

DWPF Sample Vial Insert Study-Statistical Analysis of DWPF Mock-Up Test Data

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DWPF Sample Vial Insert Study


Statistical Analysis of DWPF Mock-up Test Data(U)

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DWPF Sample Vial Insert Study

Statistical Analysis of DWPF Mock-up Test Data(U)

S.P. Harris

1. Introduction

This report is prepared as part of Technical/QA Task Plan WSRC-RP-97-351 which was issued in response to Technical Task Request HLW/DWPF/TTR-970132 submitted by DWPF. Presented in this report is a statistical analysis of DWPF Mock-up test data for evaluation of two new analytical methods which use insert samples from the existing Hydragard™ sampler. The first is a new hydrofluoric acid based method⁽¹⁾ called the Cold Chemical Method(Cold Chem) and the second is a modified fusion method.

Either new DWPF analytical method could result in a two to three fold improvement in sample analysis time. Both new methods use the existing Hydragard™ sampler to collect a smaller insert sample from the process sampling system. The insert testing methodology applies to the DWPF Slurry Mix Evaporator(SME) and the Melter Feed Tank(MFT) samples.

The "insert" sample is named after the initial trials which placed the container inside the sample (peanut) vials. Samples in small 3 ml containers(Inserts) are analyzed by either the cold chemical method or a modified fusion method. The current analytical method uses a Hydragard™ sample station to obtain nearly full 15 ml peanut vials. The samples are prepared by a multi-step process for Inductively Coupled Plasma (ICP) analysis by drying, vitrification, grinding and finally dissolution by either mixed acid or fusion. In contrast, the insert sample is placed directly in the dissolution vessel, thus eliminating the drying, vitrification and grinding operations for the Cold chem method. Although the modified fusion still requires drying and calcine conversion, the process is rapid due to the decreased sample size and that no vitrification step is required.

A slurry feed simulant material was acquired from the TNX pilot facility from the test run designated as PX-7. The Mock-up test data were gathered on the basis of a statistical design presented in SRT-SCS-97004(Rev. 0)^(1a). Simulant PX-7 samples were taken in the DWPF Analytical Cell Mock-up Facility using 3 ml inserts and 15 ml peanut vials. A number of the insert samples were analyzed by Cold Chem and compared with full peanut vial samples analyzed by the current methods. The remaining inserts were analyzed by the modified fusion method, for comparison to the current method, and also

to obtain a calcine correction factor. The simulant was within 40 - 42 wt% solids in order to provide a rheology within the DWPF design range. The rheology at 42 wt% was approximately 47 dynes/cm² yield stress at 25 C.

2. Parametric Studies

During preliminary testing⁽²⁾ using PX-7 samples taken with a prototypic HydragardTM in the 786-A Thermal Fluids Laboratory, the following variables were evaluated for their effect on insert samples relative to full 15ml peanut vial samples:

Hold time - the time period covering stopping the flow of the material to the time the sample is removed from the station(5 sec, 30 sec);

Vial Flow rate - the measured flow through the sample container(1, 2, 3 gpm);

Valve circulation time - the time material flushed through the sample container(2, 11, 20 sec);

The valve opening/closing rate of the HydragardTM sampler was without delay (fast/fast). Three insert sizes were considered: 1.5, 3 and 4 ml. Six dilutions of PX-7 were used ranging from 34 to 51 wt%.

Over all tests, the 3 ml insert was the smallest insert that gave comparable sampling results. No discernible difference was found for the 3 ml inserts among the study conditions^(3a, 3b).

3. Sample Size for Mock Up Testing

Reeve suggested that a minimal statistical design for evaluation of a new DWPF analytical method should include a minimum of 4 peanut vial samples taken per shift over six shifts⁽⁴⁾ for the current method. Also, the number of samples for the new method is to be determined to insure equivalent variability with regard to the current method.

Since the new method will require conversion of the sample results to a glass equivalent basis, the variation of the proposed calcine correction factor^(1a) was incorporated into the insert size selection.

4. Sampling and Analyses

Samples were pulled according to the insert study Mock-up test design^(1a). Insert samples within each sample block were prepared by either Cold Chem, which is a concentrated hydrofluoric acid digestion carried out at room temperature, or the modified fusion method, which is a scaled up version of the current peroxide/hydroxide method. Two sets of standards were used to mimic the reagents used for the preparations. Instrumental analysis by ICP-AES was under the same calibration per block per dissolution.

Both Dip & HydragardTM 15 ml samples were prepared using the current methods, microwave assisted mixed acid and hydroxide/peroxide fusion. Two set of standards (mixed acid & fusion) were used to mimic the reagents in the preparation. Instrumental analysis by ICP-AES was under the same calibration per block per dissolution.

A minimum of three to four lab technicians were to be rotated for sampling, preparation and analysis. The requirement was easily met due to shift environment and the flow of work. For each preparation set, at least two reagent blanks and two analytical reference glass (ARG-1) powders were included with the sample dissolutions.

The DWPF test was performed in 717-10S using a Mock-up facility with physical dimensions similar to analytical cells located in 221-S. All sampling and sample handling was performed in the mock-up cells using remote tools and master slave manipulators. Test conditions were nominally:

- Vial Flow Rate (1 gpm target)
- Valve Circulation Time(5-10 sec target)
- Valve Opening & Closing Rate: Nominal (without delay)
- Hold Time: Nominal (without delay)
- One Feed Type: PX-7 ~40-42wt%

The test was to include 6 blocks of samples. Each block represented a discrete event involving startup, operation and shutdown of the sampling system located in 717-10S adjacent to the analytical cells. The test totaled:

12 Dip 15 ml samples

24 HydragardTM 15 ml samples

108 Insert 3 ml samples: (54- Cold Chem, 54- Fusion & Wt% cal)

(Two samples were identified as outliers & deleted, Ref. Section 8.)

One cold chem insert sample and one fusion insert sample were identified as statistical outliers.

5. Statistical Comparisons

The statistical analysis includes the following items for satisfying the TTR HLW/DWPF/TTR-970132.

1. Determine the Hydragard™ sampler bias using the Li/Fe ratio for each method and compare to the 15 ml dip sample results.
2. Compare the elemental wt% of the new method insert samples versus the current method 15 ml Hydragard™ samples. Compute the bias between the new method and the current method.
3. Statistical study of sum of Oxides for Inserts vs Hydragard™ samples.

As further investigation of any calculated biases, statistical analysis of the check standard, ARG-1, elemental weight percentages and statistical analysis of blank elemental data were conducted. Variability comparisons, calculation of variance-covariance matrices, study of primary properties, determination of number of insert samples such that the variance of the new method is comparable to the variance of the current method are presented in a companion document by C.P. Reeve⁽⁷⁾.

6. Statistical Criteria for the New Method

The new method, for statistical comparison purposes, should satisfy multiple criteria applied to both the elemental wt% data and also to the primary property models. The new method's bias should be of no practical significance within the DWPF's Product Composition Control System(PCCS). A statistical test for bias is applied to the elemental wt% data in this report. Also, the impact of the calcine or vitrification factor is evaluated. An observed bias could be a result of the sampling method, the analytical method or the result of the correction factor when applied to the insert elemental results.

7. Analytical Standards

The ARG-1 check standard is a multicomponent glass standard developed by Corning for use by participants in the Material Characterization Center(MCC) at Pacific Northwest Laboratories. ARG-1 was analyzed at least twice along with each sample

preparation to evaluate systematic preparation errors. ARG-1 has been well characterized by the MCC. Also, it has been extensively analyzed at the DWPF analytical lab ⁽⁵⁾. Results from ARG-1 analyzed at DWPF, using the current method, in the time frame of March 1993 to September 8, 1995 are shown in Table 7 for comparison with the Mock-up test data.

8. Data Outputs

Data were supplied to SRTC-SCS by the DWPF Analytical Services both electronically and in hard copy(OPS-DTX-970023). The electronically transferred data was validated by SCS using the hard copy.

Data were supplied for microwave assisted mixed acid and peroxide/hydroxide fusion for the current methods, cold chem and fusion along with wt% calcined correction factor for the new methods. Sample data were statistically screened for outliers with only one sample being eliminated in the cold chem insert sample(Block 2, Pull 7) and one sample being eliminated in the fusion insert samples(Block 4, Pull 6). Statistical checks for valid data should be developed for routine production use of any new method. Two full blocks of data(Block 3 and Block 6) were not supplied by DWPF Engineering due to known errors in handling of wt% solids measurements. Two additional blocks were run: Block 7 & 8 to replace Blocks 3 and 6.

9. Assumptions

1. The statistical design centers on testing PX-7 feed at one rheology. It is inferred that the results apply to general SME and MFT sample analysis. Parametric testing has shown that varying feed rheologies within the design basis have no adverse effect on the quality of the insert samples when compared to 15 ml Dip and HydragardTM samples^(3a, 3b).
2. Procedural difficulties associated with Blocks 3 and 6 involved the weight % determinations. Handling delays in taking filled zirconium insert weights greatly expanded the range of values. It is unsure how much of the variation in blocks 1,2,4 and 5 are related to the handling. Blocks 7 and 8 were weighed without delay once the filled insert was transferred into the zirconium crucible.
3. A constant wt% solids was expected across all six blocks of data regardless of the method of analysis. This does not appear to be the case using summary statistics from Tables 5 and 6. Plot 7 shows the graphical comparisons block by block.

Chart 1

	Solids	Calcined	Vitrified
	<u>wt%</u>	<u>wt%</u>	<u>wt%</u>
Dip	40.6	-	34.4
Hyd	40.1	-	33.1
Dip & Hyd	40.2	-	33.5
Fusion Inserts	42.3	36.4	-

Based on close agreement between the zirconium inserts and additional vials pulled during block 4 which are not included in this report, DWPF Engineering suspects a systematic error in the Dip and Hydragard™ means. The correct value for wt% solids is expected to be 42.3 wt% solids taken from the fusion inserts.

10. Summary of Statistical Analyses

Summary statistics and Box-and-Whisker plots have been produced for the PX-7 samples, ARG-1 and blanks for the Mock-up test data. The analysis aids in this report can be studied with regard to specific comparisons of interest.

Separate statistical analyses were done using indicator ratios for the PX-7 samples and also calcine corrected insert data, ARG-1 elementals and elementals for blanks. The ARG-1 summary statistics are shown in Table 8. The established Corning values and DWPF historical ARG-1 results are shown in Table 7. Table 7 contains the statistical results for the current method fusion and mixed acid preparations. These data can be used for comparison purposes with the Mock-up test data. The summary statistics for the blanks are shown in Table 9.

The Box-and-Whisker plots were done for Li/Fe ratios(Plot 1), Si/Li ratios(Plot 2) for PX-7 samples and also the calcine corrected elemental wt% data(Plot 3). Box-and-Whisker plots were also done for the insert fusion data(Plot 4) and the ARG-1 elemental wt%'s(Plot 5). The plots were produced by Statgraphics⁽⁶⁾.

Each Box-and-Whisker plot displays the minimum and maximum values, the 25th, 50th(median) and 75th percentiles. The box is aligned vertically and encloses the interquartile range(the 25th to 75th percentile). The upper part of the box represents the 75th percentile while the lower part represents the 25th percentile. Extreme points, indicated by small boxes or +'s, are shown extending from the box.

10.1 Elemental wt% Relative Differences:

Hydragard™ 15 ml samples vs 3 ml insert cold chem samples

Hydragard™ 15 ml samples vs 3 ml insert fusion samples

Cold chemical inserts

The percent difference between the Hydragard™ 15 ml and the cold chemical insert data lies within the range -6.4% to -2.2% for the calcine corrected data and across the major elements as defined as greater than 1.0 wt% (Table 3). A negative bias (corrected insert elemental wt%'s are lower than the peanut vial Hydragard™ sample wt%'s) is typically seen for the calcine corrected data(ref. Sec. 11). This bias can be the result of the correction factor, the sampling, and/or the analytical method.

Using a vitrification correction factor and reweighting to a constant wt% solids, the percent differences lies in the range -3.1% to 1.2%(Table 3). The constant wt% vitrification corrected data do not typically show a significant bias for the major elementals.

The calcine correction factors and vitrification correction factors are shown in Table 6. The vitrification factors were based on the Hydragard™ peanut vial samples while the calcine correction factors were determined from the fusion inserts.

Fusion inserts

The percent difference between the Hydragard™ 15 ml using the fusion preparation method and the fusion insert data lies within the range -2.7 to -0.8% for the calcine basis data and from 0.7% to 2.7% for the constant wt% vitrification corrected data across the major elements(greater than 1.0 wt% in Table 4). However, the bias is not significant for either case. Comparisons with Hydragard™ 15 ml analyses using the mixed acid preparation are shown in Table 4.1. The calcine insert data typically have a significant negative bias ranging between -5.6% to -2.5% for the major elements except for Si which is positively biased at +3.4%.

10.2 Li/Fe Ratios

The Li/Fe ratio is an indicator for the uniformity of the sample which is insensitive to conversion factors. It is a sensitive measurement of the frit to sludge composition since the Li is exclusively from the Frit and the Fe is exclusively from the sludge component.

Over all blocks, for insert cold chemical Li/Fe ratios, the difference in means between the 3 ml insert samples and the current method(Mixed Acid) dip 15 ml samples was marginally significant. The average relative difference across all blocks was -6.3% (Table 1.1) with a 95% confidence interval of -12.7% to +0.1%. Over all blocks, for insert fusion Li/Fe ratios, there was also a marginally significant difference in means

between the insert samples and the fusion dip 15 ml samples. The average relative difference across all blocks was -6.8%(Table 2.1) with a 95% confidence interval of -12.7% to -0.1%. The relative difference between the insert fusion Li/Fe ratios and the current method Mixed Acid dip samples was -8.1%(Table 2.1.1) with a 95% confidence interval of -14.1% to -2.0%.

The Li/Fe ratios for the Hydragard™ samples were about 6% lower than the dip samples for both fusion and mixed acid preparations. This is within expectations based on the Stiemke studies in both magnitude and directions⁽²⁾ implying that the sample loop and sampling valve provide a technically equivalent sample as compared to the mezzanine installed system in use in 221-S.

The average insert cold chemical Li/Fe ratio lines up very well with the ratio from the current method Hydragard™ peanut vial samples. This implies that the frit was fully dissolved by the Cold Chem method and that the insert is not impacting the composition of the sample.

10.3 Si/Li Ratios

Silicon and lithium are present in the feed predominately as frit components. The ratio of the two elements should be a constant with some variation associated with the noise of the elemental analysis. The changes in the ratio may indicate losses of silicon as SiF₄ or precipitation of silicon as silica or silicon etched from the plasma torch contributing to the analysis.

Over all blocks, for insert cold chemical Si/Li ratios, there was no significant difference in means between the 3 ml insert samples and the mixed acid dip 15 ml samples. The average relative difference across all blocks was -0.6%(Table 1.2) with a 95% confidence interval of -4.6% to +3.5%. The insert fusion Si/Li ratios were significantly different than the fusion dip 15 ml samples. The average relative difference across all blocks was 1.8%(Table 2.2) with a 95% confidence interval of 0.4% to 3.3%. The relative difference between the insert fusion Si/Li ratios and the mixed acid dip samples was 10.1%(Table 2.2.1). The Si/Li ratios, for the fusion or mixed acid dip samples, were not statistically different than the Hydragard™ peanut vial samples. However, the dip fusion Si/Li ratios(11.769) are typically higher than the dip mixed acid Si/Li ratios (10.997) . Similar results hold for the peanut vial Hydragard™ Si/Li ratios(11.82 for fusion, 10.95 for mixed acid). This suggests that any difference is germane to the current methods.

10.4 ARG-1

ARG-1 has been used as a check standard for the insert cold chemical method. However, it does not provide an assessment of the reliability of the calcine factor used to adjust Cold Chemical elemental concentrations. In addition, it does not cover the dissolution process on the compounds originating in the simulant feed.

Box-and-Whisker plots are shown in Plot 5 along with the Corning elemental wt% means. The ARG-1 average elemental wt%'s, from Mock-up testing, are comparable to the Corning values except for Si using the Cold Chemical preparation. The Cold Chemical Si wt% is $7.4\% \pm 0.9\%$ lower (95% confidence limits) than the Corning value (Chart 2). The Si for Insert Cold Chem is also 3.3% below the Si from the Hydragard™ peanut vial mixed acid samples which is historically biased low. The variability for the Insert Fusion preparations(Table 8) is somewhat greater than historical values (Table 7) while the mixed acid variability is lower. The variability for the insert cold chemical ARG-1 data is comparable to ARG-1 fusion data.

Chart 2

	<u>ARG1: Si</u>		
	<u>N</u>	<u>Average</u>	<u>Standard</u>
		<u>wt%</u>	<u>Deviation</u>
			<u>wt%</u>
Corning		22.4	-
Historical⁽⁵⁾			
DWPF Fusion	287	23.0	0.670
DWPF Mixed Acid	386	21.8	0.908
Parametric Test^(3a)			
Insert Cold Chemical ^(*)	37	21.7	0.809
Mock-up			
Insert Fusion	24	22.7	0.933
Peanut Vial Mixed Acid	13	21.4	0.515
Insert Cold Chemical	12	20.7	0.859

(*) Tests: 1, 2, 3, 4, reTEST 5

10.5 Blanks

The elemental concentrations for these reagent blanks are known to be zero. The average measurement from a group of such samples processed via an analytical procedure provides an indication of the potential bias in that analytical procedure.

The sample statistics are shown in Table 9 and Box-and-Whisker plots for the various elemental concentrations are shown in Plot 6. The averages are not large enough to be considered to be of practical importance, except for insert cold chemical Si(0.65 mg/L) noted in Chart 3. Thus, the cold chem method may be providing a Si value slightly biased high while the results from section 10.4 indicate that Si values are biased low compared to the current method.

Chart 3

	Blanks: Si		
	N	Average	Standard Deviation
		mg/L	mg/L
Historical ⁽⁵⁾			
DWPF Fusion	102	0.0514	0.0954
DWPF Mixed Acid	114	-0.1935	0.2072
Parametric Test ^(3a)			
Insert Cold Chemical ^(*)	35	0.135	0.568
Mock-up			
Insert Fusion	24	-0.0169	0.0794
Peanut Vial Mixed Acid	13	-0.0106	0.2029
Insert Cold Chemical	12	0.6457	0.2714

(*) Tests 1, 2, 3, 4, reTEST 5

11. Corrected Insert wt%'s

The wt% total solids is obtained from the difference between the slurry weight and the weight of the solids after drying at 115° C. A calcine correction factor (f_c) was obtained by taking the difference between the weight of the slurry before drying and the weight of the oxides after converting most of the compounds to oxides, by heating the sample to between 600° and 650° C. Vitrification is the conversion of compounds to a glass

between 1050° and 1150° C. Samples composed of metal salts of hydroxides and nitrates can be calcined easily and the correction factor is proposed as equivalent to vitrification factor. On this basis the calcine factor can be used to convert the elemental results from a slurry basis(I_s) to a calcined basis(I).

The correction factor for the insert elemental wt% data is applied as $I = I_s / f$ where $f = f_c$ or f_v , in this report, depending on if a calcine correction or a vitrification correction is desired. The vitrification correction factor was derived from the Hydragard™ 15 ml peanut vial samples using the current fusion analytical method.

Vitrification results may only be available for the new method as a check on the calcine correction factor. The correction factors can be determined by averaging the wt% vitrified or the wt% calcined using the mock-up test data. This assumes that the solids contents of both the peanut vials and inserts are the same. However, this does not appear to be the case in examining the data(Chart 1).

C.P. Reeve has suggested an adjustment for conversion to a vitrified basis. Specifically, let S_{pv} and S_i be the average total wt% solids for the peanut vials and the inserts, respectively. The correction factor, for adjusting the fusion insert data to a constant weight percent solids, can be computed as

$$f = (f_v / S_{pv}) / (f_i / S_i)$$

where f_v is a vitrification factor from the peanut vial data and f_i is from the fusion insert data. The correction factor $f=0.966$ for the fusion insert data. Similarly, for the cold chemical insert data $f=0.352$.

12. Oxide Sum

The sums of oxides for various correction factors applied to the insert cold chemical and insert fusion preparations, appear in Tables 3 and 4 and are summarized in Chart 4. The cold chem dissolution method provides a low sum of oxide recovery (91%) when calcine corrected. However, the wt% total solids varies between the different preparation methods. Using a vitrification correction, to constant wt% solids, increases the sum of oxides for the cold chemical inserts to 94.3%.

Based on the data supplied by TNX, PX-7 is expected to contain small concentrations of materials which were not analyzed for in this test. The sum of oxides may be slightly higher than reported if the non-analyzed materials were determined for the sample.

Chart 4

Mock-up Sum of Oxides

Insert Cold Chemical	Insert	Hydra (**)	Dip (**)
Calcine Corrected Block to Block	91.1%	96.4%	96.9%
Calcine Corrected to Overall Block Average	91.0%		
Vitrification Corrected to Constant wt% Solids	94.3%		

Fusion Inserts

Calcined Elemental Data	96.1% (**)	97.8%	98.5%
Vitrification Corrected to Constant wt% Solids	99.5%		

(*) Na & Zr by Cold Chem & the remaining elements by Fusion

(**) B by Fusion & the remaining elements by Mixed Acid, Vitrification results are only available for the peanut vial Hydragard™ and Dip samples.

The correction factor was applied to the insert Cold Chem samples before the oxide gravimetric factors were applied.

13. Conclusions

The PX-7 simulant samples from the DWPF Mock-up facility were taken within the range 40 - 42 wt% solids in order to simulate process rheology. Preliminary testing⁽²⁾ using PX-7 samples taken with a prototypic Hydragard™ in the 786-A Thermal Fluids Laboratory has indicated that varying feed compositions and feed rheologies have no adverse effect on the quality of the insert samples.

Statistical analysis of Mock-up testing indicator ratios(Li/Fe, Si/Li) has indicated that consistent sampling and analytical performance, relative to the current method, was obtained using 3 ml inserts and the cold chemical preparation method. Over all blocks, for insert cold chemical Li/Fe ratios, there was a marginally significant difference (-6.3%) in means between the 3 ml insert samples and the current method(Mixed Acid) 15ml dip samples. For insert fusion Li/Fe ratios, there also was a marginally significant difference(-6.8%) in means between the insert samples and the fusion dip 15 ml samples.

For insert cold chemical Si/Li ratios, there was not a significant difference(-0.6%) in means between the 3 ml insert samples and the mixed acid dip 15 ml samples. The insert fusion Si/Li ratios were significantly different than the fusion dip 15 ml samples.

The ARG-1 average elemental wt%'s, from Mock-up testing, are comparable to the Corning values except for Si using the Cold Chemical preparation. The Cold Chemical Si wt% is 7.4% lower than the Corning value. The cold chem method may be providing a Si value biased relatively high based on analysis of reagent blanks.

The cold chem dissolution method seems to provide a low sum of oxide recovery (91%) when calcine corrected. However, the wt% total solids varies between the different preparation methods. Using a vitrification correction, to constant wt% solids, increases the sum of oxides for the cold chemical inserts to 94.3%.

The percent difference between the calcine correction and the vitrification correction factor should be determined through experimentation and documented. As seen in this report, the choice of the correction factor can have a significant effect on the bias of the Insert Cold Chemical elemental wt% data. Low ARG-1 recovery values for Si inserts using the Cold Chem preparation method should be investigated further. In addition, the cause of high Si blank wt%'s should be determined.

Any impact of the biases seen in the comparisons between the new and current methods on the constraints in PCCS was not within the scope of this study. As such, an assessment should be included in the plan for implementation of the new method.

ATTACHMENTS

Table 1.1: Summary Statistics for Li/Fe Ratios

Insert Cold Chem(ICC) vs Peanut Vial Dip & PV HydragardTM Samples

Table 1.2: Summary Statistics for Si/Li Ratios

Insert Cold Chem(ICC) vs Peanut Vial Dip & PV HydragardTM Samples

Table 2.1: Summary Statistics for Li/Fe Ratios

Insert Fusions(IF) vs Peanut Vial Dip & PV HydragardTM Samples

Table 2.1.1: Summary Statistics for Li/Fe Ratios

Insert Fusion(IF) vs Current Method(Mixed Acid) Peanut Vial Samples

Table 2.2: Summary Statistics for Si/Li Ratios

Insert Fusions(IF) vs Peanut Vial Dip & PV HydragardTM Samples

Table 2.2.1: Summary Statistics for Si/Li Ratios

Insert Fusion(IF) vs Current Method(Mixed Acid) Peanut Vial Samples

Table 3: Bias for Insert Cold Chemical Elemental wt% Data SCREENED for Outliers

Table 4: Bias for Insert Fusion Elemental wt% Data Peanut Vial Fusion SCREENED for Outliers

Table 4.1: Bias for Insert Fusion Elemental wt% Data Peanut Vial Mixed Acid SCREENED for Outliers

Table 5: Summary of wt% Solids

Table 6: Summary of PV Vitrified Solids & Insert Calc Solids

Table 7: Historical ARG-1 from SRT-ASG-95-0081

Table 8: Sample Statistics for ARG-1 from MOCK-up Test

Table 9: Sample Statistics for BLANKS from MOCK-up Test

Plot 1: Box-and Whisker Plots: Li/Fe Ratio

Plot 2: Box-and Whisker Plots: Si/Li Ratio

**Plot 3: Box-and Whisker Plots: Calcine Corrected Insert Cold
Chemical Elemental wt% data**

Plot 4: Box-and Whisker Plots: Insert Fusion Elemental wt% data

Plot 5: Box-and Whisker Plots: ARG-1 Elemental wt% data

Plot 6: Box-and Whisker Plots: Blank Elemental wt% data

**Plot 7: Box-and Whisker Plots: wt% Total Solids, wt% Vitrified & wt%
Calcined Solids**

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Table 1.1

Summary Statistics for Li/Fe Ratios

Insert Cold Chem(ICC) vs Peanut Vial Dip & PV Hydragard™ Samples

(M): Current Method Mixed Acid

Block	ICC		Dip(M)		Hyd(M)		ICC-Dip(M)	Hyd(M)-Dip(M)
	N	Li/Fe Avg	N	Li/Fe Avg	N	Li/Fe Avg		
1	9	0.240	2	0.272	4	0.249	-0.0322*	-0.0232*
2	9	0.238	2	0.270	4	0.248	-0.0319*	-0.0218
4	8	0.261	2	0.273	4	0.263	-0.0124	-0.0101
5	9	0.256	2	0.275	4	0.251	-0.0194	-0.0240*
7	9	0.262	2	0.250	4	0.248	0.0126	-0.0012
8	9	0.252	2	0.270	4	0.250	-0.0181	-0.0195
Avg				0.2683			-0.0169(-6.3%)	-0.0166(-6.2%)

95% Conf:

Upper
 Lower

ICC-Dip(M)	Hyd(M)-Dip(M)
0.0004(0.1%)	-0.0071(-2.7%)
-0.0342(-12.7%)	-0.0262(-9.8%)

* Significant based on 95% Confidence & Least Significant Differences

Table 1.2

Summary Statistics for Si/Li Ratios

Insert Cold Chem(ICC) vs Peanut Vial Dip & PV Hydragard™ Samples

(M): Mixed Acid

Block	ICC		Dip(M)		Hyd(M)		ICC-Dip(M)	Hyd(M)-Dip(M)
	N	Si/Li Avg	N	Si/Li Avg	N	Si/Li Avg		
1	9	11.159	2	10.819	4	10.891	0.340	0.072
2	9	10.993	2	10.630	4	10.764	0.363	0.134
4	8	10.795	2	10.706	4	10.719	0.089	0.013
5	9	10.526	2	10.717	4	10.847	-0.191	0.130
7	9	10.723	2	11.496	4	11.549	-0.773*	0.054
8	9	10.721	2	10.955	4	10.922	-0.235	-0.033
Avg				10.887			-0.068(-0.6%)	0.062(+0.6%)

95% Conf:

	<u>ICC-Dip(M)</u>	<u>Hyd(M)-Dip(M)</u>
Upper	0.382(+3.5%)	0.130(+1.2%)
Lower	-0.517(-4.6%)	-0.007(-0.1%)

* Significant based on 95% Confidence & Least Significant Differences

Table 2.1

Summary Statistics for Li/Fe Ratios

Insert Fusions(IF) vs Peanut Vial Dip & PV Hydragard™ Samples

(F): Fusion

Block	IF		Dip(F)		Hvd(F)		IF-Dip(F)	Hvd(F)-Dip(F)
	N	Li/Fe Avg	N	Li/Fe Avg	N	Li/Fe Avg		
1	9	0.234	2	0.273	4	0.251	-0.0393*	-0.0222
2	6	0.241	2	0.267	4	0.245	-0.0262*	-0.0222
4	7	0.255	2	0.264	4	0.254	-0.0095	-0.0099
5	7	0.251	2	0.271	4	0.248	-0.0205	-0.0236
7	8	0.257	2	0.253	4	0.250	0.0041	-0.0022
8	8	0.243	2	0.259	4	0.244	-0.0163	-0.0155
Avg				0.2646			-0.0180(-6.8%)	-0.0159(-6.0%)

95% Conf:

	<u>IF-Dip(F)</u>	<u>Hvd(F)-Dip(F)</u>
Upper	-0.0024(-0.1%)	-0.0070(-2.6%)
Lower	-0.0335(-12.7%)	-0.0249(-9.4%)

* Significant based on 95% Confidence & Least Significant Differences

Table 2.1.1

Summary Statistics for Li/Fe Ratios

Insert Fusion(IF) vs Current Method Peanut Vial Samples

(M): Current Method Mixed Acid

Block	IF		Dip(M)		Hyd(M)		IF-Dip(M)	Hyd(M)-Dip(M)
	N	Li/Fe Avg	N	Li/Fe Avg	N	Li/Fe Avg		
1	9	0.234	2	0.272	4	0.249	-0.0379*	-0.0232
2	6	0.241	2	0.270	4	0.248	-0.0293*	-0.0218
4	7	0.255	2	0.273	4	0.263	-0.0186	-0.0101
5	7	0.251	2	0.275	4	0.251	-0.0243*	-0.0240
7	8	0.257	2	0.250	4	0.248	0.0071	-0.0012
8	8	0.243	2	0.270	4	0.250	-0.0265*	-0.0195
Avg				0.2683			-0.0216(-8.1%)	-0.0166(-6.2%)

95% Conf:

	IF-Dip(M)	Hyd(M)-Dip(M)
Upper	-0.0054(-2.0%)	-0.0071(-2.7%)
Lower	-0.0378(-14.1%)	-0.0262(-9.8%)

* Significant based on 95% Confidence & Least Significant Differences

Table 2.2

Summary Statistics for Si/Li Ratios

Insert Fusions(IF) vs Peanut Vial Dip & PV Hydragard™ Samples

(F): Fusion

Block	IF		Dip(F)		Hyd(F)		IF-Dip(F)	Hyd(F)-Dip(F)
	N	Si/Li Avg	N	Si/Li Avg	N	Si/Li Avg		
1	9	11.880	2	11.608	4	11.688	0.272*	0.080
2	6	11.944	2	11.971	4	11.882	-0.026	-0.088
4	7	12.021	2	11.695	4	11.882	0.326*	0.186
5	7	11.988	2	11.630	4	11.777	0.358*	0.147
7	8	12.043	2	11.707	4	11.720	0.336*	0.014
8	8	12.040	2	12.003	4	11.957	0.038	-0.046
Avg				11.769			0.217(1.8%)	0.049(+0.4%)

95% Conf:

Upper
 Lower

<u>IF-Dip(F)</u>	<u>Hyd(F)-Dip(F)</u>
0.393(+3.3%)	0.162(1.4%)
0.042(0.4%)	-0.065(-0.6%)

* Significant based on 95% Confidence & Least Significant Differences

Table 2.2.1

Summary Statistics for Si/Li Ratios

Insert Fusion(IF) vs Current Method Peanut Vial Samples

(M): Current Method Mixed Acid

Block	<u>IF</u>		<u>Dip(M)</u>		<u>Hyd(M)</u>		<u>IF-Dip(M)</u>	<u>Hyd(M)-Dip(M)</u>
	N	Si/Li Avg	N	Si/Li Avg	N	Si/Li Avg		
1	9	11.880	2	10.819	4	10.891	1.0613*	0.072
2	6	11.944	2	10.630	4	10.764	1.3144*	0.134
4	7	12.021	2	10.706	4	10.719	1.3149*	0.013
5	7	11.988	2	10.717	4	10.847	1.2710*	0.130
7	8	12.043	2	11.496	4	11.549	0.5469*	0.054
8	8	12.040	2	10.955	4	10.922	1.0850*	-0.033
Avg				10.887			1.0989(10.1%)	0.062(+0.6%)

95% Conf:

	<u>IF-Dip(M)</u>	<u>Hyd(M)-Dip(M)</u>
Upper	1.406(12.9%)	0.130(+1.2%)
Lower	0.791(7.3%)	-0.007(-0.1%)

* Significant based on 95% Confidence & Least Significant Differences

Table 3

**Bias for Insert Cold Chemical Elemental wt% Data
 SCREENED for Outliers**

Hyd: HydragardTM Peanut Vial Samples using the Current Method,
 B from Fusion & remaining elements from Mixed Acid

ICC: Insert Cold Chem

Bias: ICC - Hyd

Avg Percent Bias= 100(ICC-Hyd)/Hyd

Lo, Up: Lower & Upper 95% Confidence Limits

Calcine Corrected Block to Block

	<u>Averages</u>		<u>Bias</u>	<u>Percent Bias</u>		
	<u>Hyd</u> wt%	<u>ICC</u> wt%	wt%	<u>Lo</u>	<u>Avg</u>	<u>Up</u>
AL	2.387	2.263	-0.124	-10.2	-5.2	-0.3
B	2.556	2.455	-0.101	-10.2	-3.9	2.3
CA	0.729	0.749	0.019	-13.4	2.7	18.7
CR	0.079	0.036	-0.043	-61.6	-54.2	-46.8
CU	0.294	0.276	-0.018	-10.6	-6.1	-1.7
FE	7.873	7.455	-0.418	-10.0	-5.3	-0.6
K	2.519	2.464	-0.056	-6.9	-2.2	2.4
LI	1.981	1.885	-0.095	-8.7	-4.8	-0.9
MG	0.863	0.790	-0.073	-13.3	-8.5	-3.7
MN	1.579	1.496	-0.082	-9.8	-5.2	-0.6
NA	8.089	7.730	-0.359	-8.2	-4.4	-0.7
NI	0.875	0.807	-0.068	-11.9	-7.8	-3.7
SI	21.679	20.296	-1.383	-11.0	-6.4	-1.8
TI	0.084	0.078	-0.006	-12.2	-7.5	-2.7
ZR	0.867	0.799	-0.067	-13.1	-7.8	-2.5
OXSUM	96.427	91.060	-5.367	-9.4	-5.6	-1.7

Calcine Corrected to Overall Block Average

	<u>Averages</u>		<u>Bias</u>	<u>Percent Bias</u>		
	<u>Hyd</u> wt%	<u>ICC</u> wt%	wt%	<u>Lo</u>	<u>Avg</u>	<u>Up</u>
AL	2.387	2.262	-0.125	-9.5	-5.2	-1.0
B	2.556	2.457	-0.099	-11.4	-3.9	3.7
CA	0.729	0.749	0.019	-14.2	2.7	19.5
CR	0.079	0.036	-0.043	-61.6	-54.2	-46.8
CU	0.294	0.275	-0.018	-9.7	-6.2	-2.6
FE	7.873	7.452	-0.421	-8.9	-5.3	-1.8
K	2.519	2.463	-0.057	-5.7	-2.3	1.2
LI	1.981	1.885	-0.095	-8.1	-4.8	-1.6
MG	0.863	0.790	-0.073	-11.9	-8.5	-5.1
MN	1.579	1.496	-0.083	-8.8	-5.2	-1.7
NA	8.089	7.729	-0.360	-7.3	-4.5	-1.7
NI	0.875	0.807	-0.068	-11.4	-7.8	-4.2
SI	21.679	20.291	-1.389	-10.1	-6.4	-2.8
TI	0.084	0.078	-0.006	-11.5	-7.5	-3.5
ZR	0.867	0.799	-0.068	-12.2	-7.8	-3.5
OXSUM	96.427	91.041	-5.386	-8.7	-5.6	-2.5

**Table 3
 (Continued)**

Vitrification Corrected to Constant wt% Solids

	<u>Averages</u>		<u>Bias</u>	<u>Percent Bias</u>		
	<u>Hyd</u> wt%	<u>ICC</u> wt%	wt%	<u>Lo</u>	<u>Avg</u>	<u>Up</u>
AL	2.387	2.342	-0.045	-6.2	-1.9	2.4
B	2.556	2.543	-0.012	-8.3	-0.5	7.3
CA	0.729	0.775	0.046	-11.0	6.3	23.6
CR	0.079	0.038	-0.042	-60.1	-52.6	-45.1
CU	0.294	0.285	-0.008	-6.5	-2.9	0.8
FE	7.873	7.716	-0.157	-5.6	-2.0	1.6
K	2.519	2.550	0.030	-2.3	1.2	4.7
LI	1.981	1.952	-0.029	-4.8	-1.4	1.9
MG	0.863	0.817	-0.045	-8.7	-5.3	-1.8
MN	1.579	1.549	-0.030	-5.5	-1.9	1.7
NA	8.089	8.002	-0.087	-3.9	-1.1	1.8
NI	0.875	0.835	-0.040	-8.2	-4.6	-0.9
SI	21.679	21.008	-0.671	-6.8	-3.1	0.7
TI	0.084	0.081	-0.004	-8.3	-4.2	-0.2
ZR	0.867	0.827	-0.040	-9.0	-4.6	-0.1
OXSUM	96.427	94.260	-2.167	-5.4	-2.2	0.9

Table 4

**Bias for Insert Fusion Elemental wt% Data
 SCREENED for Outliers**

Hyd: Hydragard™ Fusion Peanut Vial Samples using the Current Method
 IF: Insert Fusion

Bias: IF - Hyd
 Avg Percent Bias= 100(IF-Hyd)/Hyd
 Lo, Up: Lower & Upper 95% Confidence Limits

Fusion Inserts(Calcine Basis) Elemental Data

	<u>Averages</u>		<u>Bias</u>	<u>Percent Bias</u>		
	Hyd wt%	IF wt%	wt%	Lo	Avg	Up
AL	2.328	2.265	-0.063	-5.6	-2.7	0.2
B	2.555	2.486	-0.068	-5.3	-2.7	-0.1
CA	0.656	0.603	-0.053	-19.9	-8.0	3.8
CR	0.084	0.085	0.001	-33.6	1.1	35.7
CU	0.281	0.278	-0.003	-4.9	-1.0	2.8
FE	7.730	7.666	-0.064	-6.1	-0.8	4.4
K	2.442	2.397	-0.045	-6.5	-1.8	2.8
LI	1.922	1.870	-0.052	-5.4	-2.7	0.0
MG	0.818	0.822	0.004	-3.3	0.5	4.4
MN	1.541	1.524	-0.017	-4.6	-1.1	2.5
NA	8.055	7.887	-0.169	-3.5	-2.1	-0.7
NI	0.843	0.829	-0.014	-6.3	-1.6	3.1
SI	22.712	22.424	-0.288	-3.8	-1.3	1.3
TI	0.083	0.080	-0.003	-6.9	-3.4	0.1
ZR	0.858	0.816	-0.042	-9.9	-4.9	0.0
OXSUM	97.752	96.137	-1.615	-4.2	-1.7	0.9

Fusion Inserts Vitrification Corrected to Constant wt% Solids

	<u>Averages</u>		<u>Bias</u>	<u>Percent Bias</u>		
	Hyd wt%	IF wt%	wt%	Lo	Avg	Up
AL	2.328	2.345	0.017	-2.2	0.7	3.7
B	2.555	2.574	0.020	-1.8	0.8	3.4
CA	0.656	0.624	-0.031	-16.9	-4.8	7.3
CR	0.084	0.088	0.004	-30.8	4.7	40.1
CU	0.281	0.288	0.007	-1.4	2.5	6.4
FE	7.730	7.937	0.207	-2.7	2.7	8.1
K	2.442	2.482	0.040	-3.0	1.6	6.3
LI	1.922	1.936	0.014	-2.0	0.7	3.5
MG	0.818	0.851	0.033	0.2	4.1	8.0
MN	1.541	1.578	0.037	-1.2	2.4	6.1
NA	8.055	8.166	0.111	-0.1	1.4	2.8
NI	0.843	0.859	0.016	-2.9	1.9	6.7
SI	22.712	23.217	0.506	-0.4	2.2	4.8
TI	0.083	0.083	0.000	-3.5	0.0	3.5
ZR	0.858	0.845	-0.013	-6.7	-1.6	3.5
OXSUM	97.752	99.540	1.788	-0.8	1.8	4.4

Table 4.1

**Bias for Insert Fusion Elemental wt% Data
 SCREENED for Outliers**

Hyd: HydragardTM Mixed Acid Peanut Vial Samples using the Current Method
 IF: Insert Fusion

Bias: IF - Hyd
 Avg Percent Bias= 100(IF-Hyd)/Hyd
 Lo, Up: Lower & Upper 95% Confidence Limits

Fusion Inserts(Calcine Basis) Elemental Data

	<u>Averages</u>		<u>Bias</u>	<u>Percent Bias</u>		
	Hyd wt%	IF wt%	wt%	Lo	Avg	Up
AL	2.387	2.265	-0.122	-8.9	-5.1	-1.4
B	2.556	2.486	-0.069	-5.0	-2.7	-0.4
CA	0.729	0.603	-0.126	-26.2	-17.3	-8.4
CR	0.079	0.085	0.006	-28.6	7.2	43.1
CU	0.294	0.278	-0.015	-9.2	-5.3	-1.3
FE	7.873	7.666	-0.207	-7.8	-2.6	2.6
K	2.519	2.397	-0.122	-8.8	-4.9	-0.9
LI	1.981	1.870	-0.111	-8.5	-5.6	-2.7
MG	0.863	0.822	-0.041	-7.1	-4.7	-2.3
MN	1.579	1.524	-0.055	-7.1	-3.5	0.2
NA	8.089	7.887	-0.202	-4.3	-2.5	-0.7
NI	0.875	0.829	-0.046	-9.4	-5.2	-1.0
SI	21.679	22.424	0.744	0.0	3.4	6.9
TI	0.084	0.080	-0.004	-9.2	-4.6	0.1
ZR	0.867	0.816	-0.051	-11.2	-5.9	-0.5
OXSUM	96.427	96.137	-0.291	-3.4	-0.3	2.8

Fusion Inserts Vitrification Corrected to Constant wt% Solids

	<u>Averages</u>		<u>Bias</u>	<u>Percent Bias</u>		
	Hyd wt%	IF wt%	wt%	Lo	Avg	Up
AL	2.387	2.345	-0.042	-5.6	-1.8	2.1
B	2.556	2.574	0.019	-1.6	0.7	3.1
CA	0.729	0.624	-0.105	-23.4	-14.4	-5.3
CR	0.079	0.088	0.009	-26.0	11.0	48.0
CU	0.294	0.288	-0.006	-5.9	-1.9	2.1
FE	7.873	7.937	0.064	-4.5	0.8	6.2
K	2.519	2.482	-0.037	-5.5	-1.5	2.5
LI	1.981	1.936	-0.044	-5.2	-2.2	0.7
MG	0.863	0.851	-0.011	-3.8	-1.3	1.1
MN	1.579	1.578	-0.001	-3.7	0.0	3.7
NA	8.089	8.166	0.077	-0.8	1.0	2.7
NI	0.875	0.859	-0.017	-6.2	-1.9	2.4
SI	21.679	23.217	1.538	3.6	7.1	10.6
TI	0.084	0.083	-0.001	-5.9	-1.2	3.5
ZR	0.867	0.845	-0.022	-8.0	-2.5	2.9
OXSUM	96.427	99.540	3.112	0.1	3.2	6.4

Table 5

Summary of wt% Total Solids

The coefficient of variation: $CV = 100 \text{ (Standard Deviation) / Average}$

Peanut Vial Dip & Hydragard™ data were combined since there is no statistical difference in wt% data.

Block	<u>Peanut Vial Dip</u>			<u>Peanut Vial Hyd</u>			<u>Dip & Hyd</u>			<u>Fusion Inserts</u>		
	N	Avg	CV	N	Avg	CV	N	Avg	CV	N	Avg	CV
		wt%			wt%			wt%			wt%	
		<u>Sol</u>	<u>%</u>		<u>Sol</u>	<u>%</u>		<u>Sol</u>	<u>%</u>		<u>Sol</u>	<u>%</u>
1	2	38.95	4.3%	4	40.12	1.1%	6	39.73	2.5%	9	41.28	1.7%
2	2	40.57	1.9%	4	39.91	8.5%	6	40.13	6.7%	8	43.95	4.8%
4	2	40.88	0.3%	4	37.71	8.5%	6	38.77	7.7%	9	42.33	4.3%
5	2	40.96	1.4%	4	41.38	6.2%	6	41.24	4.9%	9	42.47	2.9%
7	2	41.62	0.6%	4	40.95	0.5%	6	41.17	1.0%	9	41.99	1.1%
8	2	40.68	0.2%	4	40.26	1.0%	6	40.40	0.9%	8	41.68	1.0%
Avg	12	40.61	2.5%	24	40.06	2.3%	36	40.24	4.8%	52	42.26	3.5%

<u>Block</u>	<u>Diff(F -DH)</u> wt%
1	1.55
2	3.82
4	3.56
5	1.23
7	0.82
8	1.28
Avg	2.04

95% Conf Limits:

Upper	3.41
Lower	0.68

Table 6

Summary of Peanut Vial Vitrified Solids & Insert Calc Solids

The coefficient of variation: CV= 100 (Standard Deviation) / Average

Block	VITRIFIED SOLIDS									CALCINED SOLIDS		
	Dip			Hyd			Dip& Hyd			Fusion Inserts		
	N	Avg wt%	CV %	N	Avg wt%	CV %	N	Avg wt%	CV %	N	Avg wt%	CV %
		Sol	%		Sol	%		Sol	%		Sol	%
1	2	33.02	4.1%	4	33.54	1.2%	6	33.37	2.2%	9	35.43	1.7%
2	2	35.42	5.3%	4	32.92	8.8%	6	33.75	8.1%	8	36.97	4.7%
4	2	33.92	0.8%	4	30.74	9.0%	6	31.80	8.5%	9	36.97	4.6%
5	2	34.34	0.4%	4	34.16	7.1%	6	34.22	5.5%	9	36.80	3.7%
7	2	35.08	0.7%	4	34.03	0.7%	6	34.38	1.7%	9	36.42	1.5%
8	0			0						8	35.96	1.6%
Avg	10	34.36	3.5%	20	33.08	6.9%	30	33.51	6.1%	52	36.42	3.5%

Table 7

(SRT-ASG-95-0081)

ARG-1

DWPF Historical Data from the Current Method

SCREENED for Outliers

Sample statistics based on individual analysis results.
 Std: Sample standard Deviation

	CORNING wt%	FUSION			MIXED ACID		
		N	Avg wt%	Std wt%	N	Avg wt%	Std wt%
Al	2.50	279	2.45	0.071	393	2.43	0.071
B	2.69	286	2.57	0.095			
Ca	1.02	278	0.98	0.067	392	1.05	0.046
Cr	0.06	272	0.070	0.006	389	0.071	0.005
Cu	0	276	0.009	0.006	398	0.006	0.006
Fe	9.79	277	9.6	0.253	393	9.8	0.271
K	2.25	277	2.22	0.076	391	2.30	0.085
Li	1.49	276	1.46	0.039	387	1.49	0.039
Mg	0.52	275	0.51	0.015	397	0.52	0.017
Mn	1.46	280	1.43	0.039	392	1.45	0.040
Na	8.53				392	8.53	0.246
Ni	0.83	279	0.82	0.026	392	0.85	0.028
Si	22.4	287	23.0	0.670	386	21.8	0.908
Ti	0.69	280	0.68	0.018	392	0.70	0.020
Zr	0.10				391	0.102	0.004

Table 8

ARG-1

MOCK-up Test Data

Sample statistics based on individual ARG-1 analysis results

	CORNING wt%	INSERT FUSION			PEANUT VIAL FUSION		
		N	Avg wt%	Std wt%	N	Avg wt%	Std wt%
Al	2.50	12	2.46	0.118	12	2.45	0.077
B	2.69	12	2.60	0.146	12	2.65	0.098
Ca	1.02	12	0.96	0.059	12	0.97	0.089
Cr	0.06	12	0.075	0.012	12	0.077	0.014
Cu	0	12	0.007	0.006	12	0.007	0.002
Fe	9.79	12	9.71	0.491	12	9.66	0.386
K	2.25	12	2.22	0.138	12	2.27	0.092
Li	1.49	12	1.43	0.081	12	1.45	0.053
Mg	0.52	12	0.52	0.027	12	0.51	0.026
Mn	1.46	12	1.42	0.082	12	1.43	0.063
Na	8.53						
Ni	0.83	12	0.83	0.043	12	0.831	0.048
Si	22.4	12	22.74	1.103	12	22.59	0.741
Ti	0.69	12	0.69	0.033	12	0.688	0.026
Zr	0.10						

	CORNING wt%	MIXED ACID			COLD CHEM		
		N	Avg wt%	Std wt%	N	Avg wt%	Std wt%
Al	2.50	13	2.44	0.037	12	2.40	0.094
B	2.69				12	2.64	0.166
Ca	1.02	13	1.07	0.038	12	1.06	0.102
Cr	0.06	13	0.076	0.005	12	0.068	0.004
Cu	0	13	0.010	0.002	12	0.005	0.001
Fe	9.79	13	9.80	0.174	12	9.71	0.362
K	2.25	13	2.35	0.074	12	2.34	0.096
Li	1.49	13	1.50	0.022	12	1.49	0.059
Mg	0.52	13	0.54	0.010	12	0.52	0.037
Mn	1.46	13	1.46	0.029	12	1.45	0.055
Na	8.53	13	8.51	0.105	12	8.48	0.324
Ni	0.83	13	0.861	0.015	12	0.83	0.030
Si	22.4	13	21.43	0.515	12	20.74	0.859
Ti	0.69	13	0.706	0.014	12	0.70	0.027
Zr	0.10	13	0.104	0.003	12	0.102	0.004

Table 9

BLANKS(x 10⁻³)

MOCK-up Test Data

Sample statistics based on individual blank analysis results(x 10⁻³)

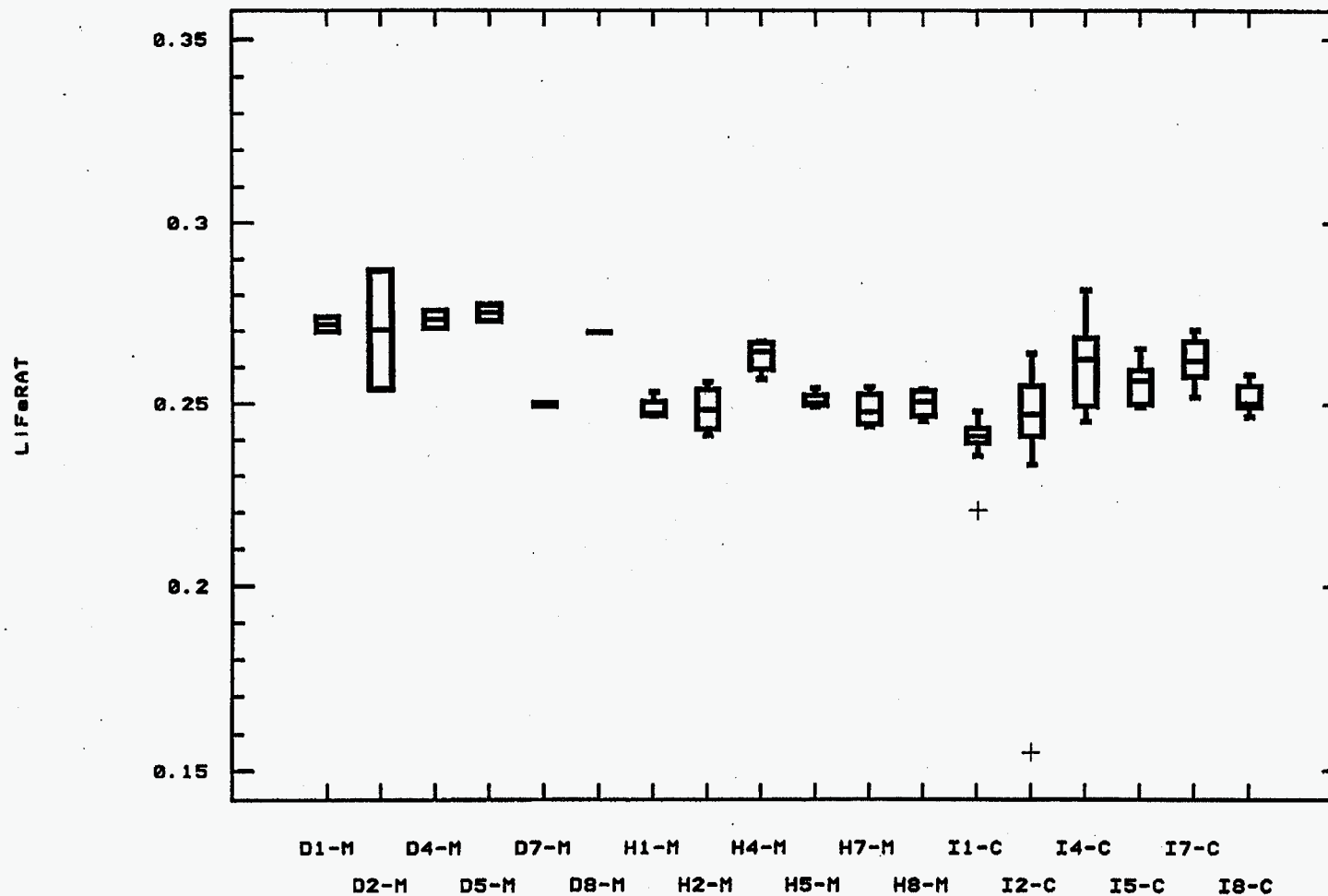
	<u>INSERT FUSION</u>			<u>PEANUT VIAL FUSION</u>		
	N	Mean mg/L	Std mg/L	N	Mean mg/L	Std mg/L
Al	12	11.8	9.7	12	15.8	7.9
B	12	6.8	28.6	12	9.3	43.5
Ca	12	-61.1	21.9	12	-4.6	24.1
Cr	12	11.7	13.0	12	12.8	11.4
Cu	12	4.4	2.8	12	5.3	3.6
Fe	12	96.1	188.1	12	57.3	80.0
K	12	29.4	24.2	12	44.1	61.7
Li	12	1.8	1.0	12	5.1	6.9
Mg	12	0.0	2.1	12	3.5	3.4
Mn	12	2.5	3.2	12	5.0	6.4
Na						
Ni	12	5.7	8.1	12	15.4	10.2
Si	12	-8.5	50.9	12	-25.3	102.2
Ti	12	4.9	2.7	12	6.5	3.5
Zr						

	<u>MIXED ACID</u>			<u>COLD CHEM</u>		
	N	Mean mg/L	Std mg/L	N	Mean mg/L	Std mg/L
Al	13	18.4	18.8	12	61.8	32.9
B				12	-46.1	76.2
Ca	13	17.7	33.9	12	103.7	64.7
Cr	13	6.5	4.2	12	1.8	3.0
Cu	13	5.8	3.0	12	1.3	1.7
Fe	13	-1.8	12.2	12	29.0	34.5
K	13	121.4	42.8	12	42.7	43.9
Li	13	3.4	1.4	12	0.4	1.9
Mg	13	0.5	3.5	12	5.4	3.7
Mn	13	1.0	0.6	12	0.8	0.8
Na	13	21.0	19.3	12	43.1	17.8
Ni	13	0.5	10.9	12	-0.5	11.9
Si	13	-10.6	202.9	12	645.7	271.4
Ti	13	3.3	2.2	12	1.3	1.5
Zr	13	2.2	2.4	12	1.2	1.1

Li/Fe Ratios for Insert Cold Chem

Plot 1

Multiple Box-and-Whisker Plot

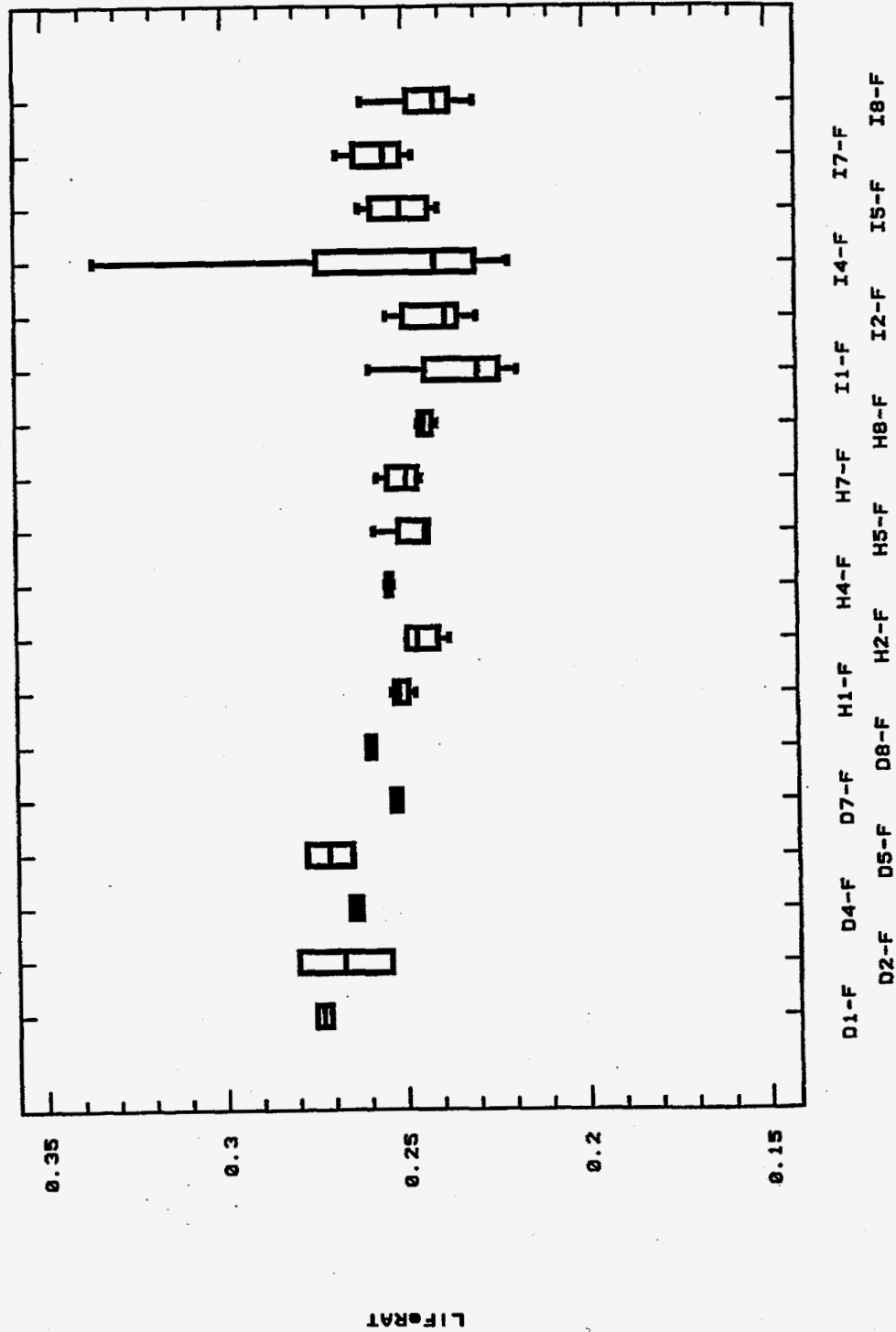


Blocks: 1 through 8, excluding Block 3 & 6
D1-M,...,D8-M: Peanut Vial Dip Samples by Mixed Acid
H1-M,...,H8-M: Peanut Vial Hydrgard™ Samples by Mixed Acid
I1-C,...,I8-C: Insert Samples by Cold Chemical

Plot 1

Li/Fe Ratios for Fusion Inserts

Multiple Box-and-Whisker Plot

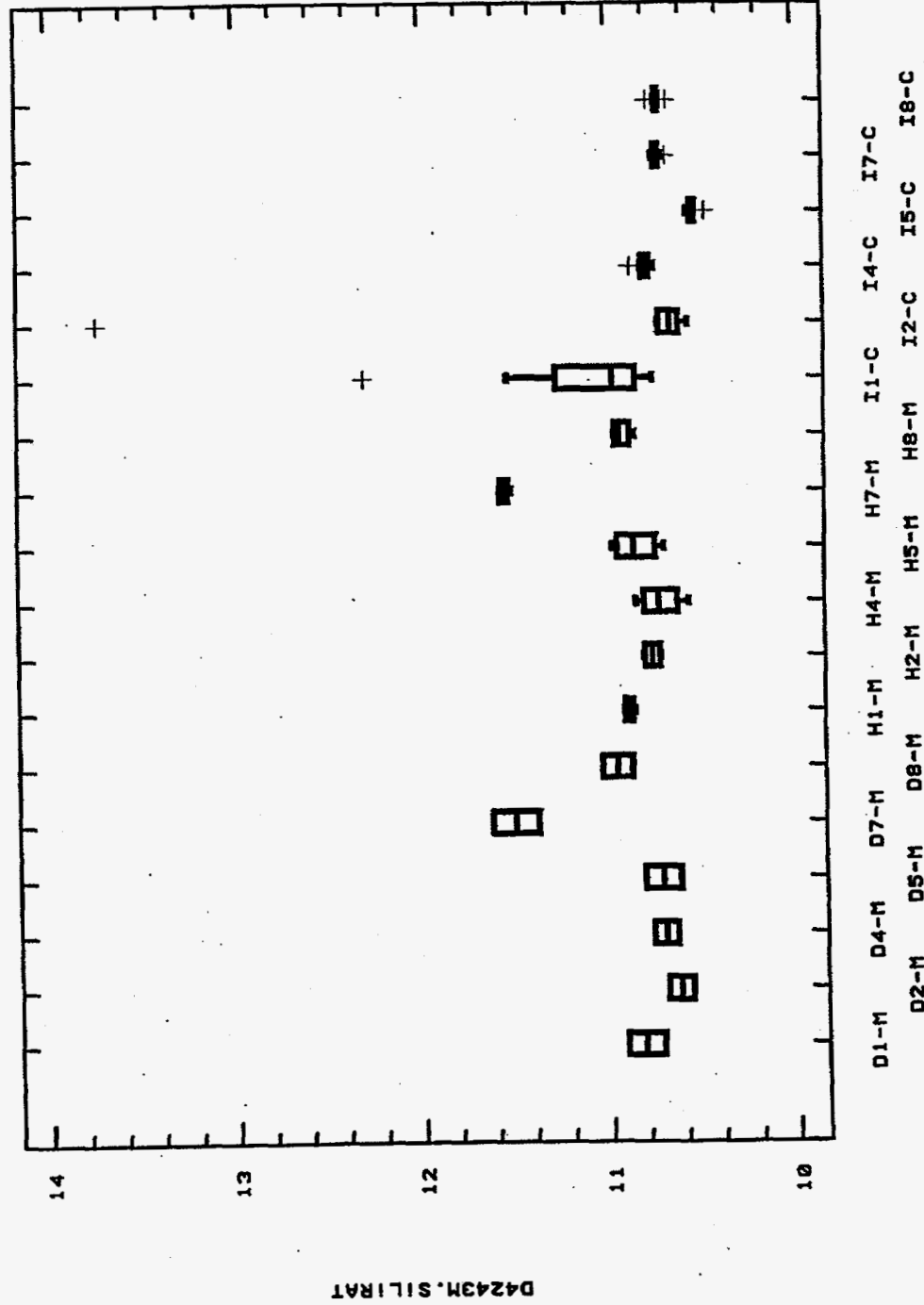


Blocks: 1 through 8, excluding Block 3 & 6
 D1-F, ..., D8-F: Peanut Vial Dip Samples by Fusion
 H1-F, ..., H8-F: Peanut Vial Hydragard™ Samples by Fusion
 I1-F, ..., I8-F: Insert Samples by Fusion

Plot 2

Si/Li Ratios for Insert Cold Chem

Multiple Box-and-Whisker Plot

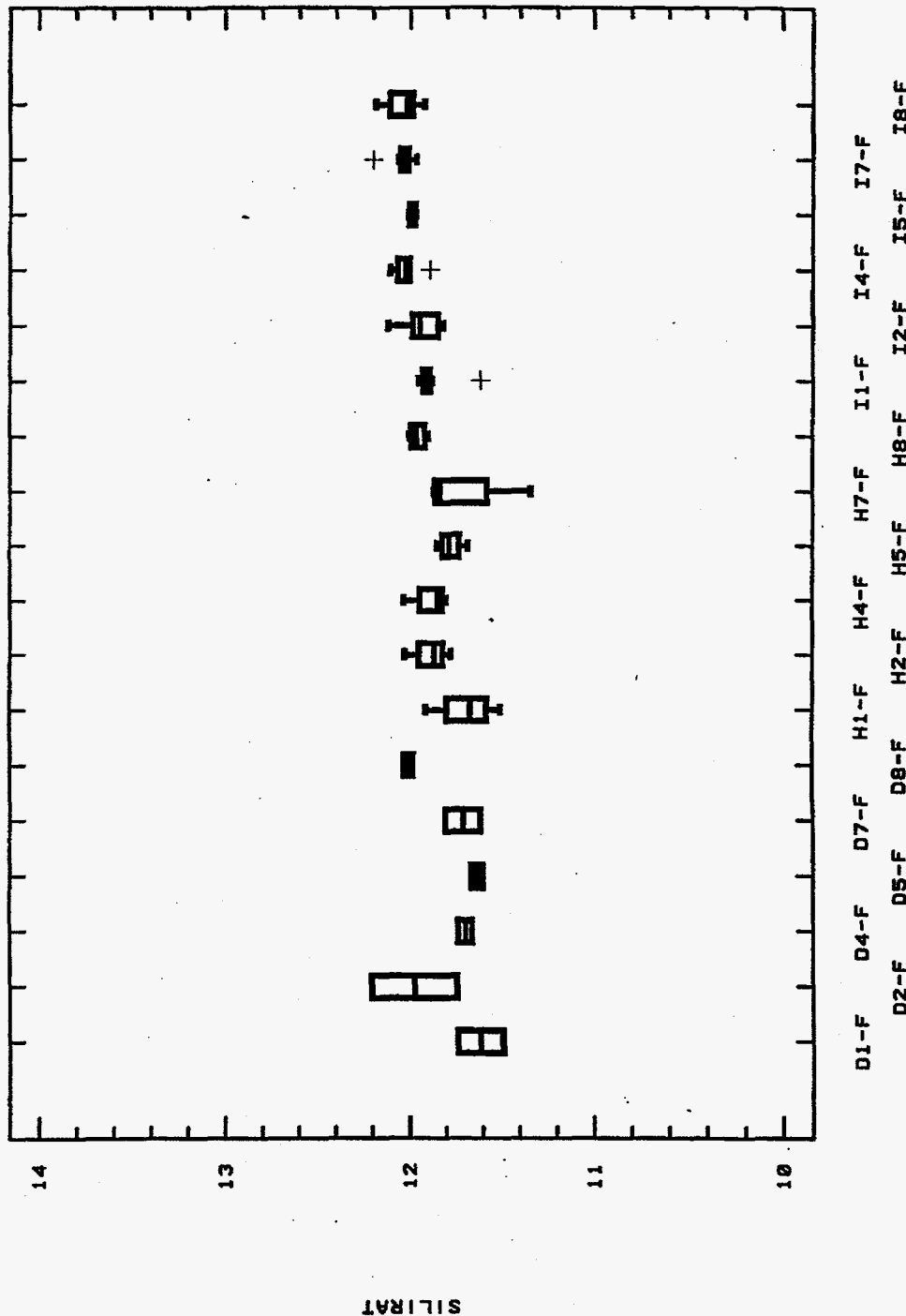


Blocks: 1 through 8, excluding Block 3 & 6
 D1-M, ..., D8-M: Peanut Vial Dip Samples by Mixed Acid
 H1-M, ..., H8-M: Peanut Vial Hydragard™ Samples by Mixed Acid
 I1-C, ..., I8-C: Insert Samples by Cold Chemical

Plot 2

Si/Li Ratios for Fusion Inserts

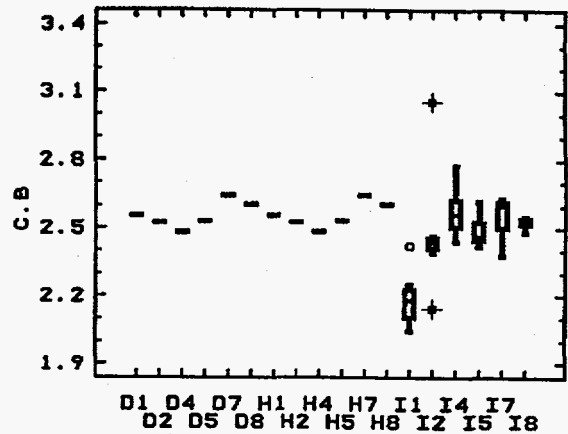
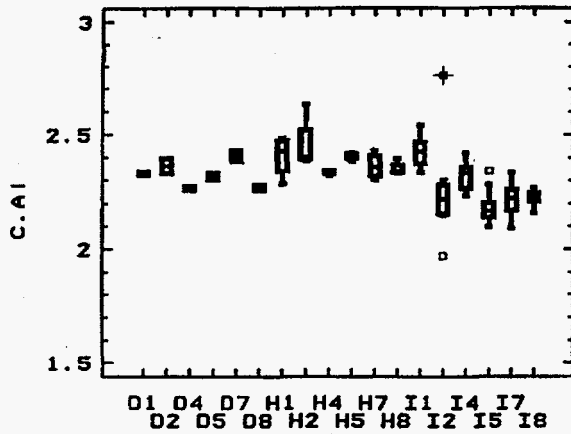
Multiple Box-and-Whisker Plot



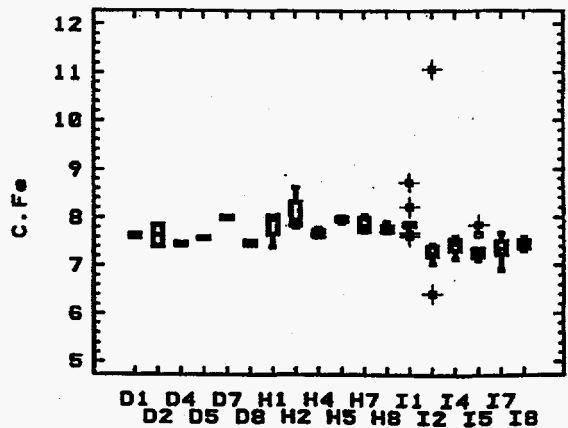
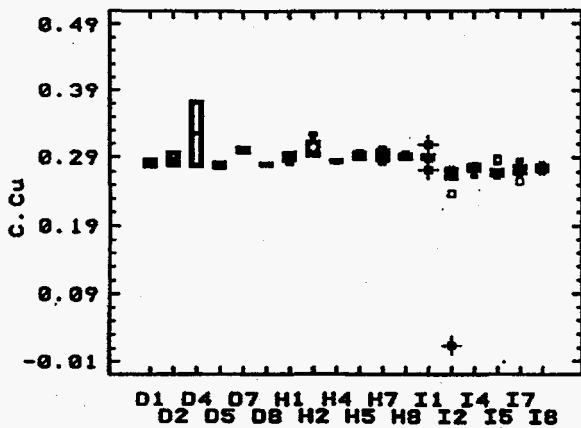
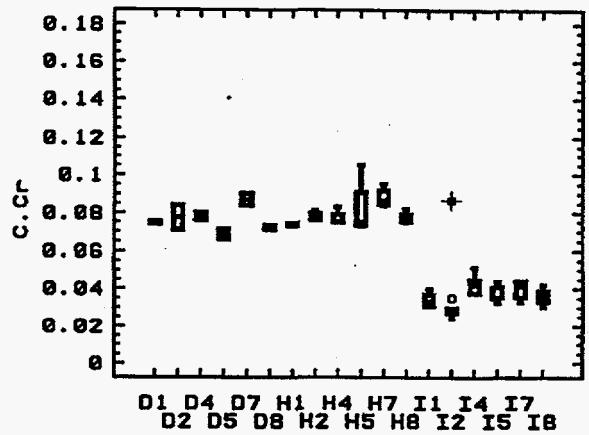
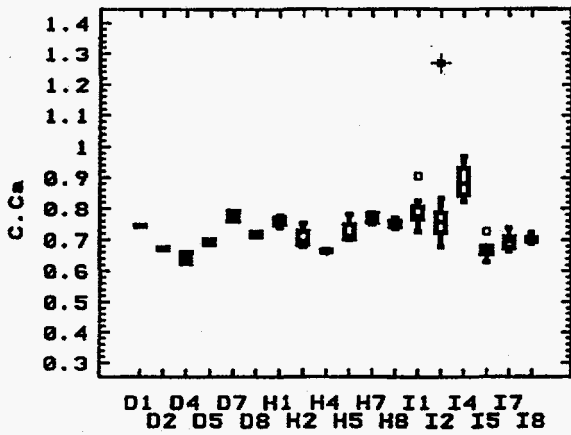
Blocks: 1 through 8, excluding Block 3 & 6
 D1-F, ..., D8-F: Peanut Vial Dip Samples by Fusion
 H1-F, ..., H8-F: Peanut Vial Hydragard™ Samples by Fusion
 I1-F, ..., I8-F: Insert Samples by Fusion

Plot 3

**Box-and Whisker Plots: Insert Cold Chemical(Calcine Corrected) Elemental wt% data
 Dip & Hydragard Peanut Vials by Mixed Acid**

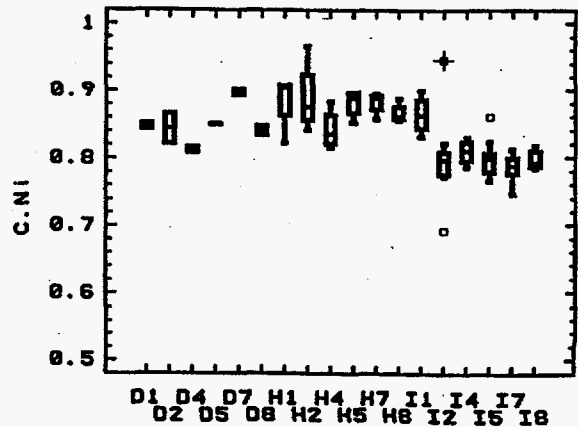
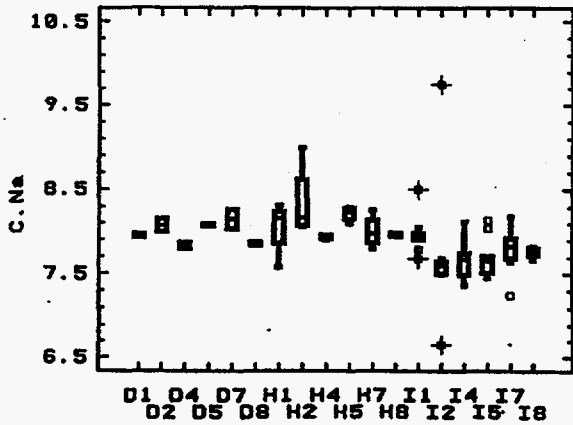
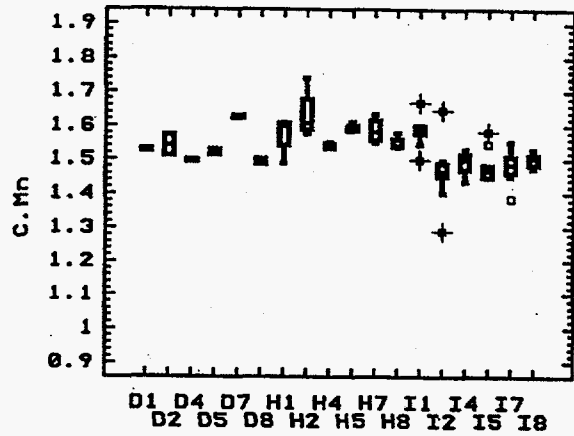
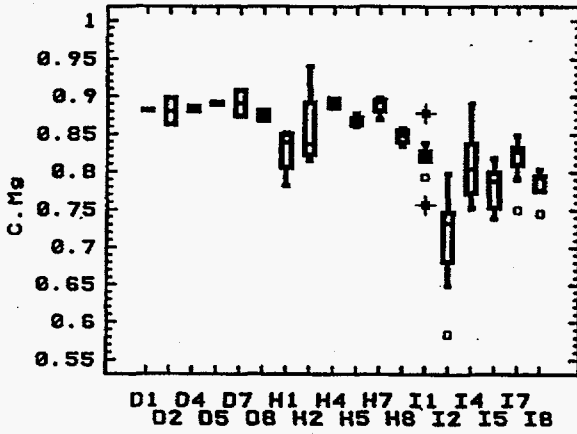
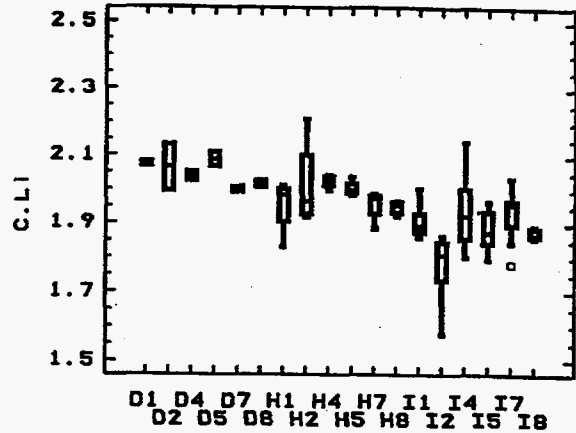
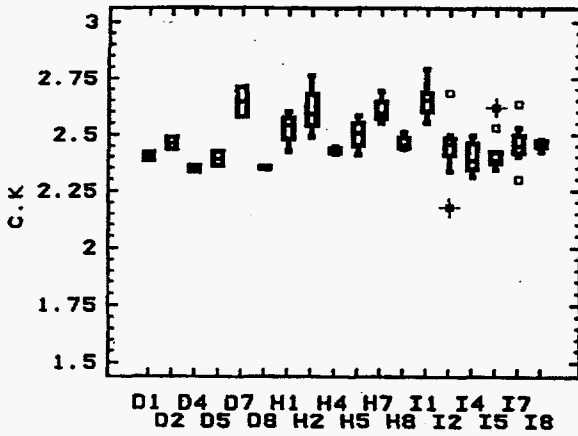


D & H from Peanut Vial Fusion



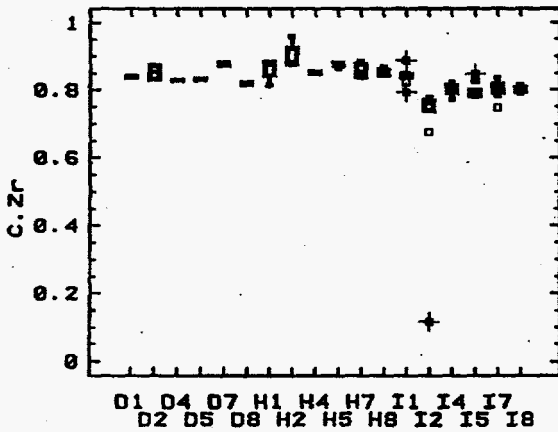
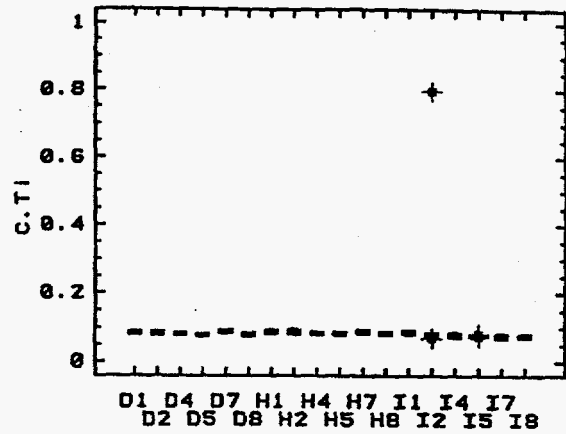
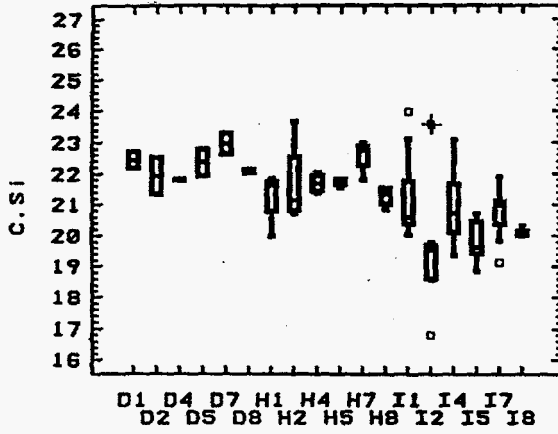
Plot 3

Box-and Whisker Plots: Insert Cold Chemical (Calcine Corrected) Elemental wt% data
Dip & Hydragard Peanut Vials by Mixed Acid



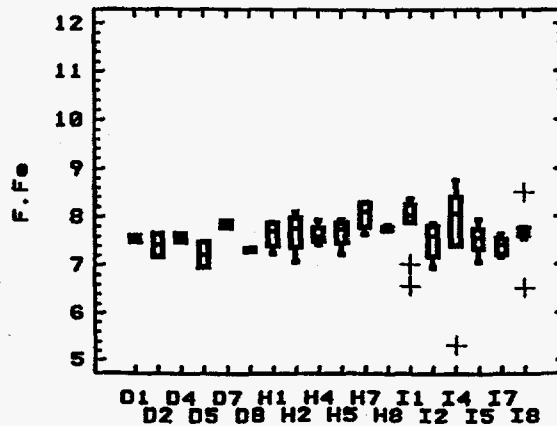
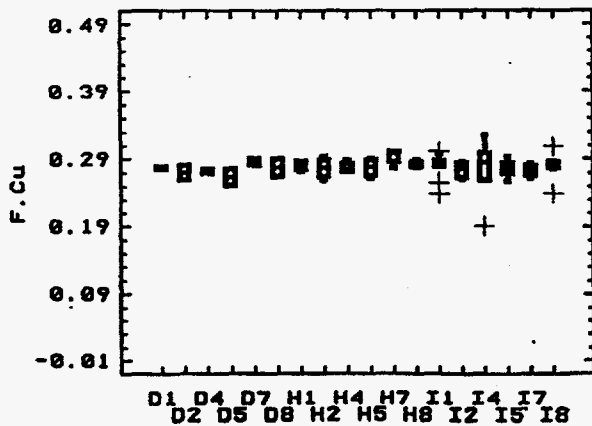
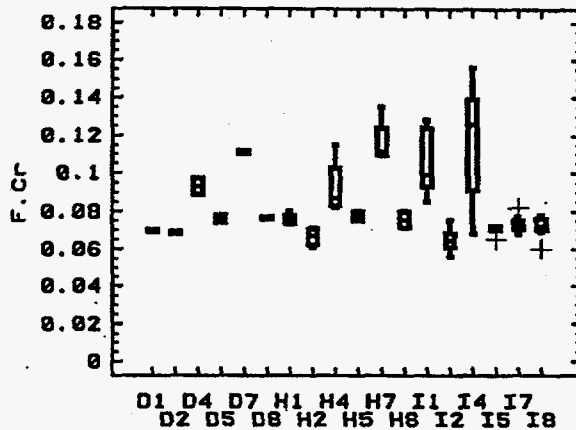
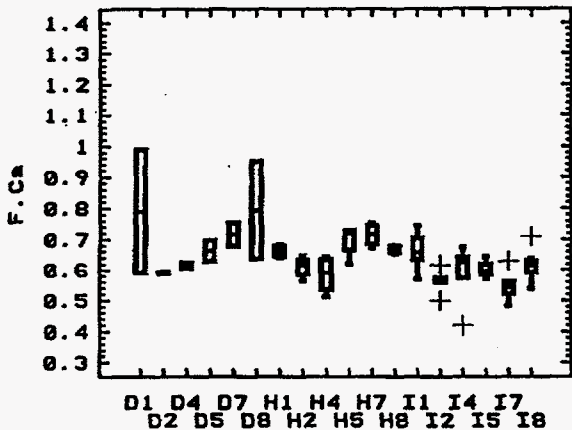
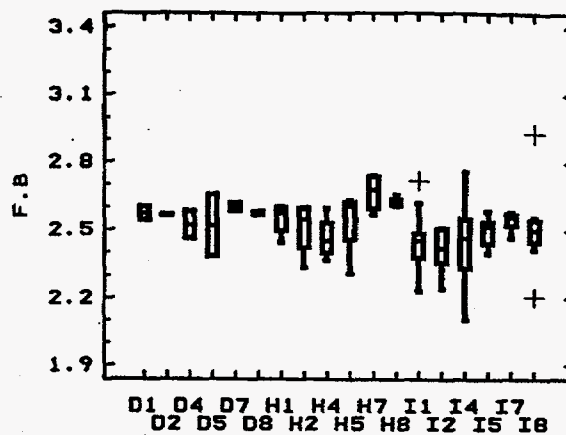
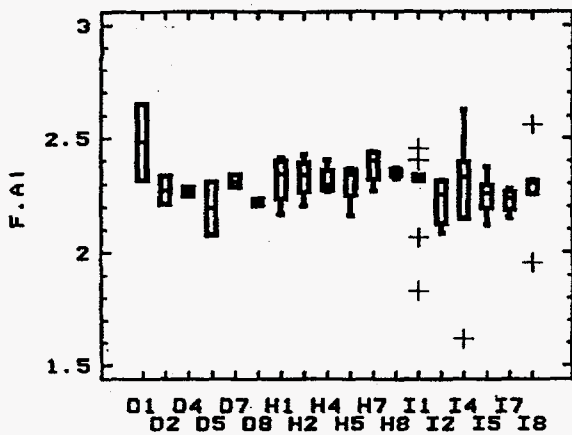
Plot 3

**Box-and Whisker Plots: Insert Cold Chemical(Calcine Corrected) Elemental wt% data
 Dip & Hydragard Peanut Vials by Mixed Acid**



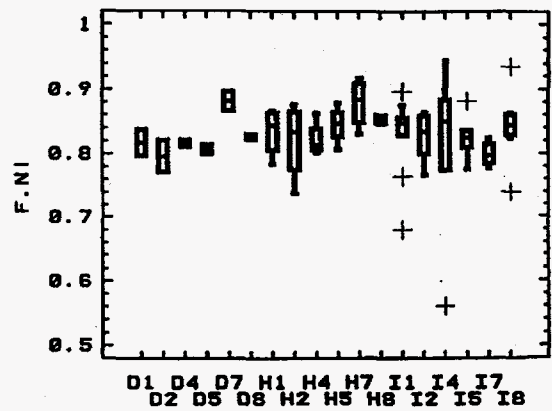
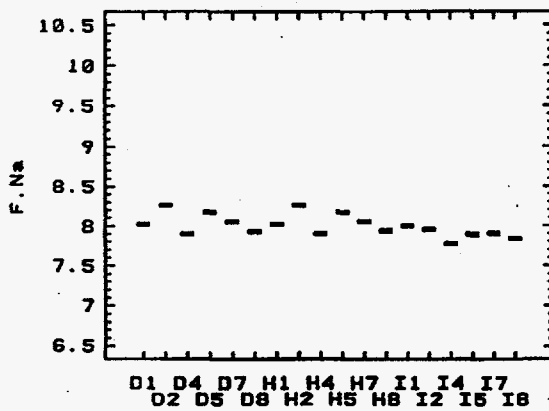
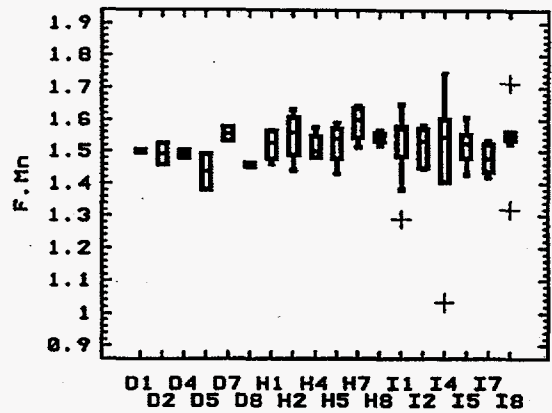
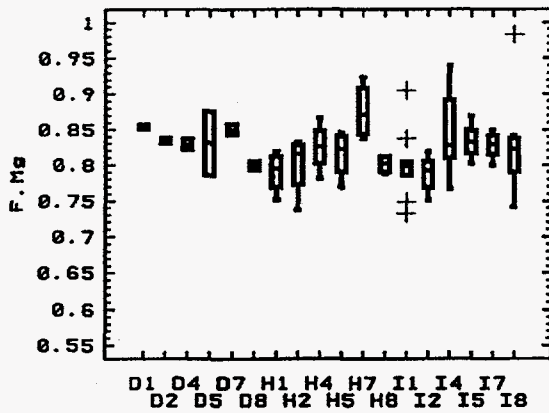
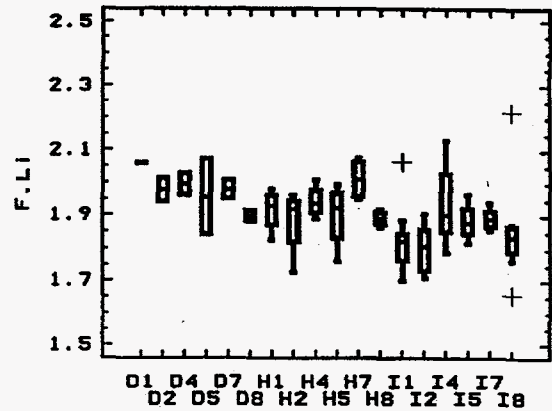
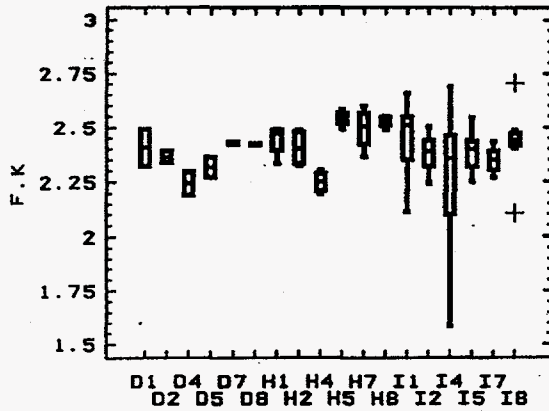
Plot 4

Box-and Whisker Plots: Insert Fusion(Calcined) Elemental wt% data
 Dip & Hydragard Peanut Vials by Fusion



Plot 4

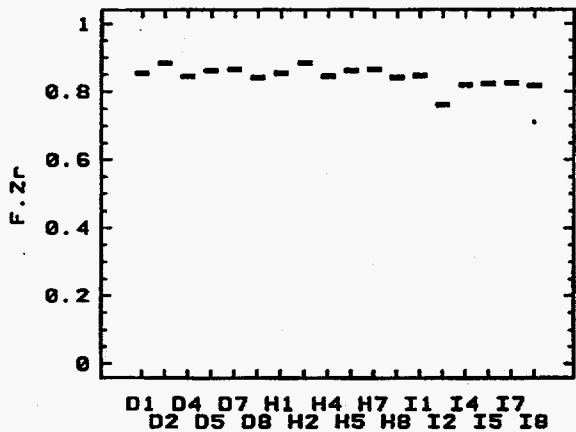
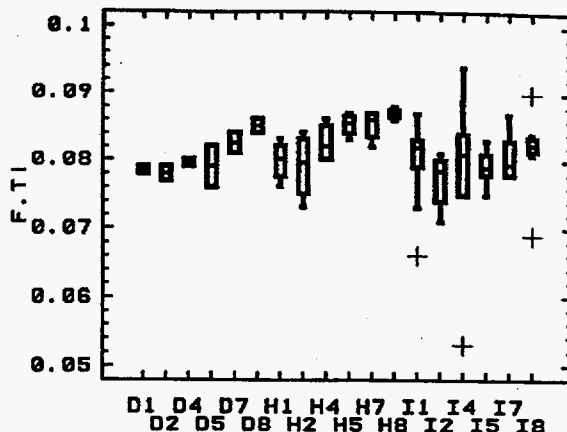
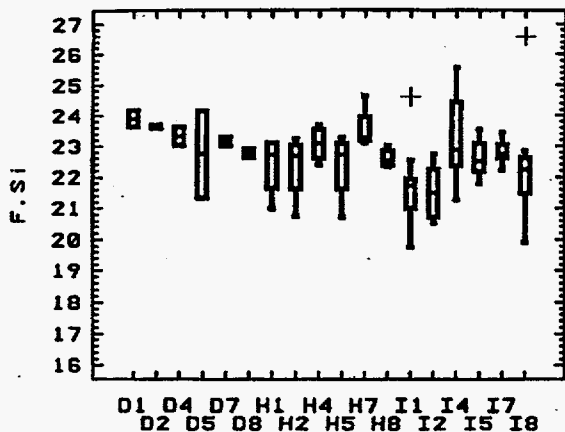
Box-and Whisker Plots: Insert Fusion(Calcined) Elemental wt% data
 Dip & Hydragard Peanut Vials by Fusion



D & H from Mixed Acid, I from Cold Chem

Plot 4

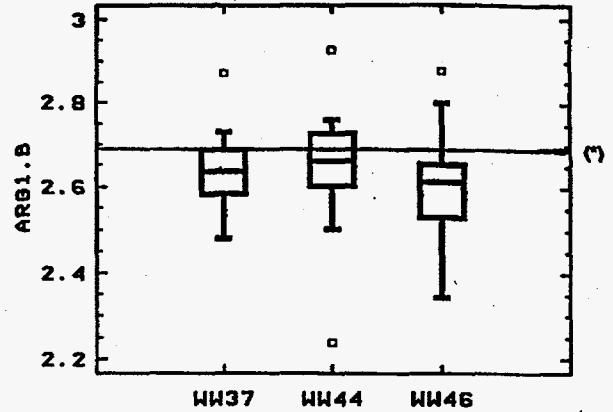
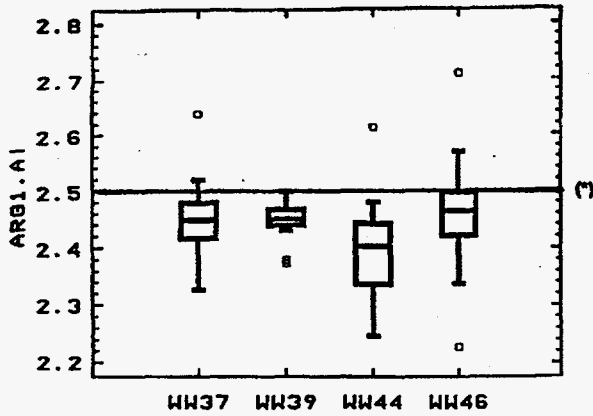
Box-and Whisker Plots: Insert Fusion(Calcined) Elemental wt% data
Dip & Hydragard Peanut Vials by Fusion



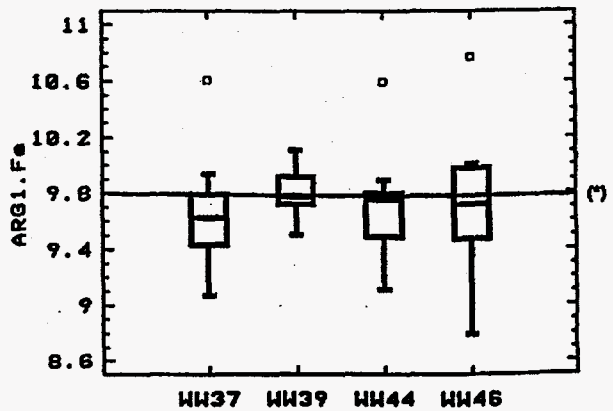
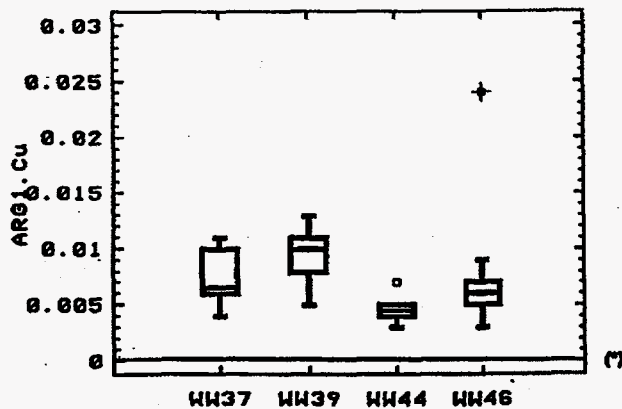
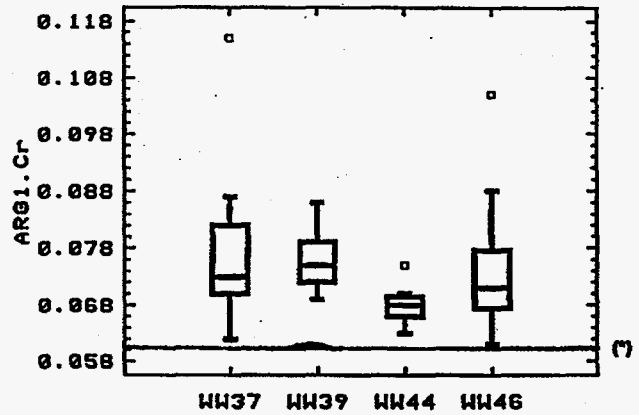
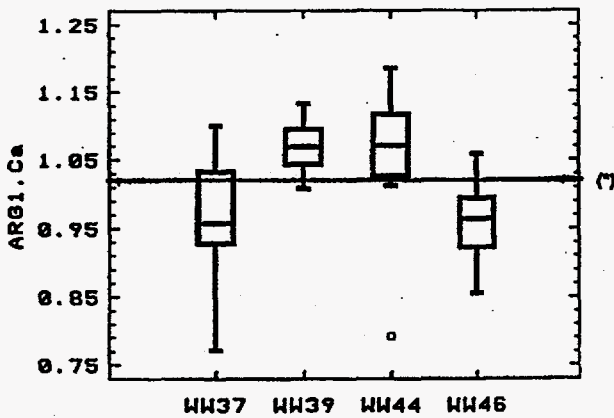
D & H from Mixed Acid, I from Cold Chem

Plot 5

Box-and Whisker Plots: ARG-1 Elemental wt% data & Corning wt%⁽¹⁾

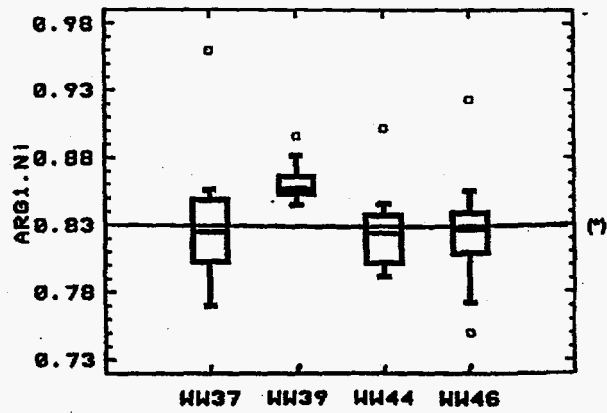
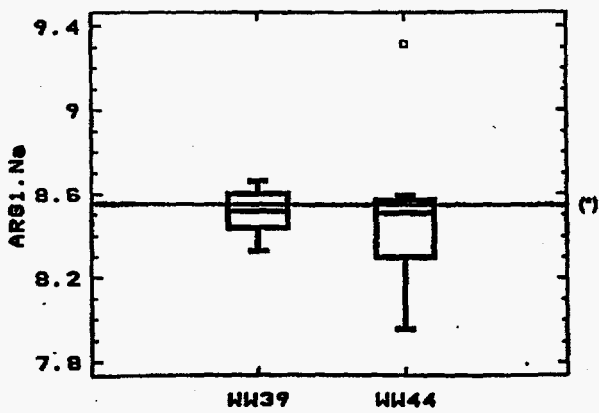
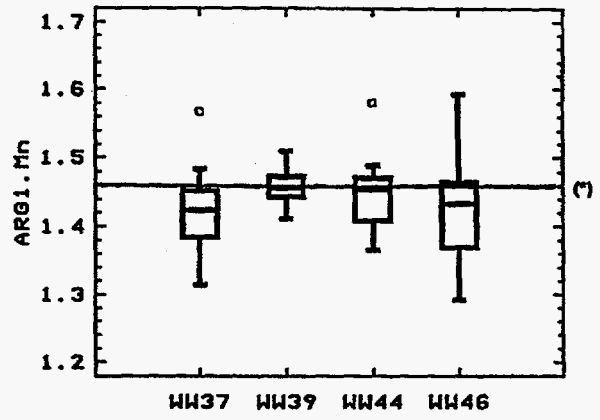
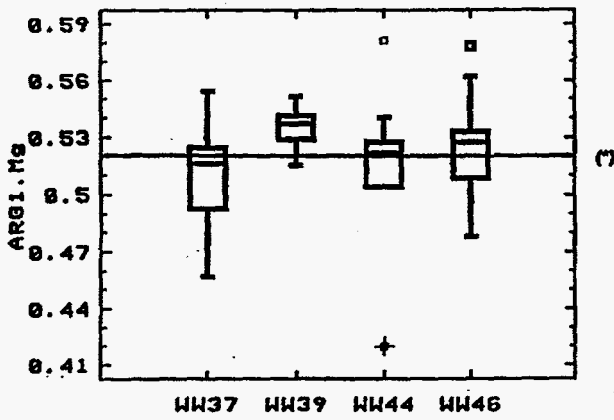
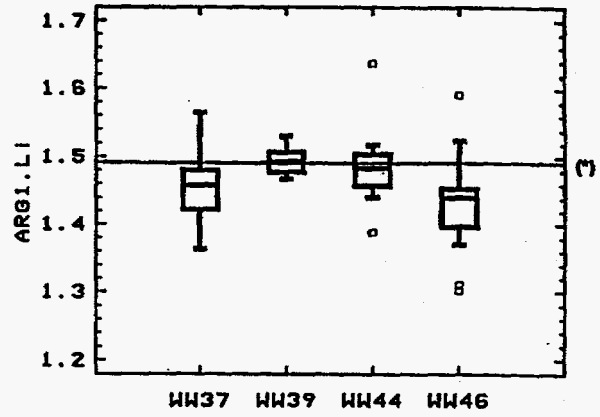
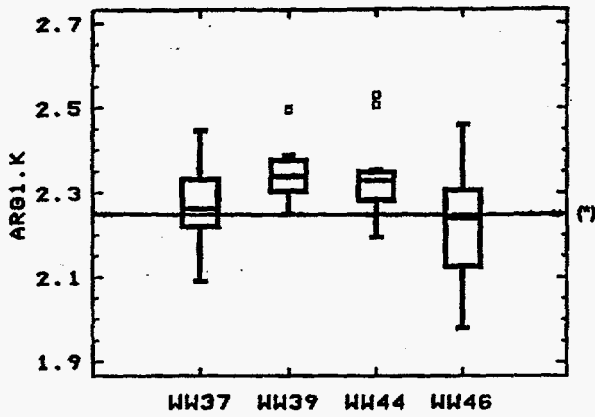


WW37: ARG-1, by Fusion, run with Peanut Vial Samples
 WW39: ARG-1, by Mixed Acid, run with Peanut Vial Samples
 WW44: ARG-1, by Cold Chem, run with Insert Cold Chem Samples
 WW46: ARG-1, by Fusion, run with Fusion Inserts



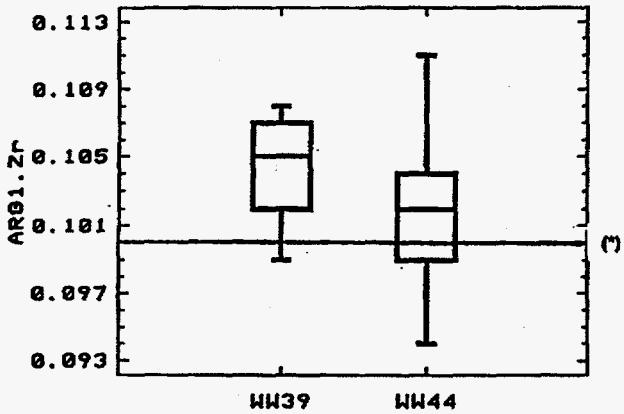
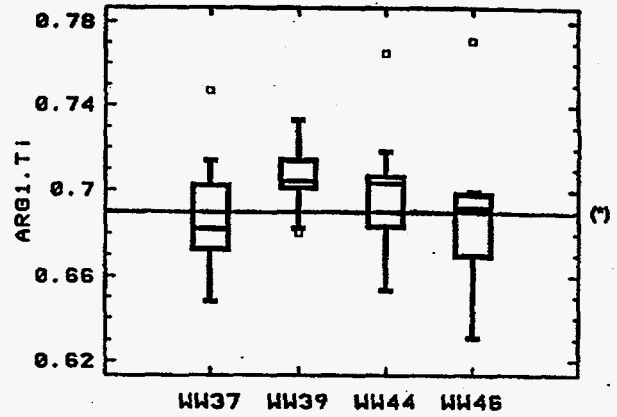
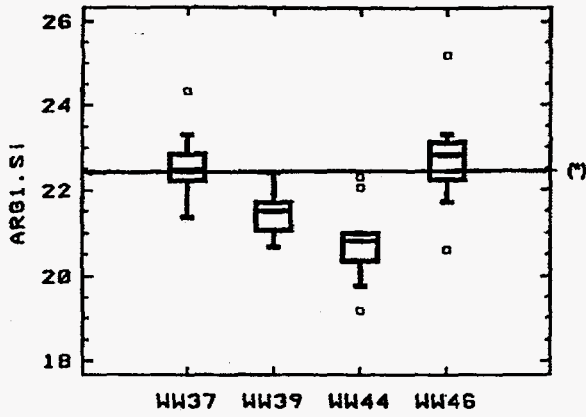
Plot 5

Box-and Whisker Plots: ARG-1 Elemental wt% data & Corning wt%⁽⁷⁾



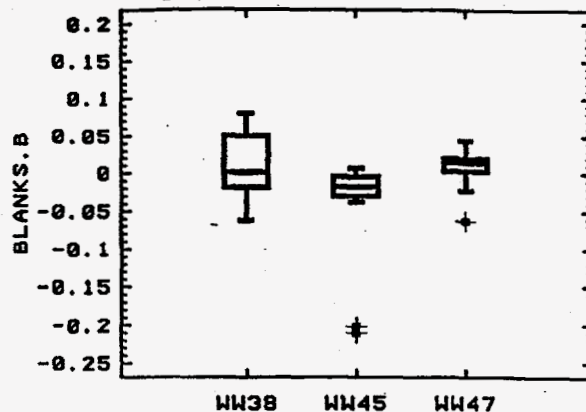
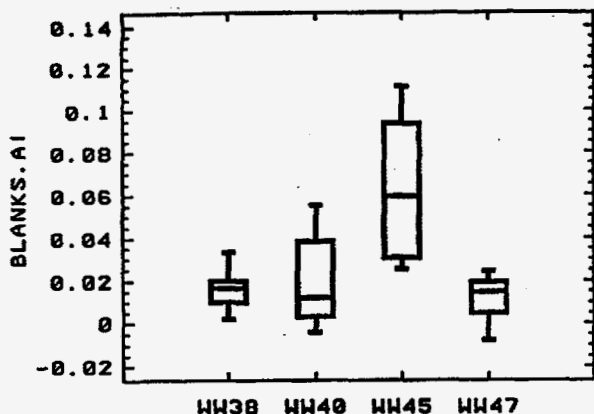
Plot 5

Box-and Whisker Plots: ARG-1 Elemental wt% data & Corning wt%^(*)

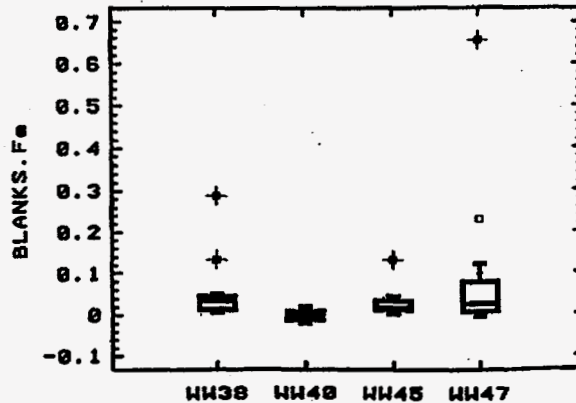
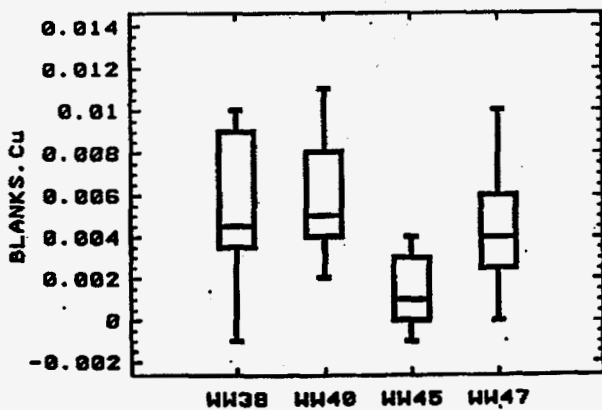
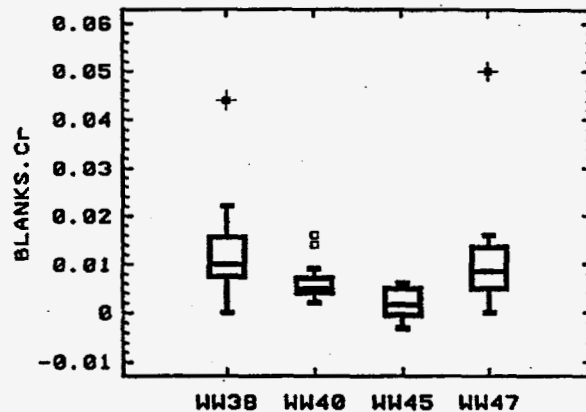
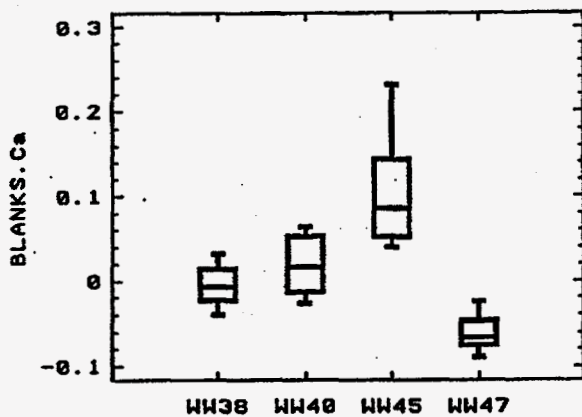


Plot 6

Box-and Whisker Plots: Blank Elemental data(mg/L)

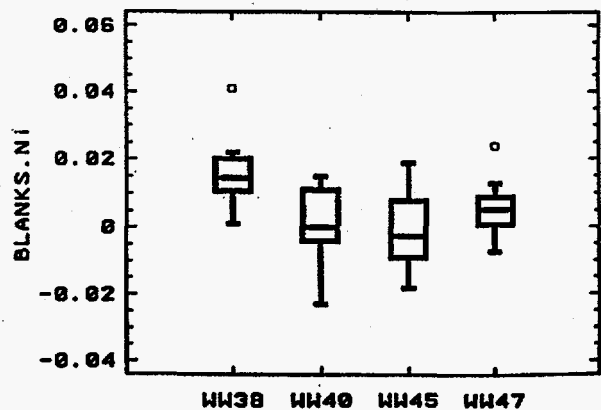
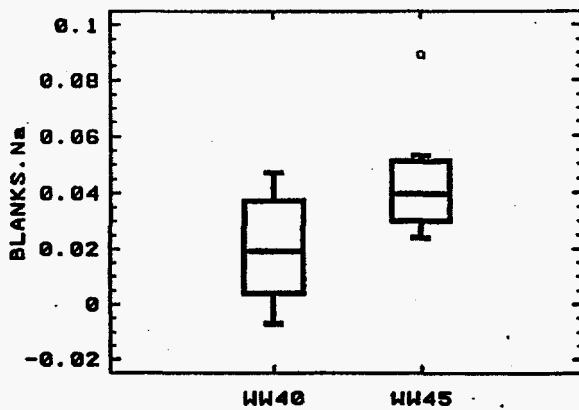
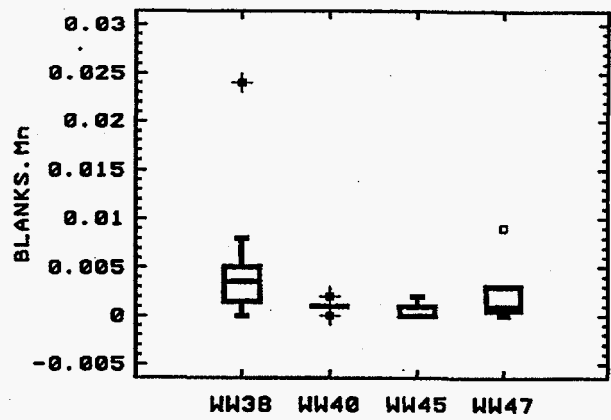
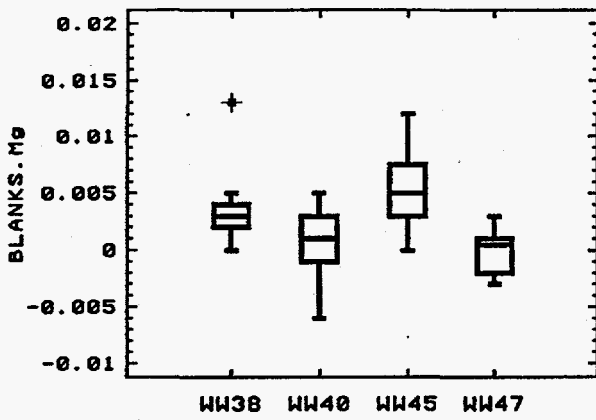
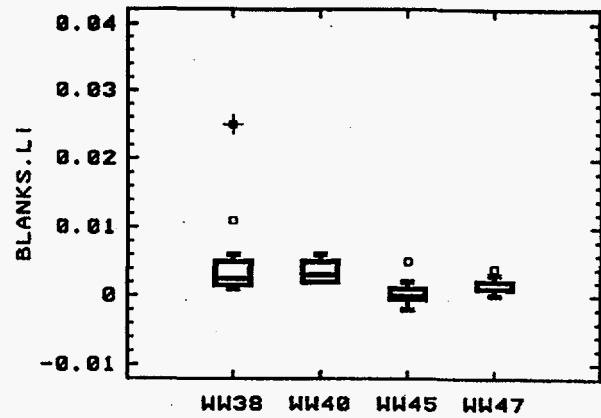
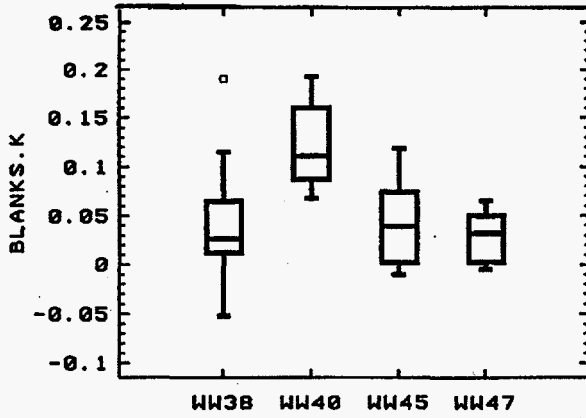


WW38: Blanks, by Fusion, run with Peanut Vial Samples
WW40: Blanks, by Mixed Acid, run with Peanut Vial Samples
WW45: Blanks, by Cold Chem, run with Insert Cold Chem Samples
WW47: Blanks, by Fusion, run with Fusion Inserts



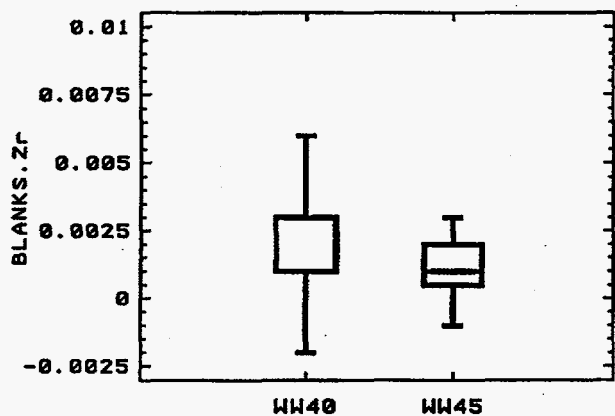
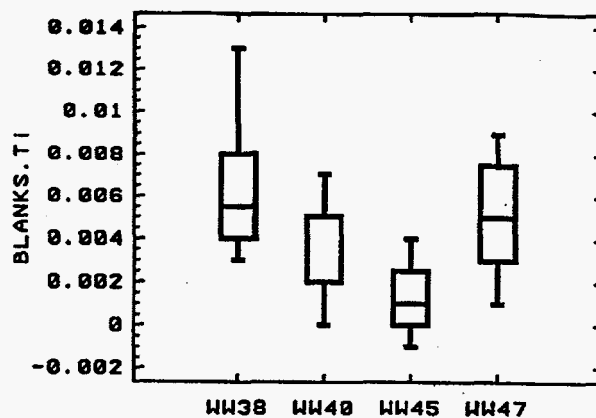
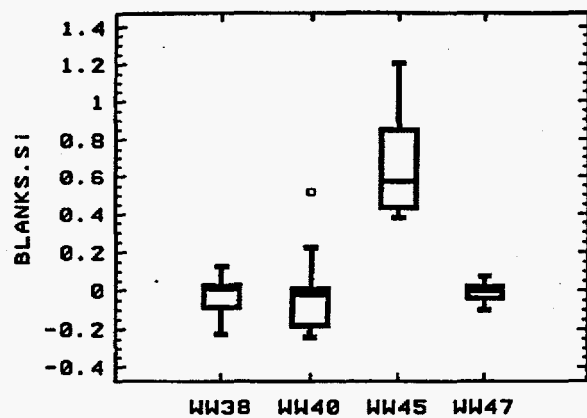
Plot 6

Box-and Whisker Plots: Blank Elemental data(mg/L)



Plot 6

Box-and Whisker Plots: Blank Elemental data(mg/L)

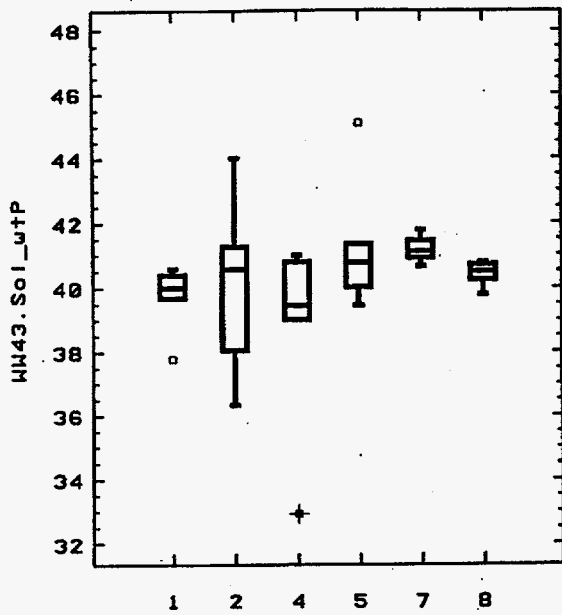


Plot 7

Box-and Whisker Plots: wt% Total Solids, wt% Vitrified and wt% Calcined data

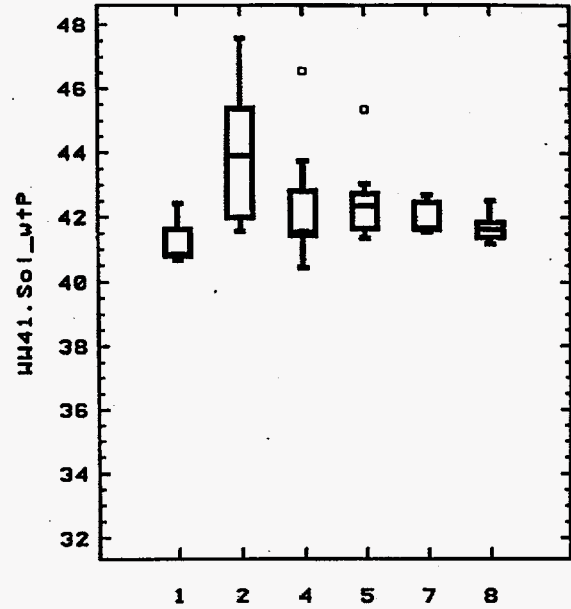
WW41: Fusion Inserts

WW43: Peanut Vials- Dip & Hydragard



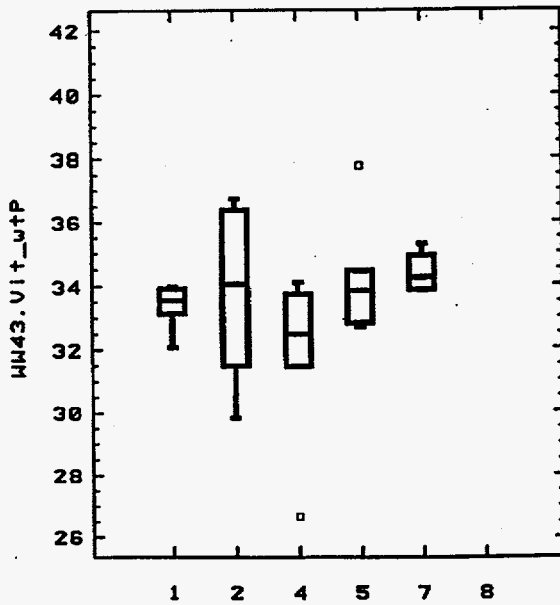
Block

WW43: Peanut Vials- Dip & Hydragard



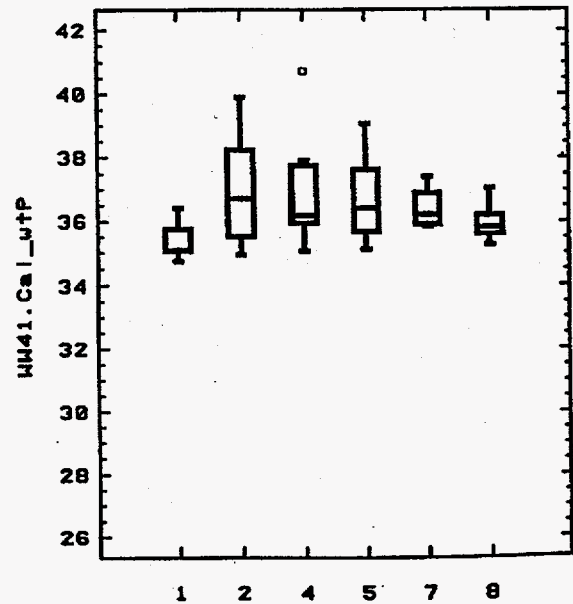
Block

WW41: Fusion Inserts



Block

WW43: Peanut Vials- Dip & Hydragard



Block

WW41: Fusion Inserts