO_2 bc (wt%)						-1.6%						1.4%						0.4%
PM_1 bc (wt%)						-3.0%						0.1%						3.0%
PM_2 bc (wt%)						-3.6%						-1.6%						3.7%

Sludge	CST	Frit

bc => bias-corrected measurements; Na_2O is bolded to indicate that it is present in sludge, frit, and CST; and SiO_2 is bolded to indicate that it is in the frit and CST.

4.2 Hydragard® Sampling

For sampling at DWPF, the slurry is pumped out of the tank and returned to the tank through a recirculation loop. DWPF maintains this recirculation flow between 4 and 10 gpm with a nominal average recirculation flow rate of 7 gpm. The Hydragard® valve draws a side stream from this recirculation flow and directs the stream through the sampling valve. Before comparing the results of the grab sample to the Hydragard® sample, it is important to demonstrate that the contents of the sample do not depend significantly on this recirculation flow rate. Therefore, samples were taken using the Hydragard® at 4, 7 and 10 gpm and analyzed for chemical composition.

Exhibit A2 in Appendix A provides plots and statistical graphics of the samples taken with the Hydragard® at the different flowrates by test phase. These results are summarized in Tables 4.2, 4.3, and 4.4.

Table 4.2 Summary of Comparisons Among the Hydragard® Samples at Different Flow Rates for Phase 1

	Phase 1: Sludge-Frit-CST (High Solids)							
	4 gpm		7 gpm		10 gpm		Differences	
Oxide	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	(Max-Min)	(Mx-Mn)/Mn
Al_2O_3 (wt%)	6.342	0.156	6.279	0.204	6.440	0.213	0.161	2.6%
B_2O_3 (wt%)	5.565	0.123	5.411	0.127	5.341	0.210	0.224	4.2%
CaO (wt%)	1.354	0.068	1.345	0.083	1.396	0.077	0.051	3.8%
Cr_2O_3 (wt%)	0.108	0.003	0.105	0.006	0.115	0.011	0.010	9.1%
CuO (wt%)	0.014	0.006	0.010	0.005	0.015	0.005	0.005	52.8%
Fe_2O_3 (wt%)	13.319	0.218	13.608	0.232	13.863	0.291	0.544	4.1%
K_2O (wt%)	0.143	0.008	0.141	0.005	0.150	0.016	0.008	6.0%
Li_2O (wt%)	4.027	0.070	3.969	0.064	3.877	0.121	0.150	3.9%
MgO (wt%)	2.027	0.058	1.992	0.042	2.022	0.046	0.035	1.8%
MnO (wt%)	1.580	0.021	1.601	0.021	1.641	0.039	0.061	3.9%
Na_2O (wt%)	8.533	0.263	8.499	0.155	8.619	0.254	0.120	1.4%
NiO (wt%)	0.176	0.004	0.181	0.011	0.186	0.012	0.010	5.6%
SiO_2 (wt%)								3.1%
TiO_2 (wt%)								1.9%
PM_1 (wt%)								3.1%
PM_2 (wt%)								3.4%
wt% Solids	45.167	0.312	44.756	0.340	44.144	0.500	1.022	2.3%
Al_2O_3 bc (wt%)	6.543	0.154	6.606	0.185	6.679	0.172	0.136	2.1%
B_2O_3 bc (wt%)	5.461	0.073	5.327	0.111	5.270	0.132	0.191	3.6%
CaO bc (wt%)	1.271	0.069	1.263	0.083	1.312	0.075	0.050	3.9%
Cr_2O_3 bc (wt%)	0.117	0.002	0.121	0.003	0.124	0.009	0.006	5.5%
CuO bc (wt%)	0.015	0.006	0.010	0.006	0.016	0.006	0.005	52.3%
Fe_2O_3 bc (wt%)	13.300	0.153	13.434	0.246	13.822	0.349	0.522	3.9%
K_2O bc (wt%)	0.139	0.007	0.136	0.005	0.146	0.014	0.010	7.5%
Li_2O bc (wt%)	4.102	0.066	4.037	0.061	3.976	0.077	0.127	3.2%
MgO bc (wt%)	1.967	0.034	1.962	0.018	1.951	0.023	0.016	0.8%
MnO bc (wt%)	1.554	0.016	1.565	0.023	1.609	0.043	0.055	3.6%
Na_2O bc (wt%)	8.864	0.181	9.017	0.203	8.922	0.141	0.153	1.7%
NiO bc (wt%)	0.178	0.004	0.182	0.011	0.188	0.013	0.010	5.5%
SiO_2 bc (wt%)								2.7%
TiO_2 bc (wt%)								2.0%
PM_1 bc (wt%)								3.1%
PM_2 bc (wt%)								3.8%

Sludge	CST	Frit

bc => bias-corrected measurements;
Na_2O is bolded to indicate that it is present in sludge, frit, and CST;
and SiO_2 is bolded to indicate that it is in the frit and CST.

Table 4.3 Summary of Comparisons Among the Hydragard® Samples at Different Flow Rates for Phase 2

	Phase 2 Sludge-Frit-CST (Low Solids)							
	4 gpm		7 gpm		10 gpm		Differences	
Oxide	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	(Max-Min)	(Mx-Mn)/Mn
Al_2O_3 (wt%)	6.208	0.140	6.390	0.181	6.375	0.284	0.182	2.9%
B_2O_3 (wt%)	5.560	0.153	5.594	0.171	5.446	0.167	0.148	2.7%
CaO (wt%)	1.284	0.030	1.342	0.037	1.334	0.059	0.058	4.5%
Cr_2O_3 (wt%)	0.102	0.007	0.108	0.008	0.110	0.010	0.008	7.5%
CuO (wt%)	0.019	0.007	0.021	0.004	0.021	0.006	0.002	9.3%
Fe_2O_3 (wt%)	12.953	0.265	13.386	0.375	13.405	0.550	0.452	3.5%
K_2O (wt%)	0.102	0.016	0.117	0.010	0.117	0.016	0.015	15.1%
Li_2O (wt%)	3.940	0.082	3.877	0.123	3.833	0.125	0.107	2.8%
MgO (wt%)	1.943	0.022	1.930	0.031	1.946	0.038	0.016	0.8%
MnO (wt%)	1.526	0.032	1.568	0.047	1.579	0.065	0.052	3.4%
Na_2O (wt%)	**8.617**	**0.054**	**8.729**	**0.169**	**8.775**	**0.157**	**0.158**	**1.8%**
NiO (wt%)	0.157	0.007	0.164	0.008	0.163	0.007	0.007	4.6%
SiO_2 (wt%)								**2.8%**
TiO_2 (wt%)								**2.3%**
PM_1 (wt%)								**1.7%**
PM_2 (wt%)								**1.7%**
wt% Solids	42.989	2.450	41.489	0.641	41.322	0.932	1.667	4.0%
Al_2O_3 bc (wt%)	6.470	0.142	6.647	0.199	6.629	0.304	0.177	2.7%
B_2O_3 bc (wt%)	5.497	0.158	5.530	0.155	5.380	0.181	0.150	2.8%
CaO bc (wt%)	1.252	0.029	1.300	0.047	1.289	0.058	0.048	3.8%
Cr_2O_3 bc (wt%)	0.122	0.004	0.126	0.004	0.128	0.006	0.006	4.6%
CuO bc (wt%)	0.020	0.007	0.022	0.004	0.021	0.006	0.002	9.0%
Fe_2O_3 bc (wt%)	13.411	0.308	13.839	0.424	13.851	0.595	0.440	3.3%
K_2O bc (wt%)	0.096	0.014	0.109	0.009	0.109	0.014	0.014	14.3%
Li_2O bc (wt%)	4.174	0.076	4.094	0.118	4.045	0.121	0.129	3.2%
MgO bc (wt%)	1.984	0.019	1.966	0.037	1.983	0.040	0.018	0.9%
MnO bc (wt%)	1.556	0.037	1.598	0.052	1.609	0.071	0.053	3.4%
Na_2O bc (wt%)	**8.549**	**0.162**	**8.626**	**0.212**	**8.712**	**0.099**	**0.162**	**1.9%**
NiO bc (wt%)	0.164	0.008	0.171	0.008	0.171	0.008	0.007	4.3%
SiO_2 bc (wt%)								**3.0%**
TiO_2 bc (wt%)								**1.6%**
PM_1 bc (wt%)								**1.7%**
PM_2 bc (wt%)								**4.6%**

Sludge	CST	Frit

bc => bias-corrected measurements;
Na_2O is bolded to indicate that it is present in sludge, frit, and CST;
and SiO_2 is bolded to indicate that it is in the frit and CST.

Table 4.4 Summary of Comparisons Among the Hydragard® Samples at Different Flow Rates for Phase 3

	Phase 3: Sludge-Frit Only							
Oxide	4 gpm Mean	Std Dev	7 gpm Mean	Std Dev	10 gpm Mean	Std Dev	Differences (Max-Min)	(Mx-Mn)/Mn
Al_2O_3 (wt%)	6.183	0.181	6.079	0.090	6.457	0.213	0.379	6.2%
B_2O_3 (wt%)	7.759	0.186	7.867	0.069	7.673	0.198	0.194	2.5%
CaO (wt%)	1.450	0.053	1.429	0.028	1.530	0.090	0.101	7.1%
Cr_2O_3 (wt%)	0.098	0.009	0.097	0.010	0.104	0.015	0.007	7.2%
CuO (wt%)	0.014	0.010	0.014	0.008	0.014	0.008	0.000	3.5%
Fe_2O_3 (wt%)	12.696	0.312	12.620	0.195	13.244	0.644	0.624	4.9%
K_2O (wt%)	0.127	0.007	0.125	0.006	0.130	0.007	0.005	3.6%
Li_2O (wt%)	3.312	0.071	3.370	0.050	3.268	0.089	0.051	1.6%
MgO (wt%)	2.256	0.027	2.259	0.029	2.263	0.040	0.007	0.3%
MnO (wt%)	1.603	0.057	1.594	0.024	1.668	0.069	0.075	4.7%
Na_2O (wt%)	10.490	0.260	10.532	0.220	10.534	0.234	0.044	0.4%
NiO (wt%)	0.160	0.007	0.157	0.006	0.168	0.013	0.011	6.9%
SiO_2 (wt%)								2.3%
TiO_2 (wt%)								6.2%
PM_1 (wt%)								2.9%
PM_2 (wt%)								2.4%
wt% Solids	43.065	0.740	43.596	0.403	42.505	0.776	1.091	2.6%
Al_2O_3 bc (wt%)	6.408	0.180	6.294	0.110	6.693	0.225	0.399	6.3%
B_2O_3 bc (wt%)	7.673	0.197	7.765	0.093	7.588	0.222	0.177	2.3%
CaO bc (wt%)	1.425	0.054	1.402	0.027	1.504	0.091	0.102	7.3%
Cr_2O_3 bc (wt%)	0.104	0.015	0.105	0.011	0.111	0.014	0.006	5.9%
CuO bc (wt%)	0.015	0.010	0.014	0.008	0.014	0.008	0.001	4.4%
Fe_2O_3 bc (wt%)	12.776	0.345	12.690	0.245	13.325	0.629	0.635	5.0%
K_2O bc (wt%)	0.130	0.008	0.127	0.008	0.133	0.009	0.006	5.0%
Li_2O bc (wt%)	3.500	0.094	3.508	0.064	3.453	0.100	0.056	1.6%
MgO bc (wt%)	2.238	0.024	2.244	0.028	2.246	0.039	0.007	0.3%
MnO bc (wt%)	1.603	0.056	1.593	0.022	1.668	0.069	0.075	4.7%
Na_2O bc (wt%)	10.823	0.242	10.785	0.198	10.871	0.217	0.086	0.8%
NiO bc (wt%)	0.166	0.007	0.164	0.006	0.175	0.013	0.011	7.0%
SiO_2 bc (wt%)								2.2%
TiO_2 bc (wt%)								6.4%
PM_1 bc (wt%)								2.9%
PM_2 bc (wt%)								2.1%

Sludge	CST	Frit

bc => bias-corrected measurements;

Na_2O is bolded to indicate that it is present in sludge, frit, and CST;

and SiO_2 is bolded to indicate that it is in the frit and CST.

In these tables, shading is used to identify the source of each oxide, and both the original and bias-corrected measurements are given. The wt% solids values are also investigated in these exhibits and tables.

The last two columns (under the heading "Differences") of these tables give the difference between the largest and smallest concentration values over the flowrates for each oxide and % difference (relative to the smallest value). Phases 1 and 2 had % differences less than 5% for all the major oxides (those in the glass at concentrations greater than 0.5 wt%). In Phase 3 (the sludge-frit-only phase), Al_2O_3, Al_2O_3 bc, CaO, CaO bc, and Fe_2O_3 bc all had differences greater than 5%, but none of these were above 7.3%. Thus, the impact of flow rate on the chemical composition of CST slurries is less than that observed for the baseline case.

4.3 Comparisons of Grab and Hydragard® Samples

The comparisons of the feed compositions from the grab sample vs. the Hydragard® sample are provided in Exhibit A3 of Appendix A and in Table 4.5. For these comparisons, the grab sample compositions for each phase are determined by averaging the low and high grab samples while the Hydragard® sample compositions are determined by averaging the analyses at 4, 7 and 10 gpm recirculation flow rates. For the baseline case of sludge (at 26 wt% oxide) and frit (Frit 200 at 64 wt% oxide), the results show a clear and consistent difference between grab and Hydragard® samples. Relative to the grab sample, the sludge components increased by ~ 9.5 % in the Hydragard® sample. This corresponds, for each 100 grams of feed (in terms of oxides) to 2.5 g (9.5% of 26 g) of sludge oxides. For mass balance, the 100 g of feed on an oxide basis must lose 2.5 g of frit. This corresponds to 2.5/74 or 3.4 wt%. The data revealed a 3.4 wt% loss of frit in the Hydragard® vs. the grab sample. Therefore, the data for Phase 3 are consistent and reveal that Hydragard® samples are frit poor and sludge rich relative to the grab samples. These results indicate a somewhat larger bias toward frit depletion than was observed in the 1994 testing.

Table 4.5 Summary of Grab vs Hydragard® Comparisons

	Phase 1: Sludge-Frit-CST (High Solids)						Phase 2: Sludge-Frit-CST (Low Solids)						Phase 3: Sludge-Frit Only (High Solids)					
	Grab Sampler		Hydragard Sampler		Differences		Grab Sampler		Hydragard Sampler		Differences		Grab Sampler		Hydragard Sampler		Differences	
Oxide	Mean	Std Dev	Mean	Std Dev	(H-G)	(H-G)/G	Mean	Std Dev	Mean	Std Dev	(H-G)	(H-G)/G	Mean	Std Dev	Mean	Std Dev	(H-G)	(H-G)/G
Al_2O_3 (wt%)	5.798	0.157	6.354	0.197	0.556	9.6%	5.977	0.111	6.324	0.219	0.347	5.8%	5.677	0.055	6.240	0.230	0.563	9.9%
B_2O_3 (wt%)	5.455	0.180	5.439	0.180	-0.016	-0.3%	5.655	0.136	5.533	0.170	-0.122	-2.2%	8.034	0.134	7.767	0.175	-0.267	-3.3%
CaO (wt%)	1.265	0.042	1.365	0.076	0.100	7.9%	1.256	0.033	1.320	0.050	0.065	5.2%	1.340	0.026	1.470	0.074	0.130	9.7%
Cr_2O_3 (wt%)	0.101	0.005	0.109	0.008	0.008	8.3%	0.100	0.006	0.106	0.009	0.007	6.8%	0.094	0.019	0.100	0.012	0.006	6.8%
CuO (wt%)	0.010	0.005	0.013	0.006	0.003	25.9%	0.020	0.007	0.020	0.006	0.000	0.3%	0.010	0.007	0.014	0.008	0.004	38.0%
Fe_2O_3 (wt%)	12.651	0.339	13.596	0.329	0.946	7.5%	12.558	0.266	13.248	0.451	0.690	5.5%	11.697	0.178	12.854	0.500	1.156	9.9%
K_2O (wt%)	0.129	0.006	0.144	0.011	0.015	11.6%	0.100	0.008	0.112	0.016	0.013	12.7%	0.122	0.004	0.127	0.007	0.005	4.1%
Li_2O (wt%)	3.962	0.124	3.957	0.106	-0.005	-0.1%	3.961	0.110	3.883	0.116	-0.078	-2.0%	3.400	0.040	3.300	0.073	-0.100	-2.9%
MgO (wt%)	2.012	0.042	2.014	0.050	0.002	0.1%	1.960	0.027	1.940	0.031	-0.020	-1.0%	2.239	0.028	2.259	0.032	0.020	0.9%
MnO (wt%)	1.506	0.036	1.607	0.037	0.102	6.7%	1.476	0.025	1.558	0.053	0.082	5.5%	1.481	0.019	1.622	0.061	0.141	9.5%
Na_2O (wt%)	**8.614**	**0.215**	**8.550**	**0.226**	**-0.064**	**-0.7%**	**8.678**	**0.128**	**8.707**	**0.148**	**0.029**	**0.3%**	**10.517**	**0.196**	**10.518**	**0.230**	**0.001**	**0.0%**
NiO (wt%)	0.164	0.005	0.181	0.010	0.017	10.4%	0.150	0.005	0.161	0.008	0.011	7.2%	0.147	0.004	0.162	0.010	0.015	10.3%
SiO_2 (wt%)						-1.0%						-2.0%						-3.2%
TiO_2 (wt%)						8.4%						6.3%						11.4%
PM_1 (wt%)						6.6%						6.9%						13.9%
PM_2 (wt%)						8.2%						6.3%						3.3%
wt% Solids	45.856	0.167	44.689	0.571	-1.167	-2.5%	42.367	0.158	41.933	1.681	-0.433	-1.0%	45.534	0.361	43.055	0.781	-2.478	-5.4%
Al_2O_3 bc (wt%)	6.084	0.147	6.610	0.174	0.526	8.6%	6.225	0.119	6.582	0.231	0.356	5.7%	5.890	0.065	6.465	0.242	0.575	9.8%
B_2O_3 bc (wt%)	5.429	0.147	5.353	0.132	-0.076	-1.4%	5.598	0.149	5.469	0.171	-0.129	-2.3%	7.956	0.138	7.675	0.188	-0.281	-3.5%
CaO bc (wt%)	1.191	0.042	1.282	0.076	0.091	7.6%	1.221	0.029	1.280	0.049	0.059	4.8%	1.321	0.035	1.444	0.075	0.122	9.3%
Cr_2O_3 bc (wt%)	0.110	0.003	0.121	0.006	0.011	9.6%	0.118	0.003	0.125	0.005	0.008	6.7%	0.101	0.019	0.107	0.013	0.005	5.4%
CuO bc (wt%)	0.011	0.005	0.014	0.006	0.003	25.2%	0.021	0.007	0.021	0.006	0.000	0.2%	0.010	0.006	0.014	0.008	0.004	37.7%
Fe_2O_3 bc (wt%)	12.552	0.322	13.519	0.338	0.967	7.7%	13.005	0.227	13.700	0.487	0.695	5.3%	11.838	0.233	12.930	0.509	1.093	9.2%
K_2O bc (wt%)	0.127	0.006	0.140	0.010	0.013	10.3%	0.094	0.007	0.105	0.014	0.011	11.9%	0.125	0.005	0.130	0.008	0.005	4.0%
Li_2O bc (wt%)	4.093	0.088	4.038	0.084	-0.054	-1.3%	4.193	0.111	4.105	0.116	-0.088	-2.1%	3.609	0.055	3.487	0.088	-0.122	-3.4%
MgO bc (wt%)	1.936	0.043	1.960	0.026	0.024	1.2%	1.998	0.026	1.977	0.033	-0.021	-1.1%	2.220	0.024	2.243	0.030	0.023	1.0%
MnO bc (wt%)	1.469	0.036	1.576	0.037	0.107	7.3%	1.505	0.022	1.588	0.058	0.082	5.5%	1.480	0.019	1.621	0.061	0.142	9.6%
Na_2O bc (wt%)	**8.934**	**0.192**	**8.934**	**0.182**	**0.000**	**0.0%**	**8.582**	**0.134**	**8.629**	**0.172**	**0.047**	**0.5%**	**10.838**	**0.249**	**10.826**	**0.214**	**-0.012**	**-0.1%**
NiO bc (wt%)	0.166	0.005	0.183	0.011	0.017	10.5%	0.157	0.005	0.169	0.008	0.011	7.1%	0.153	0.003	0.168	0.010	0.015	9.9%
SiO_2 bc (wt%)						-1.7%						-2.1%						-3.3%
TiO_2 bc (wt%)						8.2%						6.1%						10.6%
PM_1 bc (wt%)						6.6%						6.9%						13.9%
PM_2 bc (wt%)						7.4%						7.3%						0.6%

Sludge	CST	Frit

bc => bias-corrected measurements; Na_2O is bolded to indicate that it is present in sludge, frit, and CST; and SiO_2 is bolded to indicate that it is in the frit and CST.

These baseline results were used to determine whether or not the addition of CST to the feed affects the 9.5 % increase in sludge and 3.4% decrease in frit for the Hydragard® sample relative to the grab sample. Phase 1 results are considered first. This phase used a 46 wt% total solids slurry, the same total solids as used in Phase 3. However, this feed has 10 wt% CST, 26 wt% sludge and 64 wt% Frit 200. The results reveal that the sludge is enriched in the Hydragard® sample relative to the grab sample by ~ 8.1 wt%. This is slightly less than the baseline case and suggests that CST does not lead to greater enrichment of sludge in the Hydragard® sample.

CST also exhibited a difference in concentration between the grab and Hydragard® sample. This difference was ~ 7.7 wt%, a value essentially equivalent to the sludge enrichment. Therefore, CST and sludge are both enriched by the same percentage at the expense of depletion in frit. The measured frit depletion was only 1.5% and the mass balance was therefore not as good as in Phase 1. The lack of mass balance is addressed later in this Section.

The results for Phase 1 demonstrate that CST does not lead to further differentiation of the sludge and frit by the Hydragard® sampler and in fact may reduce the differentiation slightly. These data also reveal that the CST behaves like sludge in that it is enriched at the same rate relative to the frit for the Hydragard® vs. grab samples.

Phase 2 results reveal the same pattern observed in Phase 1. In this case, the total solids wt % in the slurry was 42% and the sludge enrichment (~5.5%) was slightly less than that observed in Phase 1. Similarly, the CST enrichment (6.7%) was also less than measured in Phase 1. The measured frit depletion was higher at 2.2% and therefore, the mass balance was better than Phase 1, but still not as good as Phase 3. Nevertheless, the pattern is the same with both sludge and CST being enriched in the Hydragard® sample relative to the grab sample and the frit being depleted in the Hydragard® sample. Again, the measured enrichment of the sludge and the measured depletion of the frit in the Hydragard® were less than observed in the baseline case (Phase 1).

In both Phases 1 and 2, the mass balance between frit depletion and sludge enhancement was not as good as Phase 3. If a mass balance is forced on Phase 1 and 2 by using the enrichments observed for sludge and CST as correct, then Phase 1 would have a frit depletion of 4.5% and Phase 2 a frit depletion of 3.2 wt%.

In summary, the presence of CST in the slurry did not increase (and, may slightly decrease) differentiation of the sludge from the frit relative to the baseline case. These data also reveal that the CST behaves like the sludge in being enriched at approximately the same rate relative to the frit.

4.4 Effect of Hydragard® Valve Opening time.

The dependence of the chemical composition of the feed sample as a function of the valve opening time was also performed. The time of valve opening was varied between 5 and 60 seconds. The results from the samples taken to support this investigation over the 3 phases are plotted by time and phase in Exhibit A4 in Appendix A. Both measured and bias-corrected values are included in these plots.

In the detailed, statistical analysis of each phase [7, 8, & 9], both linear trends and quadratic behaviors were explored for these data. A few of the oxides appeared to demonstrate statistically significant (at the 5% level) behavior, but there was no indication of a systematic problem over a group of components (i.e., for the sludge components or the CST components).

Tables 4.6, 4.7, and 4.8 provide summaries of these data for Phases 1, 2, and 3, respectively. An indication of the variation in the oxide concentrations over the 4 sampling time intervals for each phase is provided by the last 2 columns of each of the tables. These columns present the maximum differences and % differences (relative to the values at 40 seconds) between the average concentrations at 40 seconds and the average concentrations at the other sampling times. For Phase 1 (ignoring the Na_2O results {see footnote at the bottom of the next page}), all of the major oxides (measured and bias corrected) had differences less than 5% except for CaO (5.9%), CaO bc (6.8%), PM_2 bc (13.2%). For Phase 2 (which demonstrated the greatest amount of variation in these results over the three test phases), the measured and bias-corrected values for Al_2O_3, CaO, Fe_2O_3, MnO, TiO_2, PM_1 and PM_2 all had differences greater than 5%, but none of the differences were greater than 10%. None of the differences for Phase 3 were greater than 5%.

In summary, few statistically significant trends in the oxide concentrations were seen in the data from any test phase; however, compositional variation was seen among samples taken at different flow loop times. This was true primarily for Phase 2 and to a lesser extend for Phase 1. Little compositional variation was seen in the samples taken at different flow loop times during Phase 3.

Table 4.6 Chemical Composition as a Function of Sample Time for Phase 1

	Phase 1: Sludge-Frit-CST (High Solids)											
	5 Seconds		10 Seconds		20 Seconds		40 Seconds		60 Seconds		Differences from 40 sec	
Oxide	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	Max Diff	Max Diff/40s
Al_2O_3 (wt%)	6.166	0.400	6.228	0.295	6.224	0.446	6.246	0.306	6.154	0.141	-0.092	-1.5%
B_2O_3 (wt%)	5.389	0.290	5.377	0.174	5.434	0.161	5.319	0.346	5.440	0.269	0.121	2.3%
CaO (wt%)	1.368	0.113	1.452	0.213	1.330	0.071	1.385	0.119	1.303	0.041	-0.082	-5.9%
Cr_2O_3 (wt%)	0.068	0.004	0.068	0.003	0.073	0.011	0.069	0.004	0.071	0.009	0.004	5.6%
CuO (wt%)	0.039	0.005	0.038	0.005	0.039	0.006	0.040	0.007	0.039	0.006	-0.001	-3.7%
Fe_2O_3 (wt%)	13.460	0.401	13.583	0.247	13.745	0.674	13.548	0.286	13.466	0.263	0.197	1.5%
K_2O (wt%)	0.070	0.003	0.065	0.009	0.067	0.008	0.080	0.006	0.070	0.009	-0.015	-18.9%
Li_2O (wt%)	3.941	0.243	3.890	0.227	3.913	0.231	3.891	0.234	3.908	0.277	0.050	1.3%
MgO (wt%)	1.919	0.062	1.965	0.067	1.915	0.043	1.932	0.024	1.910	0.028	0.033	1.7%
MnO (wt%)	1.543	0.052	1.560	0.036	1.595	0.107	1.559	0.040	1.545	0.043	0.036	2.3%
Na_2O (wt%)[2]	**8.522**	**0.583**	**11.039**	**4.649**	**8.726**	**0.818**	**8.947**	**0.701**	**8.689**	**0.316**	**2.092**	**23.4%**
NiO (wt%)	0.144	0.009	0.143	0.004	0.137	0.005	0.162	0.035	0.147	0.016	-0.025	-15.3%
SiO_2 (wt%)												**0.5%**
TiO_2 (wt%)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	-1.7%
PM_1 (wt%)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	-3.7%
PM_2 (wt%)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	-4.0%
Al_2O_3 bc (wt%)	6.462	0.318	6.525	0.204	6.522	0.358	6.548	0.215	6.452	0.110	-0.096	-1.5%
B_2O_3 bc (wt%)	5.523	0.243	5.512	0.119	5.570	0.125	5.450	0.308	5.574	0.217	0.124	2.3%
CaO bc (wt%)	1.261	0.110	1.339	0.209	1.228	0.070	1.287	0.114	1.200	0.042	-0.087	-6.8%
Cr_2O_3 bc (wt%)	0.105	0.009	0.105	0.010	0.115	0.018	0.108	0.009	0.108	0.016	0.007	6.3%
CuO bc (wt%)	0.042	0.002	0.042	0.001	0.042	0.002	0.043	0.003	0.042	0.002	-0.001	-3.2%
Fe_2O_3 bc (wt%)	13.303	0.336	13.427	0.200	13.578	0.590	13.391	0.221	13.314	0.291	0.188	1.4%
K_2O bc (wt%)	0.057	0.002	0.053	0.009	0.055	0.007	0.066	0.003	0.057	0.009	-0.012	-18.9%
Li_2O bc (wt%)	4.260	0.158	4.204	0.110	4.230	0.188	4.207	0.174	4.222	0.170	0.053	1.3%
MgO bc (wt%)	2.049	0.055	2.093	0.059	2.043	0.041	2.065	0.026	2.038	0.025	0.029	1.4%
MnO bc (wt%)	1.561	0.038	1.579	0.026	1.615	0.095	1.577	0.025	1.564	0.049	0.038	2.4%
Na_2O bc (wt%)	**8.796**	**0.456**	**11.452**	**5.025**	**9.001**	**0.635**	**9.245**	**0.787**	**8.974**	**0.234**	**2.206**	**23.9%**
NiO bc (wt%)	0.150	0.008	0.149	0.003	0.142	0.006	0.170	0.039	0.154	0.016	-0.028	-16.4%
SiO_2 bc (wt%)												**0.4%**
TiO_2 bc (wt%)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	-1.7%
PM_1 bc (wt%)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	-3.7%
PM_2 bc (wt%)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	13.2%

Sludge	CST	Frit

bc => bias-corrected measurements;
Na_2O is bolded to indicate that it is present in sludge, frit, and CST;
and SiO_2 is bolded to indicate that it is in the frit and CST.

[2] The Na_2O measurements from just one preparation of one of the samples taken at 10 seconds appear to be potential outliers (on the high side), but these values were not removed from the calculations presented in this report. They contribute to the larger than expected average, standard deviation, and differences.

Table 4.7 Chemical Composition as a Function of Sample Time for Phase 2

Phase 2 Sludge-Frit-CST (Low Solids)												
	5 Seconds		10 Seconds		20 Seconds		40 Seconds		60 Seconds		Differences from 40 sec	
Oxide	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	(Max 40s)	(Mx 40s)/40s
Al_2O_3 (wt%)	6.026	0.291	6.320	0.275	6.379	0.114	6.646	0.293	6.251	0.564	-0.620	-9.3%
B_2O_3 (wt%)	5.200	0.355	5.436	0.104	5.277	0.177	5.192	0.157	4.965	0.434	0.244	4.7%
CaO (wt%)	1.419	0.113	1.336	0.044	1.469	0.145	1.420	0.096	1.553	0.155	0.133	9.4%
Cr_2O_3 (wt%)	0.045	0.004	0.068	0.025	0.050	0.009	0.047	0.011	0.049	0.014	0.021	45.2%
CuO (wt%)	0.058	0.004	0.059	0.006	0.061	0.013	0.057	0.007	0.060	0.006	0.004	7.1%
Fe_2O_3 (wt%)	12.983	0.723	13.118	0.195	13.427	0.332	13.978	0.398	13.423	1.106	-0.995	-7.1%
K_2O (wt%)	0.066	0.015	0.059	0.002	0.082	0.045	0.095	0.067	0.062	0.005	-0.036	-38.0%
Li_2O (wt%)	3.743	0.234	3.908	0.093	3.850	0.064	3.745	0.057	3.590	0.234	0.163	4.3%
MgO (wt%)	1.808	0.048	1.871	0.054	1.911	0.048	1.871	0.097	1.791	0.055	-0.080	-4.3%
MnO (wt%)	1.485	0.038	1.478	0.020	1.531	0.067	1.573	0.040	1.539	0.083	-0.094	-6.0%
Na_2O (wt%)	9.034	0.233	8.959	0.172	9.160	0.474	9.369	0.526	9.420	0.416	-0.409	-4.4%
NiO (wt%)	0.151	0.006	0.151	0.007	0.155	0.018	0.153	0.008	0.159	0.014	0.007	4.5%
SiO_2 (wt%)												1.7%
TiO_2 (wt%)												5.3%
PM_1 (wt%)												-5.6%
PM_2 (wt%)												-9.4%
Al_2O_3 bc (wt%)	6.155	0.287	6.455	0.253	6.520	0.123	6.792	0.293	6.387	0.562	-0.637	-9.4%
B_2O_3 bc (wt%)	5.216	0.320	5.455	0.105	5.293	0.154	5.208	0.096	4.977	0.378	0.246	4.7%
CaO bc (wt%)	1.316	0.107	1.241	0.042	1.367	0.137	1.320	0.095	1.449	0.152	0.129	9.8%
Cr_2O_3 bc (wt%)	0.130	0.042	0.189	0.096	0.136	0.046	0.122	0.006	0.124	0.023	0.067	55.2%
CuO bc (wt%)	0.056	0.002	0.057	0.004	0.060	0.013	0.056	0.005	0.059	0.004	0.005	8.5%
Fe_2O_3 bc (wt%)	12.715	0.706	12.848	0.173	13.154	0.350	13.696	0.407	13.141	1.061	-0.981	-7.2%
K_2O bc (wt%)	0.057	0.013	0.051	0.002	0.074	0.045	0.085	0.063	0.053	0.005	-0.034	-40.5%
Li_2O bc (wt%)	4.001	0.247	4.177	0.105	4.115	0.068	4.002	0.059	3.837	0.245	0.175	4.4%
MgO bc (wt%)	2.022	0.023	2.097	0.087	2.144	0.110	2.100	0.149	2.004	0.061	-0.096	-4.6%
MnO bc (wt%)	1.515	0.039	1.508	0.017	1.562	0.069	1.605	0.043	1.569	0.082	-0.097	-6.0%
Na_2O bc (wt%)	8.863	0.284	8.787	0.160	8.983	0.435	9.188	0.483	9.239	0.449	-0.400	-4.4%
NiO bc (wt%)	0.155	0.007	0.154	0.007	0.159	0.021	0.156	0.012	0.163	0.017	0.006	4.0%
SiO_2 bc (wt%)												2.7%
TiO_2 bc (wt%)												-7.6%
PM_1 bc (wt%)												-5.6%
PM_2 bc (wt%)												-6.2%

Sludge	CST	Frit

bc => bias-corrected measurements;

Na_2O is bolded to indicate that it is present in sludge, frit, and CST; and SiO_2 is bolded to indicate that it is in the frit and CST.

Table 4.8 Chemical Composition as a Function of Sample Time for Phase 3

Phase 3 Sludge-Frit Only (High Solids)												
	5 Seconds		10 Seconds		20 Seconds		40 Seconds		60 Seconds		Differences from 40 sec	
Oxide	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	Mean	Std Dev	(Max-40s)	(Ms-40s)/40s
Al_2O_3 (wt%)	5.891	0.220	5.960	0.233	5.971	0.044	6.087	0.125	6.075	0.205	-0.195	-3.2%
B_2O_3 (wt%)	7.543	0.244	7.726	0.102	7.589	0.140	7.647	0.067	7.575	0.109	-0.105	-1.4%
CaO (wt%)	1.346	0.016	1.328	0.055	1.364	0.029	1.384	0.028	1.389	0.042	-0.056	-4.0%
Cr_2O_3 (wt%)	0.102	0.011	0.102	0.013	0.103	0.011	0.104	0.009	0.105	0.013	-0.003	-2.5%
CuO (wt%)	0.022	0.008	0.019	0.004	0.018	0.003	0.019	0.004	0.019	0.003	0.003	15.0%
Fe_2O_3 (wt%)	12.173	0.360	12.197	0.392	12.392	0.280	12.643	0.410	12.614	0.352	-0.470	-3.7%
K_2O (wt%)	0.136	0.004	0.140	0.009	0.137	0.006	0.141	0.007	0.138	0.002	-0.005	-3.3%
Li_2O (wt%)	3.474	0.097	3.498	0.106	3.441	0.058	3.458	0.083	3.441	0.072	0.040	1.2%
MgO (wt%)	2.223	0.032	2.208	0.020	2.214	0.015	2.225	0.055	2.224	0.029	-0.018	-0.8%
MnO (wt%)	1.535	0.038	1.535	0.040	1.566	0.022	1.593	0.054	1.598	0.037	-0.059	-3.7%
Na_2O (wt%)	10.324	0.518	10.430	0.685	10.346	0.581	10.309	0.372	10.355	0.410	0.121	1.2%
NiO (wt%)	0.160	0.008	0.162	0.015	0.163	0.009	0.165	0.007	0.167	0.013	-0.004	-2.7%
SiO_2 (wt%)												-1.2%
TiO_2 (wt%)												1.3%
PM_1 (wt%)												-9.1%
PM_2 (wt%)												-2.8%
Al_2O_3 bc (wt%)	6.087	0.161	6.158	0.174	6.171	0.059	6.290	0.162	6.278	0.174	-0.203	-3.2%
B_2O_3 bc (wt%)	7.720	0.255	7.907	0.099	7.766	0.149	7.825	0.063	7.751	0.108	-0.105	-1.3%
CaO bc (wt%)	1.345	0.012	1.327	0.043	1.363	0.021	1.383	0.041	1.388	0.030	-0.056	-4.1%
Cr_2O_3 bc (wt%)	0.086	0.019	0.086	0.021	0.087	0.019	0.088	0.018	0.089	0.021	-0.002	-2.3%
CuO bc (wt%)	0.022	0.007	0.019	0.004	0.018	0.002	0.019	0.004	0.018	0.002	0.003	15.3%
Fe_2O_3 bc (wt%)	12.380	0.493	12.403	0.491	12.604	0.505	12.862	0.643	12.827	0.424	-0.482	-3.7%
K_2O bc (wt%)	0.134	0.001	0.137	0.005	0.135	0.002	0.139	0.007	0.135	0.005	-0.005	-3.4%
Li_2O bc (wt%)	3.714	0.101	3.740	0.121	3.678	0.066	3.697	0.097	3.679	0.085	0.043	1.1%
MgO bc (wt%)	2.206	0.044	2.190	0.026	2.196	0.008	2.208	0.036	2.206	0.016	-0.018	-0.8%
MnO bc (wt%)	1.538	0.034	1.537	0.044	1.569	0.016	1.596	0.043	1.601	0.041	-0.059	-3.7%
Na_2O bc (wt%)	10.416	0.087	10.516	0.086	10.435	0.071	10.411	0.268	10.454	0.206	0.105	1.0%
NiO bc (wt%)	0.159	0.009	0.161	0.016	0.162	0.010	0.164	0.008	0.166	0.013	-0.004	-2.7%
SiO_2 bc (wt%)												-1.1%
TiO_2 bc (wt%)												1.3%
PM_1 bc (wt%)												-9.1%
PM_2 bc (wt%)												-2.8%

Sludge	CST	Frit

bc => bias-corrected measurements;
Na_2O is bolded to indicate that it is present in sludge, frit, and CST
and SiO_2 is bolded to indicate that it is in the frit and CST.

5 Summary and Conclusions

The FY00 CST Sampling Study investigated the tank homogeneity by comparing high and low Grab samples and sampling efficiency of the Hydragard® system for melter feed slurries by comparing Hydragard® and Grab samples. The sampling efficiency of the Hydragard® was also assessed over a range of recirculation flows (i.e., 4, 7, and 10 gpm) for a series of special tests involving sample-valve open times of 5, 10, 20, 40, and 60 seconds. Three slurries were investigated: Phase 1–Sludge-Frit-CST at ~ 46 wt% total solids, Phase 2–Sludge-Frit-CST at ~ 42 wt% total solids, and Phase 3–Sludge-Frit Only at ~ 46 wt% total solids.

The results from these studies suggest the following conclusions:

- High and low Grab samples indicate that the tank was well mixed for each phase of testing; however, a 52 wt% total solids Sludge-Frit-CST slurry could not be agitated because the yield stress was too high to obtain motion in significant portions of the slurry.
- The slurries for all 3 phases showed a bias toward higher sludge and lower frit in the Hydragard® samples versus the Grab samples.
- CST components sampled the same as the sludge components.
- From the special samples and test runs,
    - The bias toward frit loss in the Hydragard showed some tendency to increase with increasing recirculation loop flow.
    - Valve open times between 5 and 60 seconds had no (consistently) significant effect on Hydragard® sample composition.

Thus, the data from these tests indicate no problem with sampling size-reduced CST (less than 177 μm) with the Hydragard® system in that the behavior of the CST components within the Hydragard® system mimics the behavior of the sludge components.

6 References

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4. Koopman, D. C. and Lambert, D. P., "Hydrogen Generation and Foaming During Tests in the GFPS Simulating DWPF Operations with Tank 42 Sludge and CST (U)," WSRC-TR-99-00302, September 3, 1999.

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6. SAS Institute, Inc., **JMP® Statistics and Graphics Guide**, Version 3, SAS Institute, Inc., Cary, NC, 1995.

7. Edwards, T. B., "Statistical Analyses Supporting the High-Solids Phase (Phase 1) of the CST Sampling Study," WSRC-RP-2000-00794, Revision 0, September 18, 2000.

8. Edwards, T. B., "Statistical Analyses Supporting the Low-Solids Phase (Phase 2) of the CST Sampling Study," WSRC-RP-2000-00795, Revision 0, September 18, 2000.

9. Edwards, T. B., "Statistical Analyses Supporting the Sludge-Frit-Only Phase (Phase 3) of the CST Sampling Study," WSRC-RP-2000-00796, Revision 0, September 18, 2000.

7 Appendices:

Appendix A: Statistical Exhibits

Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases
Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases
Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases
Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

Appendix B: Explanation of Statistical Graphics

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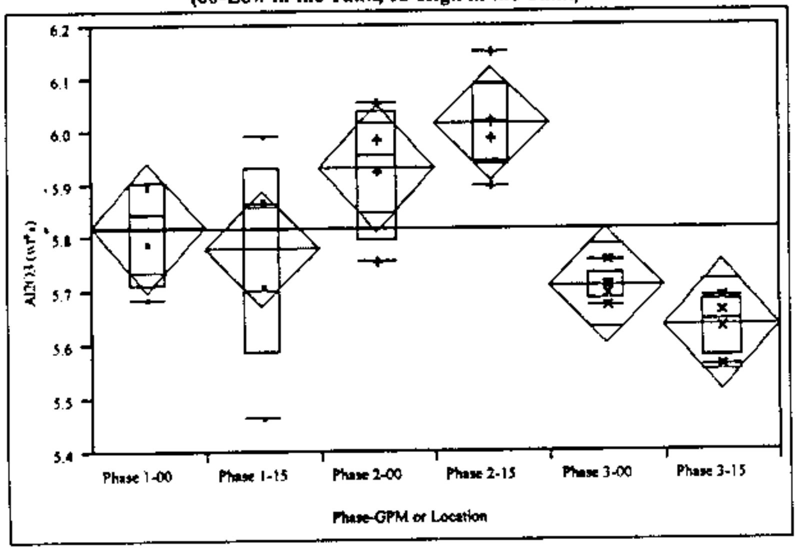
Appendix A.

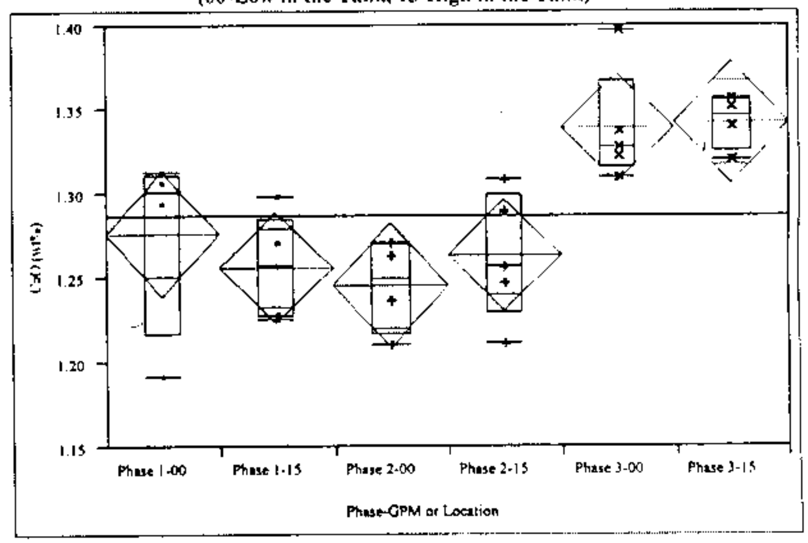
Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

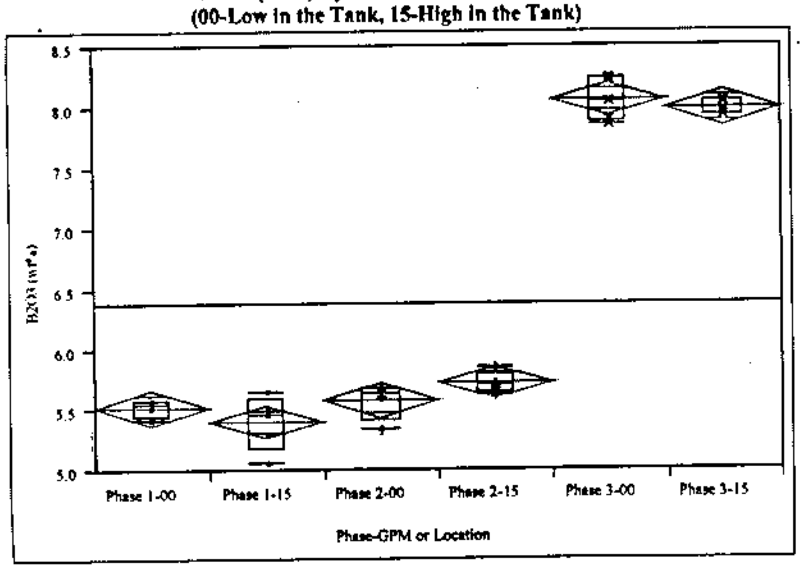
Al2O3 (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



CaO (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



B2O3 (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



Cr2O3 (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)

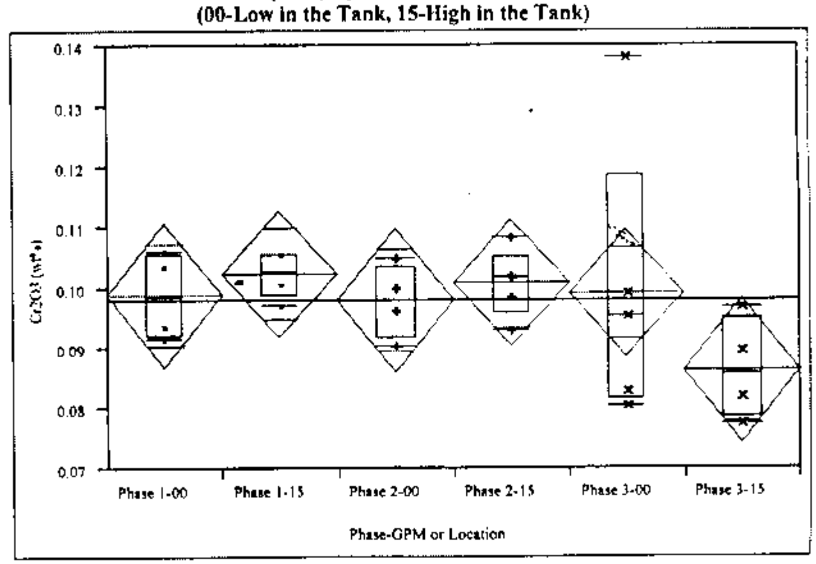
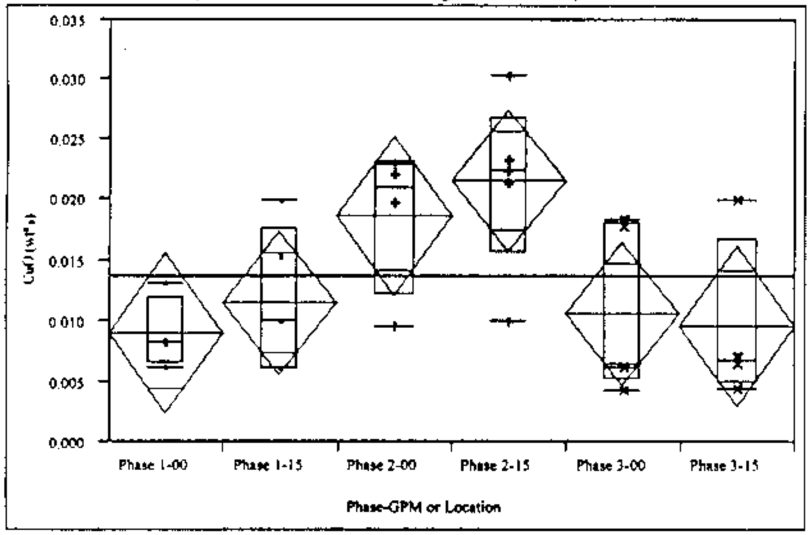
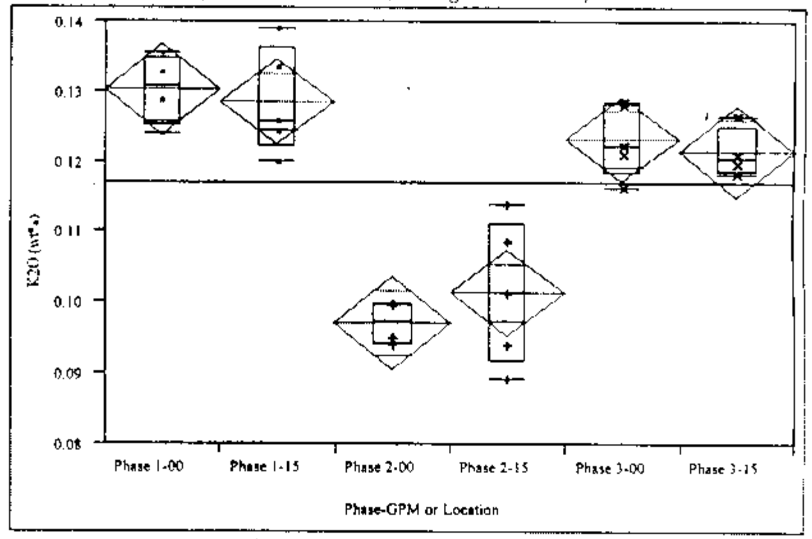


Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases
(Bias-Corrected Measurements are indicated by "bc")

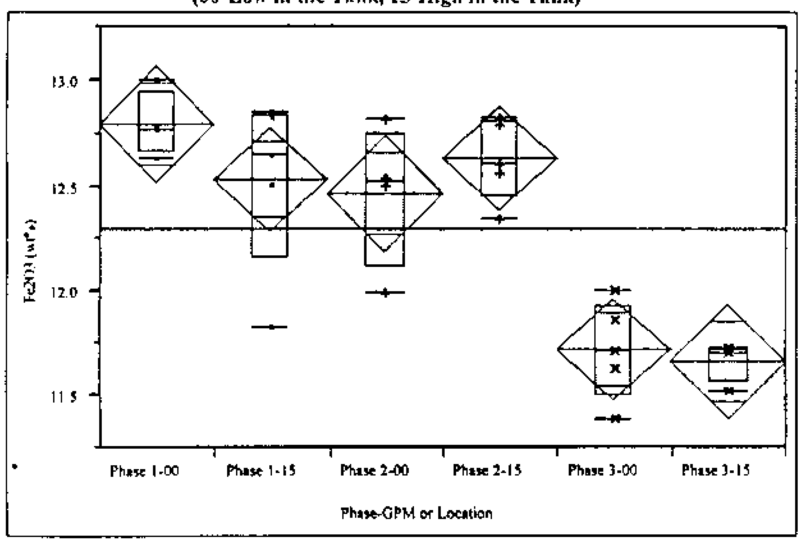
CuO (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



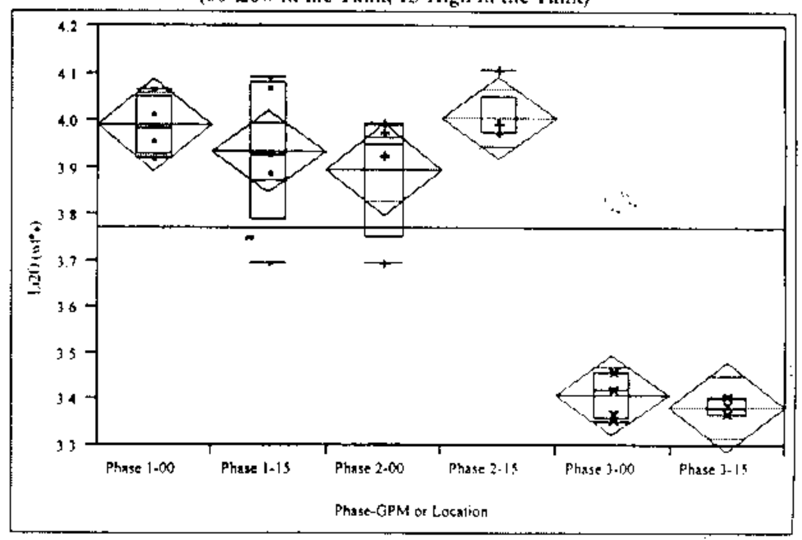
K2O (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



Fe2O3 (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



Li2O (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)

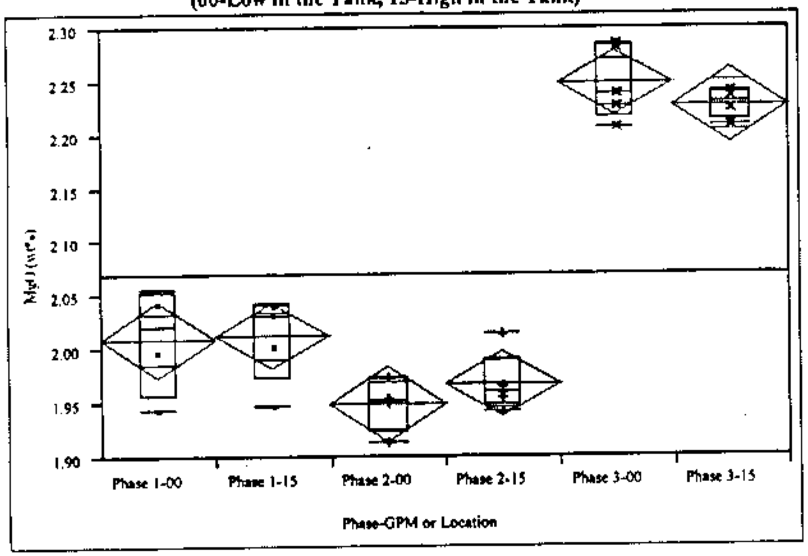


Appendix A.

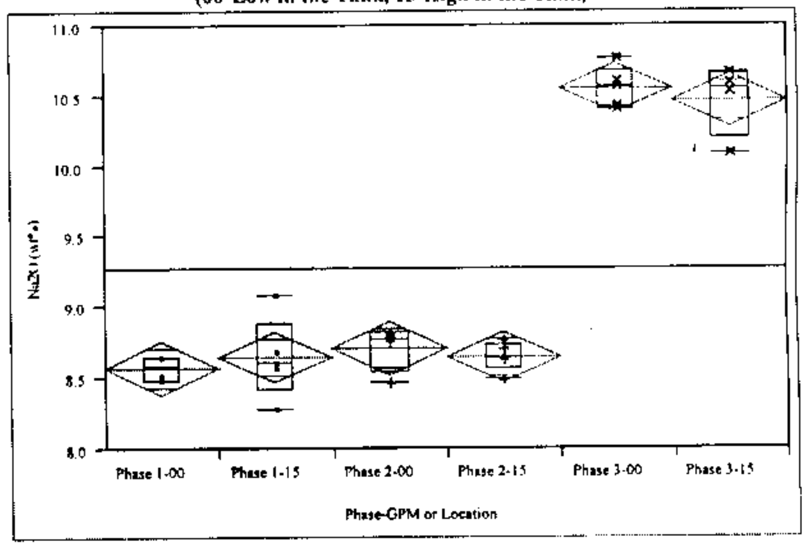
Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

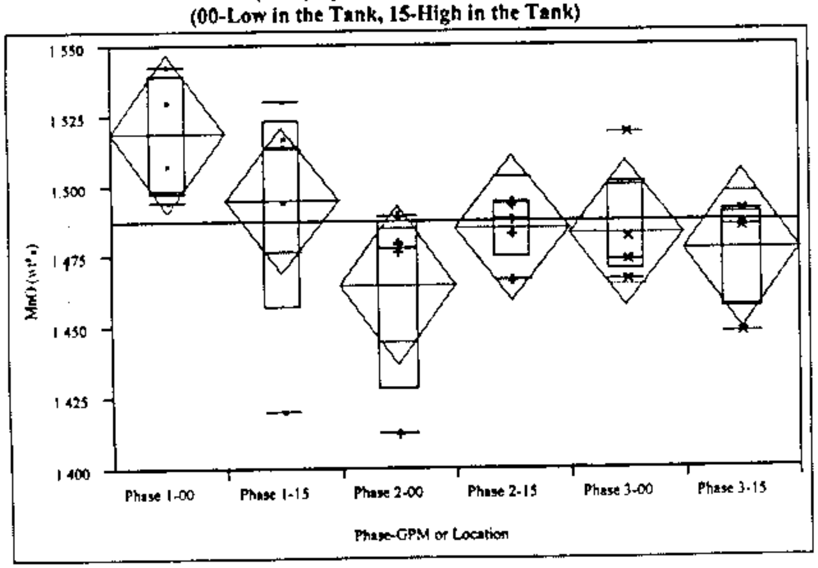
MgO (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



Na2O (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



MnO (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



NiO (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)

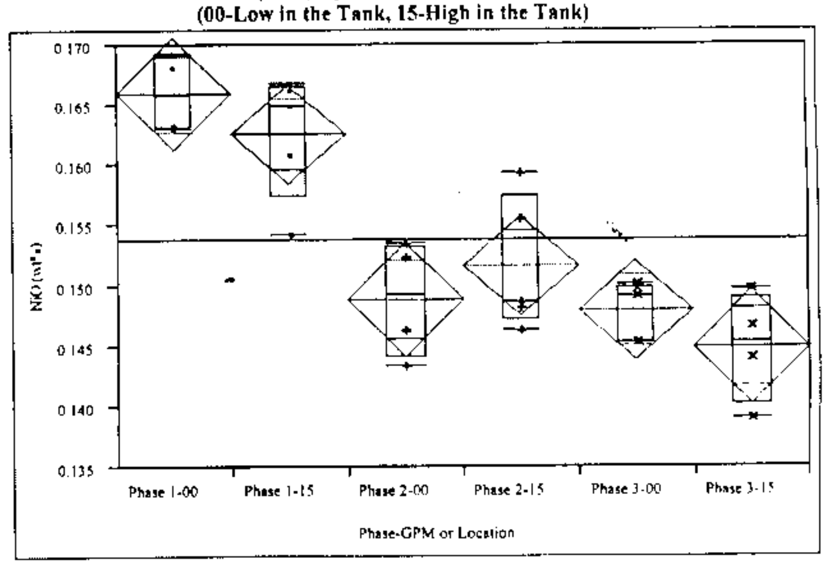
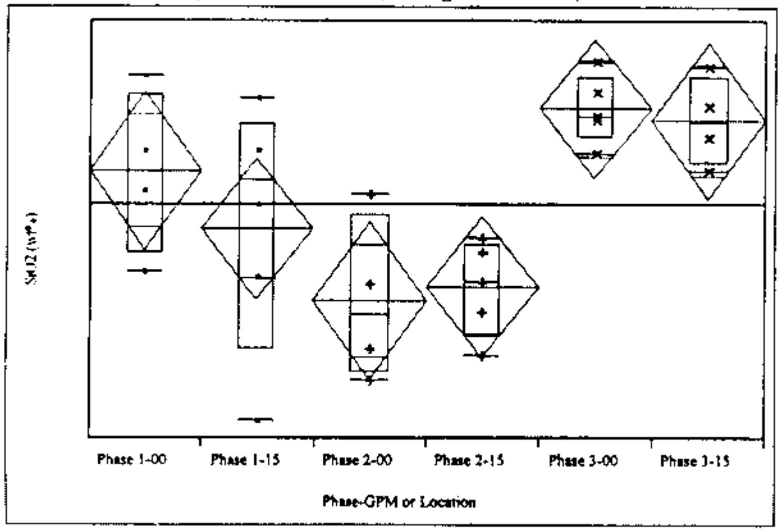
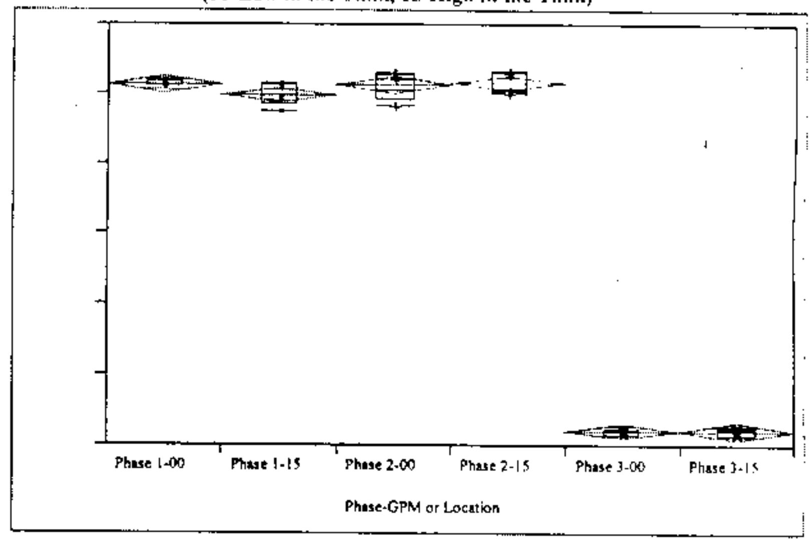


Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases
(Bias-Corrected Measurements are indicated by "bc")

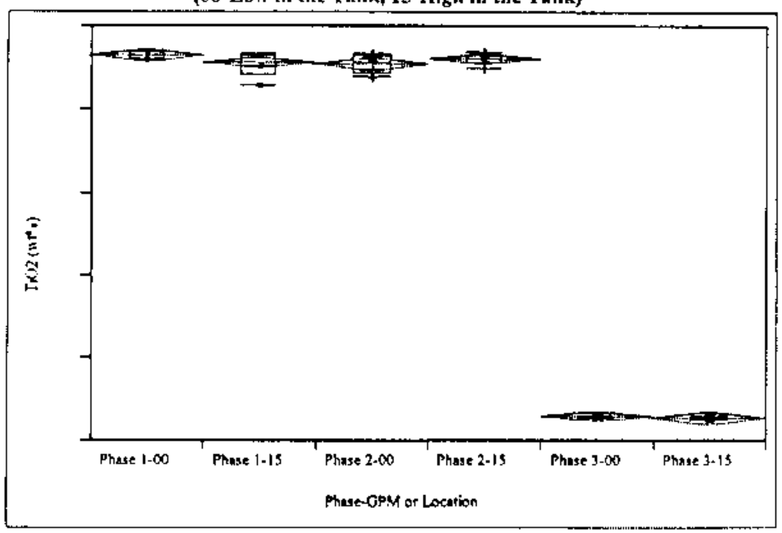
SiO2 (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



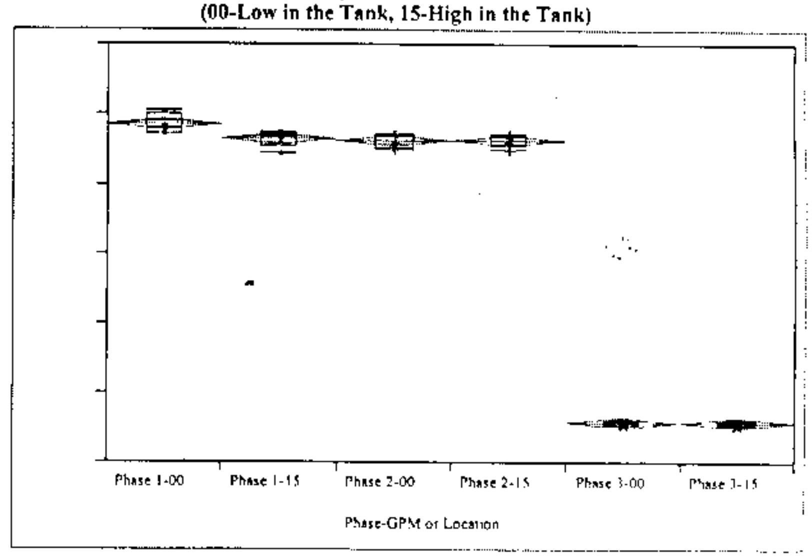
PM1 (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



TiO2 (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



PM2 (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)

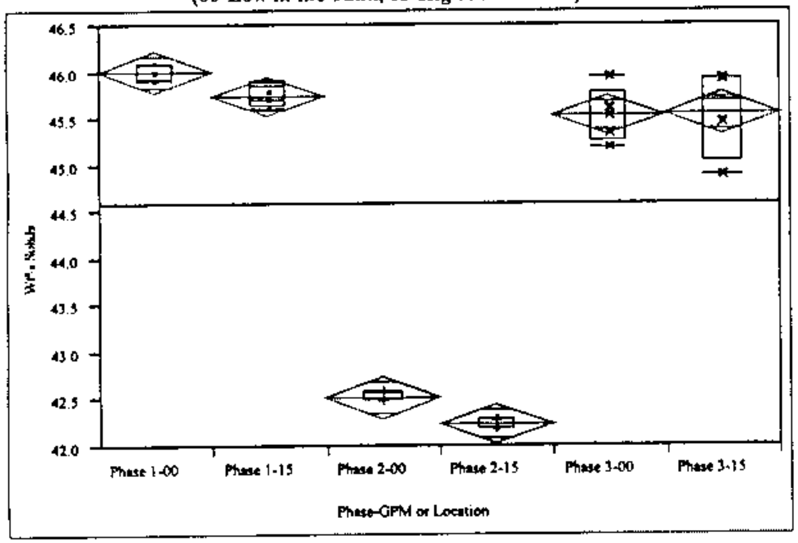


Appendix A.

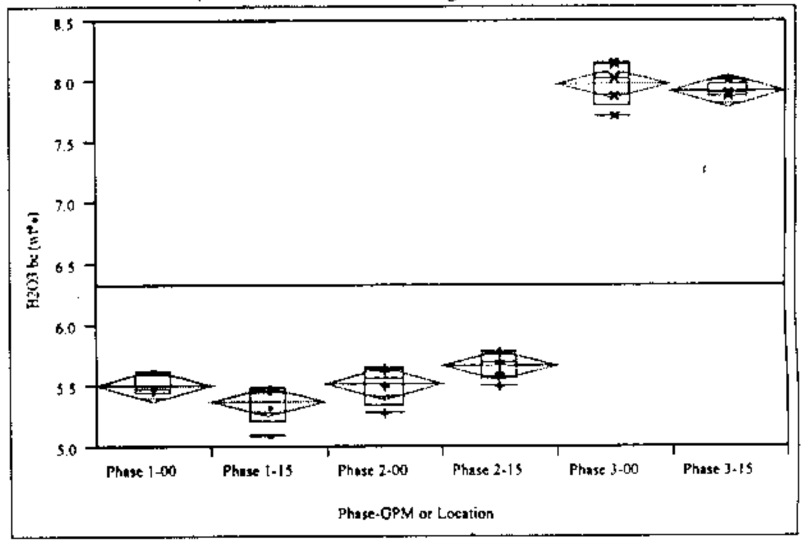
Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

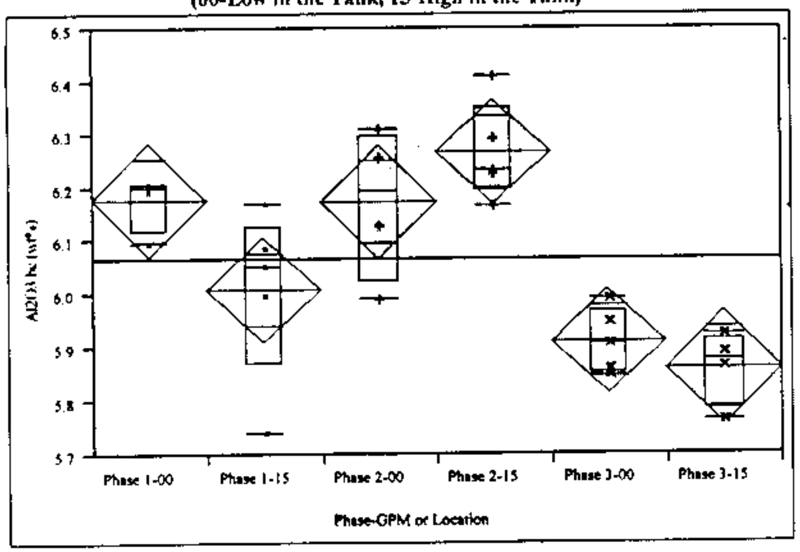
Wt% Solids By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



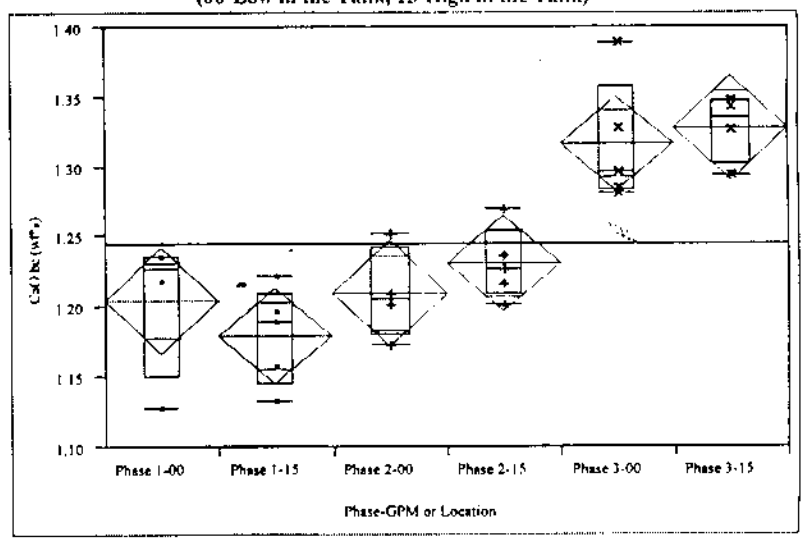
B2O3 bc (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



Al2O3 bc (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



CaO bc (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)

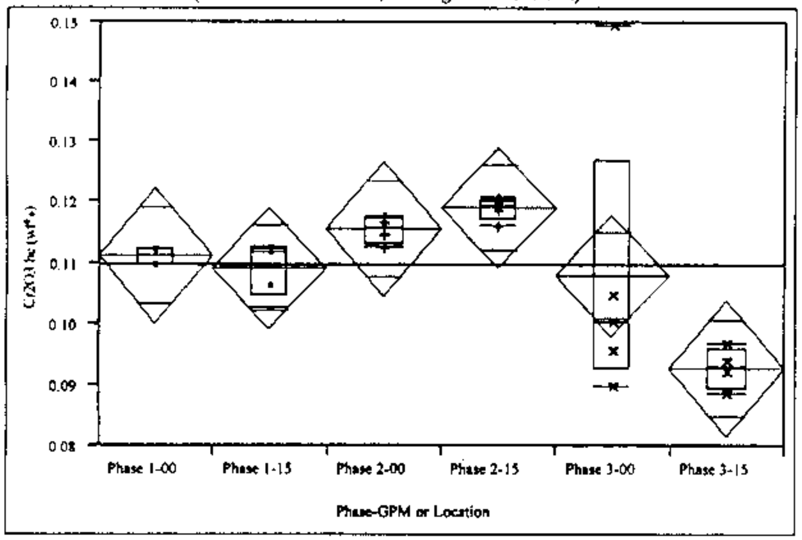


Appendix A.

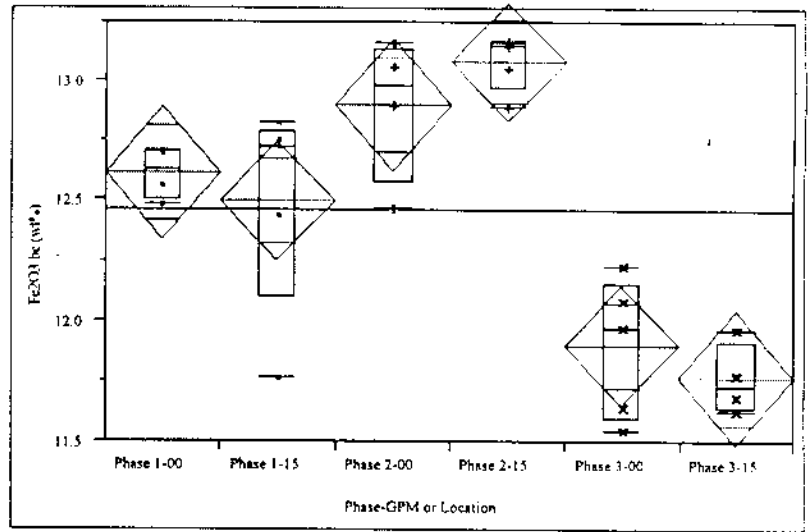
Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

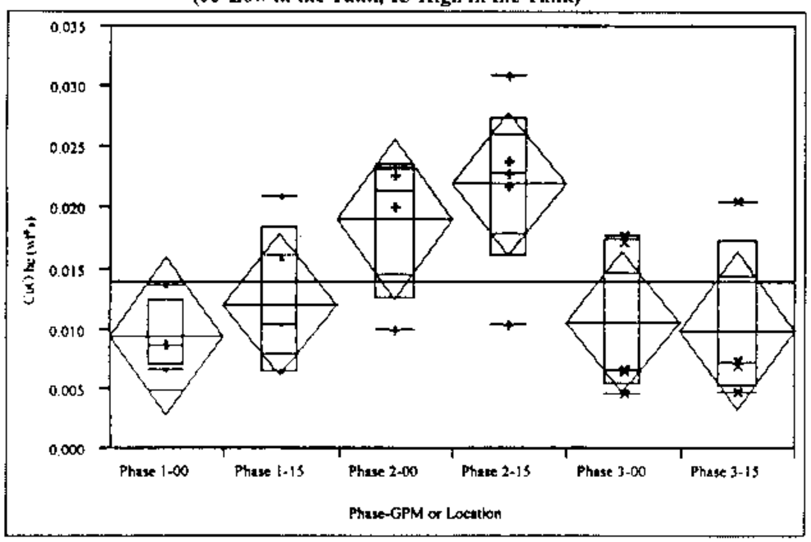
Cr2O3 bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



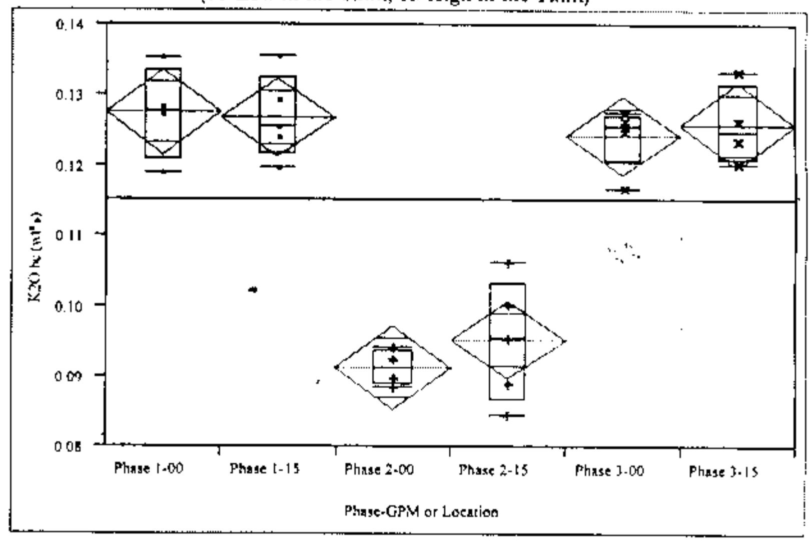
Fe2O3 bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



CuO bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



K2O bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)

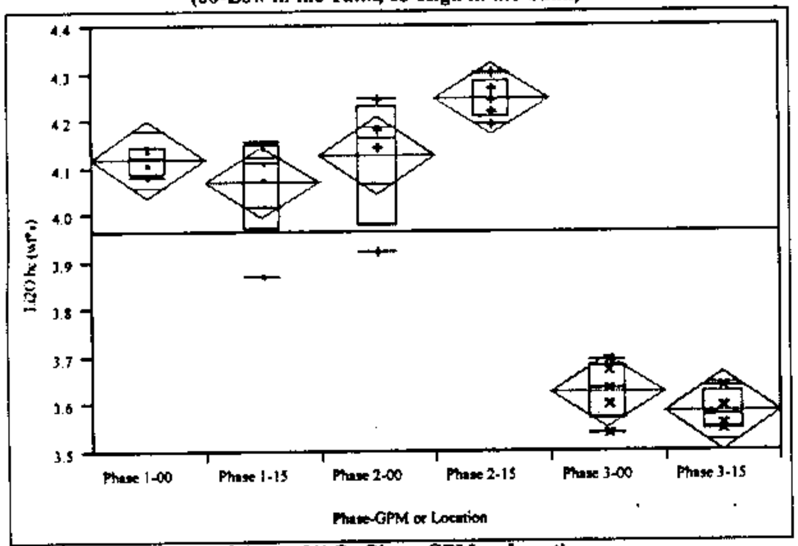


Appendix A.

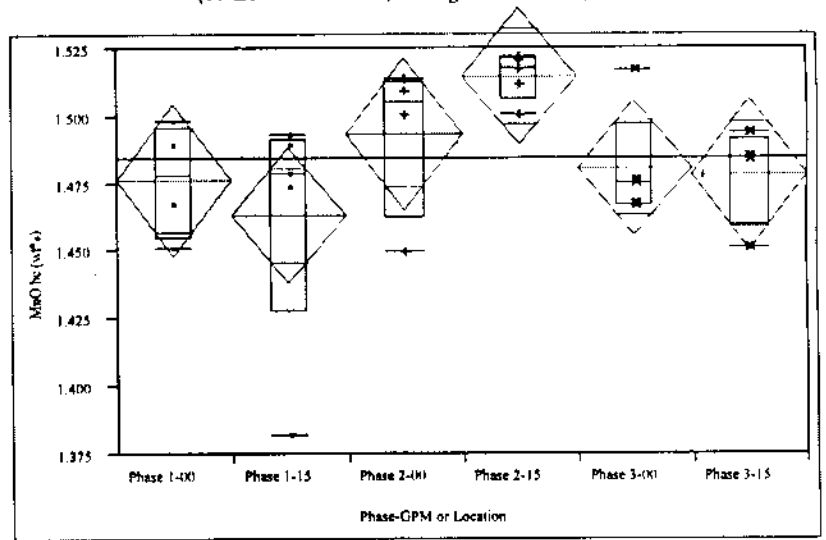
Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

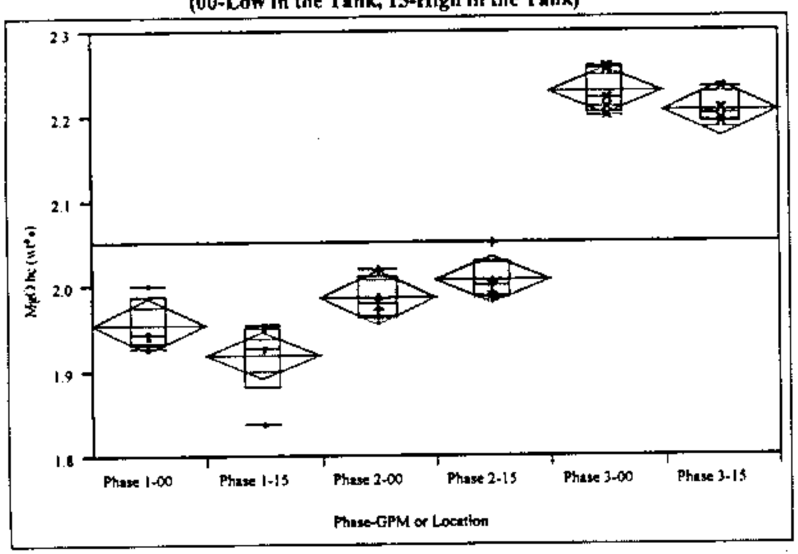
Li2O bc (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



MnO bc (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



MgO bc (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)



Na2O bc (wt%) By Phase-GPM or Location (00-Low in the Tank, 15-High in the Tank)

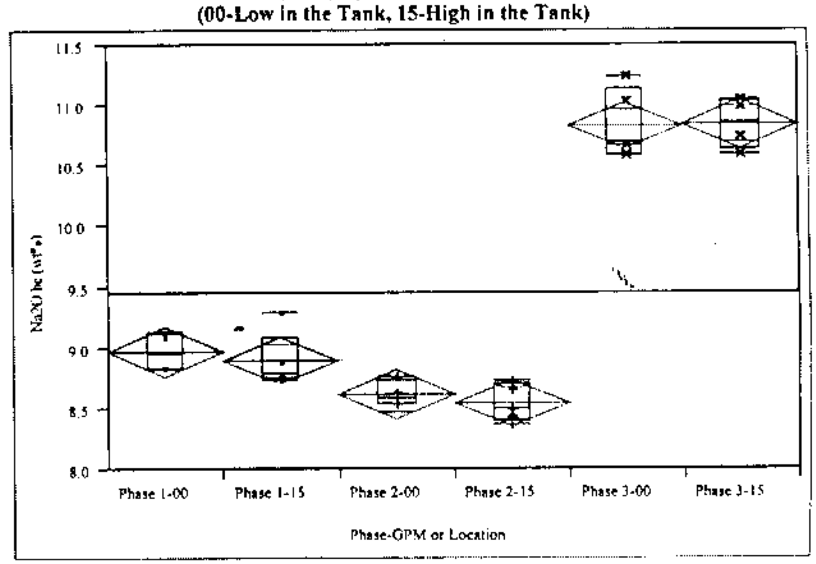
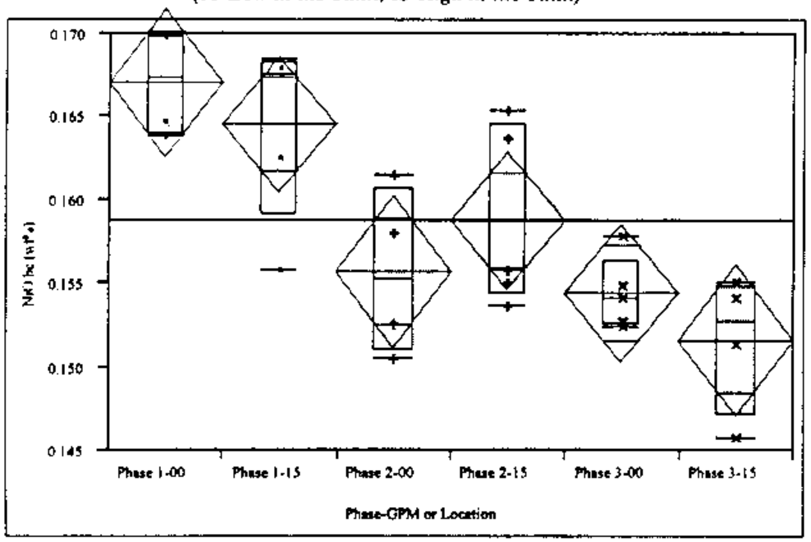


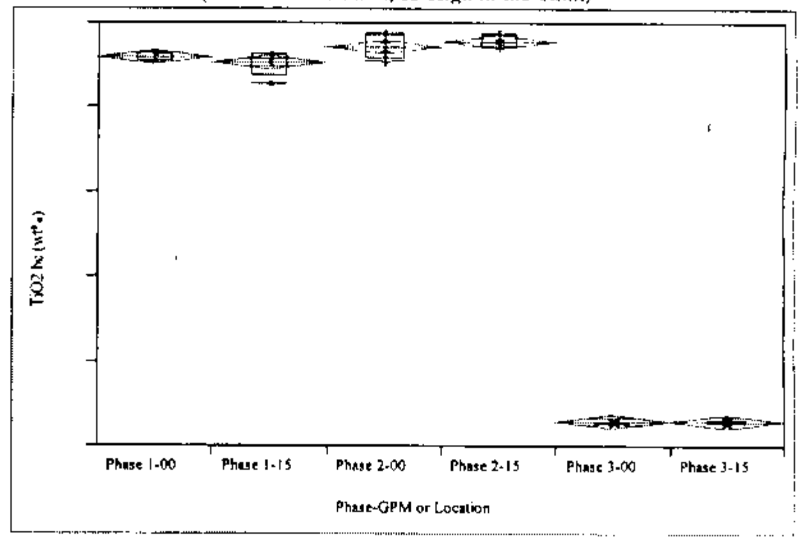
Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

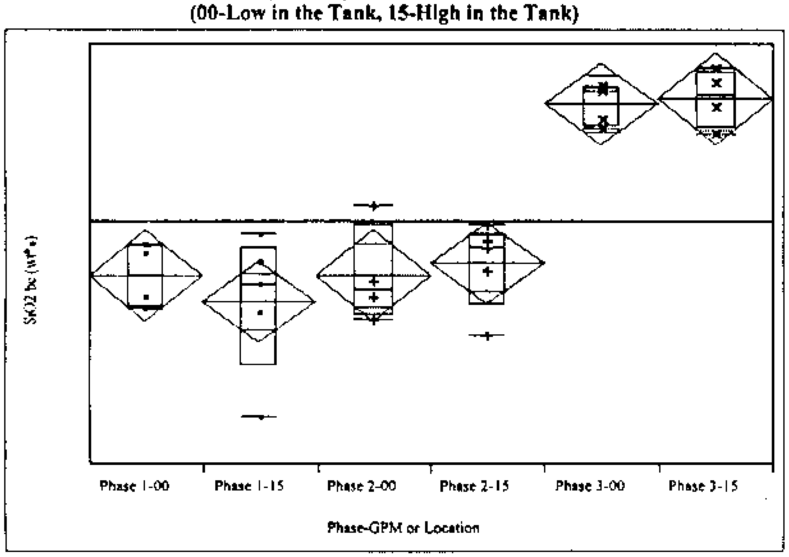
NiO bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



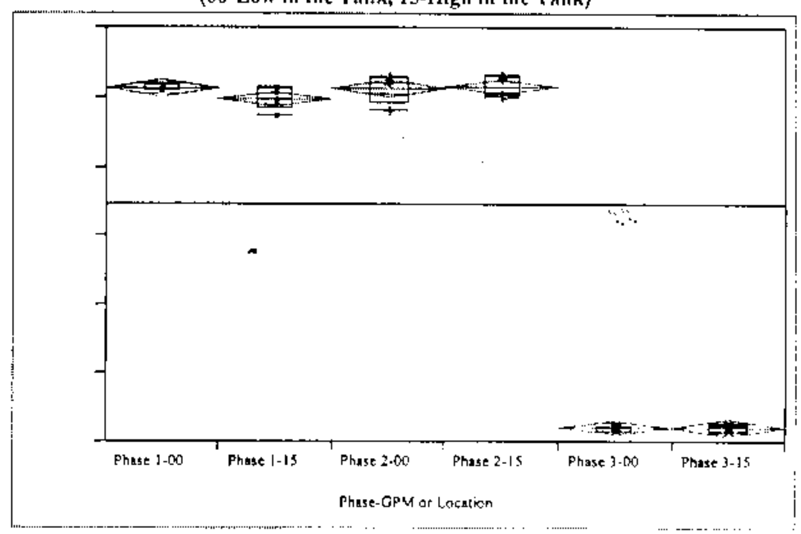
TiO2 bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



SiO2 bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



PM1 bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)



Appendix A.

Exhibit A1. Comparisons of High Versus Low Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

PM2 bc (wt%) By Phase-GPM or Location
(00-Low in the Tank, 15-High in the Tank)

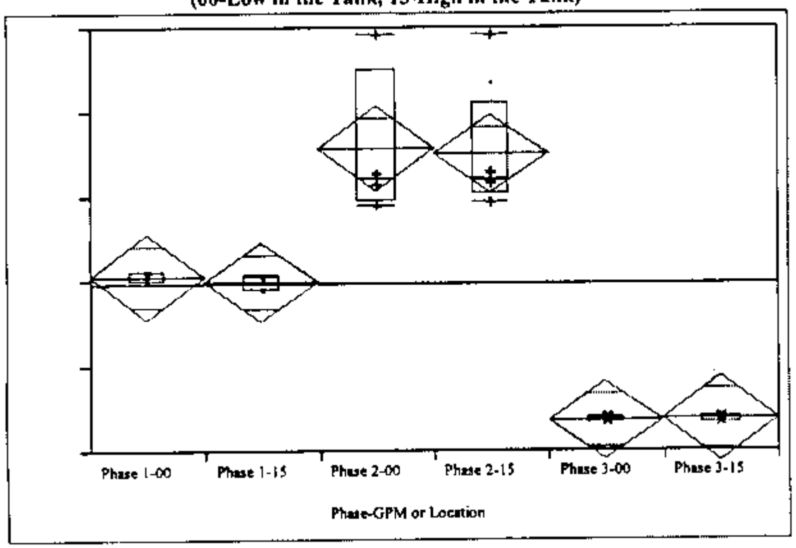
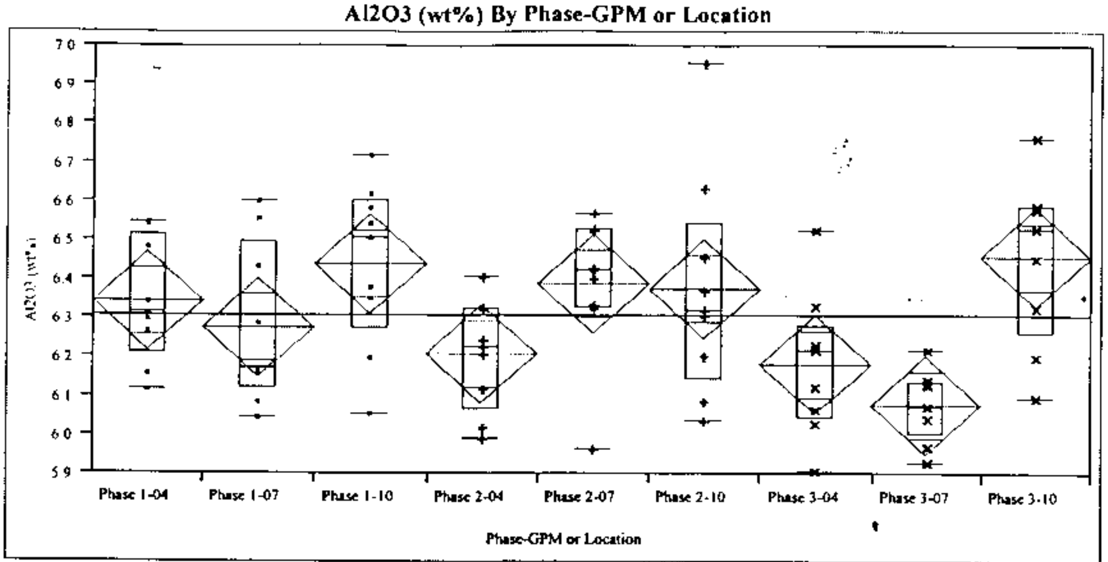
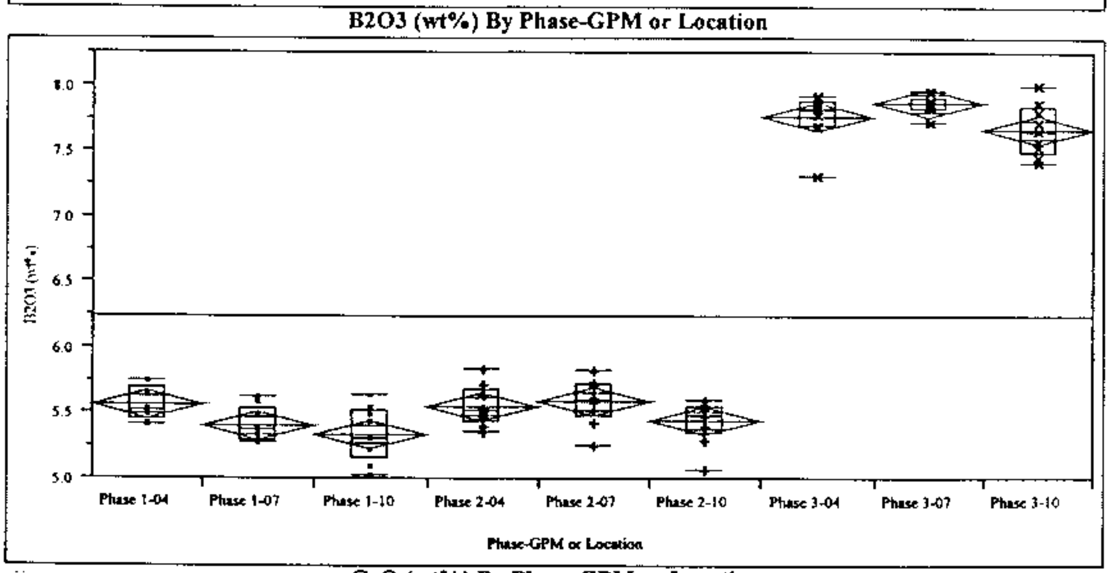


Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases

(Bias-Corrected Measurements are indicated by "bc")





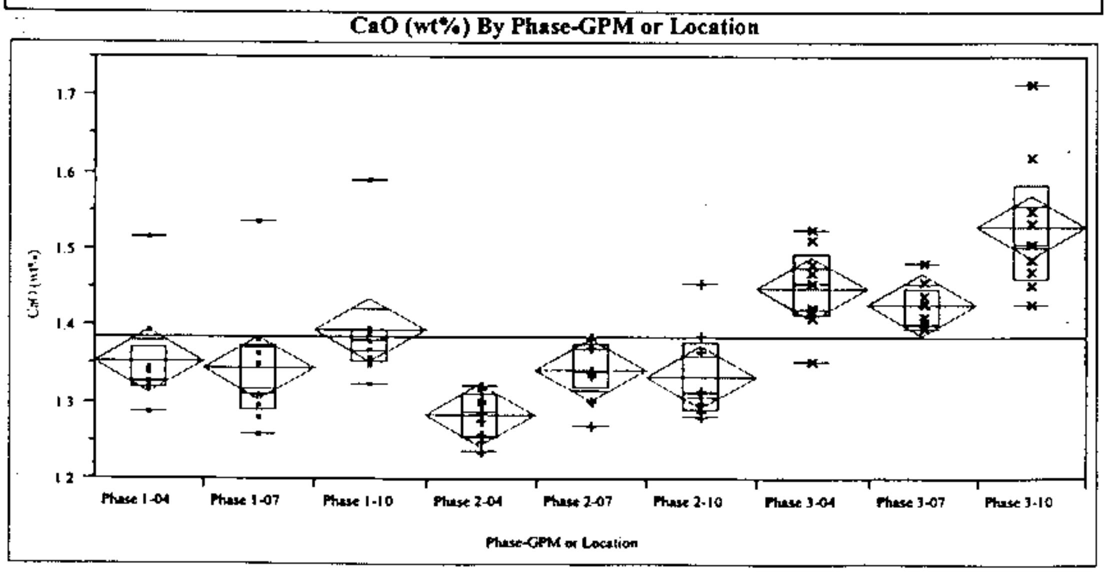
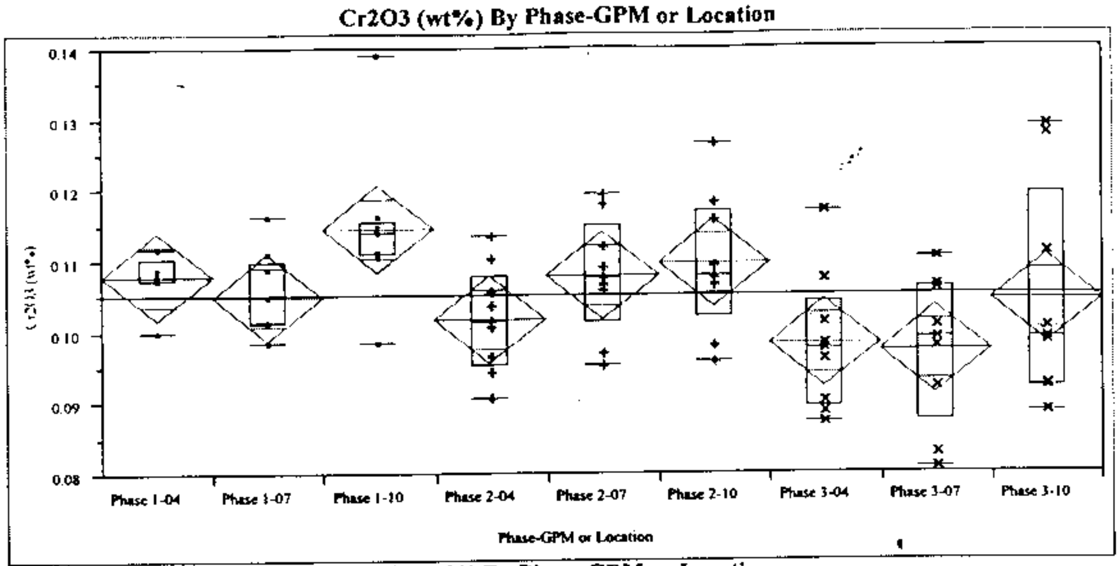
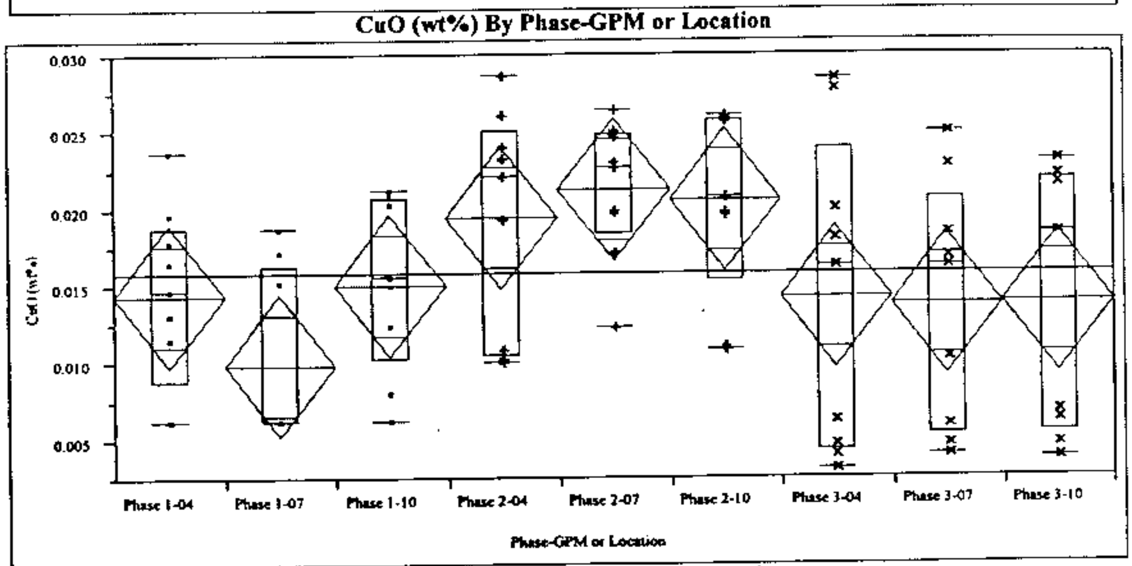


Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases

(Bias-Corrected Measurements are indicated by "bc")





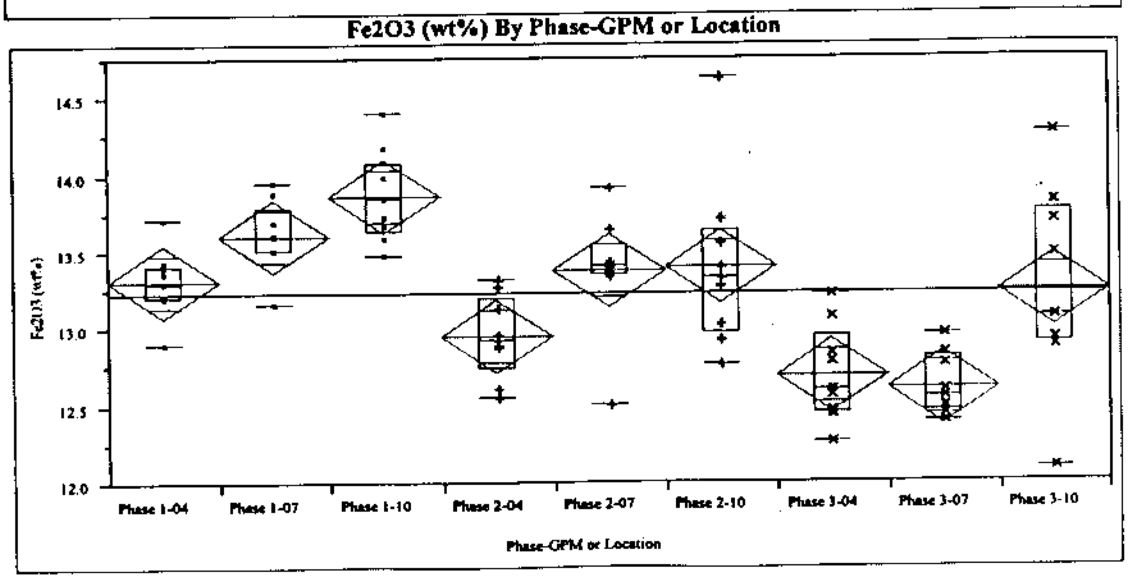
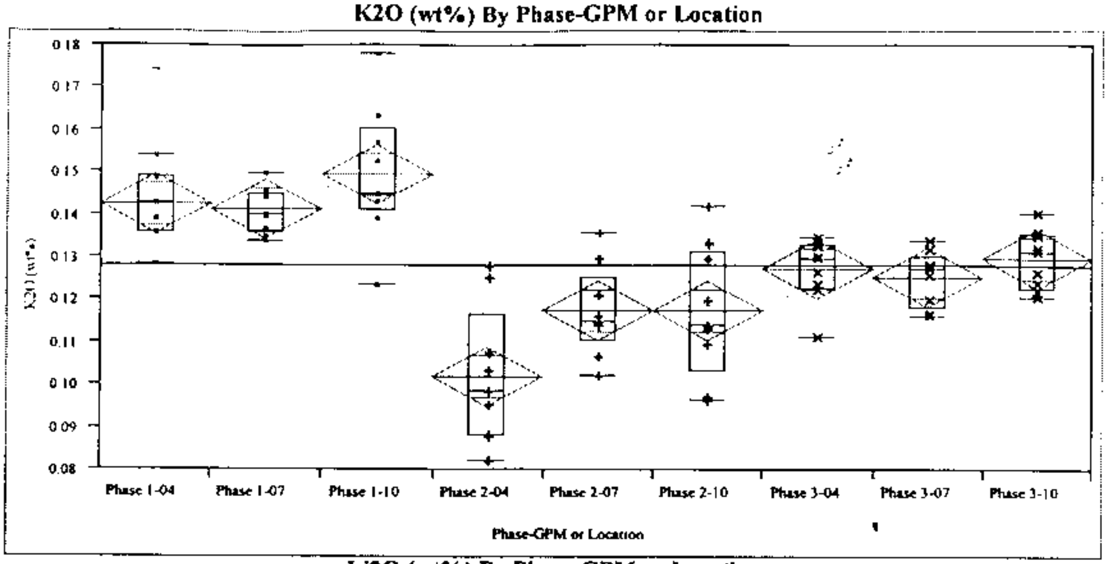


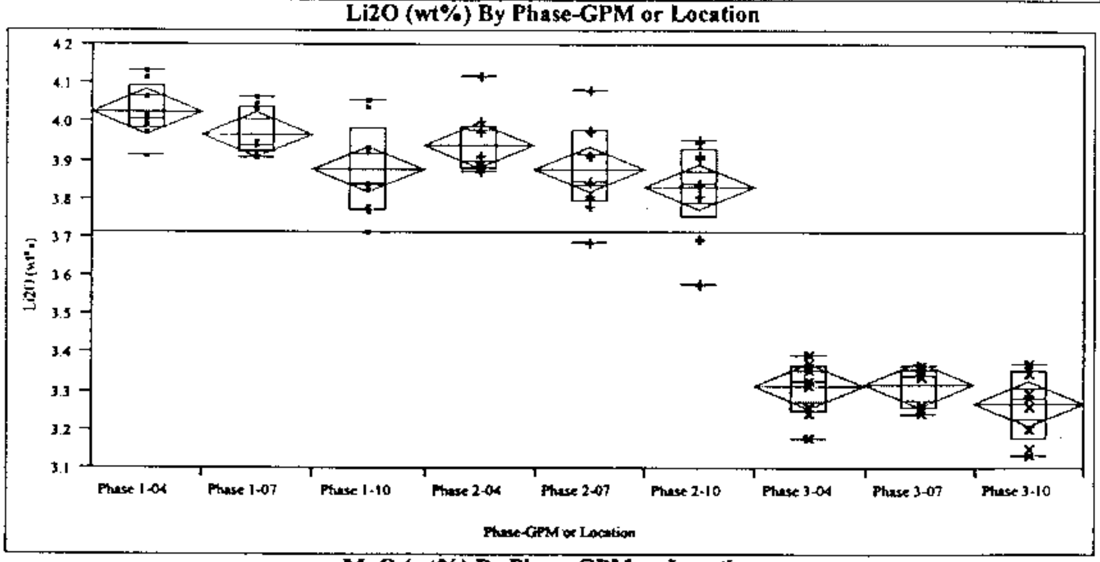
Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

K2O (wt%) By Phase-GPM or Location



Li2O (wt%) By Phase-GPM or Location



MgO (wt%) By Phase-GPM or Location

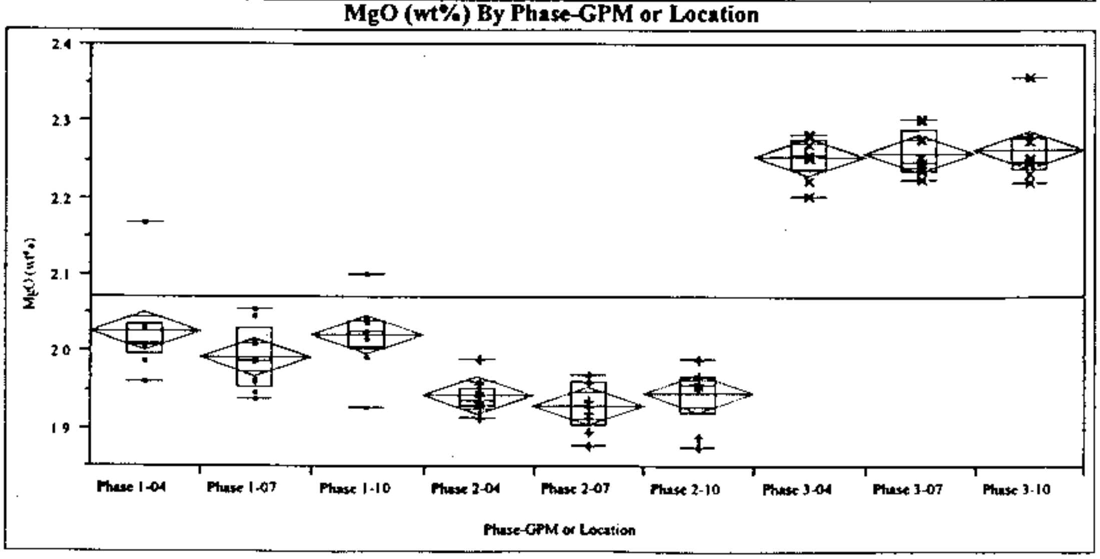
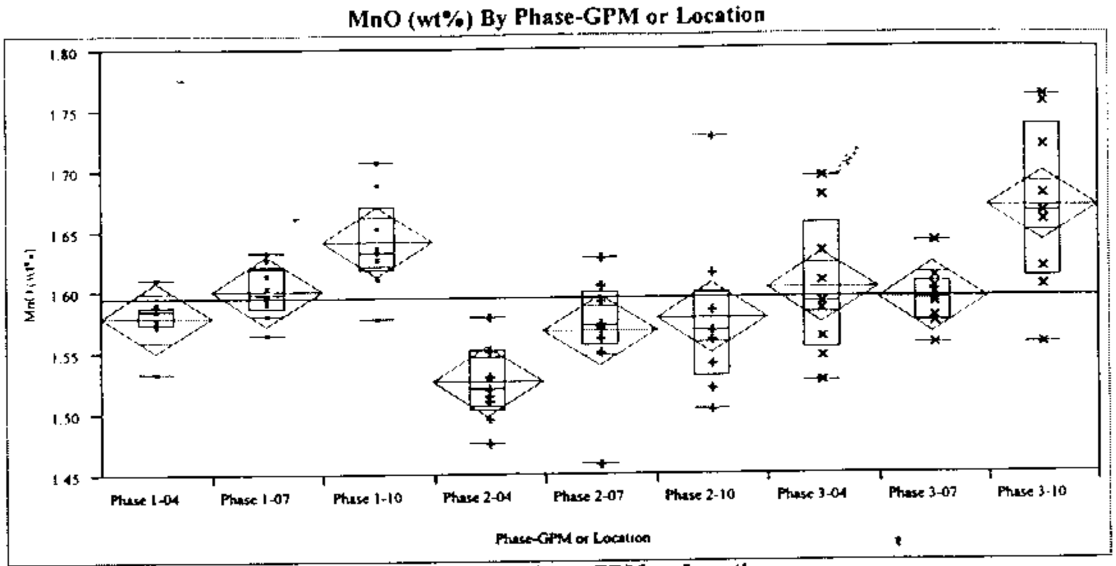
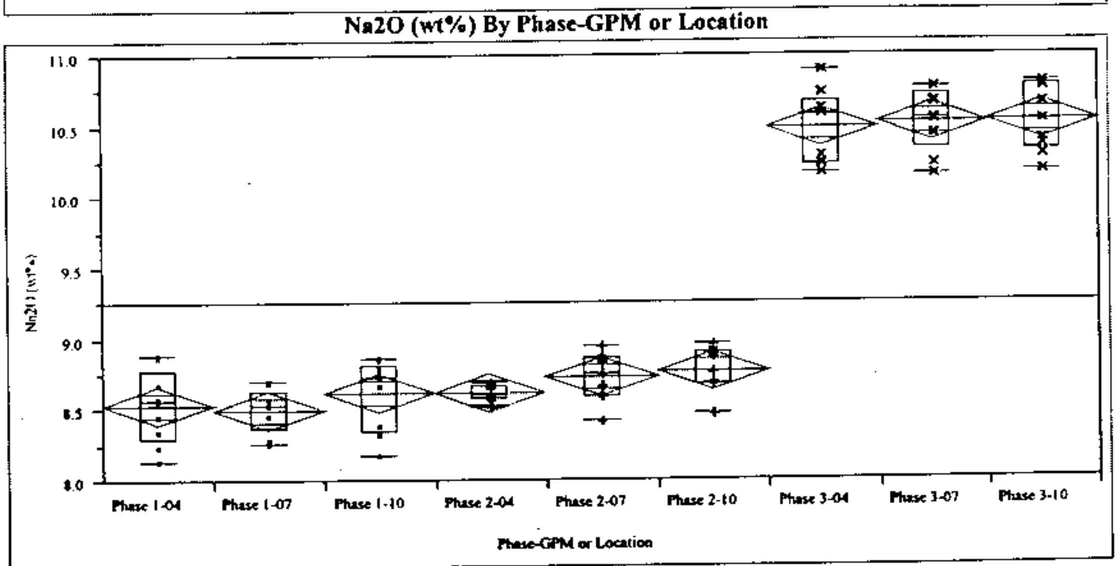


Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases

(Bias-Corrected Measurements are indicated by "bc")





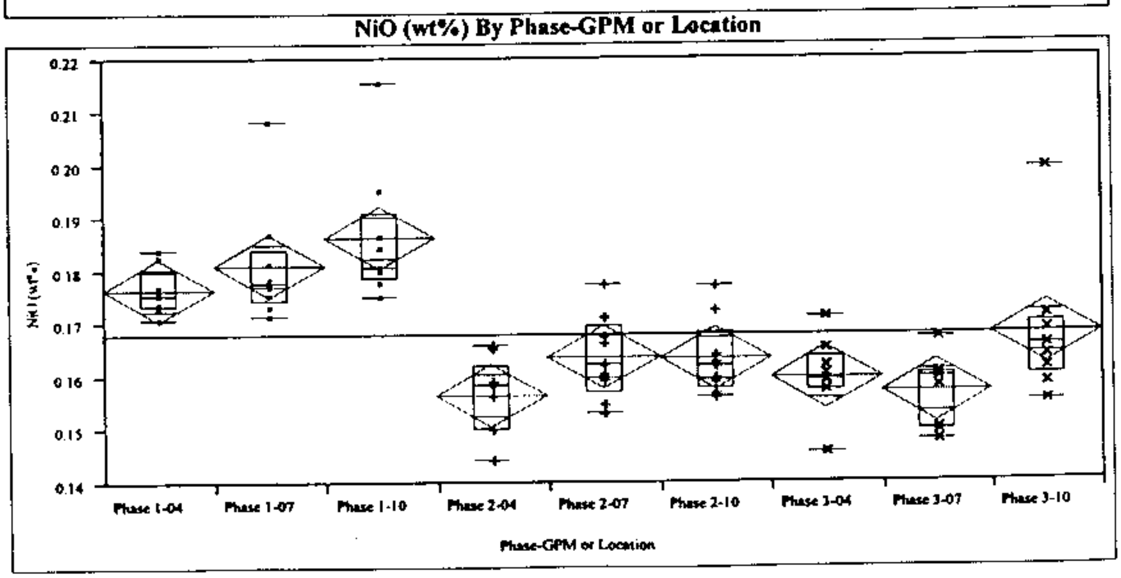
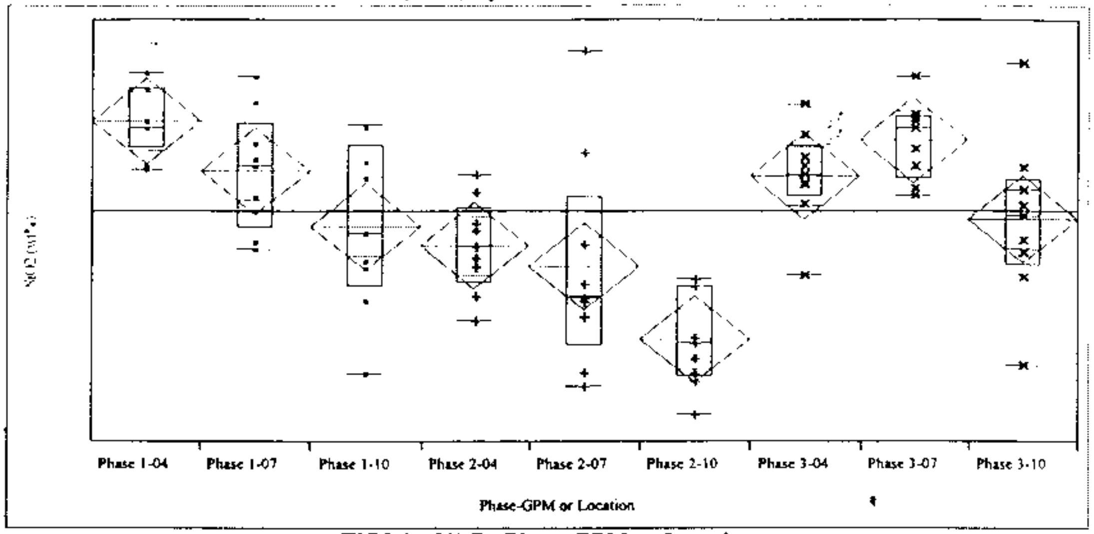


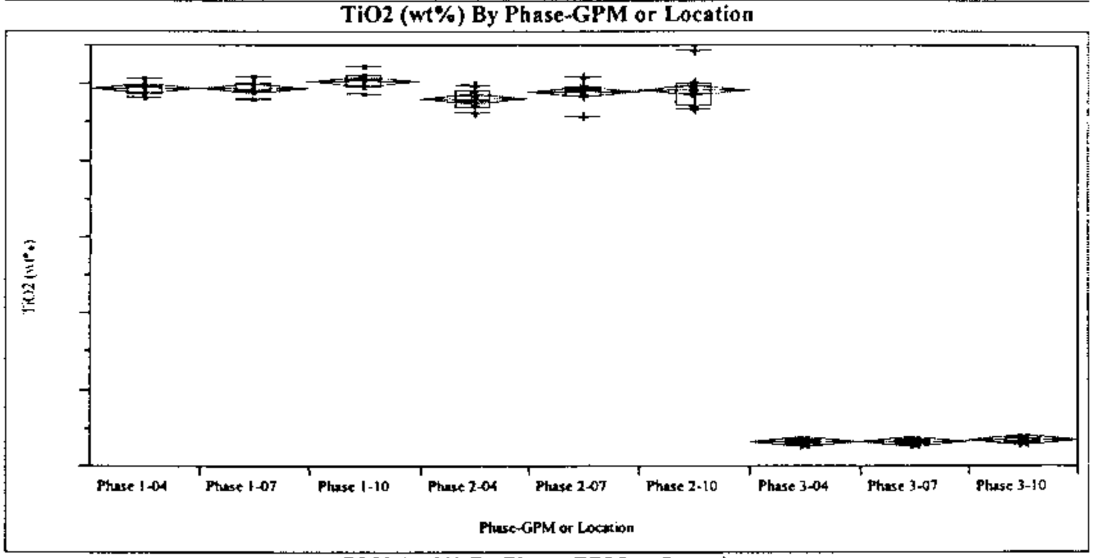
Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

SiO2 (wt%) By Phase-GPM or Location



TiO2 (wt%) By Phase-GPM or Location



PM1 (wt%) By Phase-GPM or Location

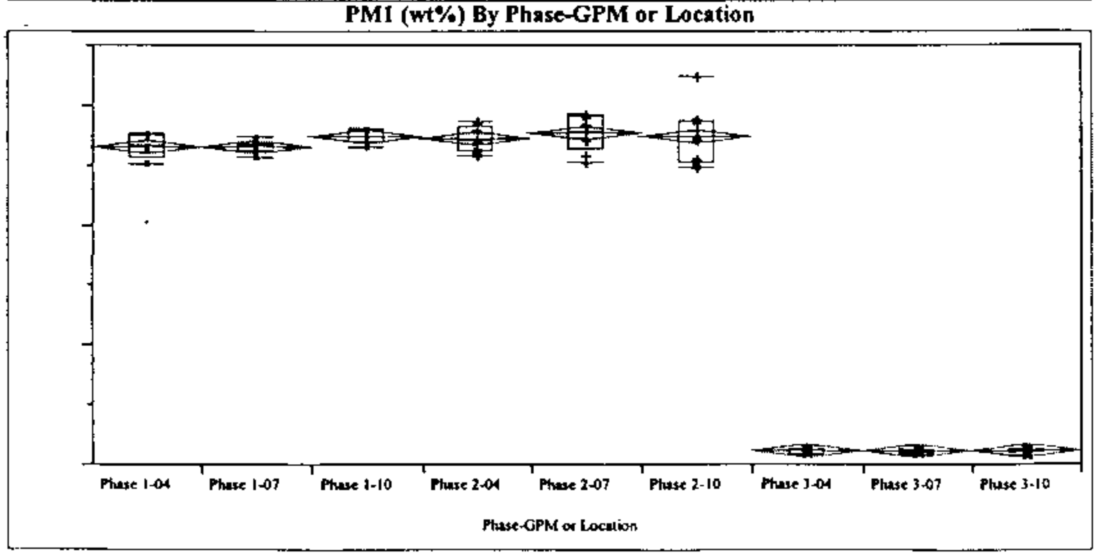
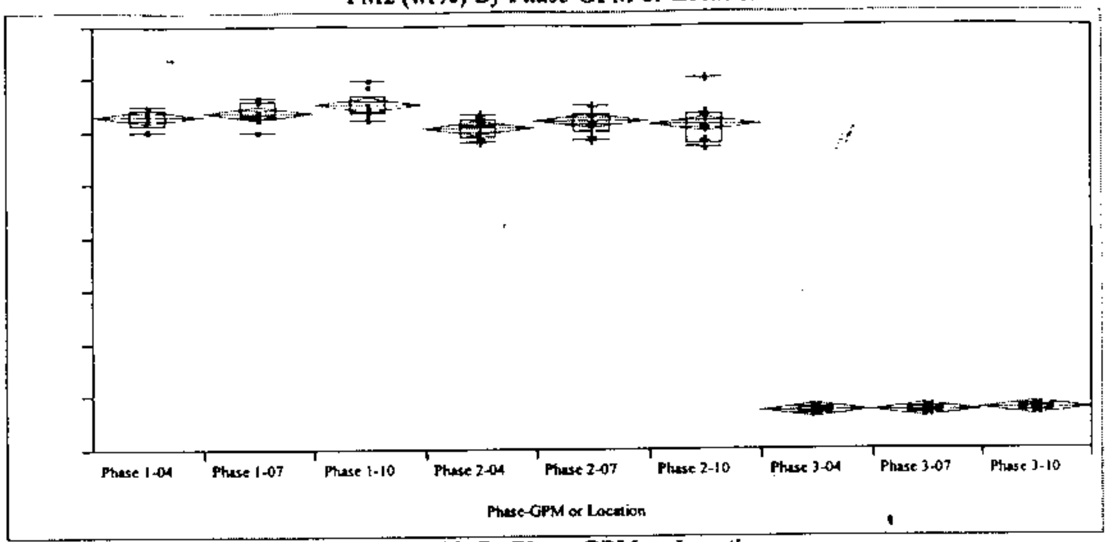
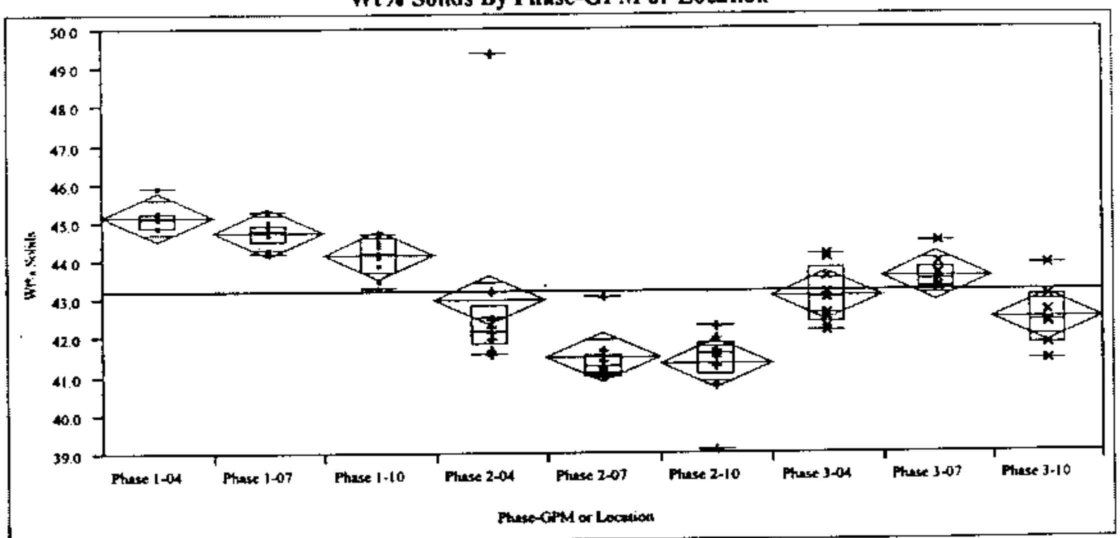


Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases
(Bias-Corrected Measurements are indicated by "bc")

PM2 (wt%) By Phase-GPM or Location



Wt% Solids By Phase-GPM or Location



Al2O3 bc (wt%) By Phase-GPM or Location

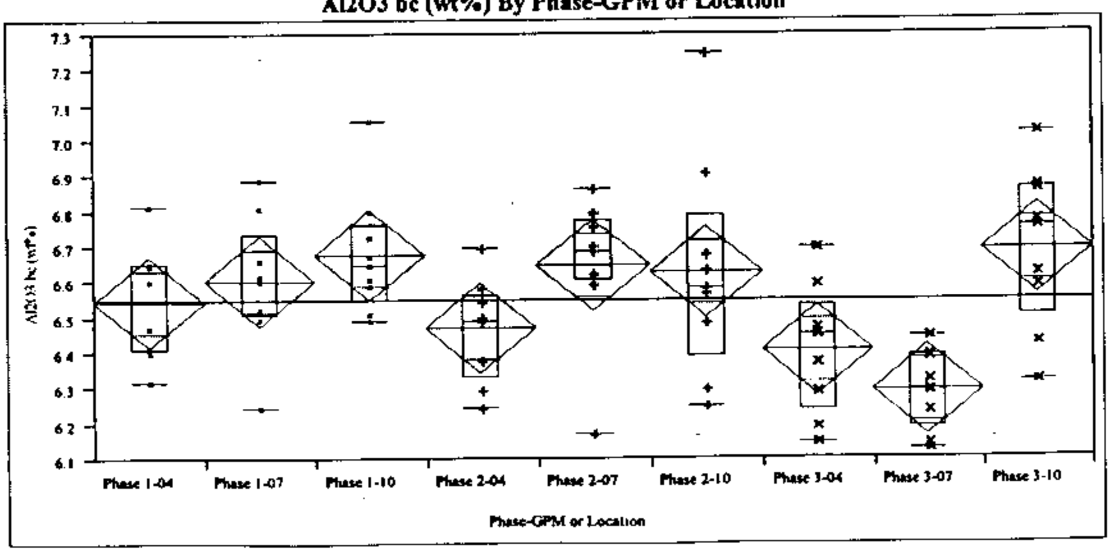
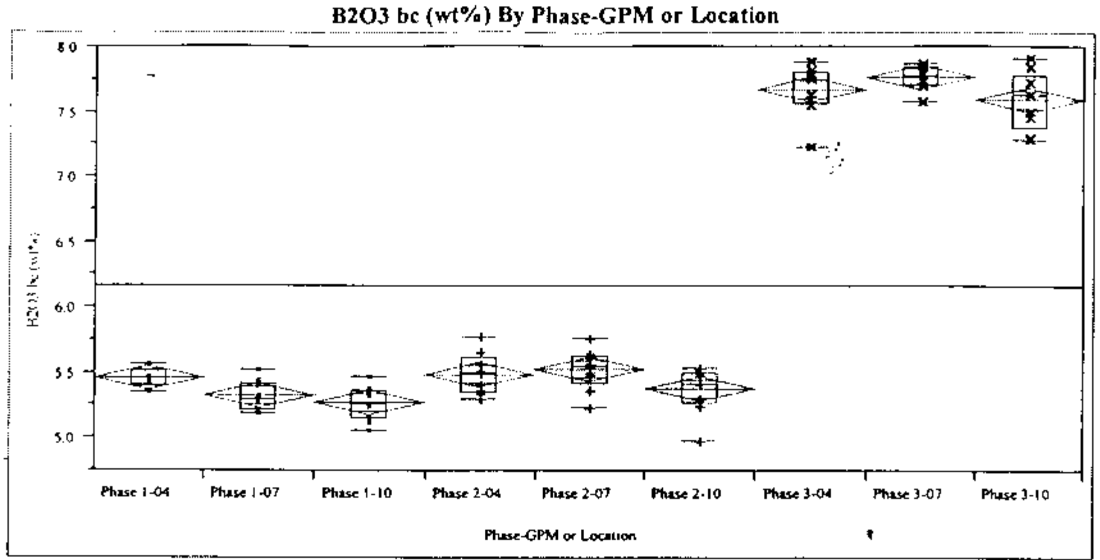
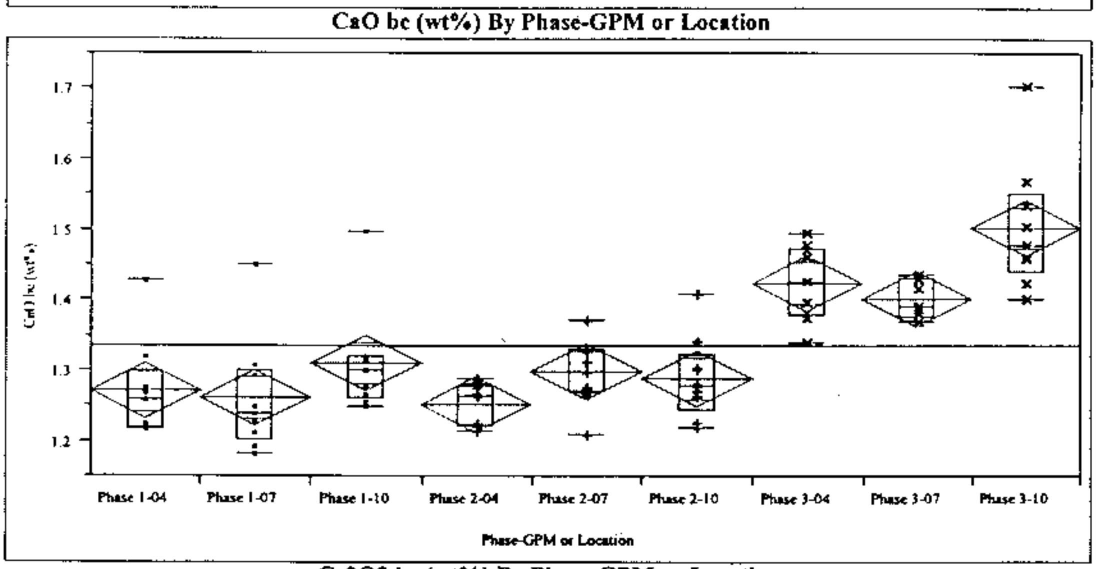


Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases
(Bias-Corrected Measurements are indicated by "bc")





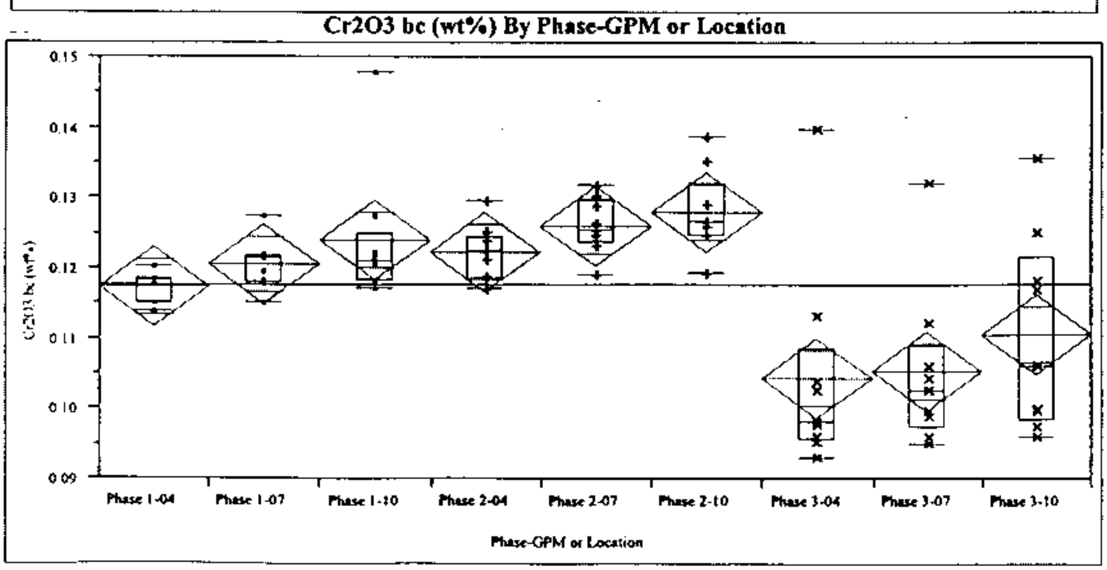
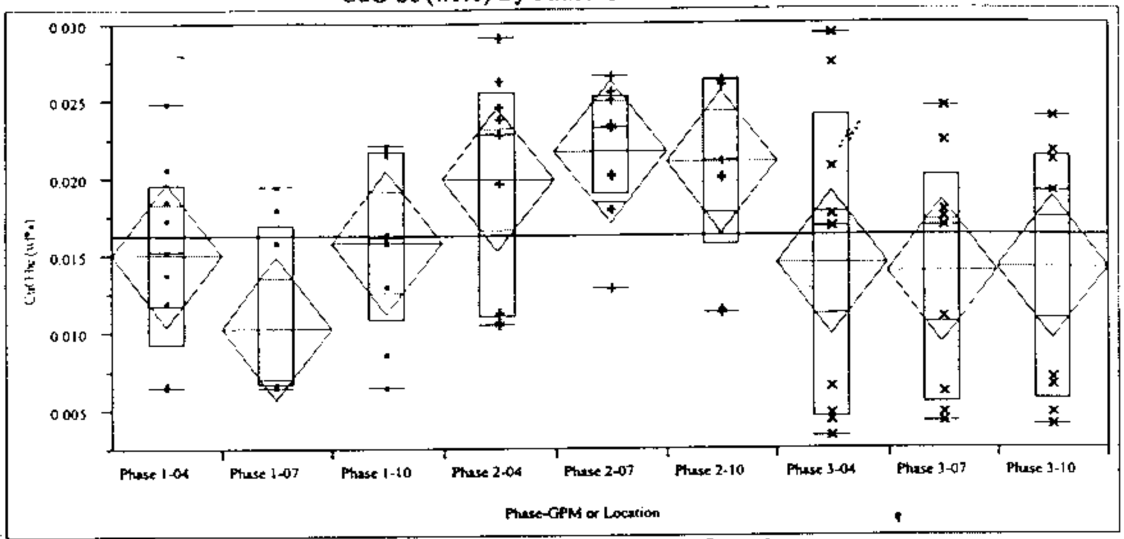


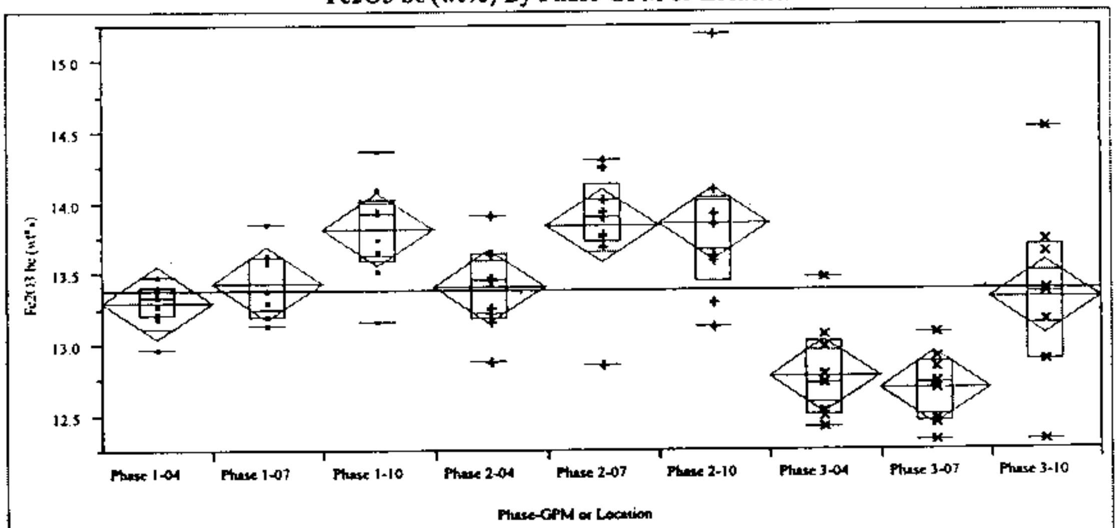
Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

CuO bc (wt%) By Phase-GPM or Location



Fe2O3 bc (wt%) By Phase-GPM or Location



K2O bc (wt%) By Phase-GPM or Location

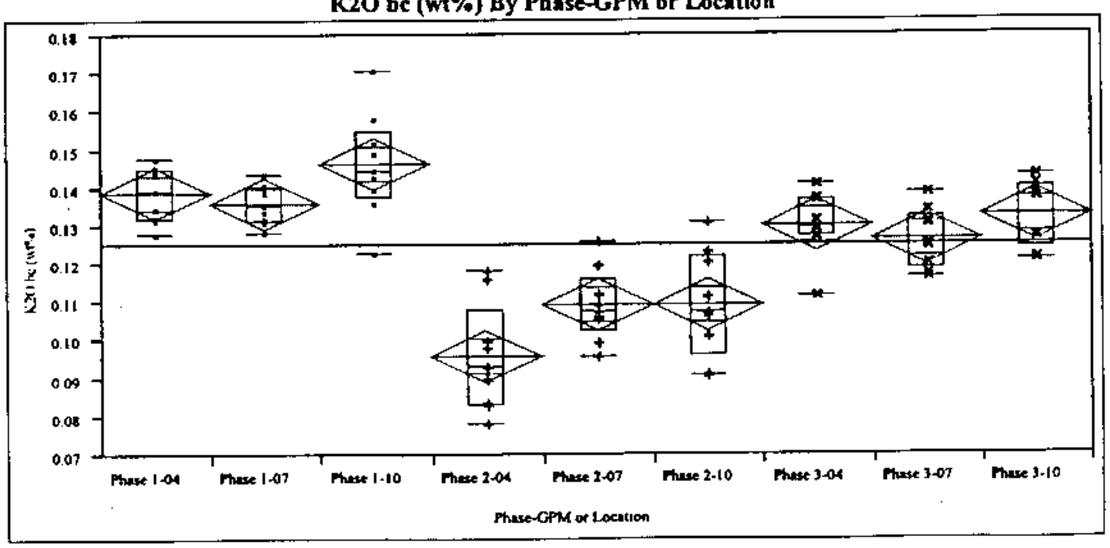
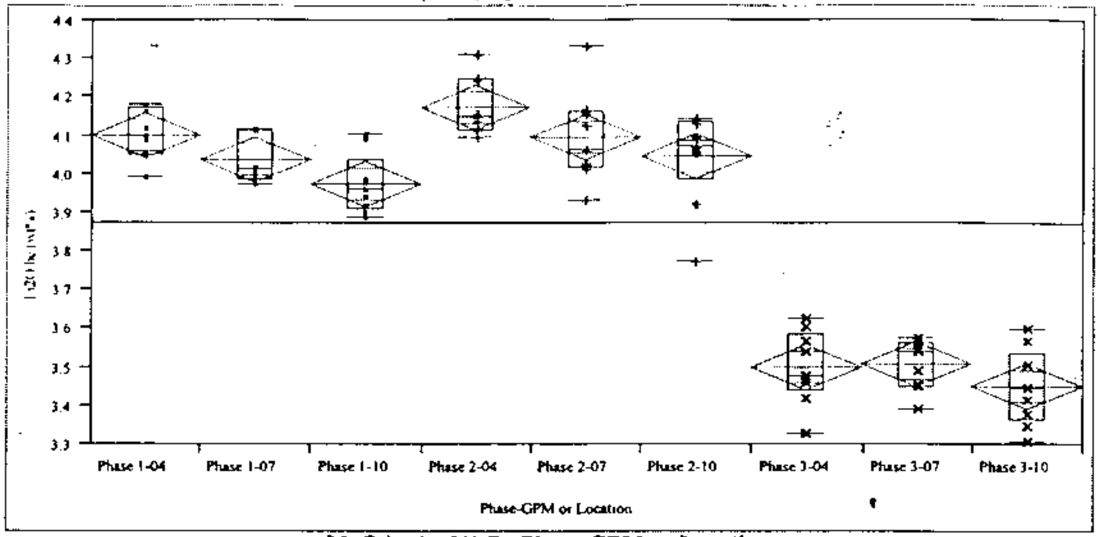
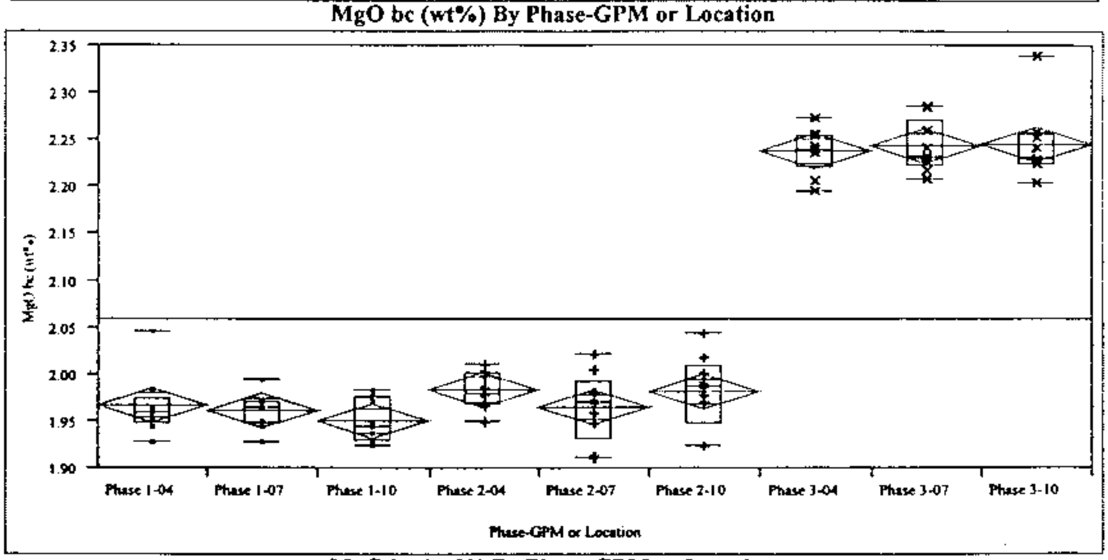


Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases
(Bias-Corrected Measurements are indicated by "bc")

Li2O bc (wt%) By Phase-GPM or Location



MgO bc (wt%) By Phase-GPM or Location



MnO bc (wt%) By Phase-GPM or Location

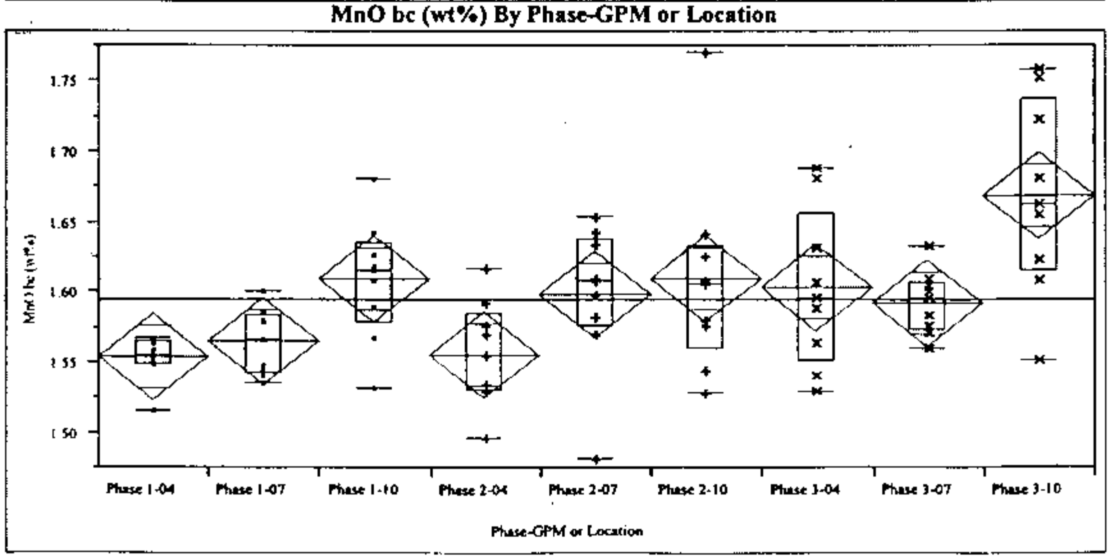
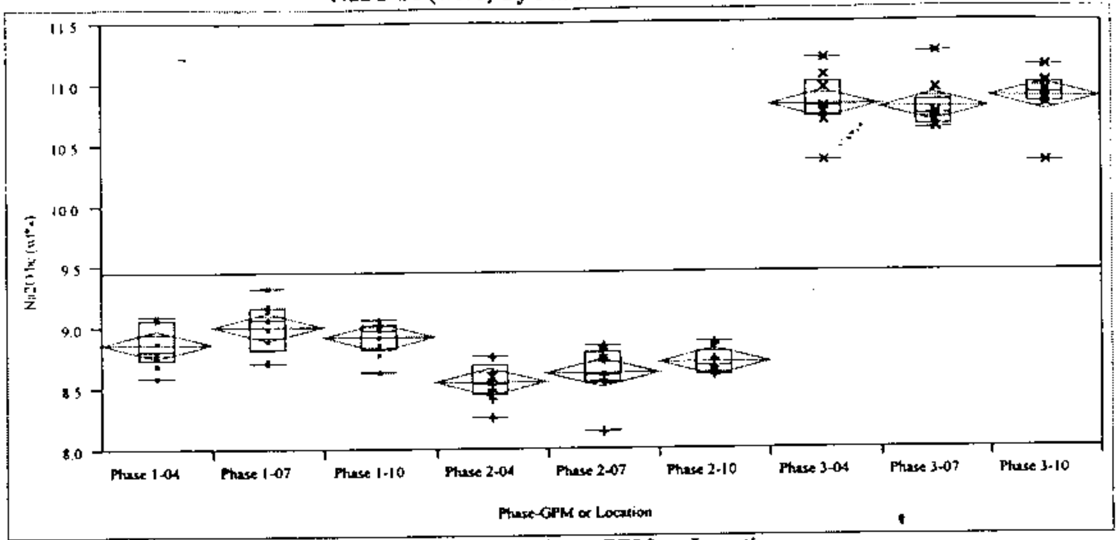
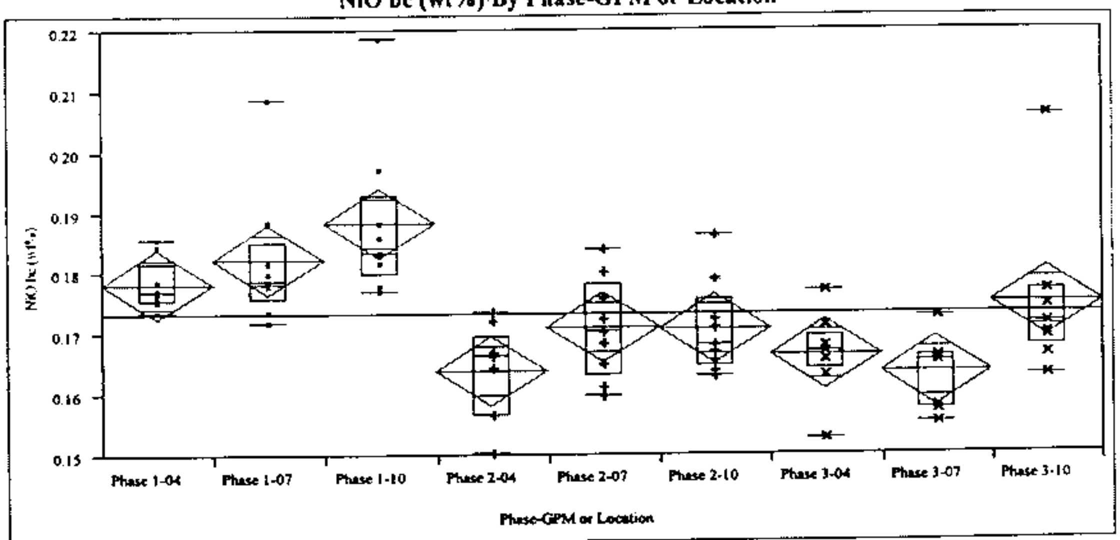


Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases
(Bias-Corrected Measurements are indicated by "bc")

Na2O bc (wt%) By Phase-GPM or Location



NiO bc (wt%) By Phase-GPM or Location



SiO2 bc (wt%) By Phase-GPM or Location

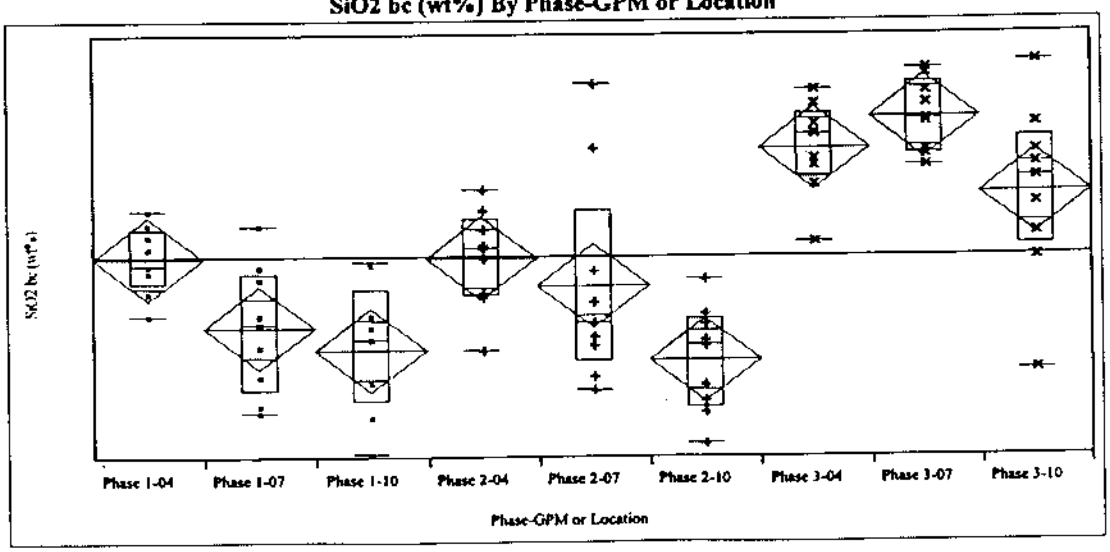
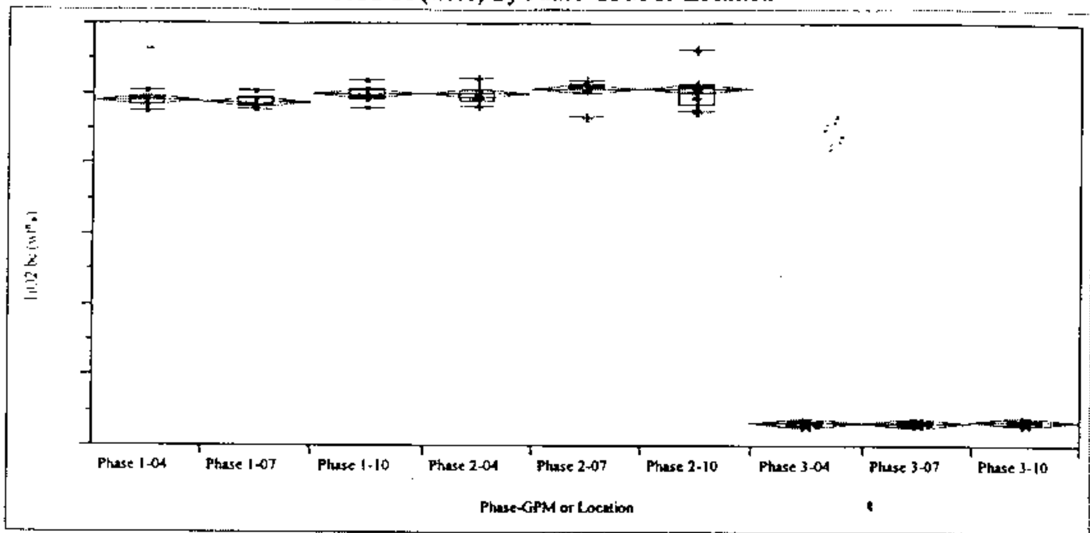


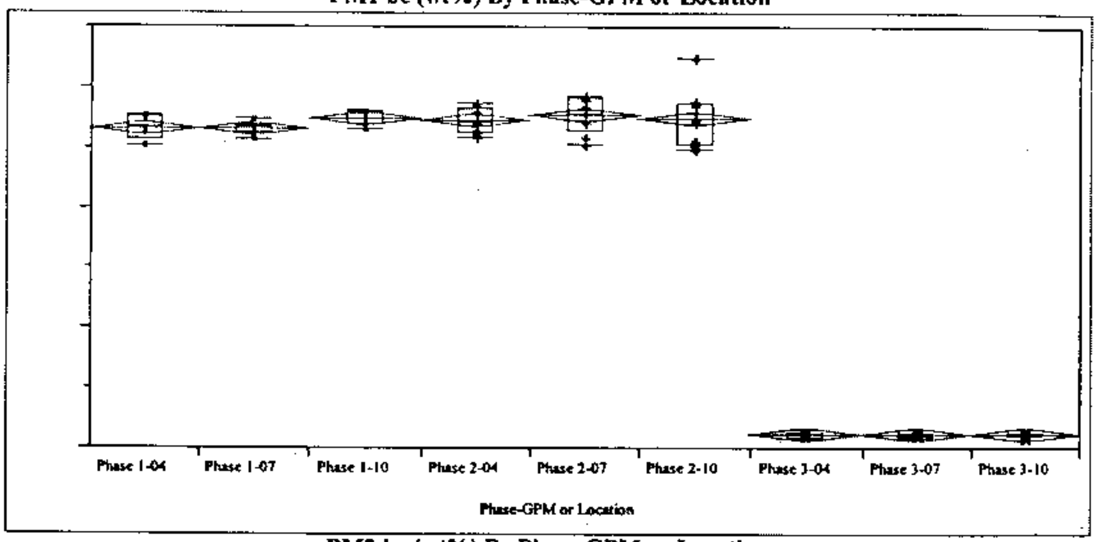
Exhibit A2. Comparisons of Samples Across Hydragard® Flowrates and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

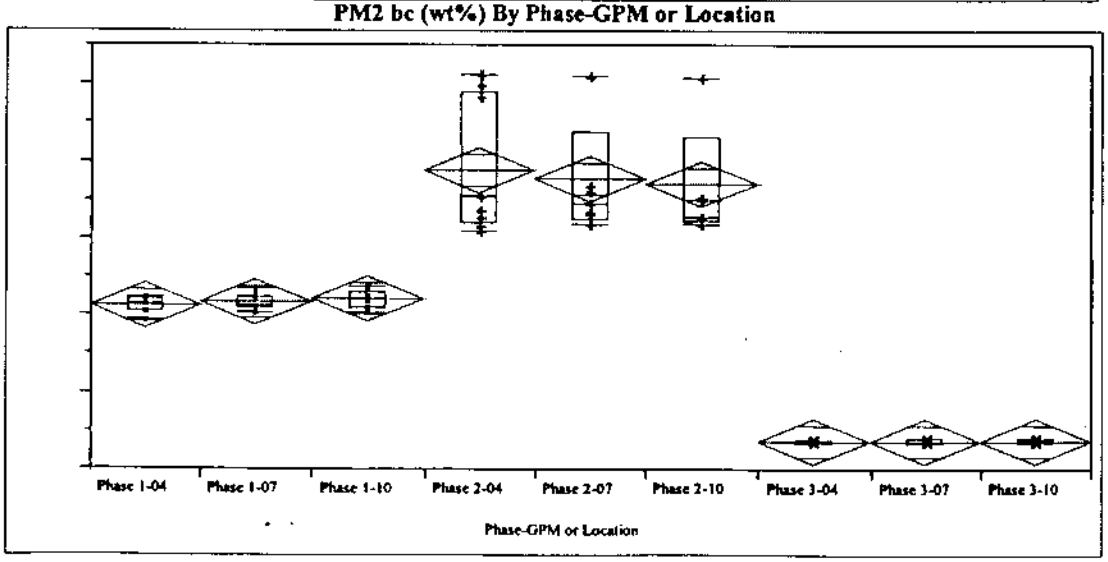
TiO2 bc (wt%) By Phase-GPM or Location



PM1 bc (wt%) By Phase-GPM or Location



PM2 bc (wt%) By Phase-GPM or Location

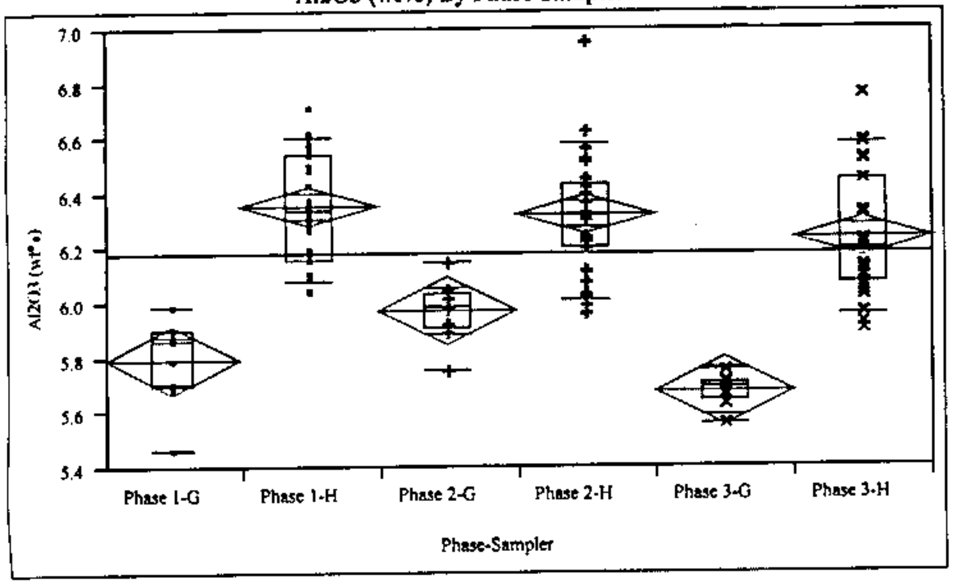


Appendix A.

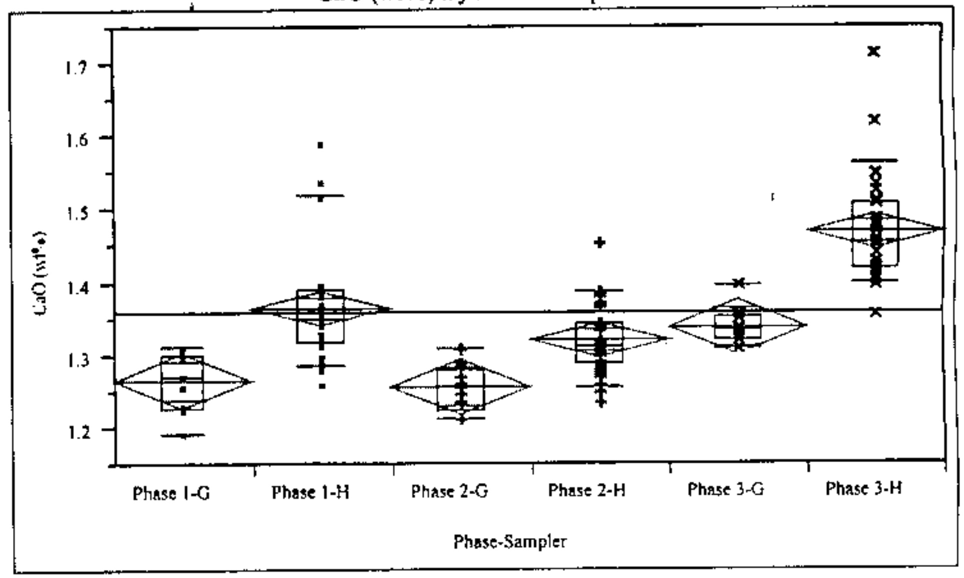
Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

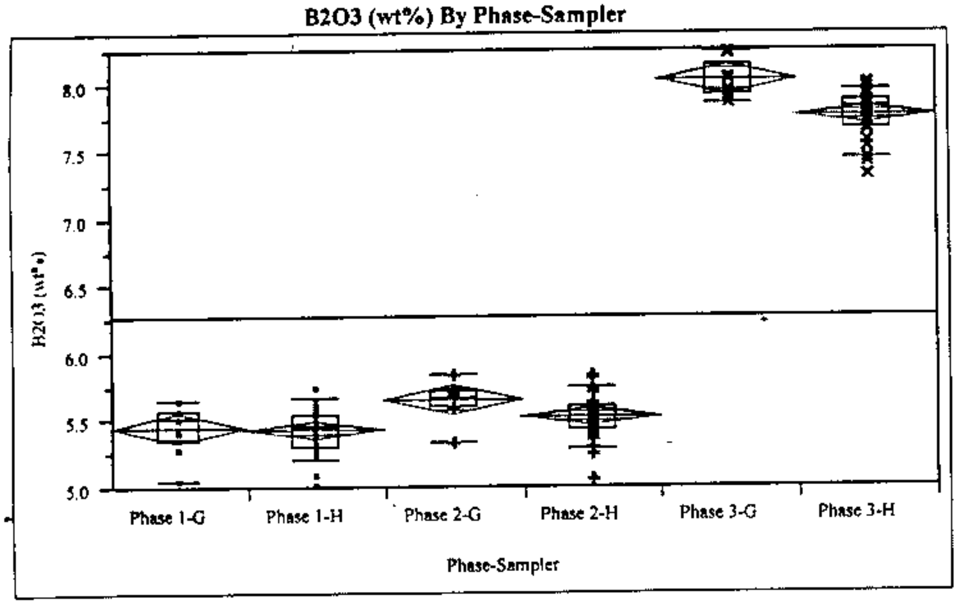
Al2O3 (wt%) By Phase-Sampler



CaO (wt%) By Phase-Sampler



B2O3 (wt%) By Phase-Sampler



Cr2O3 (wt%) By Phase-Sampler

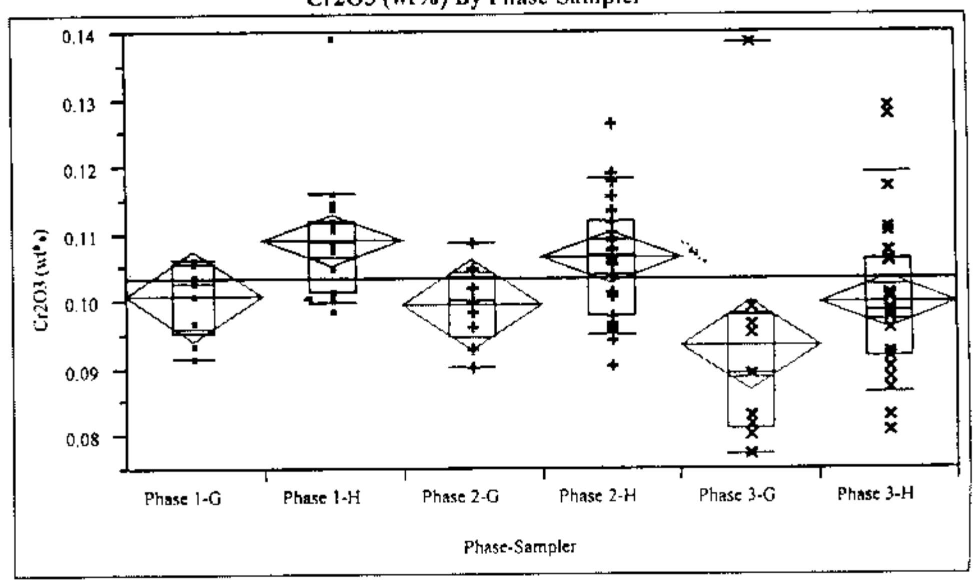
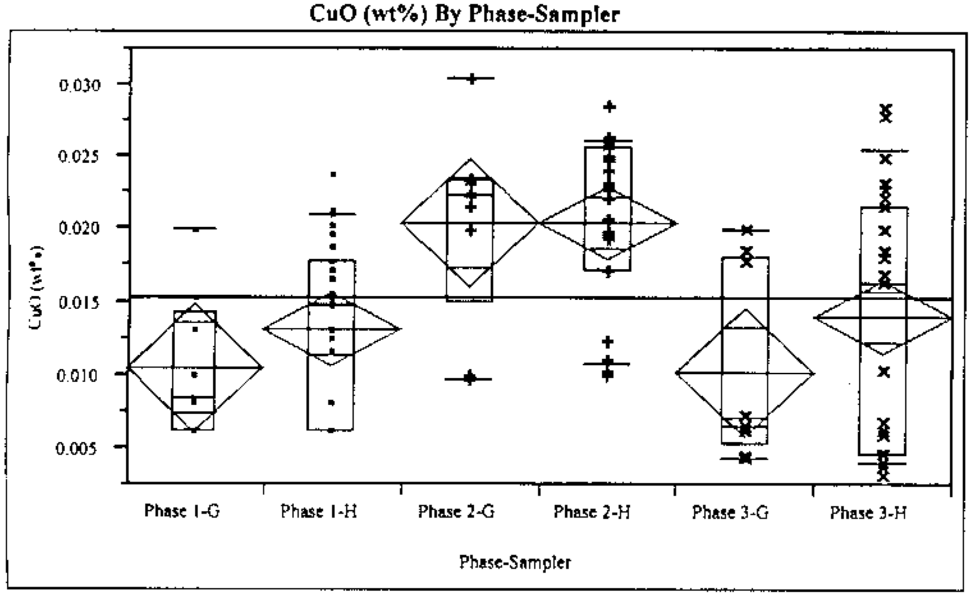
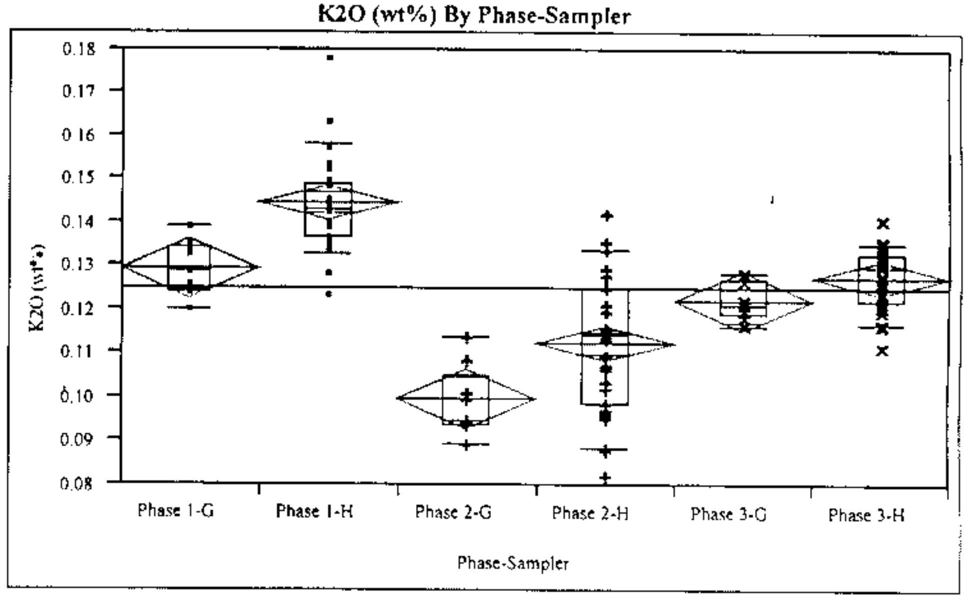
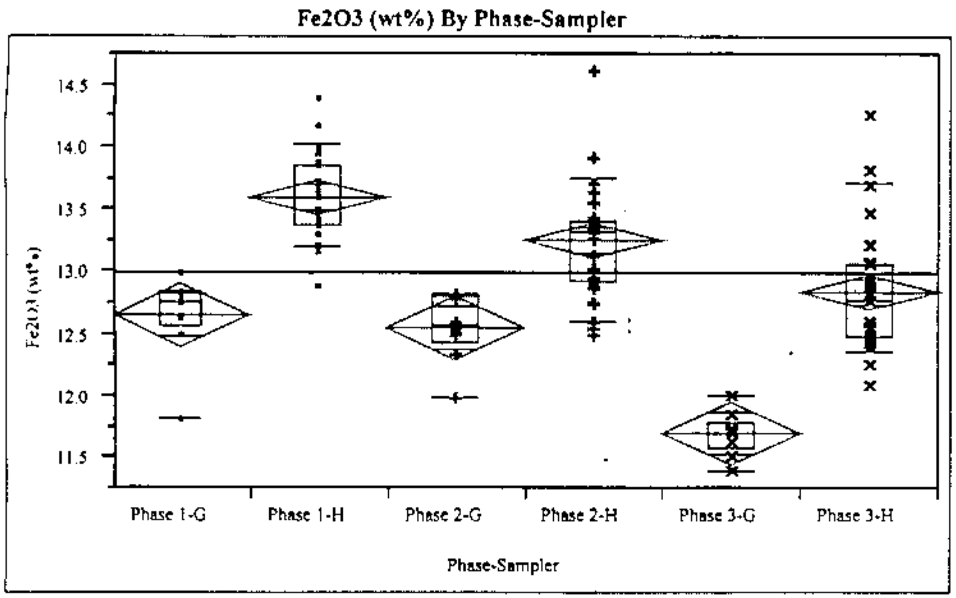
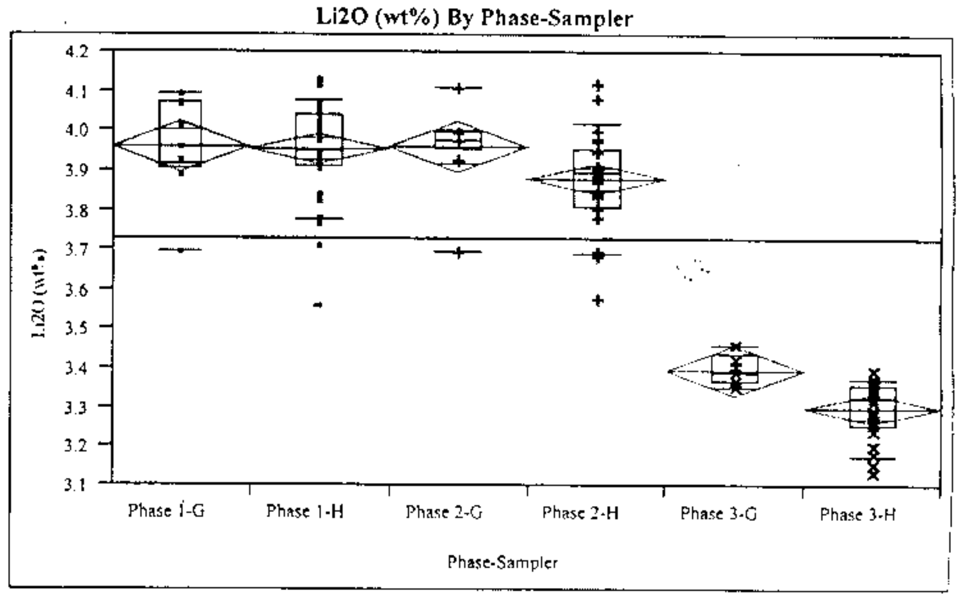


Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases
(Bias-Corrected Measurements are indicated by "bc")







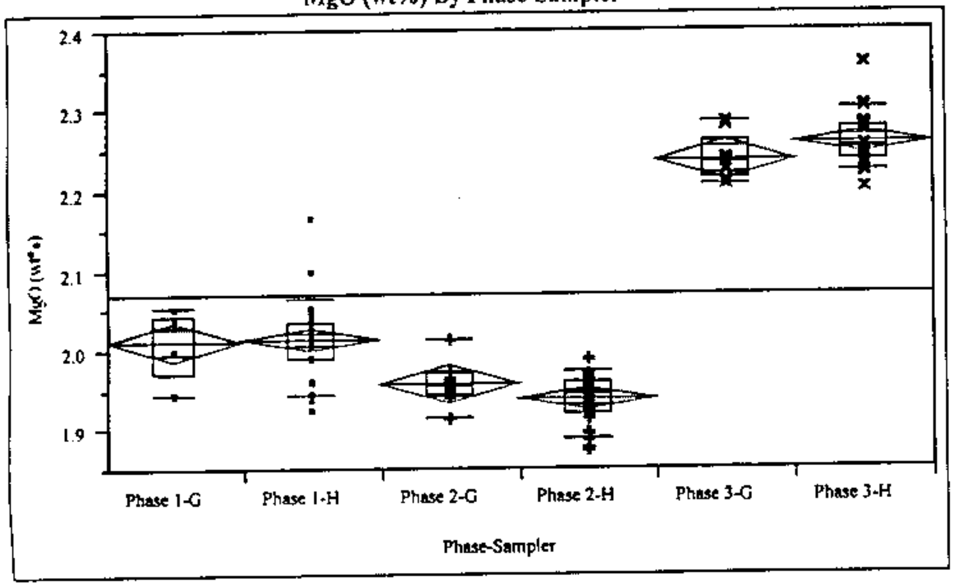


Appendix A.

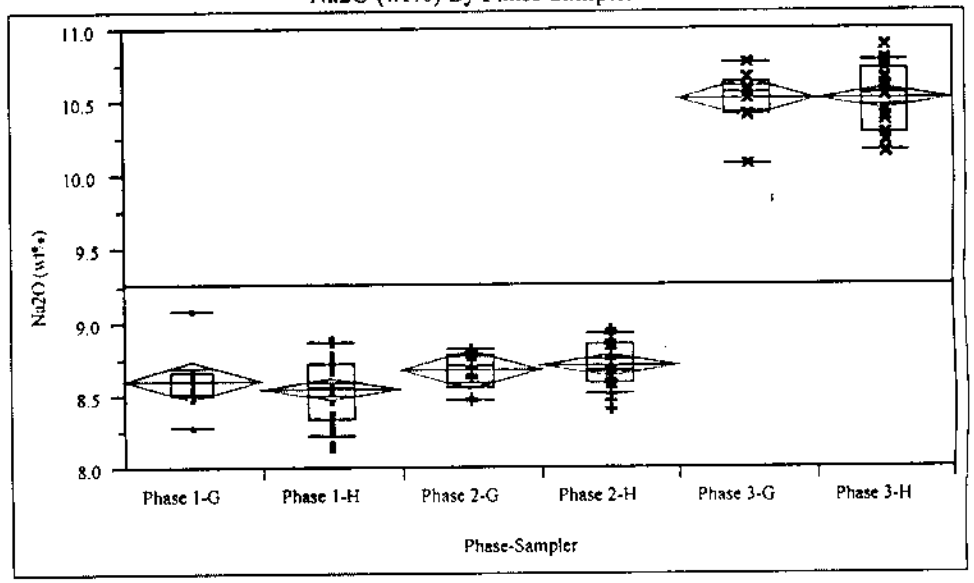
Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

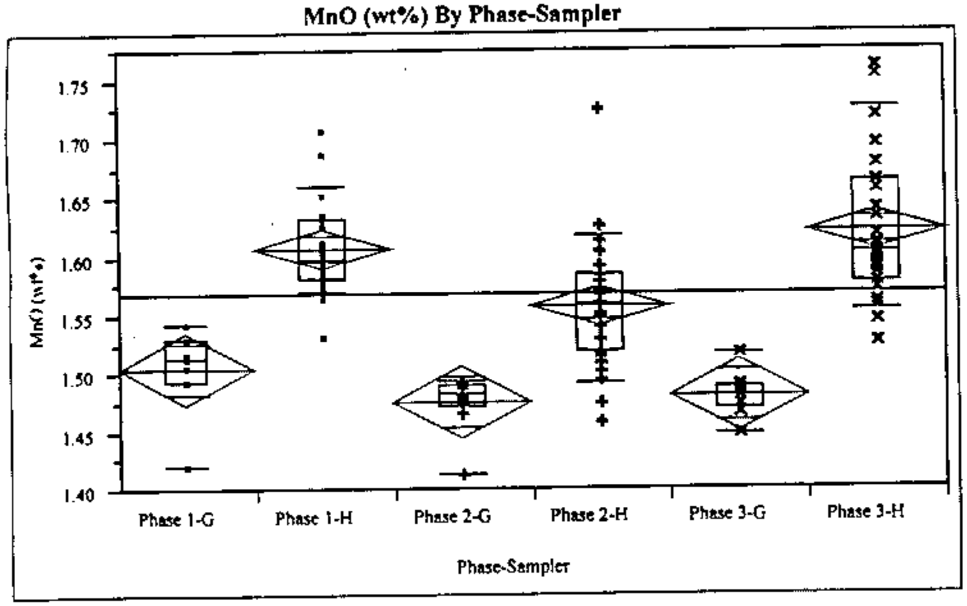
MgO (wt%) By Phase-Sampler



Na2O (wt%) By Phase-Sampler



MnO (wt%) By Phase-Sampler



NiO (wt%) By Phase-Sampler

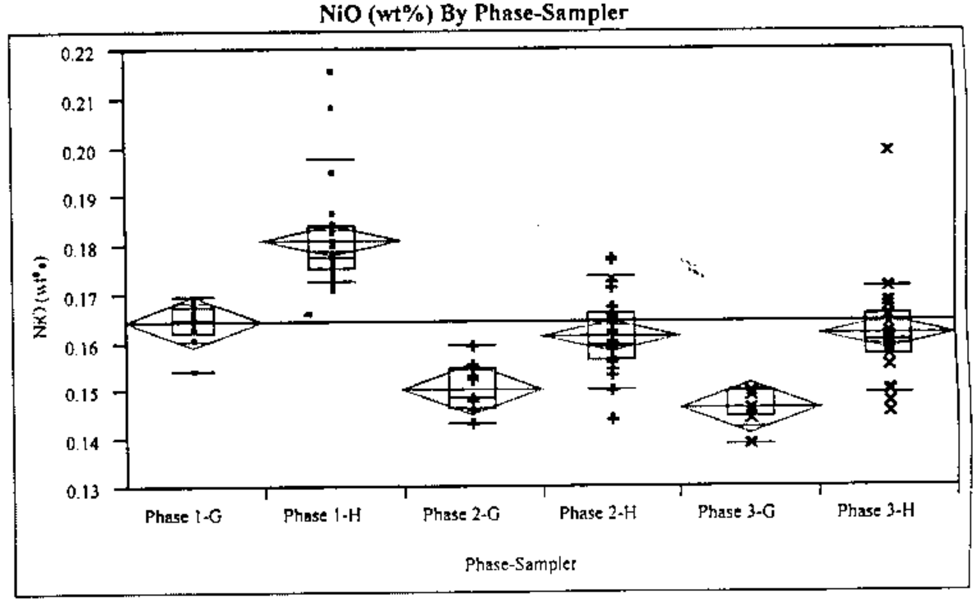
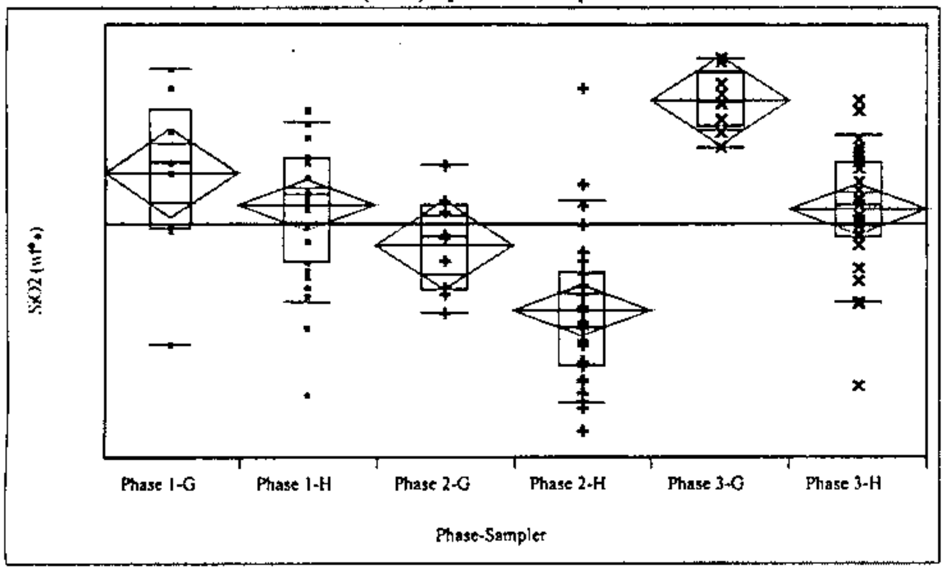
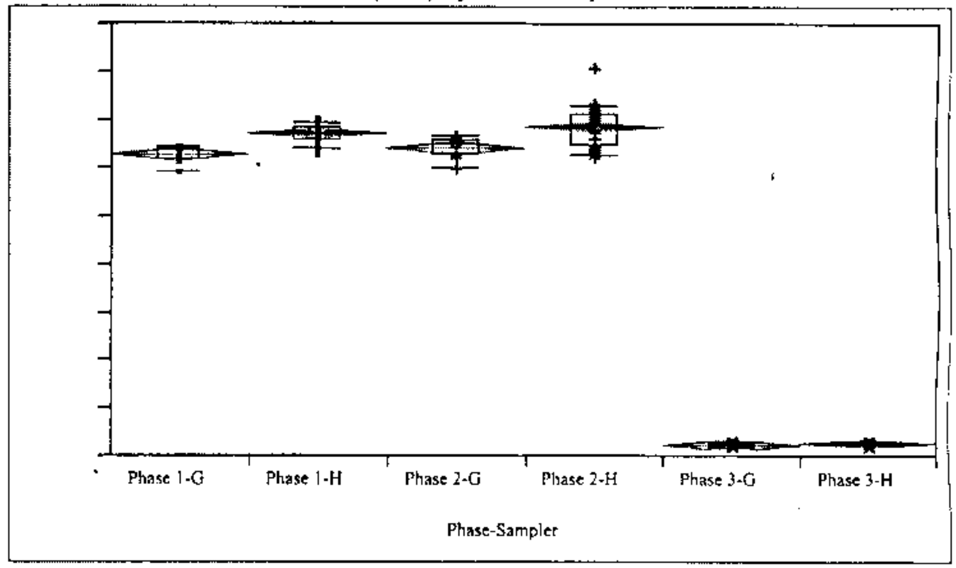


Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases
(Bias-Corrected Measurements are indicated by "bc")

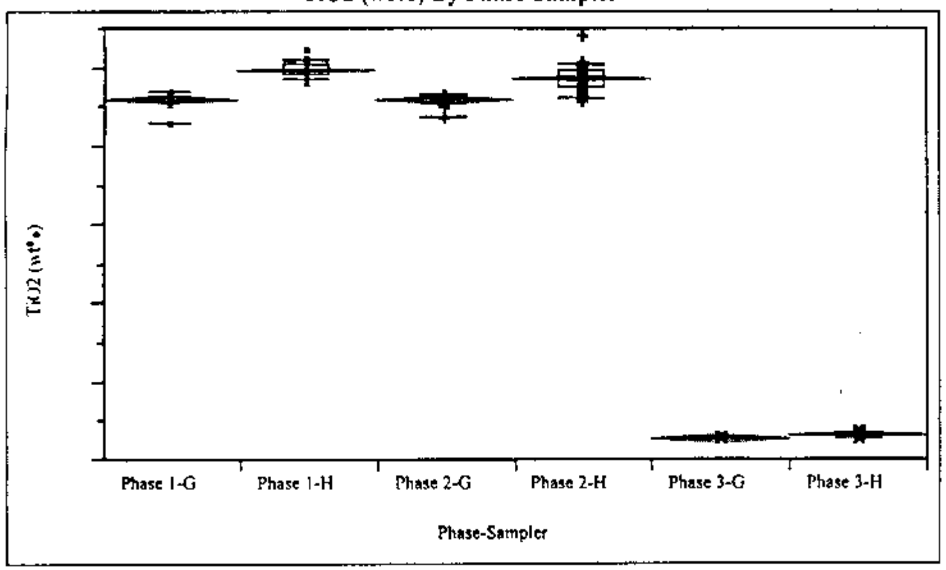
SiO2 (wt%) By Phase-Sampler



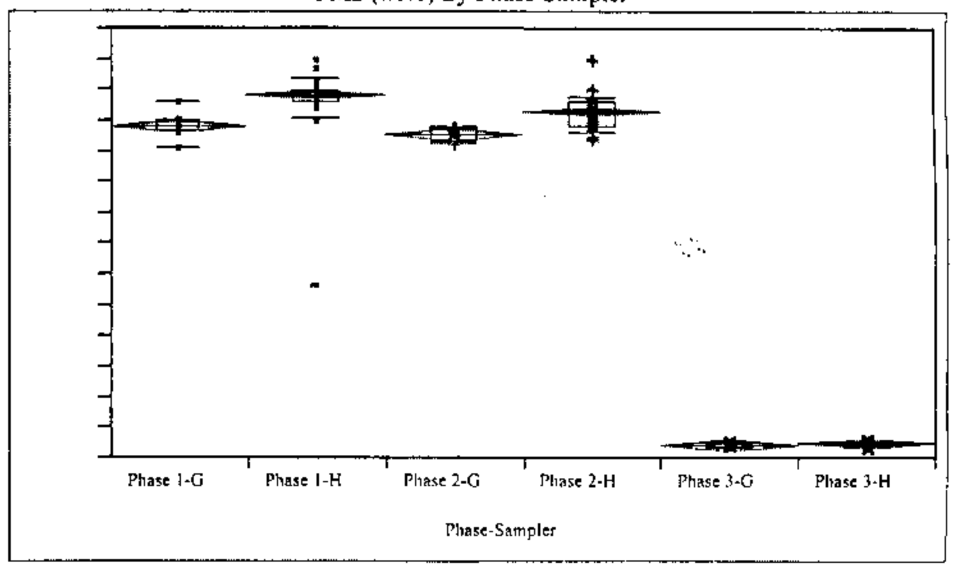
PM1 (wt%) By Phase-Sampler



TiO2 (wt%) By Phase-Sampler



PM2 (wt%) By Phase-Sampler

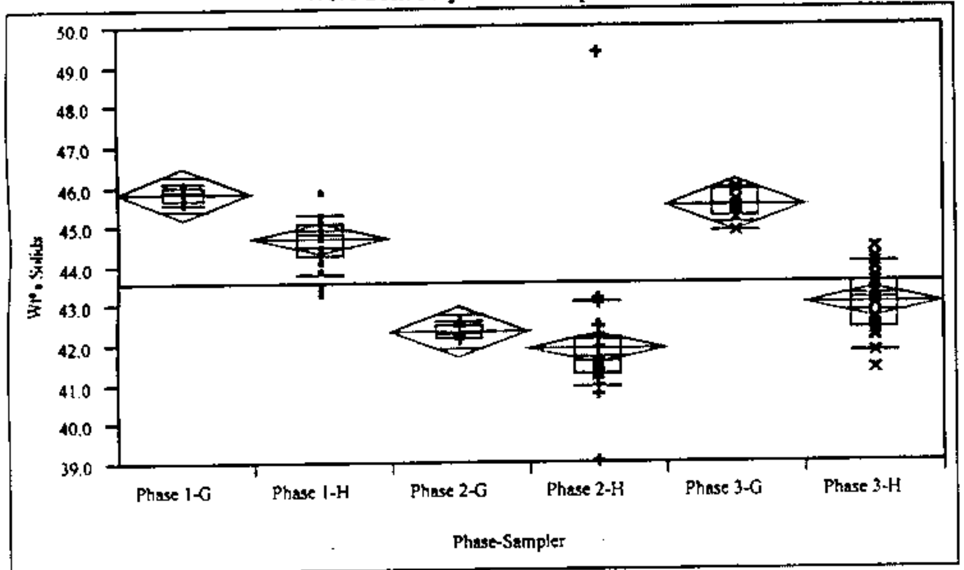


Appendix A.

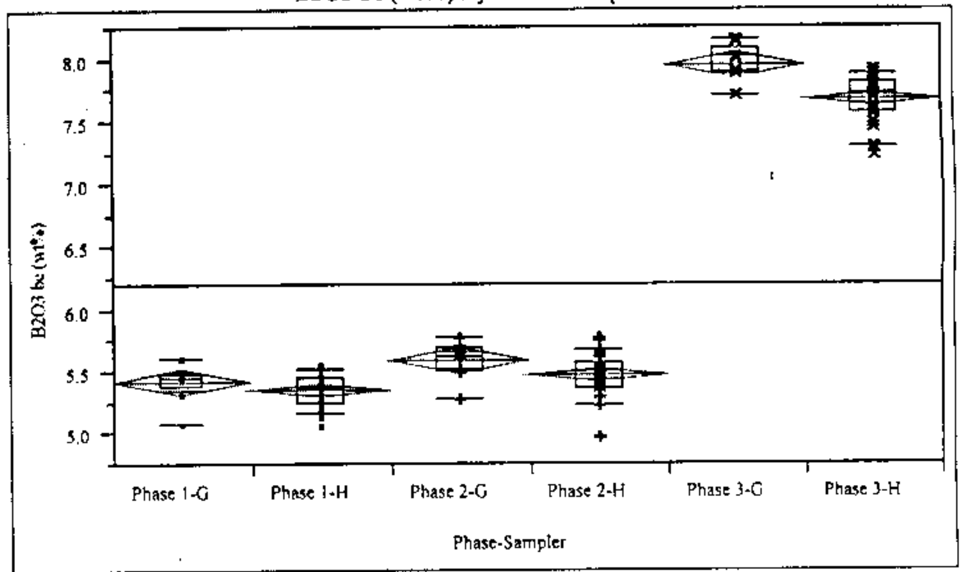
Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

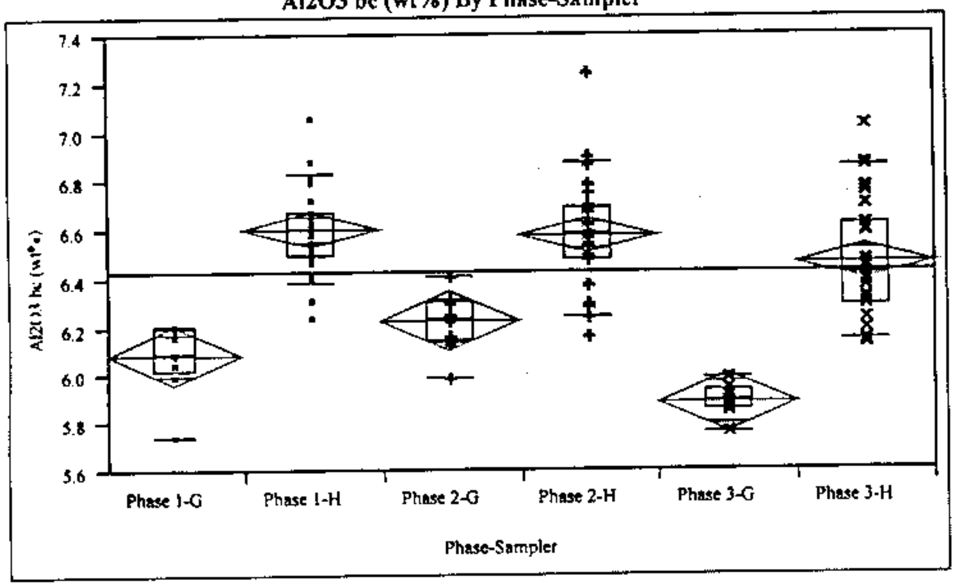
Wt% Solids By Phase-Sampler



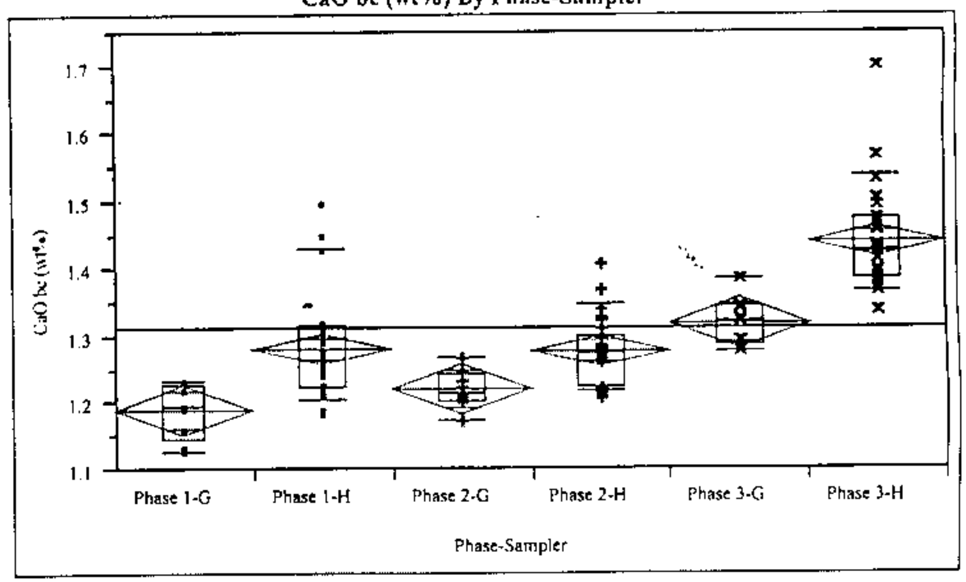
B2O3 bc (wt%) By Phase-Sampler



Al2O3 bc (wt%) By Phase-Sampler



CaO bc (wt%) By Phase-Sampler

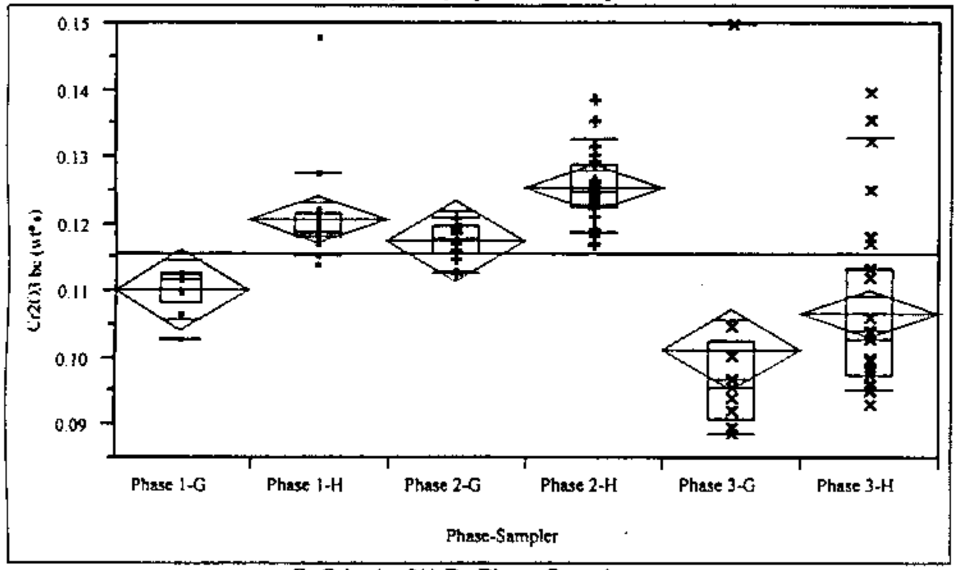


Appendix A.

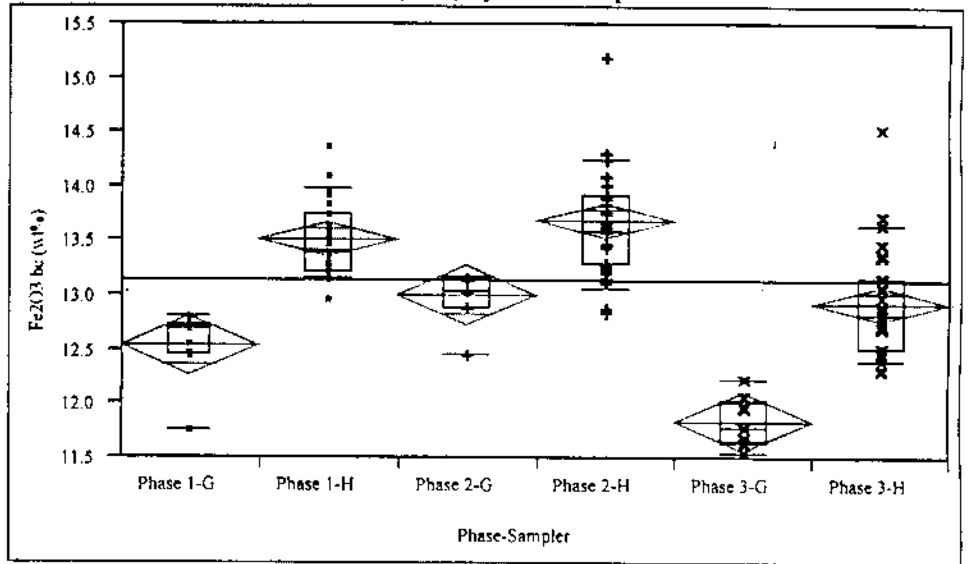
Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

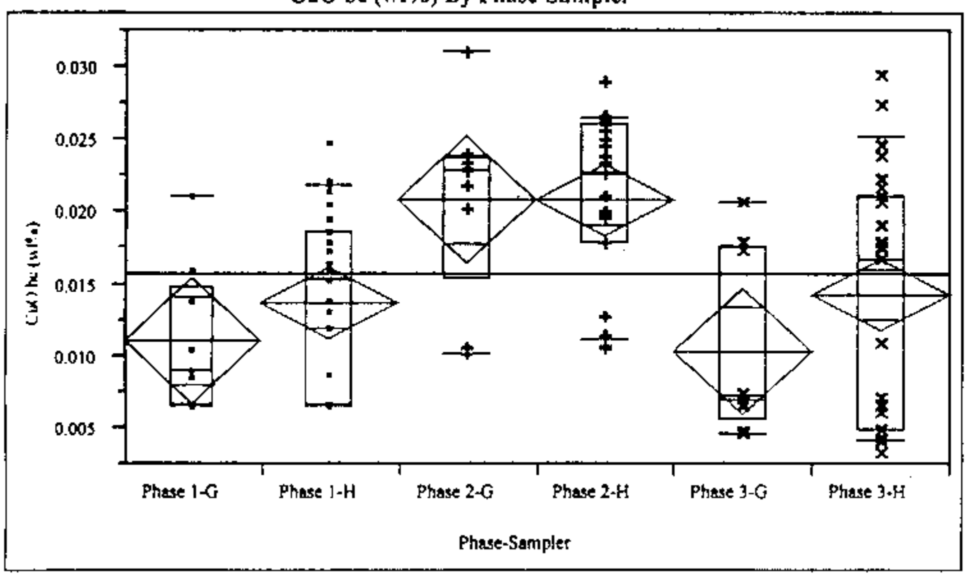
Cr2O3 bc (wt%) By Phase-Sampler



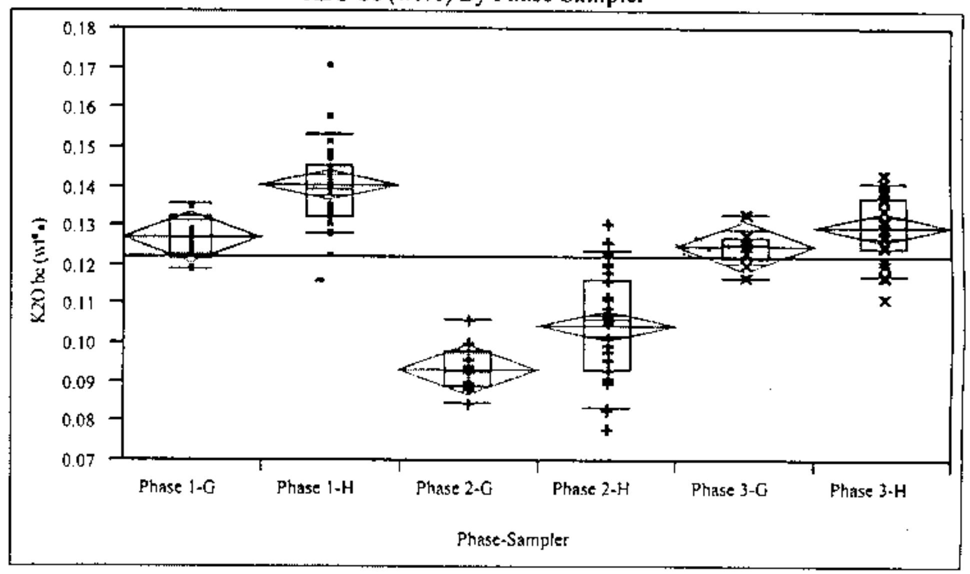
Fe2O3 bc (wt%) By Phase-Sampler



CuO bc (wt%) By Phase-Sampler



K2O bc (wt%) By Phase-Sampler

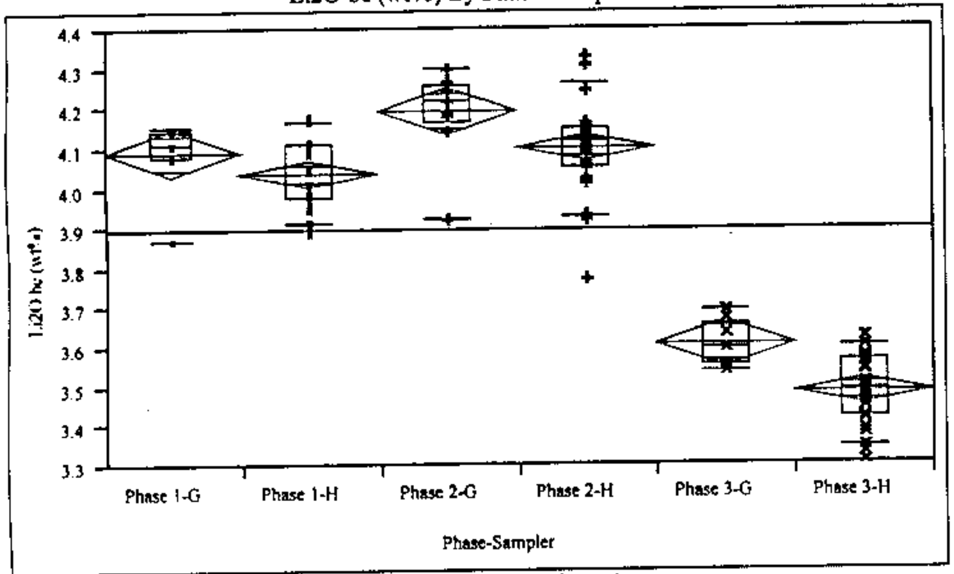


Appendix A.

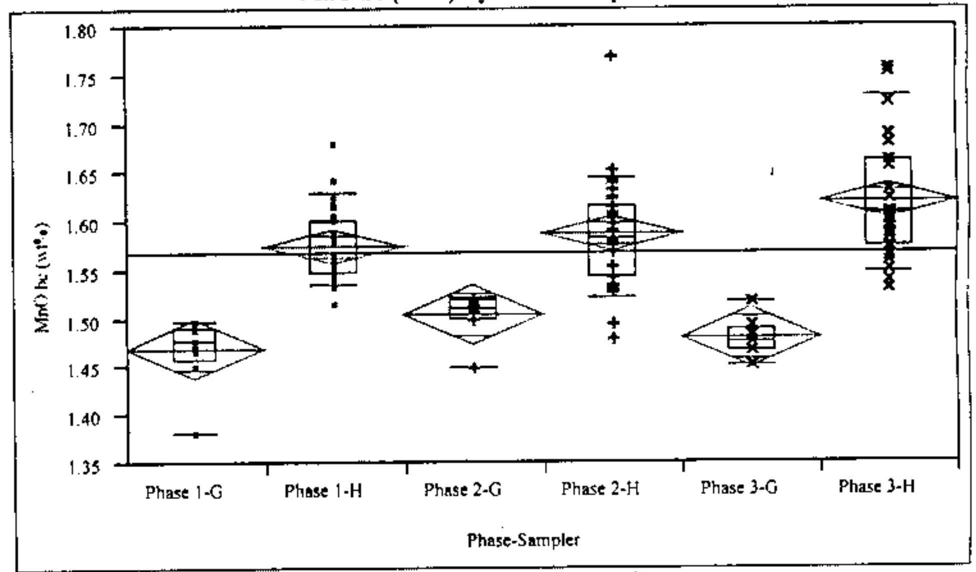
Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

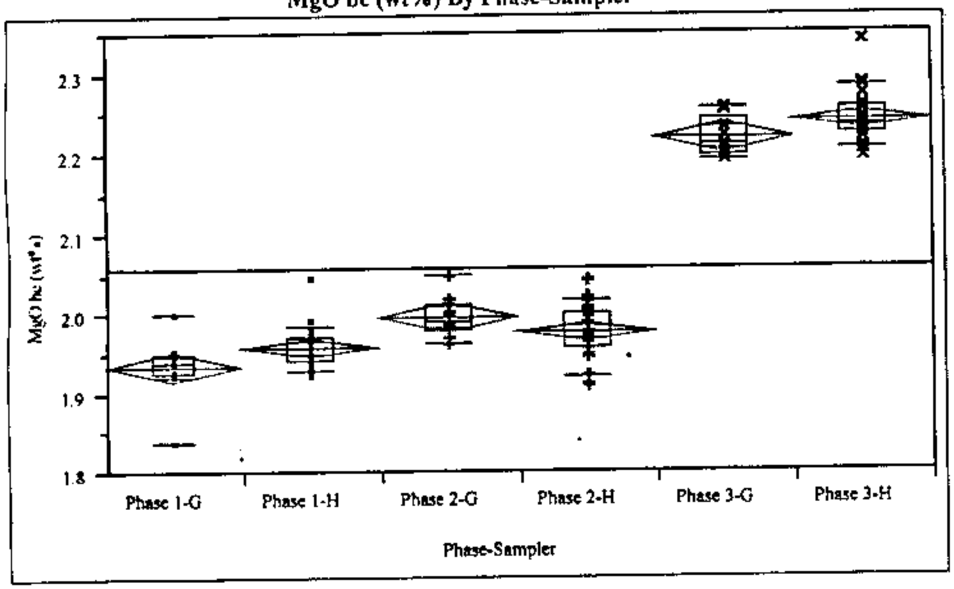
Li2O bc (wt%) By Phase-Sampler



MnO bc (wt%) By Phase-Sampler



MgO bc (wt%) By Phase-Sampler



Na2O bc (wt%) By Phase-Sampler

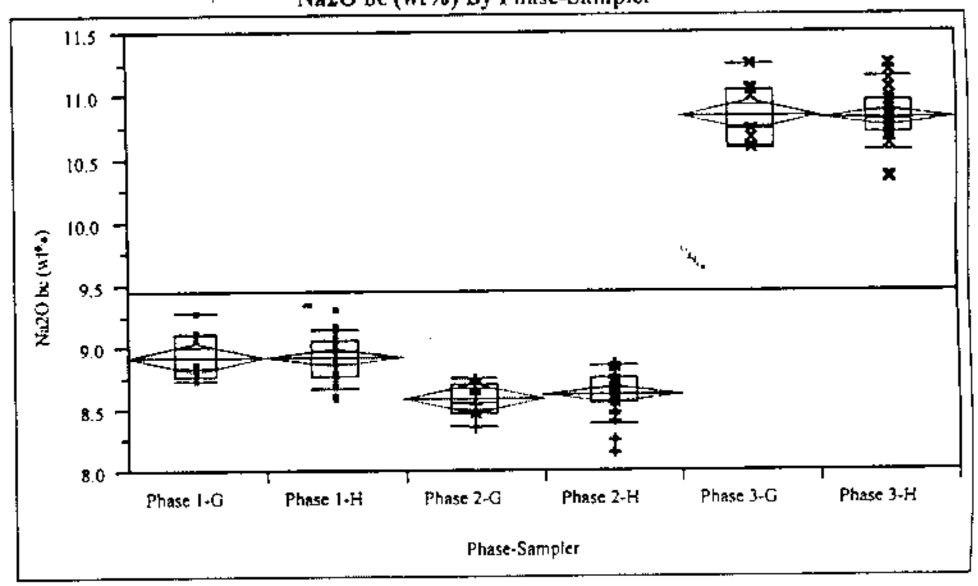
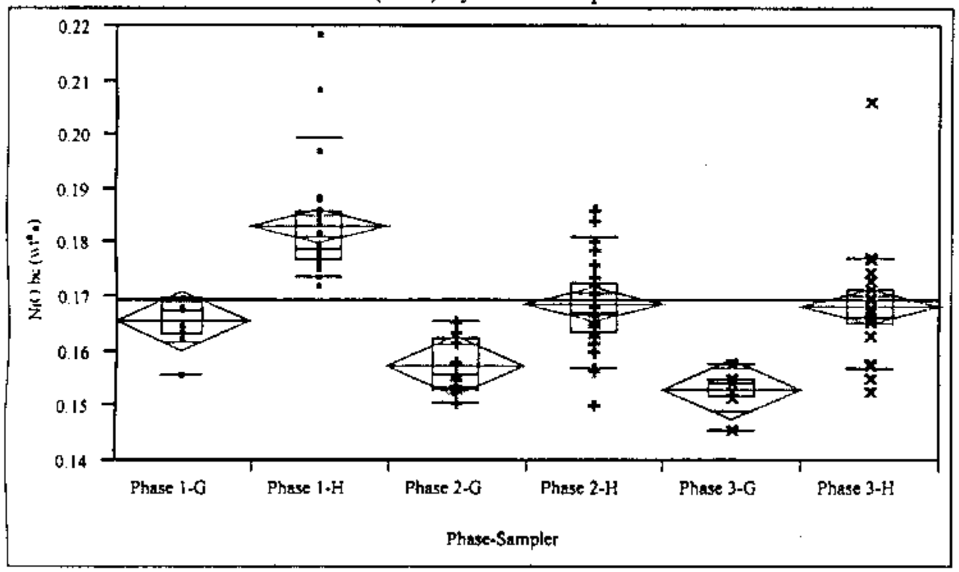
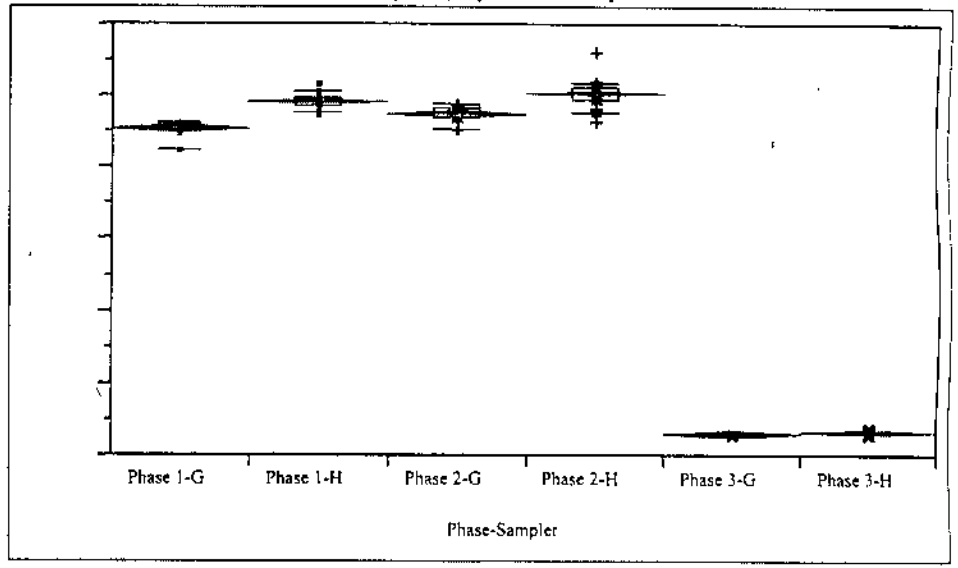


Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases
(Bias-Corrected Measurements are indicated by "bc")

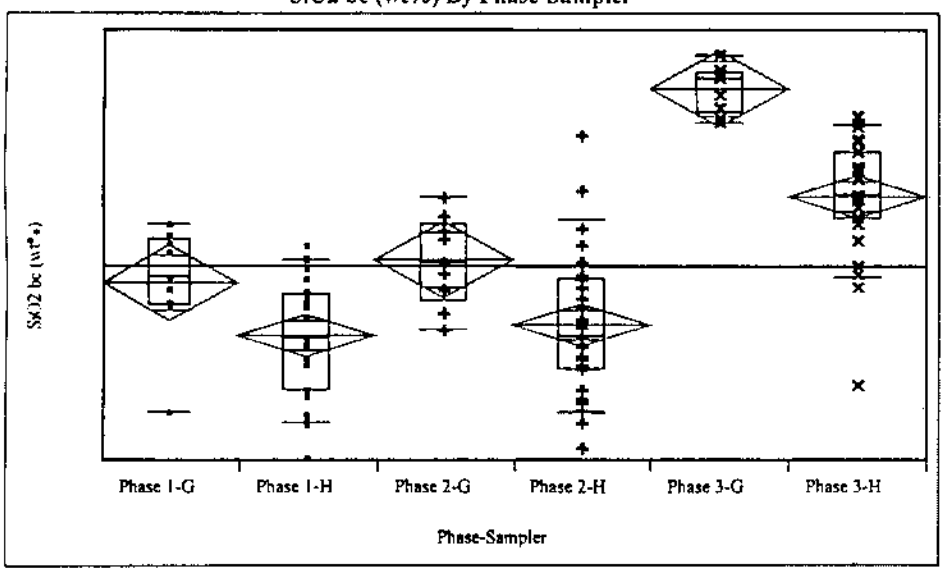
NiO bc (wt%) By Phase-Sampler



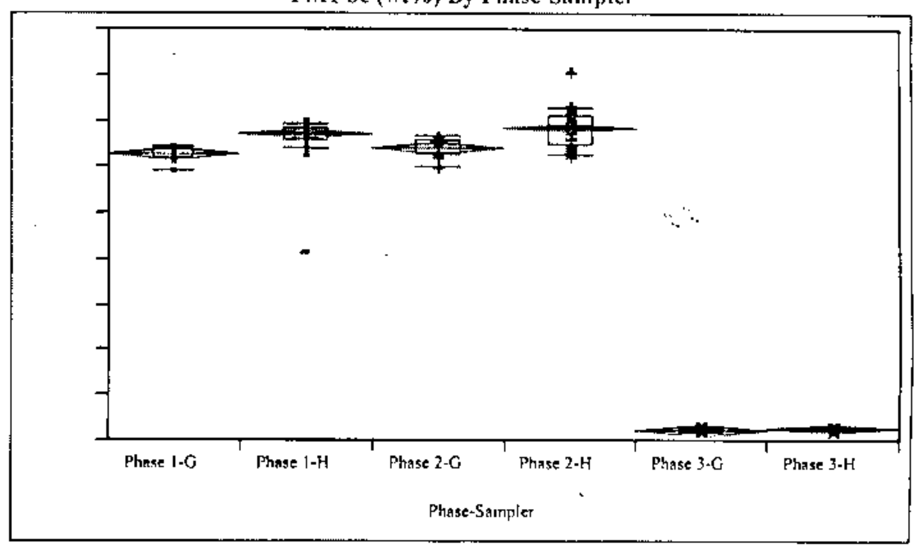
TiO2 bc (wt%) By Phase-Sampler



SiO2 bc (wt%) By Phase-Sampler



PM1 bc (wt%) By Phase-Sampler



Appendix A.

Exhibit A3. Comparisons of Hydragard® Versus Grab Samples Across Test Phases

(Bias-Corrected Measurements are indicated by "bc")

PM2 bc (wt%) By Phase-Sampler

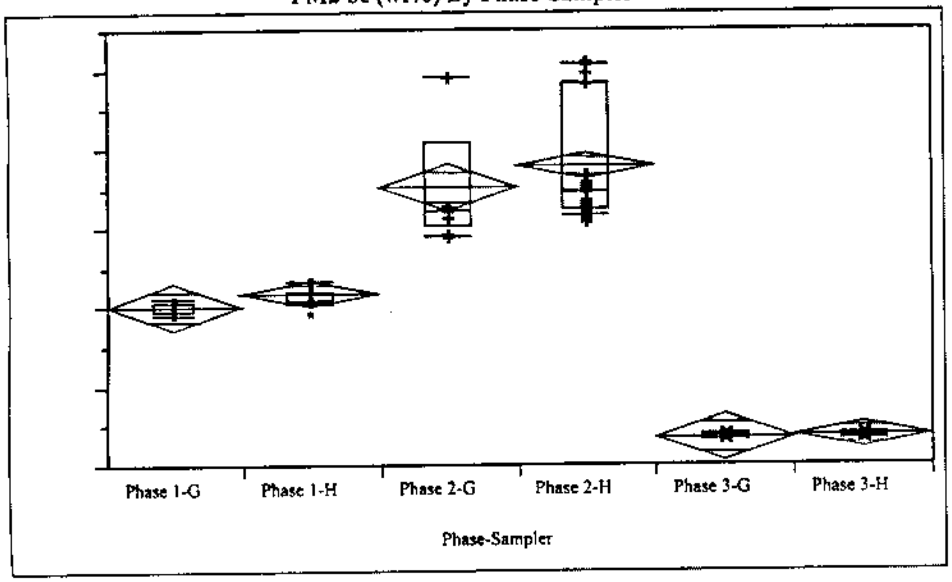
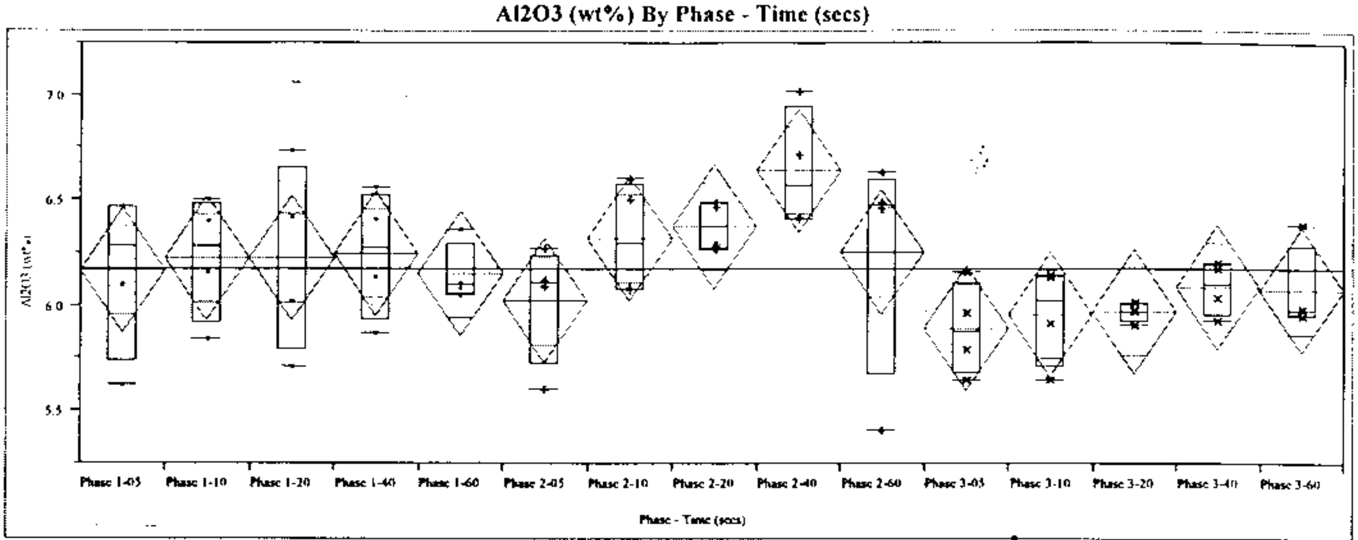
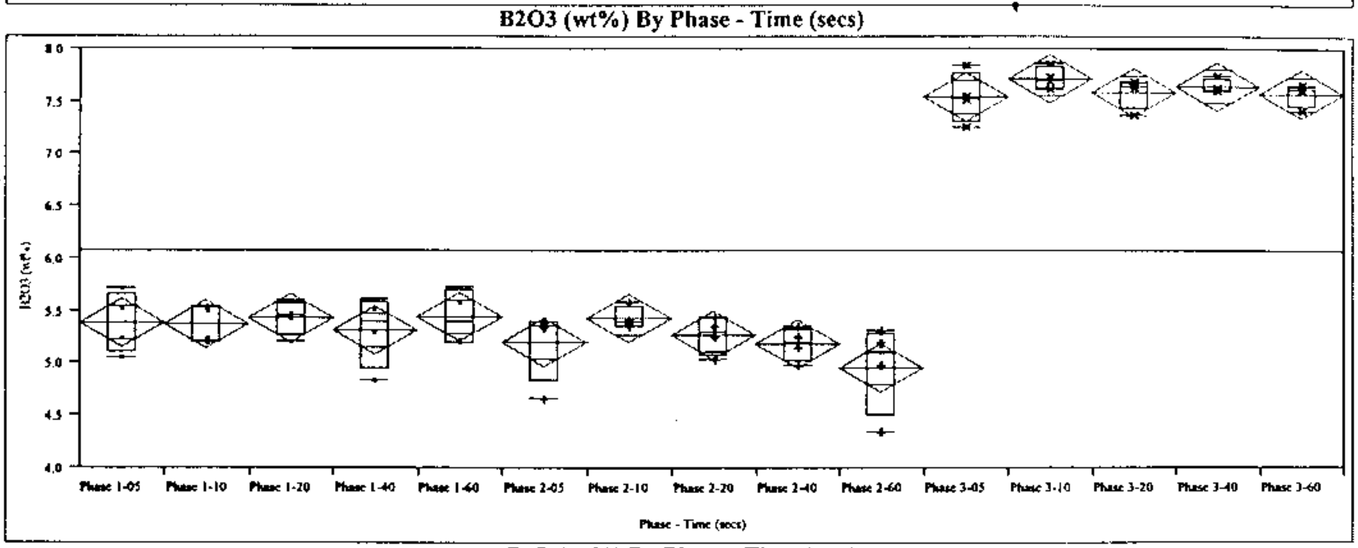


Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

(Bias-Corrected Measurements are indicated by "bc")





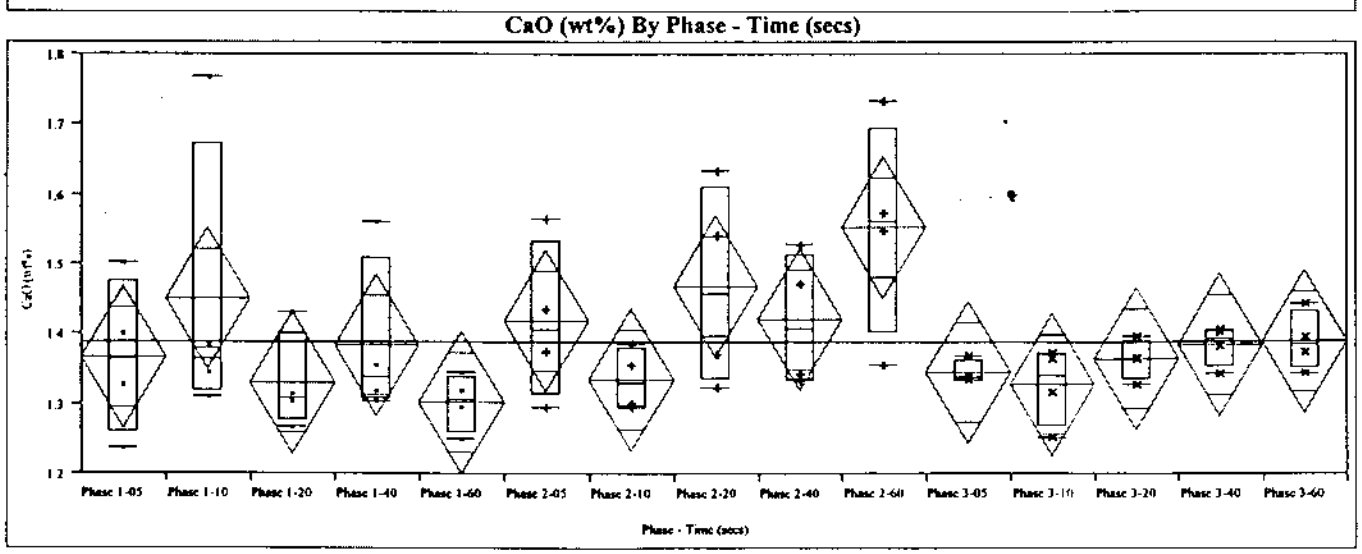
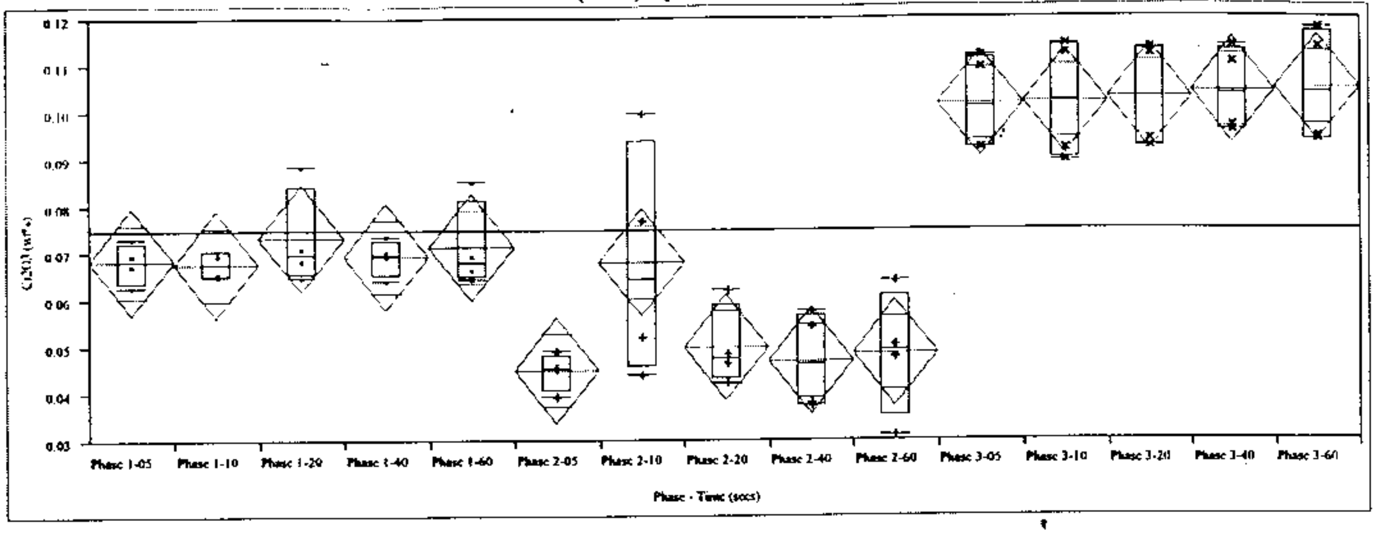


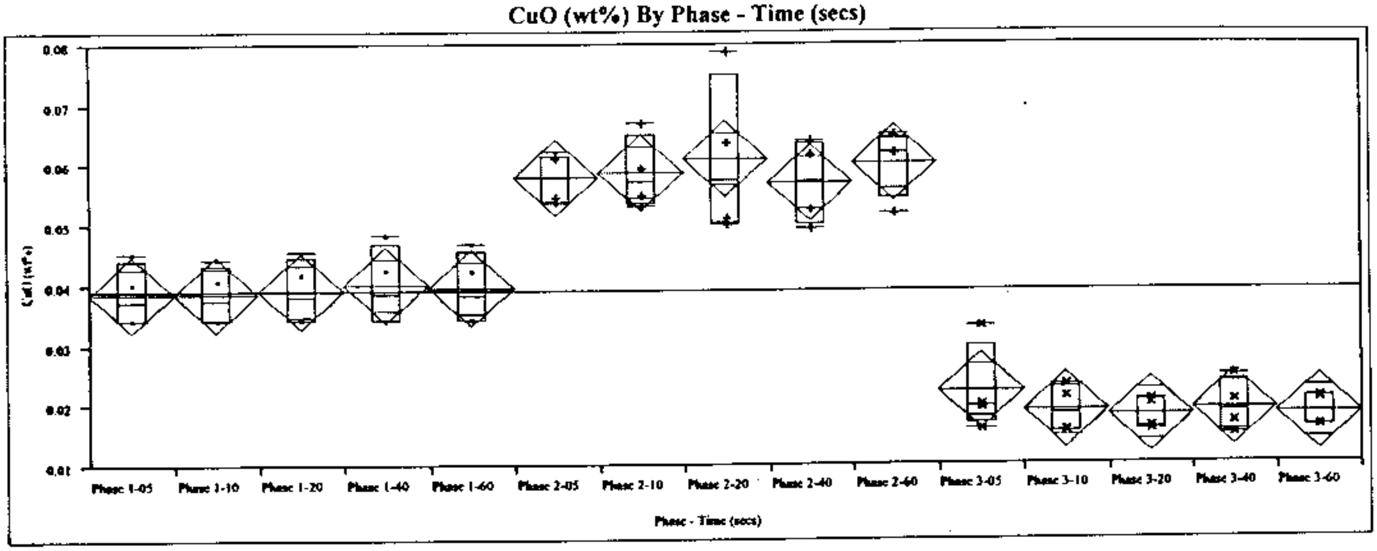
Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

Cr2O3 (wt%) By Phase - Time (secs)



CuO (wt%) By Phase - Time (secs)



Fe2O3 (wt%) By Phase - Time (secs)

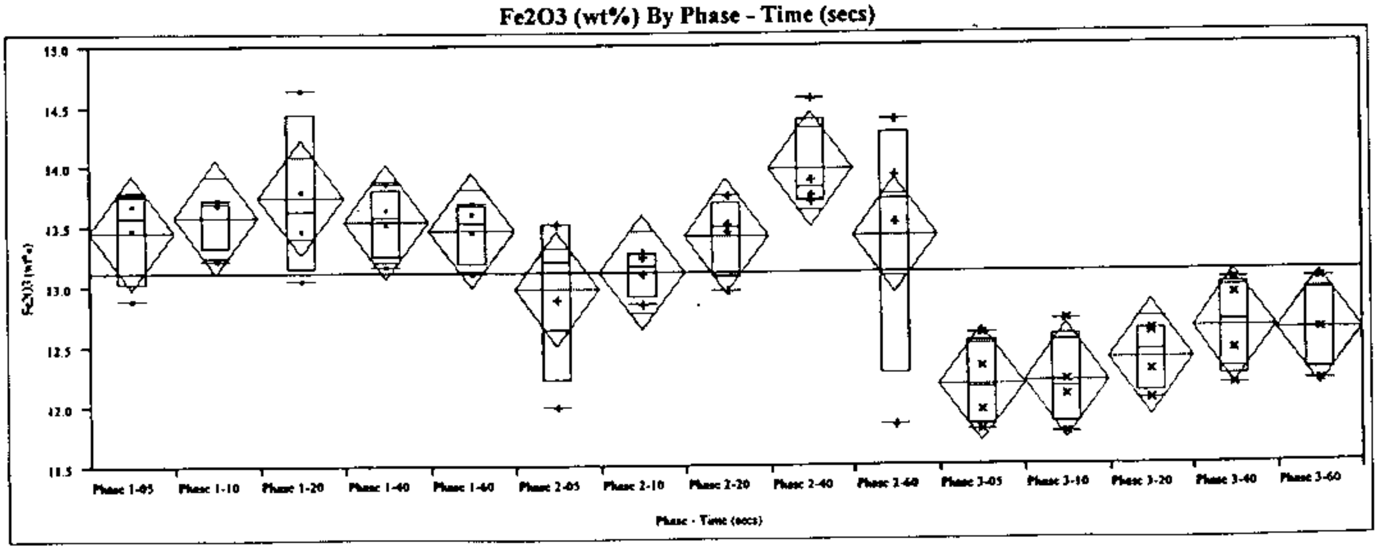
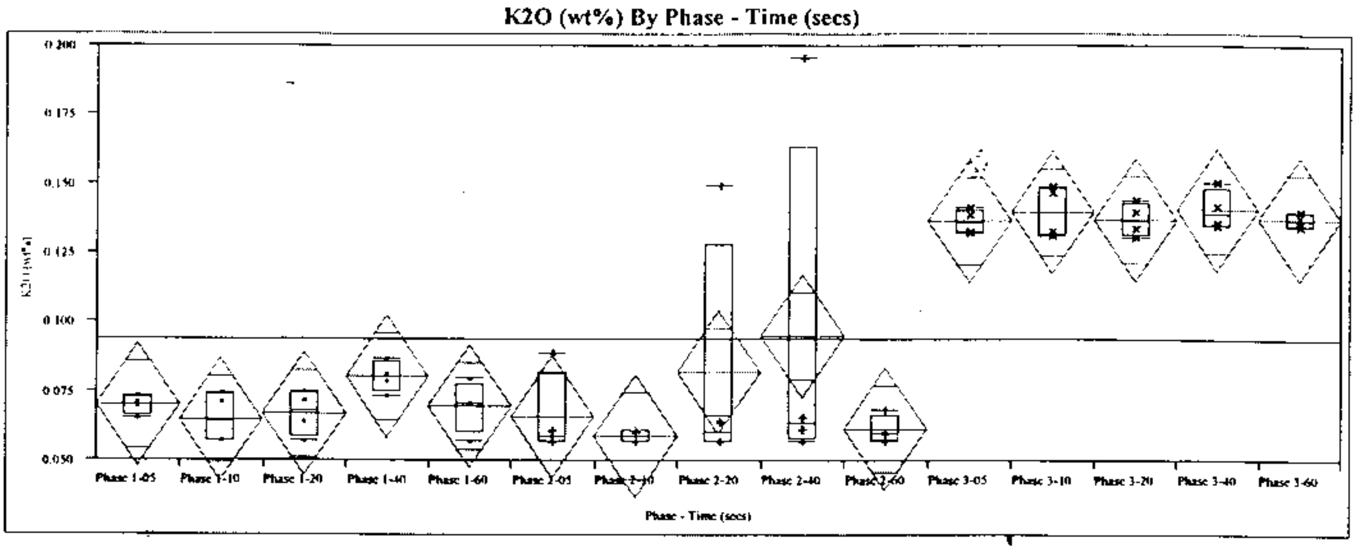
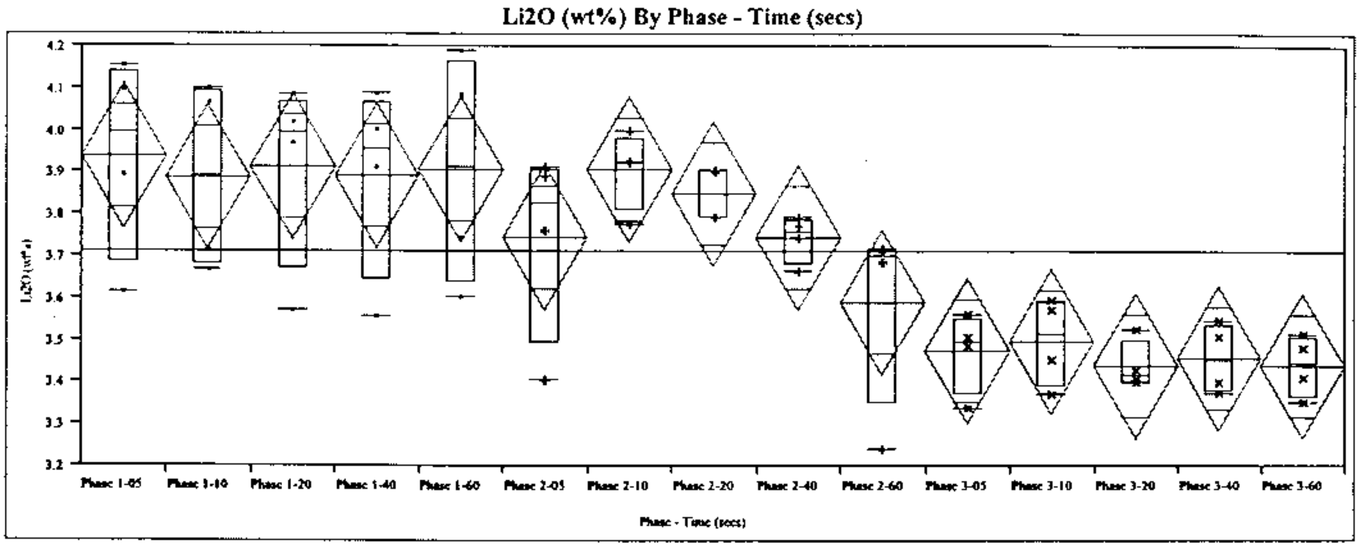


Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases
(Bias-Corrected Measurements are indicated by "bc")





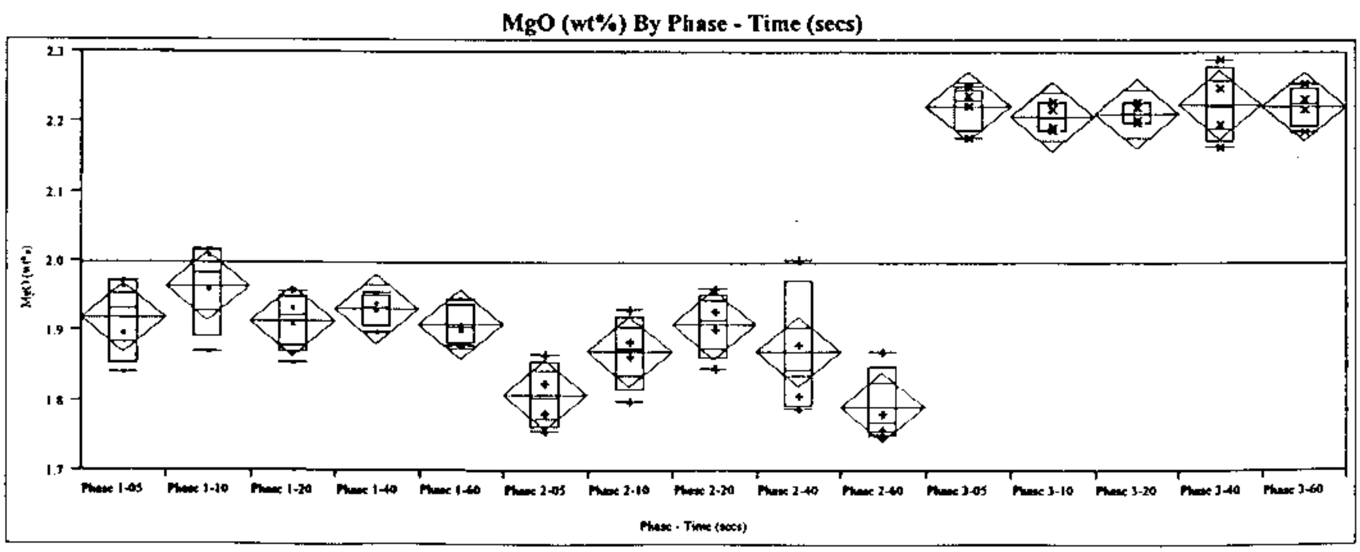
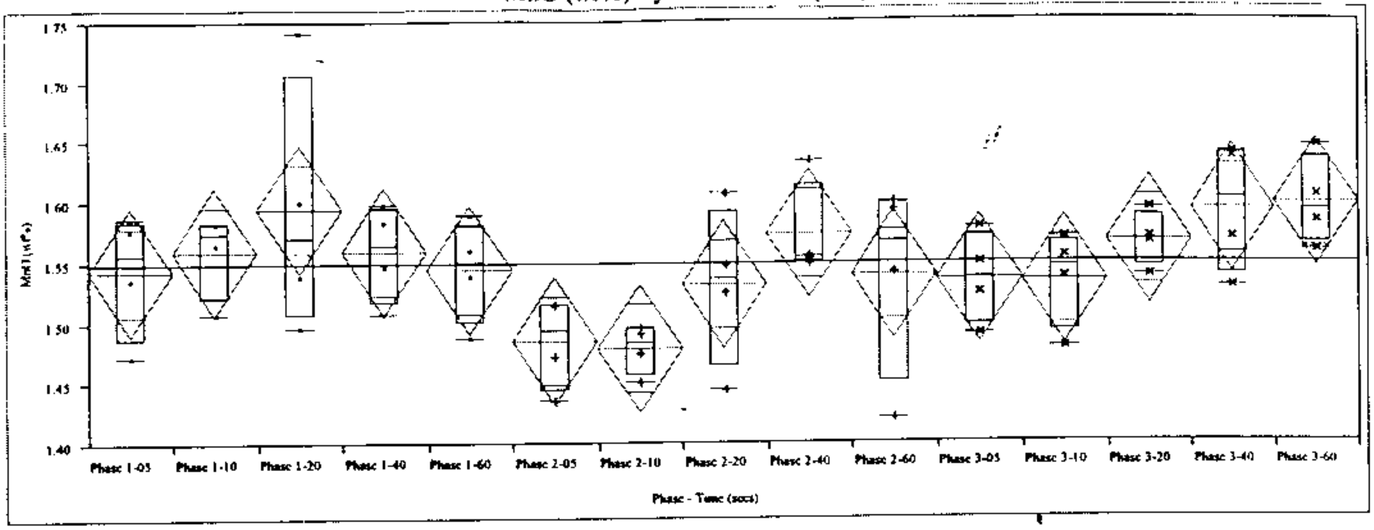
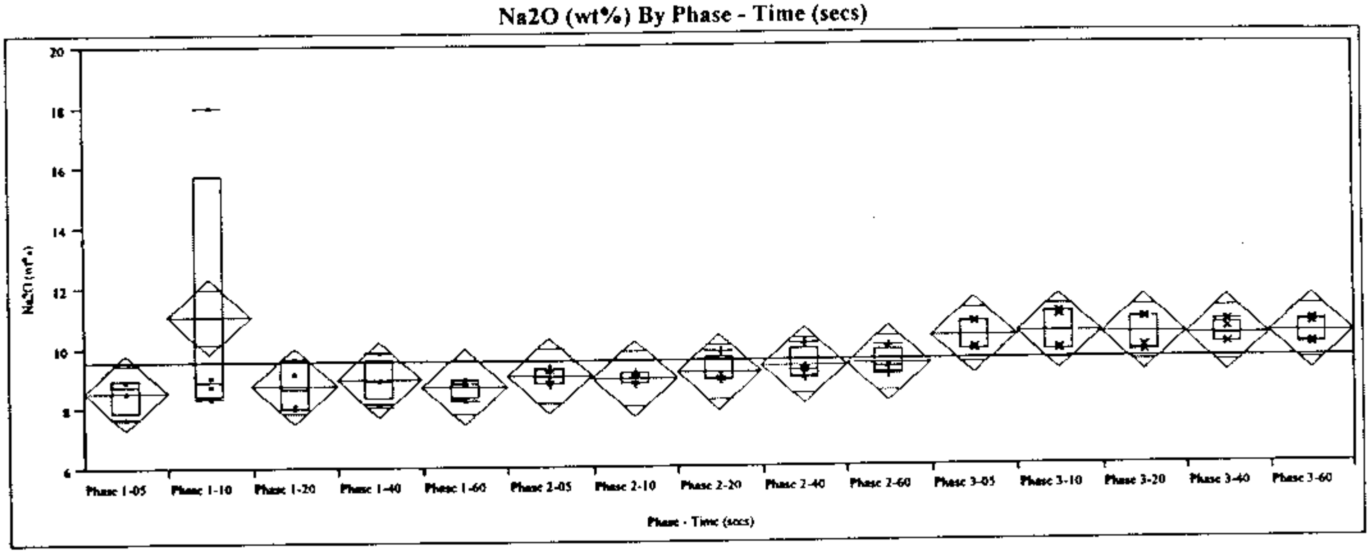


Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases
(Bias-Corrected Measurements are indicated by "bc")

MnO (wt%) By Phase - Time (secs)



Na2O (wt%) By Phase - Time (secs)



NiO (wt%) By Phase - Time (secs)

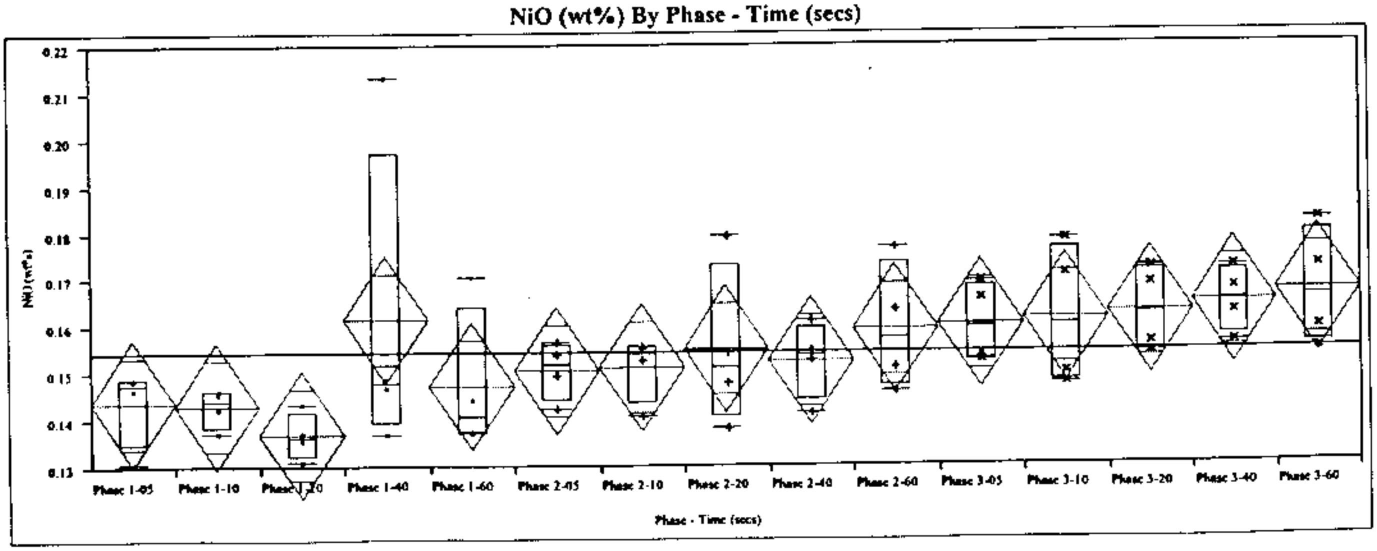
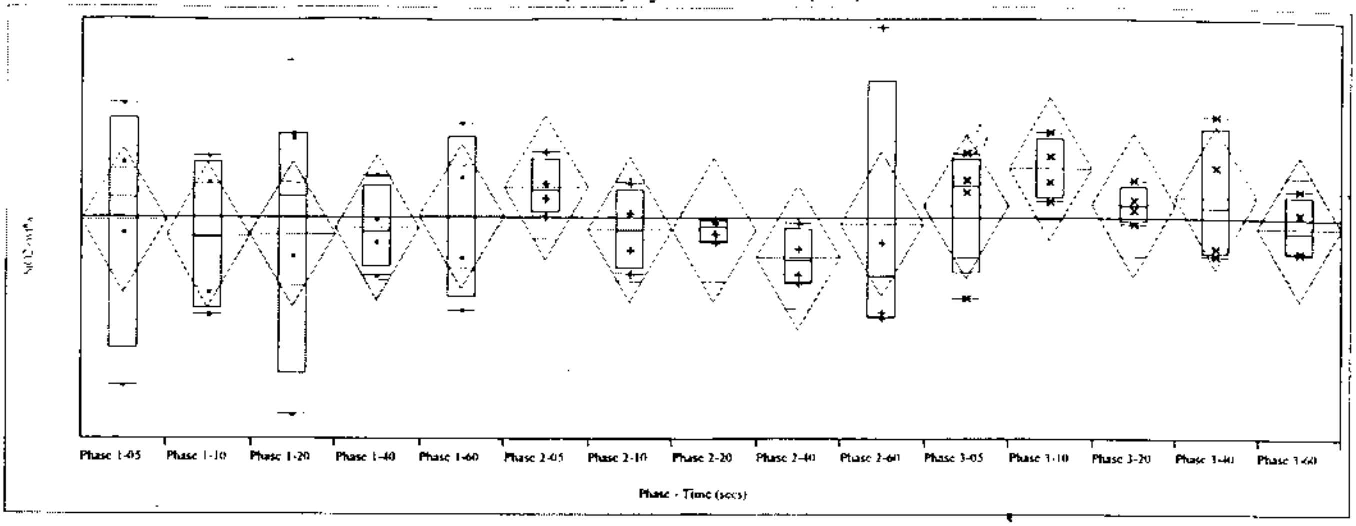


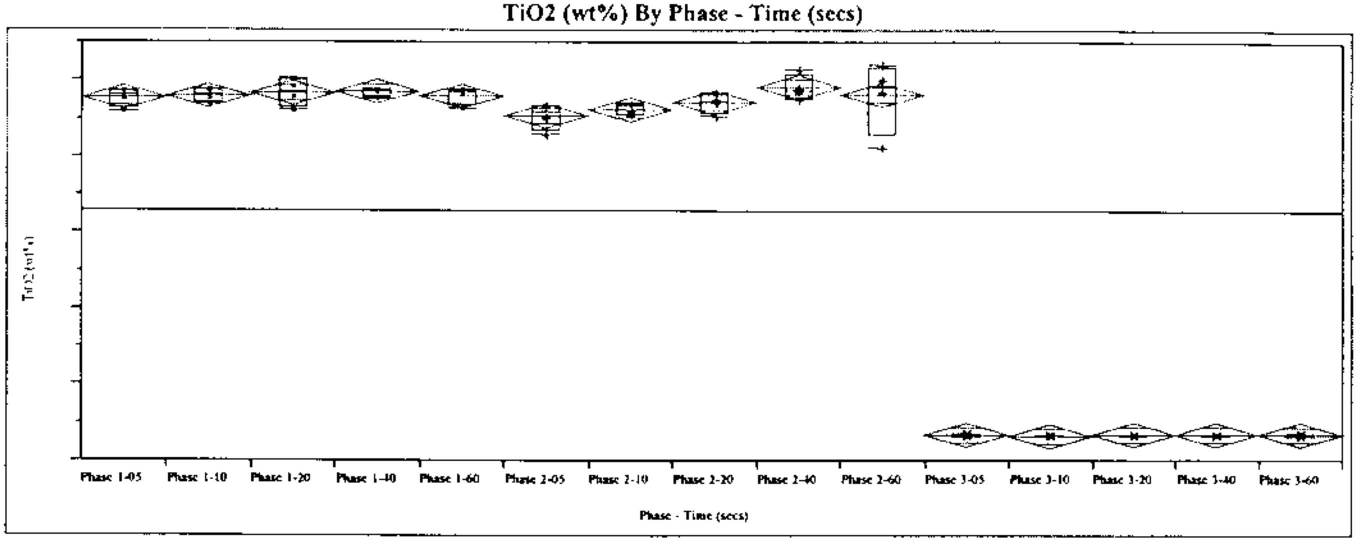
Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

SiO2 (wt%) By Phase - Time (secs)



TiO2 (wt%) By Phase - Time (secs)



PM1 (wt%) By Phase - Time (secs)

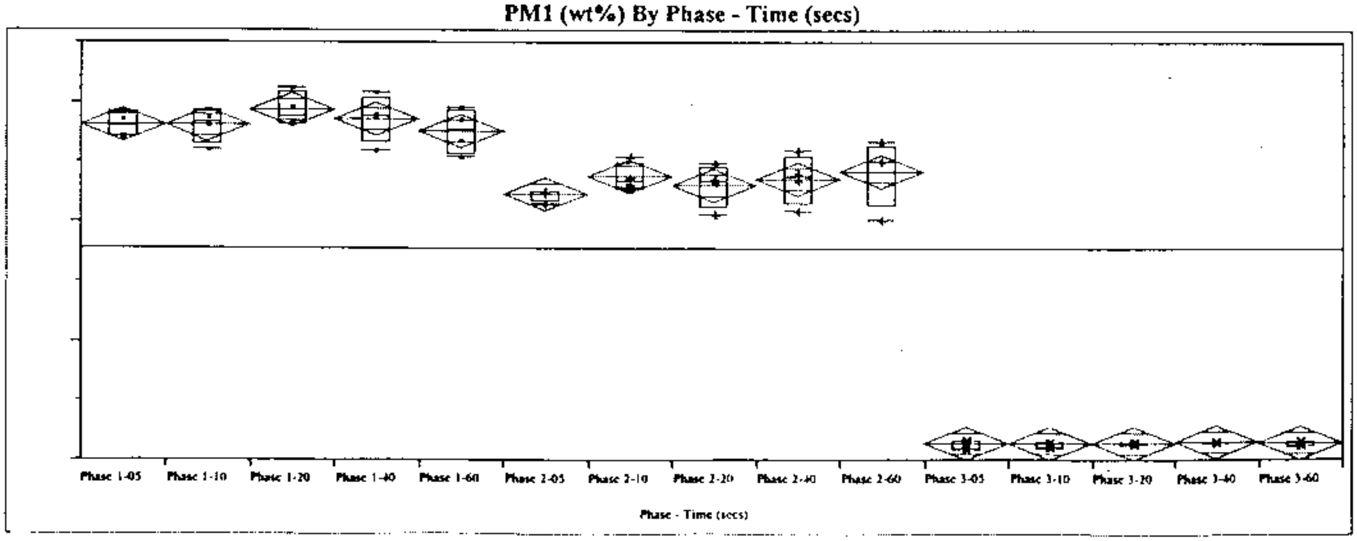
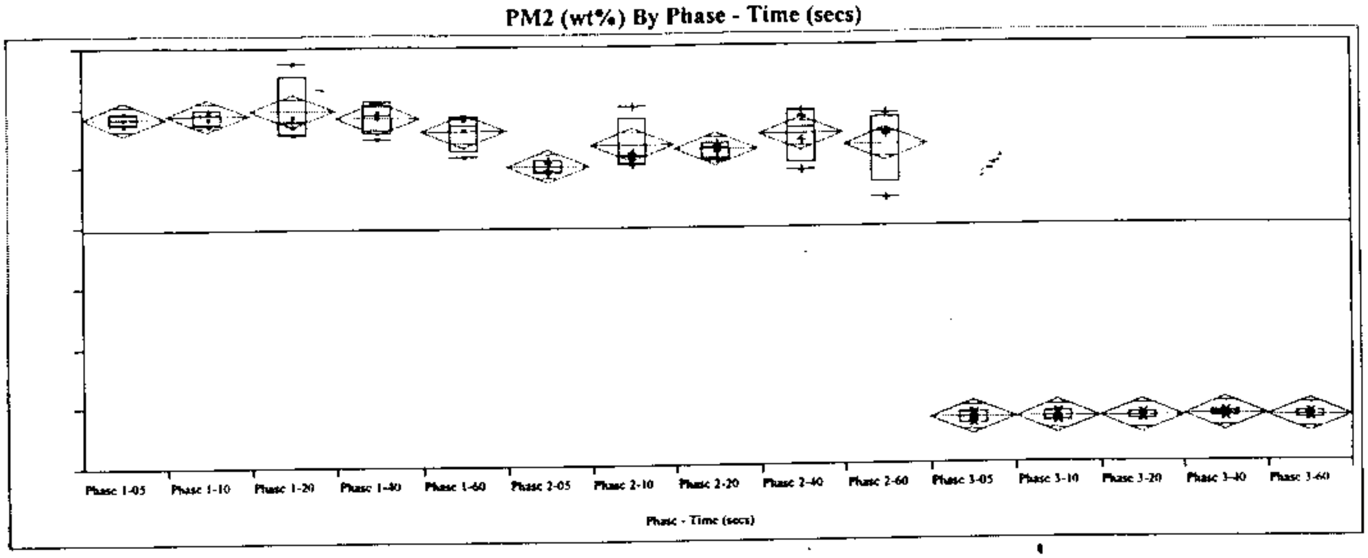
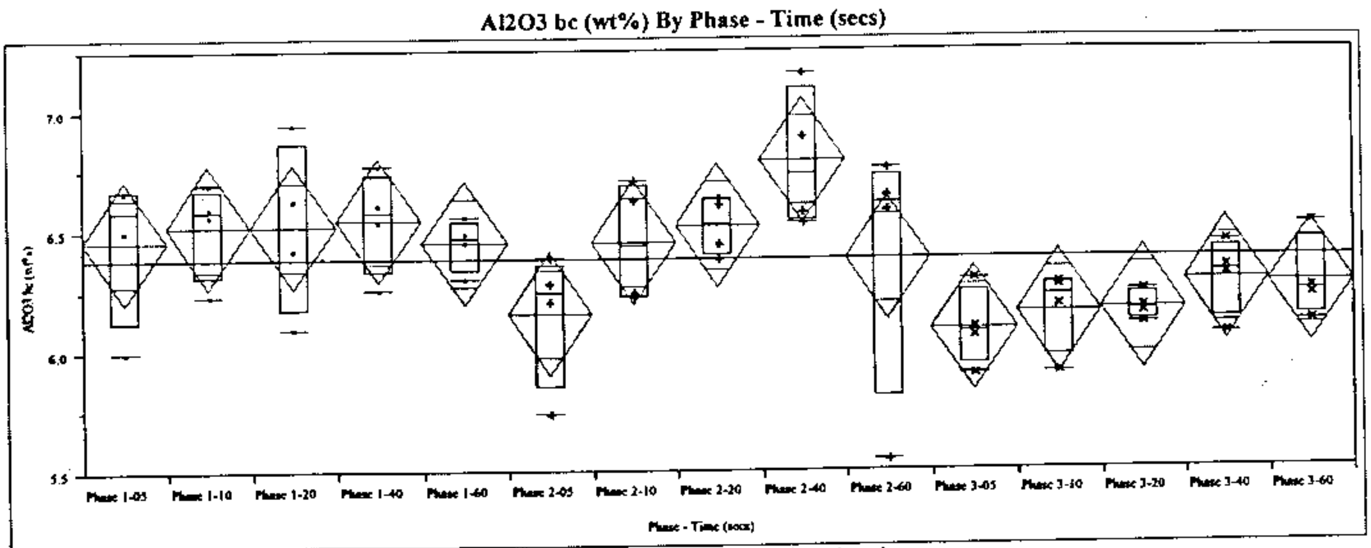


Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

(Bias-Corrected Measurements are indicated by "bc")





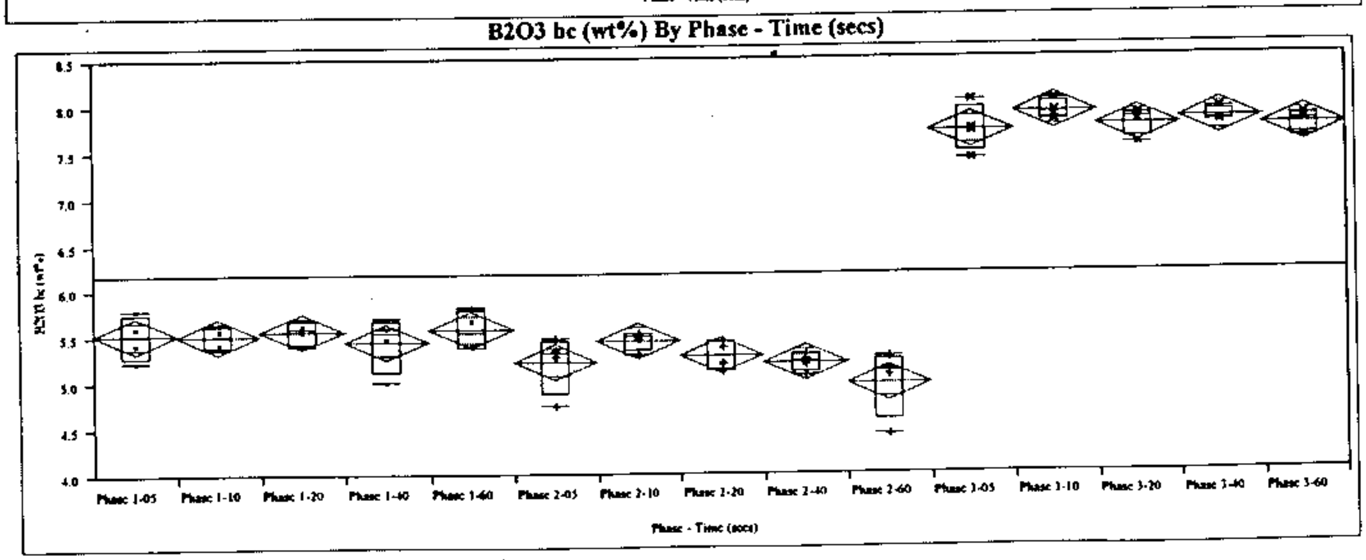
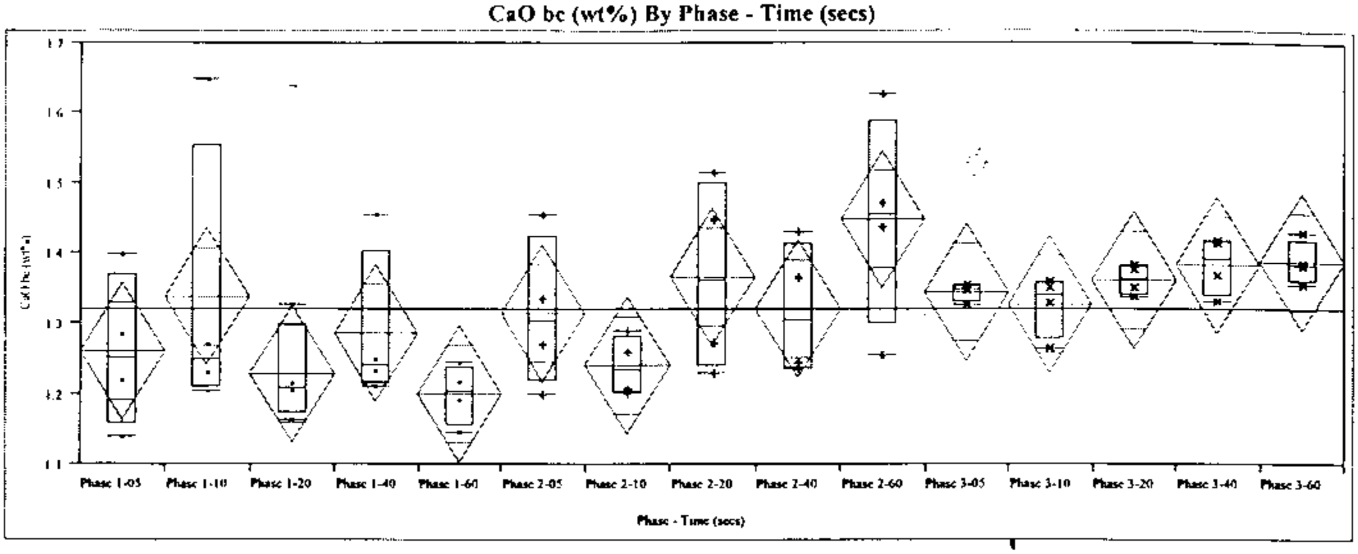
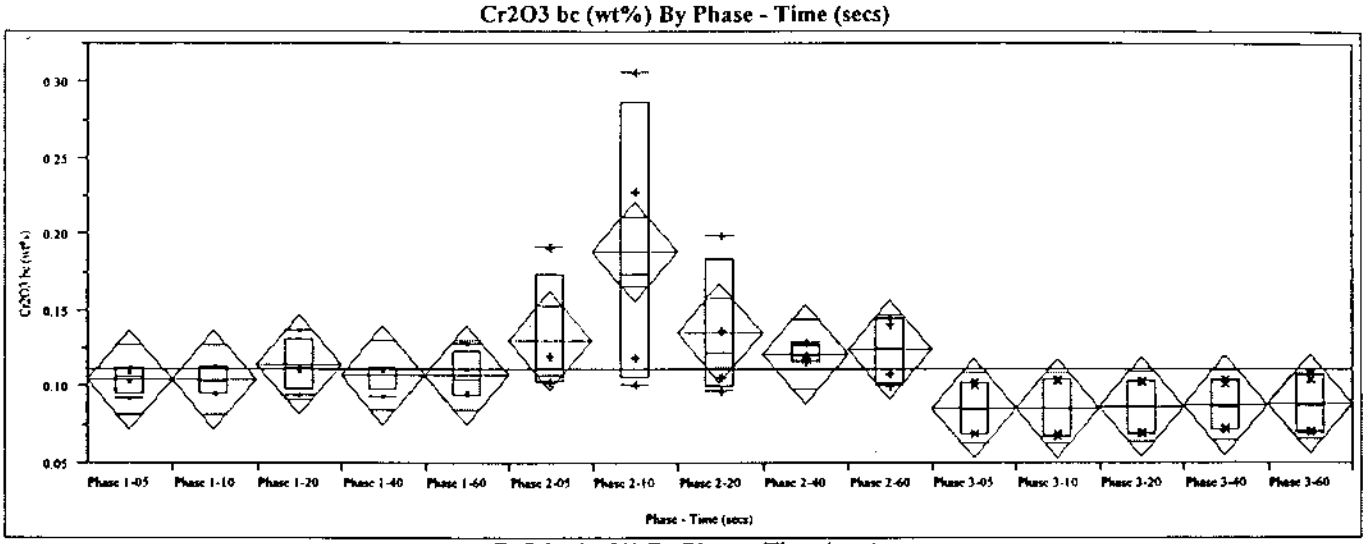


Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

(Bias-Corrected Measurements are indicated by "bc")





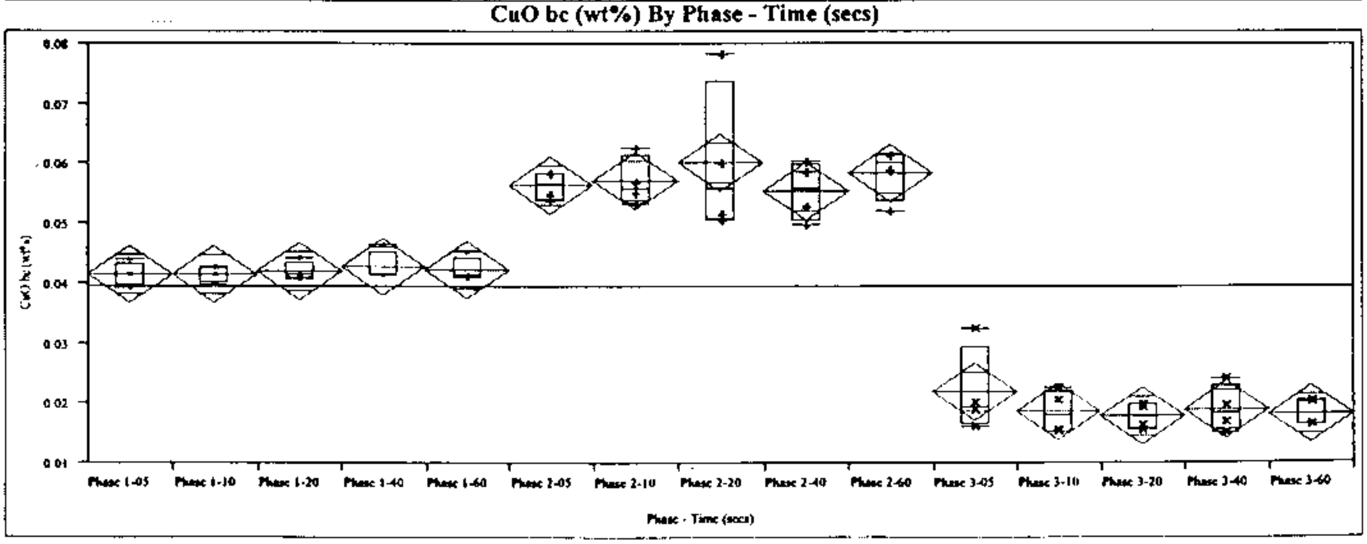
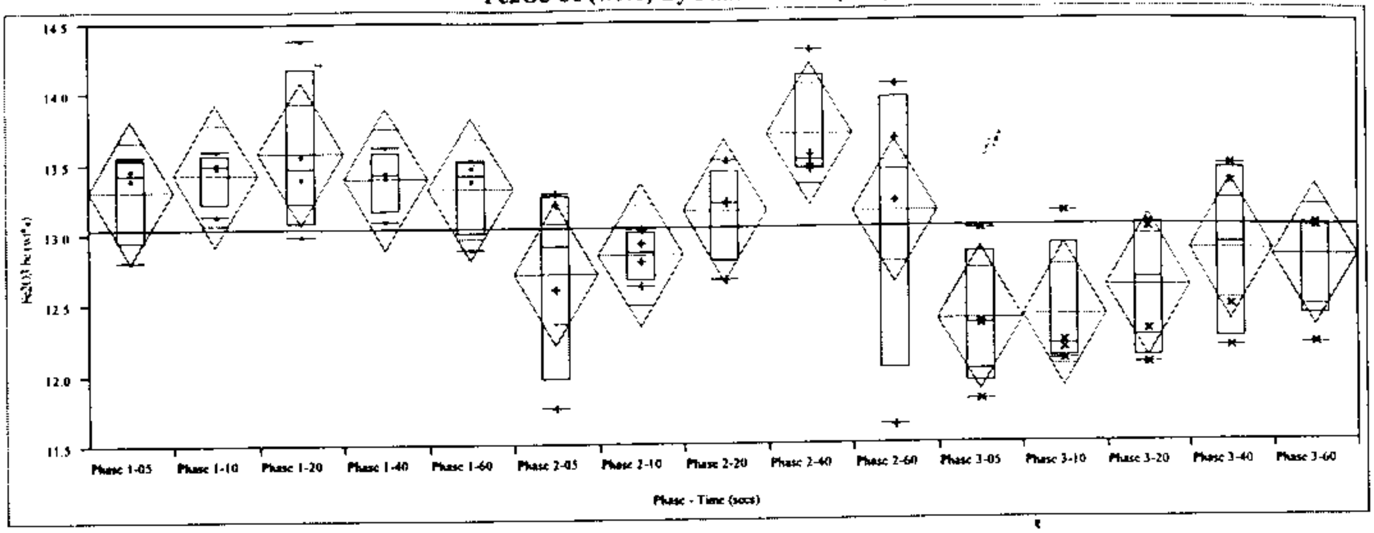


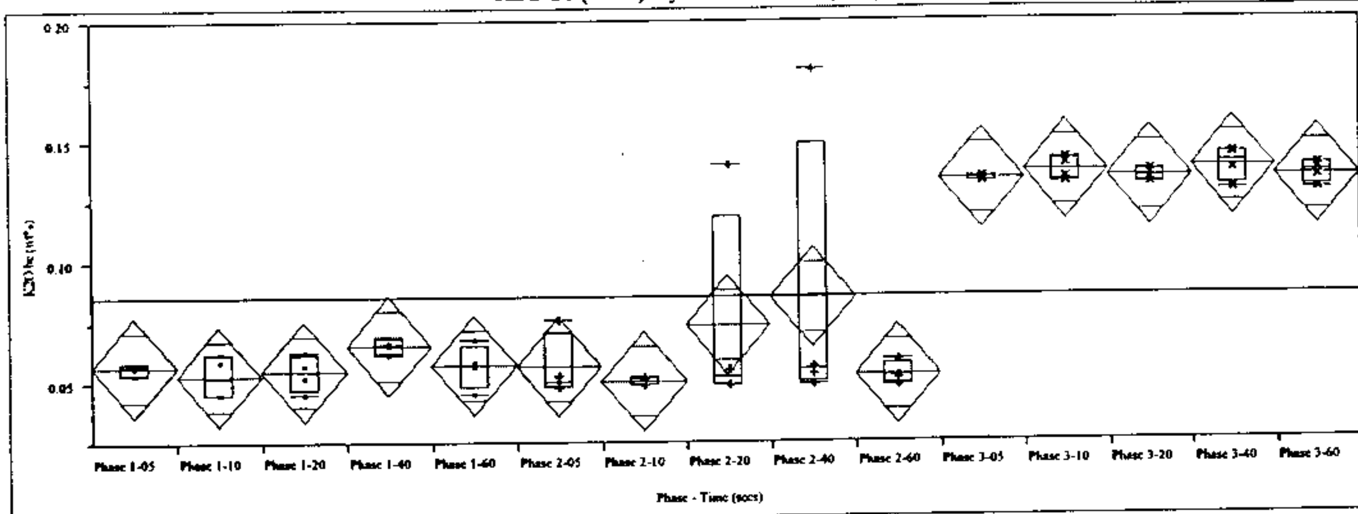
Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

Fe_2O_3 bc (wt%) By Phase - Time (secs)



K_2O bc (wt%) By Phase - Time (secs)



Li_2O bc (wt%) By Phase - Time (secs)

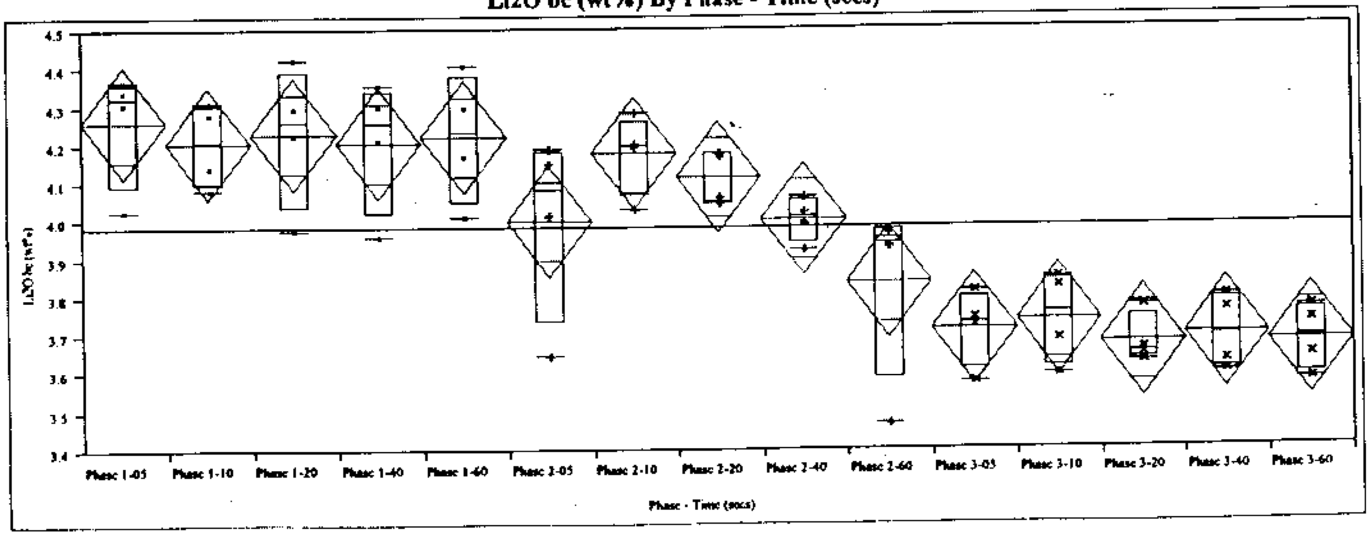
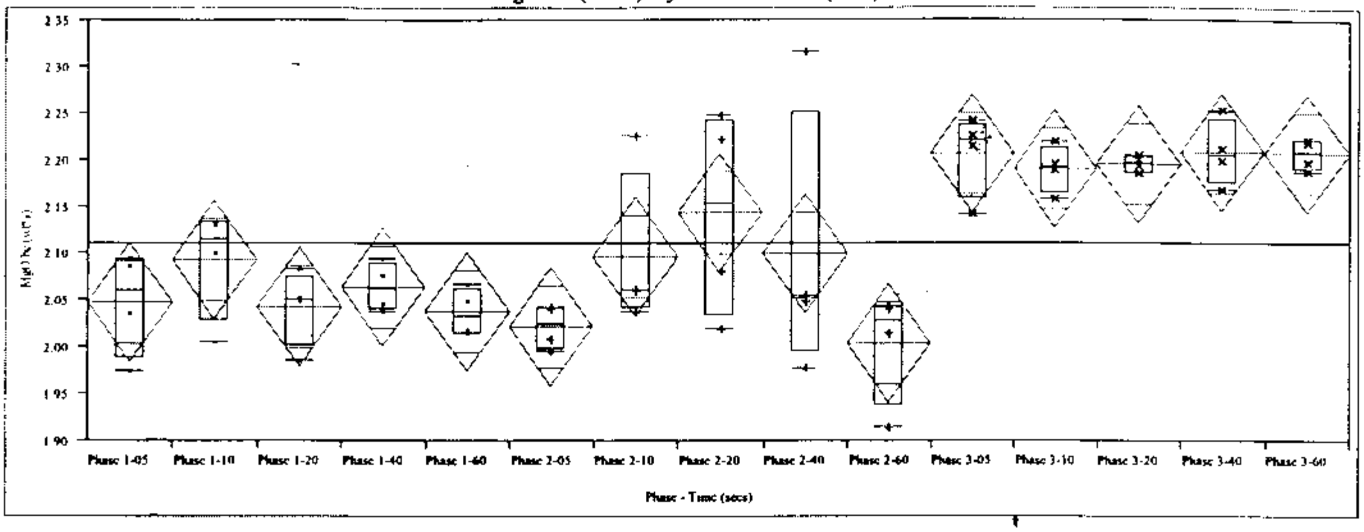
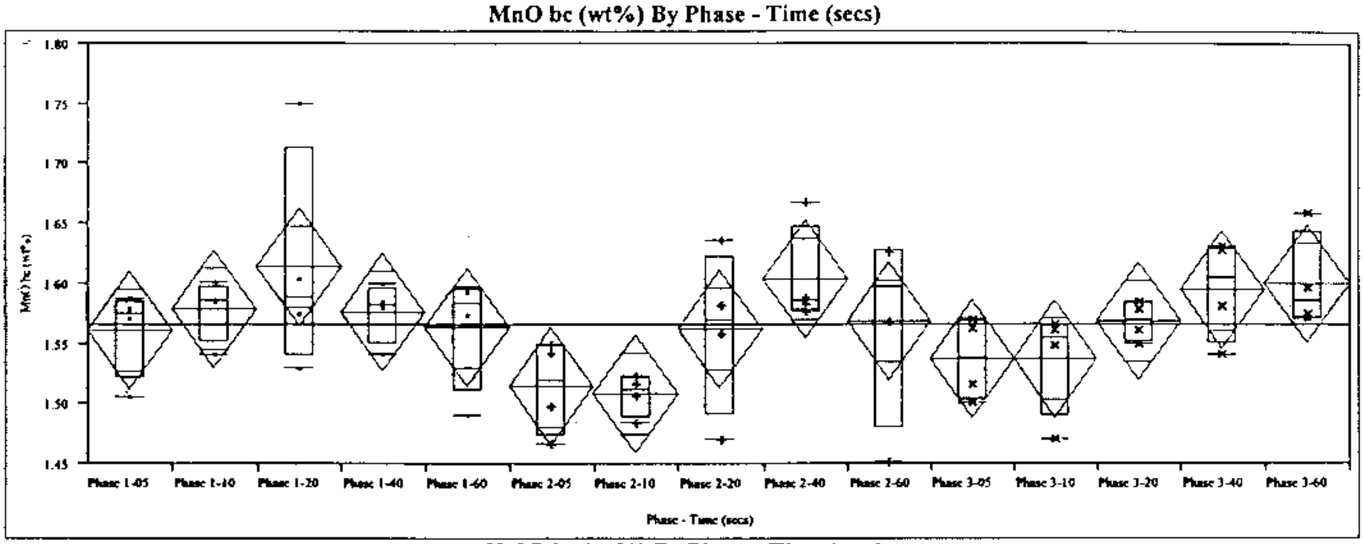


Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases
(Bias-Corrected Measurements are indicated by "bc")

MgO bc (wt%) By Phase - Time (secs)



MnO bc (wt%) By Phase - Time (secs)



Na2O bc (wt%) By Phase - Time (secs)

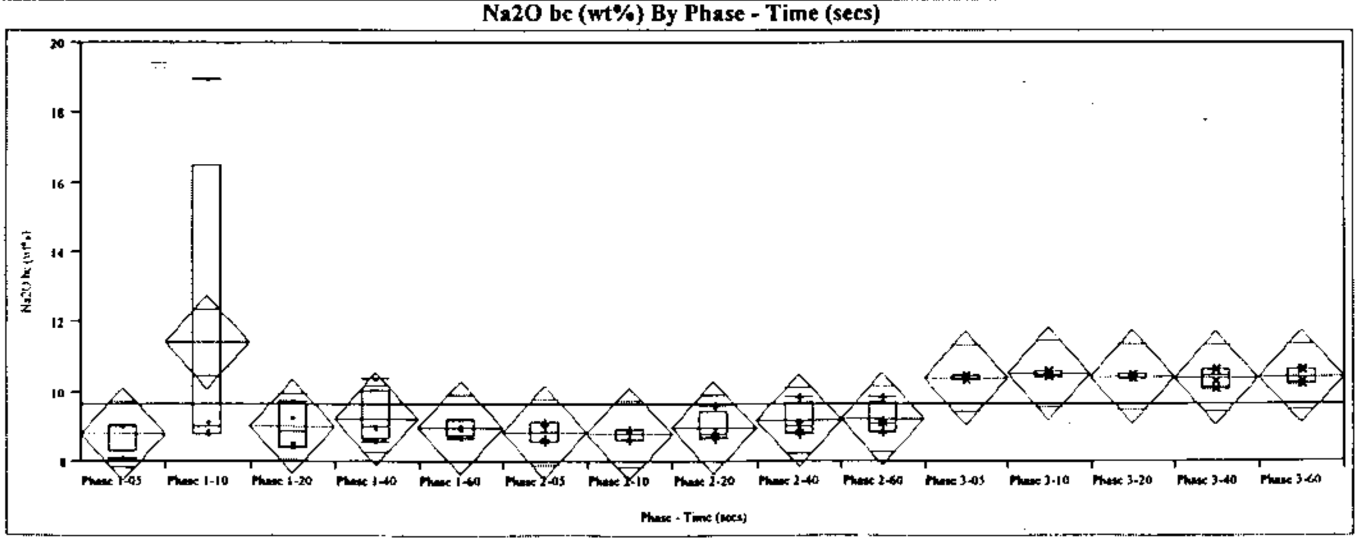
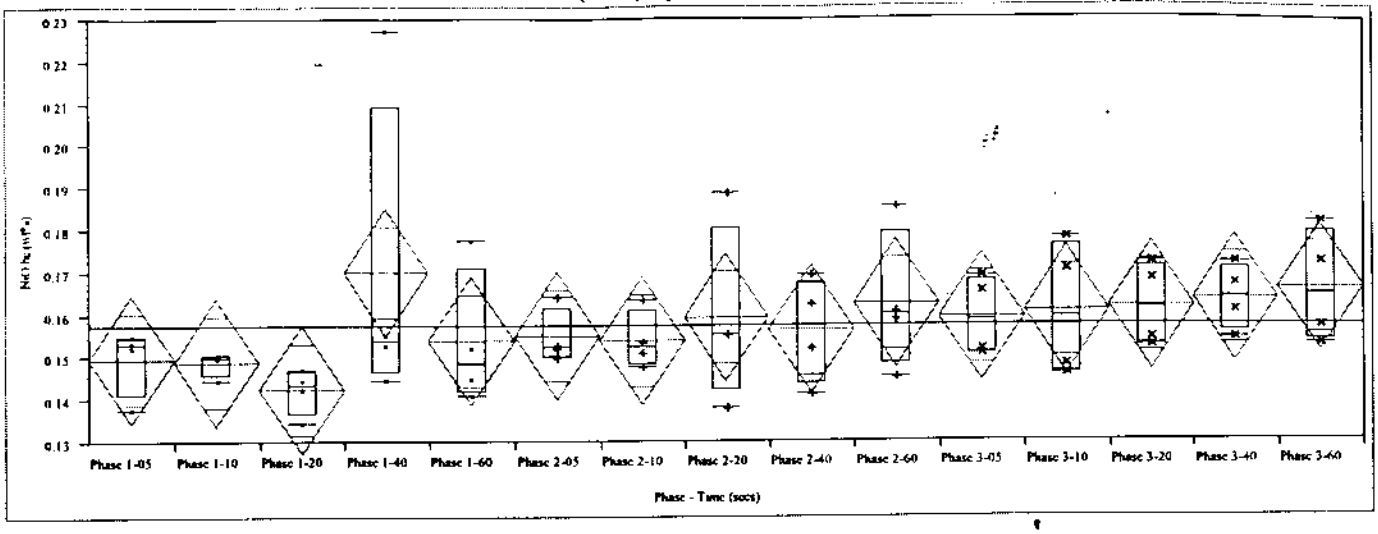


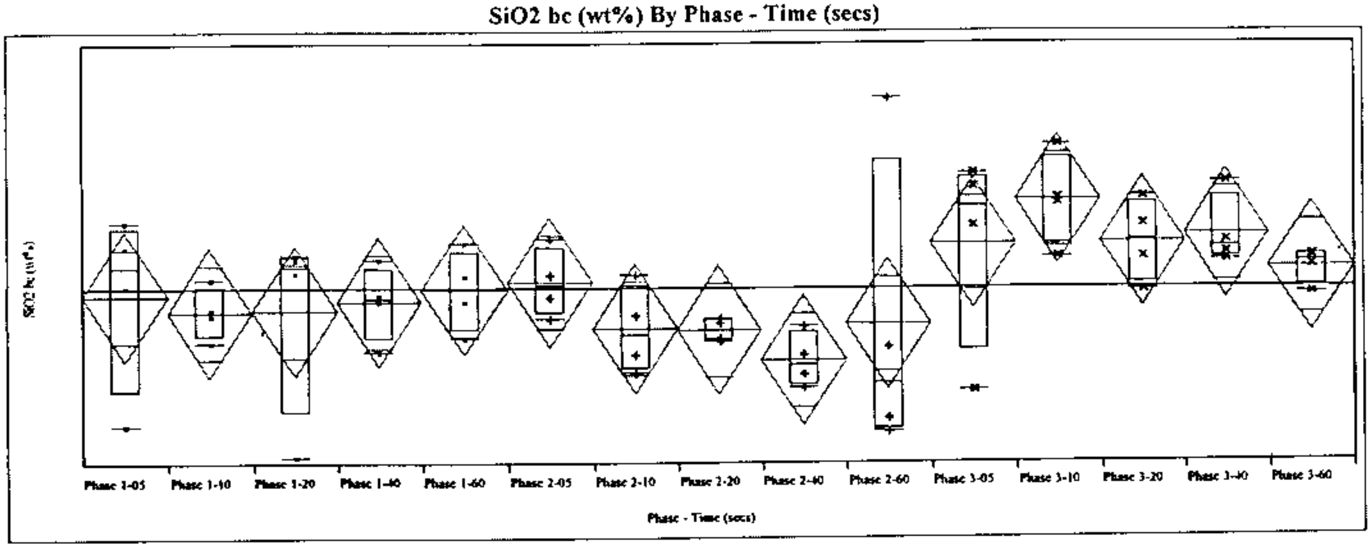
Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

NiO bc (wt%) By Phase - Time (secs)



SiO2 bc (wt%) By Phase - Time (secs)



TiO2 bc (wt%) By Phase - Time (secs)

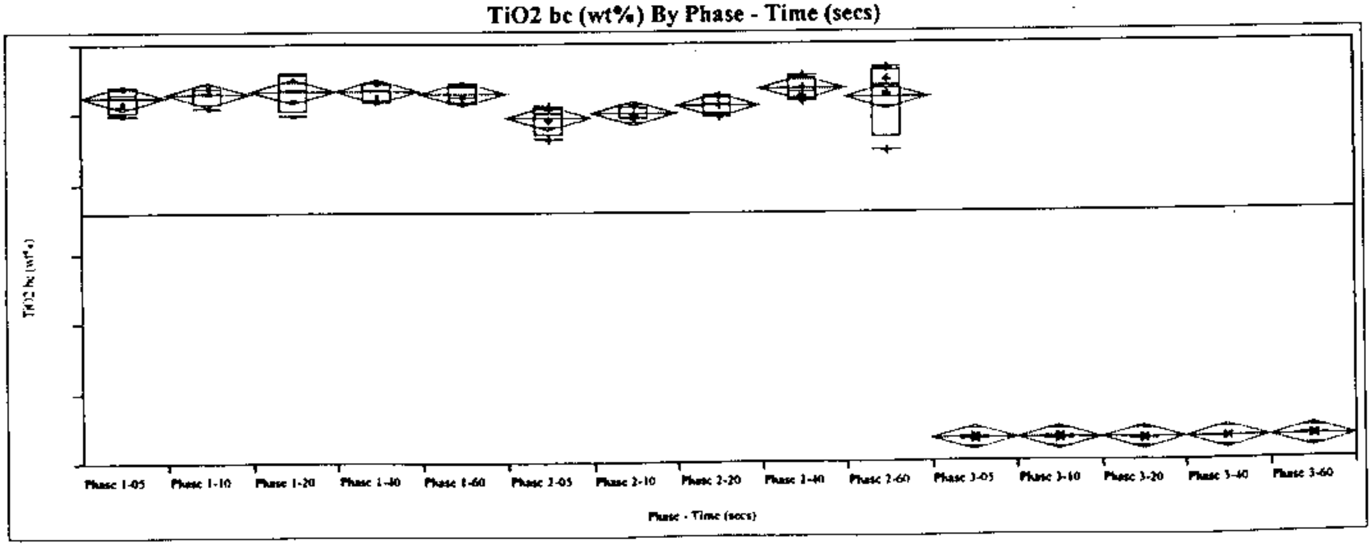
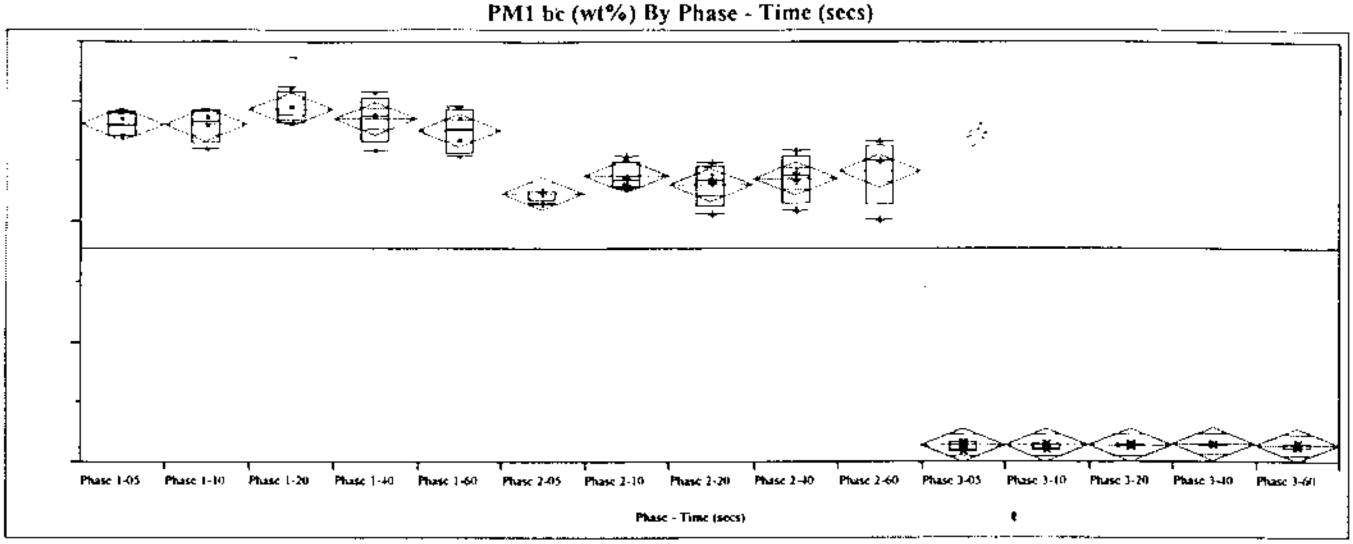


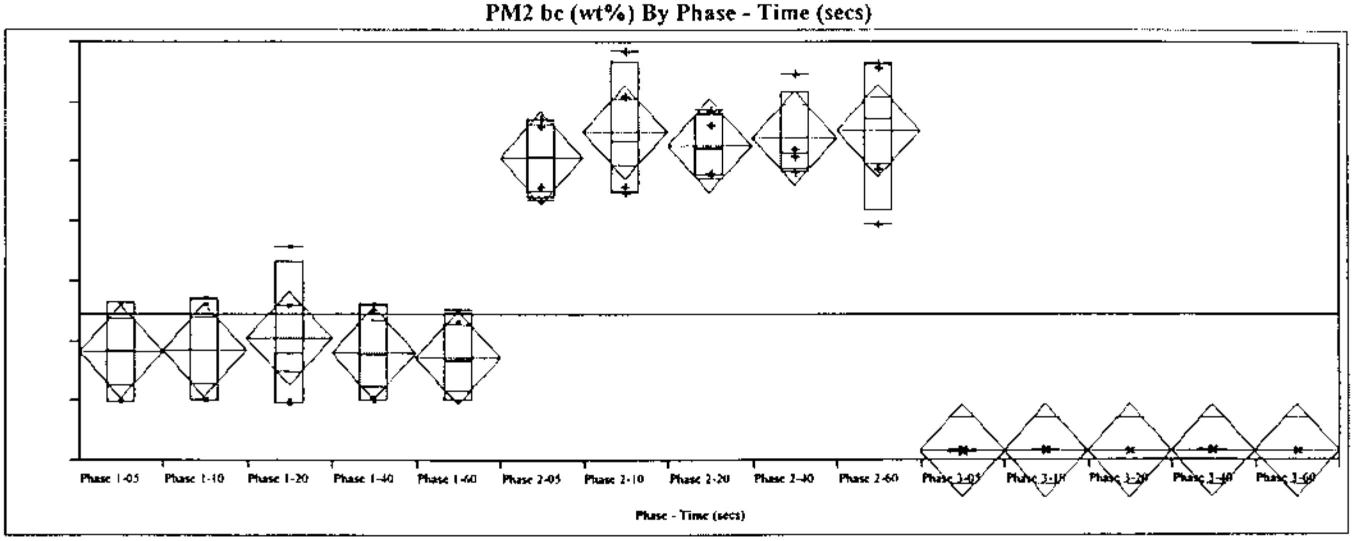
Exhibit A4. Comparisons of Samples Across Hydragard® Sample Flow Times and Test Phases

(Bias-Corrected Measurements are indicated by "bc")

PM1 bc (wt%) By Phase - Time (secs)



PM2 bc (wt%) By Phase - Time (secs)

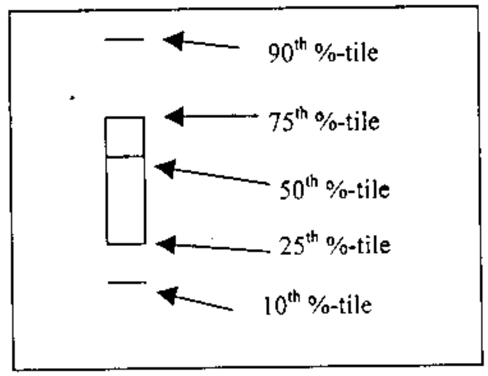


Appendix B. Explanation of JMP® Statistical Graphics

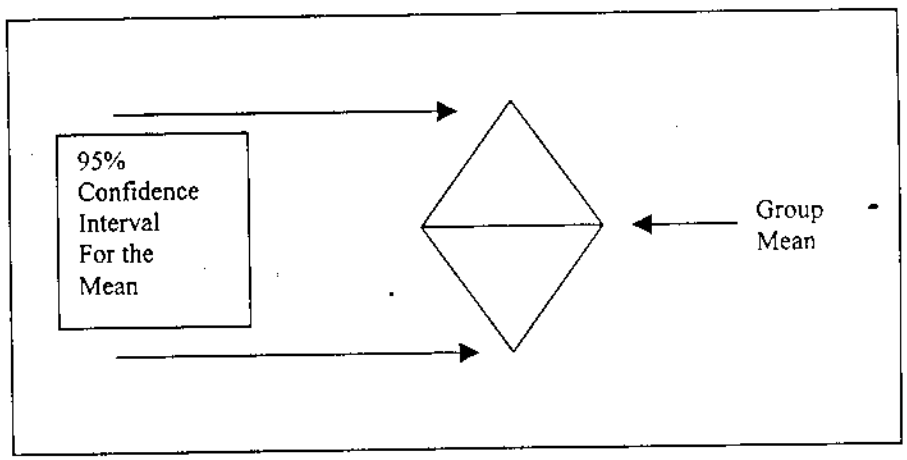
The statistical analysesreported here were conducted using JMP® Version 3.2.2 from SAS Institute, Inc., Cary, NC [6]

Data are displayed in this report using Quantile Box Plots and Means Diamonds.

Quantile Box



Means Diamonds



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