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Numbers of Samples, Analyses, and Measurements for Vitrification Estimation, Compliance, and Process Control – Methodology and Preliminary Assessment

G. F. Piepel
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April 2001

Prepared for Bechtel National, Inc.
under Project 42365

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Battelle, Pacific Northwest Division
Richland, Washington 99352

Summary

Various samples, chemical analyses of samples, and measurements will be required to monitor and control the Immobilized High Level Waste (IHLW) and Immobilized Low Activity Waste (ILAW) vitrification processes and to implement qualification and production strategies for complying with applicable specifications. Many of the process-product control and compliance strategies will be statistical in nature, meaning that variations and uncertainties will be accounted for in making decisions and demonstrating compliance.

In particular, statistical methods and formulas will be developed to determine the numbers of samples, analyses per sample, and measurements required to meet various process-product control or compliance objectives. Such objectives include estimating quantities (for example, compositions or volumes of vessels) with specified levels of precision and confidence, or detecting differences (from specification limits or between two quantities) of specified magnitudes with specified probabilities of decision errors. The term *sample size* is used to refer in general to the number of samples, analyses, or measurements, regardless of the objective.

This report presents the results of initial work to develop sample size formulas and apply the formulas to calculate sample sizes. Sample size formulas depend on several input parameters, with specific input parameters depending on the objective. The input parameters may include magnitudes of uncertainties, required levels of precision, required levels of confidence, required magnitudes of differences to be detected, and required probabilities of decision errors. The River Protection Project - Waste Treatment Plant (RPP-WTP) Project has not yet performed work to:

- **estimate uncertainties**
- **determine required levels of precision**
- **determine required levels of confidence**
- **determine magnitudes of differences that must be detected**
- **determine required limits on probabilities of decision errors.**

Hence, it was not possible in this work to calculate final sample size requirements. Rather, sample size calculations were performed for combinations of input parameters over ranges expected to cover the values that ultimately may be obtained. The results of these calculations are arranged and presented in several tables. These sample size tables are useful tools for:

- **Planning for possible sampling, analysis, and measurement requirements and costs**
- **Helping assess the advantages and disadvantages of reducing uncertainties, changing required precision levels or detectable differences, and changing required confidence levels or probabilities of decision errors.**

These two bulleted items address one of the most useful (but under-utilized) roles of statistical sample size work. Being aware of the costs and benefits of various options provides for informed choices and investing effort where the benefit is the greatest. For example, minimizing variability in the analytical methods can reduce the sampling and analyses effort. Further reductions in the sampling and analyses effort are obtainable if it is acceptable to make conclusions with lower confidence.

Many specific process-product control or compliance objectives are identified in the report, but in this initial phase of the work, it was possible to develop and apply sample size formulas for only a fraction of the objectives. Sample size work must continue in the future to address other objectives. This report will be updated in the future to include the results of subsequent work.

Acronyms, Terms, and Abbreviations

BNI	**Bechtel National, Inc.**
CHG	**CH2MHill Hanford Group, Inc.**
CRV	**Concentrate Receipt Vessel (in the IHLW or ILAW vitrification facility)**
DOE	**Department of Energy**
DQO	**Data Quality Objectives**
DQA	**Data Quality Assessment**
$D_{\alpha/2,1-\beta}$	**Percent difference between the true value of a quantity and lower and upper limits that must be detected with probability 1-β, but with only probability α of incorrectly deciding there is a difference**
$D_{\alpha,1-\beta}$	**Percent difference between the true value of a quantity and a lower limit or an upper limit that must be detected with probability 1-β, but with only probability α of incorrectly deciding there is a difference**
$\Delta_{\alpha/2,1-\beta}$	**Percent difference in two quantities that must be detected with probability 1-β, but with only probability α of incorrectly deciding there is a difference**
FCP	**Feed concentrate from pretreatment**
GFC	**Glass former chemicals**
$H_{1-\alpha}$	**Magnitude of difference (in percent) between the true value and an estimate that can be determined with 100(1 - α) percent confidence**
HLW	**High Level Waste**
ICP	**Inductively Coupled Plasma**
IHLW	**Immobilized High Level Waste**
ILAW	**Immobilized Low Activity Waste**
LAW	**Low Activity Waste**

MFPV	**Melter Feed Preparation Vessel (in the IHLW or ILAW vitrification facility)**
MFV	**Melter Feed Vessel (in the IHLW or ILAW vitrification facility)**
n_S	**Number of samples**
n_A	**Number of analyses per sample**
PCT	**Product Consistency Test**
PSWP	**Products and Secondary Wastes Plan**
$\%RSD_S$	**Percent relative standard deviation for samples**
$\%RSD_A$	**Percent relative standard deviation for analyses**
RPP-WTP	**River Protection Project – Waste Treatment Plant**
Sample size	**Generic term used to refer to the number of samples, number of chemical analyses per sample, or number of measurements**
σ_S	**Standard deviation for samples**
σ_A	**Standard deviation for analyses**
VHT	**Vapor Hydration Test**
WAPS	**Waste Acceptance Product Specifications**
WCP	**Waste Compliance Plan**

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Quality Assurance

The work and results in this report were performed according to the project QA plan (PNNL 2000), with the exception that the procedures for calculations and software control were not applied. Hence, the results are not compliant with QARD (DOE 1998). The rigorous QA procedures necessary for QARD compliance were not followed due to the preliminary nature of the work and results. QARD-compliant procedures will be followed for final versions of methods, computations, and results to be documented in a future revision of this report.

1.0 Introduction

Various process samples, chemical analyses, and measurements will be required to control River Protection Project – Waste Treatment Plant (RPP-WTP) vitrification facilities that will produce Immobilized High Level Waste (IHLW) and Immobilized Low Activity Waste (ILAW). In addition, process and/or product samples, chemical analyses, and measurements will be required to satisfy applicable compliance requirements. For example, the Waste Acceptance Product Specifications (WAPS) describe various compliance requirements for IHLW (USDOE 1996). Also, the contract between the Department of Energy (DOE) and Bechtel National, Inc. (BNI 2001) specifies compliance requirements for ILAW, as well as additional compliance requirements for IHLW.

Although the process-product control and compliance strategies are still under development, many aspects have been determined. The current status of the RPP-WTP Project's compliance strategies are given in the Waste Compliance Plan (WCP, CHG 2001a) and the Products and Secondary Wastes Plan (PSWP, CHG 2001b). Many of the compliance strategies outlined in the WCP and PSWP are statistical in nature. That is, the strategies will involve quantifying and accounting for variations and uncertainties in controlling the IHLW and ILAW vitrification processes and in satisfying compliance requirements. Statistically based strategies will be developed for pre-production (that is, qualification) activities, as well as for production and acceptance activities. Strategies for environmental regulatory compliance (e.g., plant emissions, complying with Land Disposal Restriction and de-listing criteria) will also be statistically based. However these specific regulatory needs are not addressed in this document as those aspects of the compliance strategy are being planned and carried out separately.

An important qualification activity is to determine the numbers of samples, chemical analyses per sample, and measurements necessary to

- **meet objectives of other qualification activities**
- **control the IHLW and ILAW vitrification processes**
- **satisfy compliance requirements**

with desired levels of precision, confidence, detectability, and decision errors. This report presents the results of initial work to determine appropriate statistical methods and corresponding sample size formulas to calculate the numbers of samples, analyses per sample, and measurements required to achieve specified performance. The term *sample size* is used in a general manner to refer to many situations involving numbers of samples, chemical analyses per sample, and measurements per sample or situation.

Statistical sample size formulas depend on many aspects and input parameters that influence the required number of samples, analyses per sample, or measurements. These aspects and input parameters include:

1) **<u>The objective of the sample, chemical analysis, or measurement.</u> Many specific objectives falling into two broad categories are described and discussed in Section 2.2. The two broad categories are "estimating a quantity" and "detecting whether a quantity meets a limiting value". An example objective from the first broad category is estimating the composition of slurry in an IHLW or ILAW vitrification process vessel, say the Concentrate Receipt Vessel. An example objective from the second broad category is detecting whether the IHLW or**

ILAW produced satisfies the specified limits for Product Consistency Test (PCT) releases. Specific objectives listed in Section 2.2 are addressed in more detail in Sections 3 to 6 of the report.

2) **<u>How precisely a quantity is to be estimated or how large of a difference from a limiting value is to be detected.</u> The precision, as discussed here, quantifies how much uncertainty can be tolerated in the estimate. The precision of an estimate can be improved (i.e., the uncertainty reduced) by taking multiple samples or measurements and averaging the results. Hence, the number of samples, chemical analyses, or measurements required depends in part on the required precision. Precision is often expressed by a standard deviation or relative standard deviation. However, in the sample size context, precision is expressed in terms of the desired distance from the estimate (assumed to be unbiased) to the true value. Thus, a larger sample size will be required if the objective is to estimate, with specified confidence, a quantity (such as the composition of glass samples from a canister) within 5% versus within 10%. Similarly, the detectable difference, with specified decision error probabilities, between an estimate of a quantity (say a PCT release) and a limiting value can be reduced by taking multiple samples, chemical analyses, or measurements. Thus, a larger sample size will be required to detect (with specified probabilities of decision errors) whether a quantity (such as a PCT release) is within 10% of a specified limit instead of within 20%.**

3) **<u>Desired confidence or probabilities of decision errors for the results.</u> Whereas "precision" and "detectable difference" described in Item 2 express desired closeness, "confidence" and "probabilities of decision errors" express desired confidence in the results from the sampling, analysis, or measurement. As the required level of confidence that an estimated quantity (e.g., composition in the MFPV) is within a specified precision increases (such as going from 90% to 95%), the sample size will increase. Similarly as the required probabilities of decision errors decrease, the sample size will increase. For example, if the desired probability of not detecting that the MFPV composition is unacceptable by a specified difference decreases from 5% to 1%, the sample size will increase.**

4) **<u>Magnitudes of uncertainties (for example, in composition or weight).</u> The larger the uncertainty in a sampling system, chemical analysis method, or measurement instrument, the larger the sample size needed to achieve the desired objective. The magnitude of an uncertainty will be expressed in this document as a percent relative standard deviation (%RSD), i.e., the standard deviation of a quantity divided by its average and multiplied by 100. This makes the results more general prior to determining actual means and standard deviations during qualification activities.**

As a consolidated example of the above, consider calculating the required numbers of samples and chemical analyses for the objective of estimating the chemical composition within a CRV (Step 1 above). Suppose the goal is to be 95% confident (Step 3 above) that the estimated composition is within 10% of the true composition (Step 2 above), given prior estimates of sampling and analytical uncertainty of $\%RSD_S = 10\%$ and $\%RSD_A = 5\%$, respectively (Step 4 above). These inputs are the required information to calculate the required numbers of samples and analyses.

The magnitudes of uncertainties, desired confidence levels, and desired probabilities of decision errors applicable to different vitrification process and product situations will be quantified in future qualification activities. However, based on previous experience, it was possible for the work summarized in this report to specify expected ranges for magnitudes of uncertainties. Further, it is likely that confidence levels in the range from 90% to 99% and probabilities of incorrect decisions smaller than 10% will be preferred by the RPP-WTP Project. Hence, this

report also presents the results of calculations using statistical sample size formulas developed for several combinations of reasonable uncertainty magnitudes and confidence levels or probabilities of decision errors.

The sample size formulas and the results (of applying the formulas for various combinations of uncertainties and confidence levels or probabilities of decision errors) presented in Sections 3, 4, and 5 of this report are general in the sense that they can be applied to various situations. For example, the same sample size formulas and the range of results (corresponding to the range of input parameters considered in this report) apply to IHLW and ILAW. The sample size formulas and results may also apply in pretreatment, regulatory compliance, or other situations. For example, if the objective of a sample size calculation is to determine the numbers of samples and analyses per sample required to estimate with 95% confidence the composition of a vessel within 10% of the true value, it does not matter what kind of vessel it is (for example, IHLW process, ILAW process, or pretreatment process) as long as the uncertainties and their magnitudes are included in the ranges considered in this report.

In this document it is assumed that the sampling, analytical, and measurement systems are unbiased and that the corresponding uncertainties (i.e., %RSD values) encompass all the applicable random uncertainties. Quality control (QC) issues, such as whether or not certified standards and reference materials are used to detect and correct for biases, could potentially affect the sample size calculations. The sample size formulas and results in this report can be updated in the future if needed after the RPP-WTP Project's strategy regarding these QC issues is determined.

This report does not address setting the precision and confidence levels for estimation objectives or the detectable difference and decision error probabilities for detection objectives. Those inputs to this document will take place in Data Quality Objectives (DQO) activities, or through another systematic planning process, conducted as part of other work scope within the RPP-WTP Project. This report specifically addresses determining sample sizes before the sampling, analyses, or measurements are carried out. It does not address how to do the Data Quality Assessment (DQA) once the data have been collected to verify that the numbers of samples, analyses, or measurements were, in fact, sufficient, nor how statistical data analyses are to be performed using the collected data. Work in those areas still needs to be performed as part of other qualification activities.

Section 2.1 provides a general overview of the IHLW and ILAW vitrification processes, and Section 2.2 describes four general categories of objectives for sampling, chemical analyses, or measurements. Sections 3, 4, and 5 present the statistical sample size formulas and results of applying the sample size formulas for various combinations of input parameters (for example, uncertainty magnitudes and confidence levels). However, in this initial effort, it was possible to address only a fraction of the many process control and compliance situations for which sample size formulas and determinations are needed. Section 6 outlines most of the remaining situations that will have to be addressed in future work. Additional situations likely will be identified as process-product control and compliance strategies are more fully developed. Hence, the topic coverage and results in this report should be considered preliminary. This report will need to be expanded and updated in the future to address the numbers of samples, analyses per sample, and measurements required to control and demonstrate compliance for the IHLW and ILAW vitrification processes, and possibly other portions of the RPP-WTP (such as pretreatment). Sample size considerations are also important for regulatory compliance issues, but that topic is being addressed in separate work scope, and hence is only mentioned in this report for completeness.

2.0 Vitrification Process and Sampling Objectives

Section 2.1 provides a general overview of the IHLW and ILAW vitrification processes and introduces the generic terms used to refer to IHLW and ILAW process vessels and other steps in the IHLW and ILAW vitrification processes. Section 2.2 describes various objectives for sample size calculations in the context of these processes. Future revisions of this report will identify the relationships between the objectives discussed and the RPP-WTP Project compliance strategies for IHLW and ILAW. Although many aspects of the compliance strategies have been determined in the WCP (CHG 2001a) and PSWP (CHG 2001b), many of the aspects relevant to this report have not been determined (especially for the IHLW strategy). Hence, it was considered premature to identify in this version of the report the relationships between the possible objectives discussed and the RPP-WTP Project's IHLW and ILAW compliance strategies.

2.1 IHLW and ILAW Vitrification Processes

Figure 2.1 presents a generic and simplified overview of the IHLW and ILAW vitrification processes. The purpose of the figure is to illustrate the key process vessels, the glass former chemicals system, the melter, and possible sampling and measurement points.

Sampling points being considered by the RPP-WTP Project for process-product control and compliance strategies (indicated by a circled S in Figure 2.1) include: feed concentrate from pretreatment (FCP), Concentrate Receipt Vessel (CRV), glass former chemicals (GFC), Melter Feed Preparation Vessel (MFPV), Melter Feed Vessel (MFV), and canisters of glass (subsequent to pouring from the melter and cooling). Sampling and chemical analyses are planned to verify the FCP is acceptable for transfer to the vitrification facility (IHLW or ILAW). Similarly, individual GFCs may be sampled and chemically analyzed to verify their compositions before use in the vitrification facilities.

Level and/or volume measurements will be made in the CRV, MFPV, and MFV (indicated by a diamond-enclosed L in Figure 2.1). Such measurements are important for estimating compositions in a particular vessel. Level and/or volume measurements are also important in verifying transfers to and from the MFPV, MFV, and other process vessels.

Weight measurements will be used to determine appropriate quantities of individual GFCs to add to waste feed concentrates in the MFPV. As indicated by a diamond-enclosed W in Figure 2.1, weights of individual GFCs will be determined, as well as weights of combined GFCs in the GFC Batch Makeup Hopper and the GFC Feed Hopper. Multiple weighing points provide for verifying transfers of individual and combined GFCs.

The possible sampling and measurement points in the IHLW and ILAW vitrification processes shown in Figure 2.1 are not intended as a comprehensive list of all possible sampling or measurement points that may be used for process/product control or specification compliance. Rather, these sampling and measurement points are the focus of initial efforts to statistically assess the number of samples and measurements that may be needed, depending on the objective of the sample or measurement. As the RPP-WTP Project progresses in developing and finalizing process-product control and compliance strategies, certain sampling and measurement points may be added or deleted in the future.

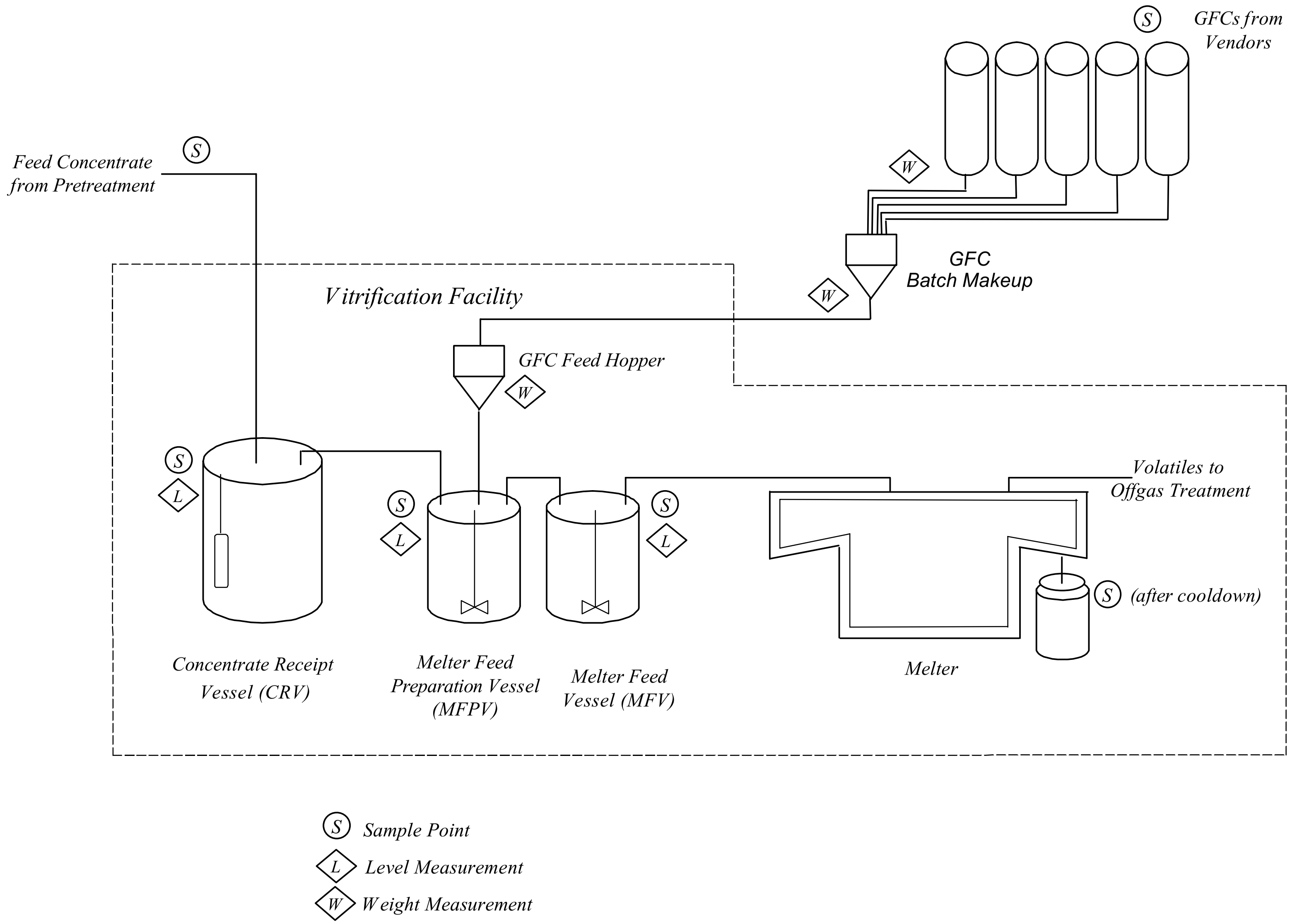

Figure 2.1. General View of the IHLW and ILAW Vitrification Processes

2.2 Sampling, Analysis, and Measurement Objectives

Four general objectives for sampling, chemical analyses, and measurement are defined as follows. Of these general objectives, three (A, B, and D) are the same as those discussed by Westsik et al. (1990).

A. Estimation. This objective is to estimate a quantity (for example, composition, weight, or level/volume) with a specified precision and a specified confidence level. Under this objective, it is assumed the sampling, chemical analysis, or measurement method is unbiased. Then, the sample size controls the effective precision and confidence of the estimate based on the sample mean of multiple determinations.

B. Detection. This objective is to decide, with specified probabilities of decision errors, whether a quantity violates its lower and upper limit by a specified amount. Two decision errors are possible: (1) failing to detect a difference larger than the specified amount, or (2) incorrectly detecting a difference as larger than the specified amount.

C. Confirmation. This objective is to confirm previous results, conclusions, or assumptions with as small a sample size as possible (maybe including a sample of size one). A confirmation objective may be applicable when it is impossible to take sufficient samples, chemical analyses, or measurements to achieve the desired precision or detectable difference with confidence, according to a statistical sample size approach. A confirmation objective may also be appropriate when it is desirable to perform limited confirmation of results obtained by a primary strategy approach. For example, a primary strategy approach for satisfying the WAPS 1.3 Product Consistency Test (PCT) requirements during production might be to collect glass samples, analyze their compositions, and apply PCT-composition models to predict PCT performance. A secondary, confirmatory strategy would be to perform the PCT on glass samples from one or more canisters per waste type. It would not be necessary to statistically select the number of samples on which to perform the PCT because the statistical considerations are addressed by the primary compliance strategy.

D. Process Monitoring. This objective is to monitor a process, using statistical methods, to detect when the process has gone outside, or is starting to go outside, its normal operating range.

For all objectives, it is assumed in this report that the sampling, chemical analysis, and measurement instruments and methods are unbiased. The RPP-WTP Project will need to demonstrate during qualification activities that all instruments and methods yield unbiased results, and have procedures in place to detect and possibly correct for biases if detected.

General objectives A, B, and C are concerned with the status at a given point in the process at a given time. General objective D is concerned with the status at a given point in the process over a range of time. At a given point in the process at a given time, it is assumed that a true (but unknown) value of a quantity is to be estimated (Objective A), detected (Objective B), or confirmed (Objective C). The assumption of an exact, true value may not be strictly correct in some cases. For example, in a process vessel, the composition throughout the vessel will not be exactly the same. However, in such cases the true value is assumed to be the mean at the given process point and given time (for example, the mean composition in a process vessel at a given time). Any such variability at the given point and given time is treated as part of the sampling uncertainty.

Specific estimation and detection objectives that may be appropriate for the various IHLW and ILAW vitrification sampling and measurement points illustrated in Figure 2.1 are listed in Table 2.1. Also included in Table 2.1 are the sources of uncertainty corresponding to each objective. These sources of uncertainty are important, because they must be factored into sample size formulas and calculations. Pulsipher (1993) provides a general discussion of many sources of uncertainty that affect vitrification processes.

The confirmation objective is not discussed further in this report because it does not involve use of statistical methods and sample size formulas. The process-monitoring objective (Objective D) is not discussed further in this report (except in Section 6 to note the need for future work) because addressing sample size issues would require knowing the specifics of the statistical process monitoring methods to be employed in the RPP-WTP IHLW and ILAW plants. The development of the RPP-WTP process-product control system has not sufficiently matured for such information to be currently available.

The objectives considered in this document are with respect to the status (for example, that of composition, level and/or volume, weight) at a specific point of the IHLW or ILAW vitrification process at a specific point in time. Other objectives may be applicable over some period of production, such as showing compliance over the course of a waste type. For example, an objective may be to show with a specified confidence that a specified high percentage of glass produced over a waste type satisfies the PCT performance limits specified in WAPS 1.3 (for IHLW) or specified in Contract Specification 2.2.2.17.2 (for ILAW). A similar objective exists for demonstrating ILAW produced from a given LAW waste type satisfies the Vapor Hydration Test (VHT) limit in Contract Specification 2.2.2.17.3. A separate technical effort addressing compliance methodology and sample size formulas for PCT and VHT compliance has proceeded simultaneously with the technical effort documented in this report, and a separate preliminary report will be issued. Other such separate efforts and reports will be needed in the future to address other aspects of process-product control or compliance over the course of processing a waste type (or some other period of production).

Table 2.1. Objectives for Sampling, Analysis, and Measurement in the IHLW and ILAW Vitrification Processes

Vessel or Process Step[(a), (b)]	Objective[(b)]	Uncertainties[(c)]
FCP or CRV	1. Estimate composition	Sampling, Analysis
	2. Estimate vessel level/volume	Level, Level-to-volume calibration[(d)]
GFC (individual)	3. Estimate composition	Sampling (individual), Analysis (individual)
	4. Confirm composition estimated by vendor	N/A—Nonstatistical
	5. Estimate weight	Weighing (individual)
GFC (combined)	6. Estimate weight	Weighing (combined)
	7. Estimate composition	Composition (individual), Weighing (individual and combined)
	8. Detect if weight of combined GFCs is different than the sum of weights of individual GFCs	Weighing (individual), Weighing (combined)
Calculated glass composition (CRV + GFC)	9. Estimate composition	CRV composition (from 1), GFC composition (from 7), Waste loading
	10. Detect if composition unacceptable	Composition (from 9)
	11. Estimate (predict) properties	Composition (from 9), Property models
	12. Detect if predicted properties unacceptable	Composition (from 9), Property models
MFPV or MPV	13. Estimate composition	Sampling, Analysis
	14. Estimate vessel level and volume	Level, Level-to-volume calibration[(d)]
	15. Detect if composition unacceptable	Composition (from 13)
	16. Estimate (predict) properties	Composition (from 13), Property models
	17. Detect if predicted properties unacceptable	Composition (from 13), Property models
Calculated (CRV + GFC) composition vs. estimated MFPV composition	18. Detect difference in compositions at the two process steps	Composition (from 9), Composition (from 13)
MFPV versus MFV	19. Detect if composition is different in the two vessels	MFPV and MFV compositions (both from 13)
	20. Detect if vessel levels/volumes different than calculated by transfers of volumes	MFPV and MFV levels and volumes (from 14)
Glass samples from canister	21. Estimate composition	Sampling, Analysis
	22. Estimate (predict) PCT or VHT performance	Composition (from 21),PCT- or VHT-composition models
	23. Detect if predicted PCT or VHT performance unacceptable	Composition (from 21), PCT- or VHT-composition models
	24. Detect if measured PCT or VHT performance unacceptable	Composition (from 21), PCT or VHT, Analysis
Not determined at this time	25. Detect if regulatory compliance achieved	Not determined at this time

(a) FCP, CRV, GFC, MFPV, and MFV are defined in the list of acronyms, terms, and abbreviations.
(b) Because process-product control and compliance strategies are still being developed, this table is not intended to include an exhaustive list of the possible process steps or objectives where samples, analyses, or measurements will be required and, hence, corresponding sample sizes determined.
(c) Uncertainty due to sampling also generally includes inherent variation in the material being sampled.
(d) It is assumed that vessel volumes will be determined via a calibration relationship relating volume to vessel level. Hence, the uncertainty in the calibration relationship must be considered.

3.0 Sample Size Methods and Preliminary Calculations for Composition Estimation Objectives

This section presents the statistical methods and formulas for calculating the sample sizes required to estimate compositions of:

- FCP, CRV, MFPV, MFV, or glass samples
- GFC.

Also presented are the results of preliminary calculations with sample size formulas for various combinations of input parameters (for example, required precision, confidence levels, and uncertainties). The methods and results presented in this section address Objectives 1, 3, 13, and 21 in Table 2.1.

Other estimation objectives are also of interest, including Objectives 2, 5, 6, 7, 9, 11, 14, 16, and 22 in Table 2.1. However, it was not possible to address all these objectives of interest in the initial effort. The estimation (and detection) objectives that will be addressed as part of future work are discussed in various subsections of Section 6.

3.1 Method and Formula Used to Determine the Numbers Of Samples and Analyses per Sample for Composition Estimation Objectives

As described in Section 2.1, the estimation objective is to estimate a quantity (for example, composition, weight, or level/volume) with a specified *precision* with a specified *confidence level*. For example, an RPP-WTP decision maker may want to be 95% confident that the estimate is within 10% of the true value (where here 10% is the desired precision). Precision is defined as the half-width of a statistical confidence interval on the true value of the quantity (or the true mean of the quantity, as discussed in Section 2.2)[a]. The half-width gives the magnitude of the difference between the true and the estimated values (that is, the precision) that can be obtained with specified confidence. The half-width with 100(1-α)% confidence is denoted by $H_{1-\alpha}$, where $0 < \alpha < 0.5$ (although typically $0 < \alpha \leq 0.1$).

Consider situations where a quantity (for example, composition in a vessel) will be estimated by collecting n_S samples and chemically analyzing each sample n_A times. In such cases, the value of $H_{1-\alpha}$ depends on the number of samples collected, the number of analyses per sample, the sampling uncertainty, the analytical uncertainty, and on the confidence level required.

The formula for $H_{1-\alpha}$ in the sampling and analysis case is given by

$$H_{1-\alpha}(\%) = t_{1-\alpha/2,\, n_S-1}[(\%RSD_S)^2/n_S + (\%RSD_A)^2/n_S n_A]^{1/2} \tag{3.1}$$

[a] This statement assumes that the sampling, chemical analysis, or measurement method is unbiased. If not, the precision is with respect to the biased (rather than unbiased) estimate.

where $\%RSD_S$ is the specified percent relative standard deviation for samples, $\%RSD_A$ is the specified percent relative standard deviation for analyses, n_S is the number of samples, n_A is the number of analyses per sample, $100(1 - \alpha)$ is the percent confidence required that using the specified number of samples and analyses per sample will result in an estimate that does not deviate from the true value by more than $H_{1-\alpha}$ percent, and $t_{1-\alpha/2, n_S-1}$ is the $(1-\alpha/2)^{th}$ quantile of the t-distribution with $n_S - 1$ degrees of freedom as read from t-distribution tables. Equation (3.1) was derived from Equation (3.27) in Bowen and Bennett (1987). Tabled values of the t-distribution are found in most statistics textbooks, including Bowen and Bennett (1987, Table A5, page 539). Values of the t-distribution are also available in many statistical software packages.

The assumptions underlying Equation (3.1) are: (1) the measurements (that is, results of sampling and analyses) are normally distributed, (2) the measurements are independent and representative of the material being sampled, and (3) no sampling or analytical bias is present in the data. The assumption of normality will be evaluated during qualification testing and cold commissioning. The assumptions of independence, representative samples, and lack of sampling and analytical bias will be assured by strict adherence to specified methods for determining where and when to collect samples, mixing the material to be sampled, sampling, analytical measurements, and quality control procedures. Testing will also be performed to investigate and demonstrate these assumptions during qualification activities.

Section 3.2 presents the results of applying (3.1) to calculate the values of n_S and n_A required to estimate a composition given the applicable sampling uncertainty ($\%RSD_S$), applicable analytical uncertainty ($\%RSD_A$), desired confidence $[100(1-\alpha)]$, and desired precision ($H_{1-\alpha}$). For example, the goal might be to estimate with $100(1-\alpha) = 95\%$ confidence the composition in a CRV within $H_{1-\alpha} = 15\%$ of the true value given $\%RSD_S = 15\%$ and $\%RSD_A = 10\%$.

Note that n_S is the number of samples at a given time from a given process or product location. For example, n_S may be the number of samples from the CRV or the MFPV at a given time. Or, n_S may be the number of glass shard samples from a given canister at a given time. Further, note that n_A is the number of chemical analyses of a given analyte for each of the n_S samples. Hence, the sample size formula (3.1) will need to be applied for each analyte, because the applicable analytical uncertainty ($\%RSD_A$), desired precision ($H_{1-\alpha}$), and desired confidence $[100(1-\alpha)]$ may change from analyte to analyte (especially if different analytical methods are used for some analytes than for others). Hence, the total number of chemical analyses for a given analyte of the n_S samples taken at a given process or product location is $N = n_S \times n_A$. The number of analyses per sample required for a given analytical method will be the maximum value of n_A over the analytes obtained by that method.

3.2 Numbers of Samples and Analyses per Sample to Estimate the Compositions of FCP, CRV, MFPV, MFV, and Waste Glass

Values of $H_{1-\alpha}$ for estimating compositions of FCP, CRV, MFPV, MFV and waste glass were calculated using Equation (3.1) for all combinations of the parameter values given in Table 3.1. The values of $\%RSD_S$ and $\%RSD_A$ in Table 3.1 are intended to cover the range of uncertainty magnitudes expected, based on past experience. The values of $\%RSD_S$ and $\%RSD_A$ for various composition estimation cases applicable to the IHLW and ILAW vitrification facilities will be estimated as a part of qualification activities. Calculations of $H_{1-\alpha}$ can be updated in the future to

reflect better estimates of %RSD_S and %RSD_A for various composition estimation situations in the IHLW and ILAW vitrification facilities.

Table 3.1. Parameter Values Used to Calculate Confidence Interval Half-Widths Via Equation (3.1) for Compositions of FCP, CRV, MFPV, MFV, and Waste Glass

Parameter [a]	Parameter Values Used
$\%RSD_S = 100\sigma_S /$ mean	2.5% 5% 10% 20%
$\%RSD_A = 100\sigma_A /$ mean	5% 10% 25%
n_S	1 2 3 4 5 6 7
n_A	1 2 3
100(1-α) Confidence	90% 95% 99%

(a) $\%RSD_S$ = percent relative standard deviation for *samples*
$\%RSD_A$ = percent relative standard deviation for *analyses*
σ_S = standard deviation for *samples*
σ_A = standard deviation for *analyses*
n_S = number of *samples*
n_A = number of *analyses per sample*
100(1 - α) = percent confidence required

In calculating $H_{1-\alpha}$ values, attention was limited to between 1 and 7 samples at a given location, and 1, 2, or 3 analyses per sample based on the desire to keep sampling and analyses at reasonable levels. However, additional calculations could be performed in the future, as desired, for more than 7 samples or more than 3 analyses per sample.

The calculated values of $H_{1-\alpha}$ were used to construct Table 3.2. Values of $H_{1-\alpha}$ were calculated for the 4 x 3 x 7 x 3 x 3 = 756 combinations of parameter values shown in Table 3.1. Rather than tabulating all of these results in a very long table, the results were summarized as shown in Table 3.2. This table displays the cases with the minimum total number of analyses (that is, the number of samples times the number of analyses per sample, n_S times n_A) needed to achieve half-widths of less than 5%, 10%, 15%, or 20% for the various combinations of $\%RSD_S$, $\%RSD_A$, and 100(1 - α) in Table 3.1. Cases achieving the same results, but having larger values of n_S times n_A than cases with the minimum total number of analyses, are not displayed in Table 3.2. It should be noted that n_A must be determined separately for each analyte and then the largest n_A across analytes measured by a particular method will be used as the number of analyses per sample for that method.

To illustrate the results in Table 3.2, suppose the $\%RSD_S$ is 10% and the $\%RSD_A$ is 5%. Then, if 6 samples are collected and 1 analysis is conducted for each sample, we would have 90% confidence that the difference between the estimated composition and the true composition would be no greater than ± 10%. Also, 6 samples with 1 analysis per sample is sufficient to provide 99% confidence of being no greater than ± 20% from the true composition. Table 3.2 also shows that if 4 samples are collected with 1 analysis per sample, the precision of ± 10% changes to ± 15%. This illustrates that Table 3.2 can be used to show how changing n_S and n_A affect the length of the half-width (in percent). Or, if a desired or required half-width (precision) for the estimate is known, Table 3.2 can be used to determine the required number of samples and analyses per sample to achieve that precision with the specified confidence. Using Table 3.2 to determine n_S and n_A requires prior estimates of $\%RSD_S$ and $\%RSD_A$, as well as some notion of what confidence level and precision (half-width length) must be achieved for the particular application. Work planned as

part of RPP-WTP qualification activities will determine $\%RSD_S$ and $\%RSD_A$ and also assess the required confidence levels and precisions needed for various situations in the IHLW and ILAW vitrification processes.

Note the lower diagonal portion of Table 3.2 contains only dashes, as do some rows of the table corresponding to 99% confidence. The dashes indicate that limiting attention to $n_S \leq 7$ and $n_A \leq 3$ is insufficient to provide the specified precision and confidence for the corresponding values of $\%RSD_S$ and $\%RSD_A$. Calculations for larger values of n_S and n_A can be performed, as needed, if and when it is determined that combinations of uncertainty magnitudes, precision, and confidence showing dashes in Table 3.2 are relevant.

Before leaving this subsection, an explanation is required regarding the univariate nature of Tables 3.1 and 3.2, and the multivariate nature of composition. That is, the chemical or radionuclide composition in a vessel consists of values (for example, concentrations) for many components (that is, element, radionuclide, and oxide), whereas Tables 3.1 and 3.2 address a single component. Different components may have different values of $\%RSD_S$ and $\%RSD_A$, and different precisions and confidence levels may be appropriate for different components. In such cases, Table 3.2 can be used separately for each component. If all components are analyzed simultaneously (for example, as in ICP), the largest combination of n_S and n_A values across components determines the required number of samples and number of analyses per sample. However, if certain components are analyzed by different methods, the largest combination of n_S and n_A values need only be determined within each group of components analyzed by a common method.

Table 3.2. Best Sampling Schemes (Values of n_S and n_A)[(a)] for the Estimation Objective that Achieve Half-Widths of Specified Size for Compositions of FCP, CRV, MFPV, MFV, and Waste Glass

			Half-widths ($H_{1-\alpha}$)[(b)]							
			<5%		<10%		<15%		<20%	
%RSD$_S$	%RSD$_A$	100(1-α)[(c)]	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A
2.5	5	90	6	1	3	1	3	1	3	1
		95	6	2	4	1	3	1	3	1
		99	--	--	6	1	5	1	4	1
	10	90	7	3	5	1	4	1	3	1
		95	--	--	7	1	5	1	4	1
		99	--	--	7	3	7	1	6	1
	25	90	--	--	--	--	6	2	7	1
		95	--	--	--	--	7	3	6	2
		99	--	--	--	--	--	--	--	--
5	5	90	7	2	4	1	3	1	3	1
		95	--	--	5	1	4	1	3	1
		99	--	--	7	1	5	1	5	1
	10	90	--	--	6	1	4	1	3	1
		95	--	--	6	2	5	1	4	1
		99	--	--	--	--	6	2	6	1
	25	90	--	--	--	--	7	2	7	1
		95	--	--	--	--	7	3	6	2
		99	--	--	--	--	--	--	--	--
10	5	90	--	--	6	1	4	1	3	1
		95	--	--	7	2	5	1	4	1
		99	--	--	--	--	7	2	6	1
	10	90	--	--	7	2	5	1	4	1
		95	--	--	--	--	6	1	5	1
		99	--	--	--	--	--	--	7	1
	25	90	--	--	--	--	7	2	7	1
		95	--	--	--	--	--	--	7	2
		99	--	--	--	--	--	--	--	--
20	5	90	--	--	--	--	7	2	5	1
		95	--	--	--	--	--	--	7	1
		99	--	--	--	--	--	--	--	--
	10	90	--	--	--	--	--	--	6	1
		95	--	--	--	--	--	--	7	2
		99	--	--	--	--	--	--	--	--
	25	90	--	--	--	--	--	--	7	2
		95	--	--	--	--	--	--	--	--
		99	--	--	--	--	--	--	--	--

(a) Combinations of n_S and n_A having the smallest total number of analyses (n_S x n_A) that achieve the specified precision (half-width) and confidence level are displayed in the body of the table. A dash indicates no combination of $n_S \leq 7$ and $n_A \leq 3$ achieves the desired precision (half-width) and confidence level.

(b) $H_{1-\alpha}$ is the precision half-width with 100(1-α)% confidence.
(c) 100(1-α) is the confidence associated with the precision half-width, $H_{1-\alpha}$.

3.3 Numbers of Samples and Analyses per Sample to Estimate the Compositions of Individual and Combined Glass Former Chemicals

Values of $H_{1-\alpha}$ obtained using Equation (3.1) for estimating compositions of individual and combined GFCs were calculated for the parameter values given in Table 3.3. The values of $\%RSD_S$ and $\%RSD_A$ in Table 3.3 are intended to cover the range of uncertainty magnitudes expected based on past experience. These quantities have smaller magnitudes in Table 3.3 compared to Table 3.1 because it is expected that GFCs will be more homogeneous and easier to sample and analyze than slurry samples. Hence, GFCs will have smaller sampling and analytical uncertainties than slurry samples. The values of $\%RSD_S$ and $\%RSD_A$ for individual and combined GFCs will be estimated as a part of qualification activities. Calculations of $H_{1-\alpha}$ can be updated as needed to reflect better estimates of $\%RSD_S$ and $\%RSD_A$ for individual and combined GFCs.

Table 3.3. Parameter Values Used to Calculate Confidence Interval Half-Widths Via Equation (3.1) for Compositions of Individual and Combined Glass Former Chemicals

Parameter [(a)]	Parameter Values Used
$\%RSD_{GFC,S} = 100\sigma_{GFC,S}$ / mean	2% 5% 10%
$\%RSD_{GFC,A} = 100\sigma_{GFC,A}$ / mean	2% 5% 10%
$n_{GFC,S}$	1 2 3 4 5 6 7
$n_{GFC,A}$	1 2 3
100(1 - α) Confidence	90% 95% 99%

(a) $\%RSD_S$ = percent relative standard deviation for *samples*
$\%RSD_A$ = percent relative standard deviation for *analyses*
$\sigma_{GFC,S}$ = standard deviation of GFC *samples* (individual or combined)
$\sigma_{GFC,A}$ = standard deviation of *analyses* for GFC (individual or combined)
$n_{GFC,S}$ = number of *samples*
$n_{GFC,A}$ = number of *analyses per sample*
100(1-α) = percent confidence required

In calculating $H_{1-\alpha}$ values, attention was limited to between 1 and 7 samples at a given location, and 1, 2, or 3 analyses per sample based on the desire to keep sampling and analyses at reasonable levels. However, additional calculations could be performed as desired for more than 7 samples or more than 3 analyses per sample.

Before proceeding, note that at this time the RPP-WTP Project does not envision directly estimating individual or combined GFC compositions by sampling and chemical analysis during production operations. Rather, the intended strategy (see CHG 2001a for IHLW, and CHG 2001b for ILAW) during production is to make use of estimated compositions and uncertainties of the individual GFCs provided by the vendor or determined as part of qualification or acceptance testing. Then, weights of the individual GFCs will be used to estimate the composition and uncertainty of the combined GFCs. That approach to estimating combined GFCs composition is

more complicated, but better, because weighing can be performed with more accuracy and precision than sampling and analysis. Still, because of the additional complexity, sample size methods, formulas, and calculations have not yet been developed for estimating combined GFC compositions via calculations involving individual GFC compositions and weights. Still, the methods, formulas, and calculations in this subsection for directly sampling and analyzing individual or combined GFC compositions should be applicable to qualification work with the GFCs and work to accept or verify vendor-provided compositions of the GFCs.

The calculated values of $H_{1-\alpha}$ were used to construct Table 3.4. Values of $H_{1-\alpha}$ were calculated for the 3 x 3 x 7 x 3 x 3 = 567 combinations of parameter values shown in Table 3.3. Rather than tabulating all of these results in a very long table, the results were summarized as shown in Table 3.4. Table 3.4 displays the cases with the minimum total number of analyses (that is, the number of samples times the number of analyses per sample, n_S x n_A) needed to achieve half-widths of less than 2.5%, 5%, and 10% for the various combinations of %RSD_S, %RSD_A, and 100(1 - α) in Table 3.3. Cases achieving the same results, but having larger values of n_S x n_A than cases achieving the minimum total number of analyses, are not displayed in Table 3.4. The results in Table 3.4 are interpreted and used in the same way as the results in Table 3.2. It should be noted that n_A must be determined separately for each analyte in a GFC, and then the largest n_A across analytes measured by a particular method will be used as the number of analyses per sample for that method.

Note the lower diagonal portion of Table 3.4 contains only dashes, as do some rows of the table corresponding to 99% confidence, and most of the column corresponding to < 2.5% half-widths. The dashes indicate that limiting attention to $n_S \leq 7$ and $n_A \leq 3$ is insufficient to provide the specified precision and confidence for the corresponding values of %RSD_S and %RSD_A. Calculations for larger values of n_S and n_A can be performed as needed if and when it is determined that combinations of uncertainty magnitudes, precision, and confidence having dashes in Table 3.4 are relevant. Also, future calculations for larger half-width values can be performed, if they are needed.

Table 3.4. Best Sampling Schemes (Values of n_S and n_A)[(a)] for the Estimation Objective that Achieve Half-Widths of Specified Size for the Compositions of Individual and Combined Glass Former Chemicals

			Half-widths ($H_{1-\alpha}$)[(b)]					
			<2.5%		<5%		<10%	
%RSD_S	%RSD_A	100(1-α)[(c)]	n_S	n_A	n_S	n_A	n_S	n_A
2	2	90	6	1	3	1	3	1
		95	7	2	4	1	3	1
		99	--	--	6	1	4	1
	5	90	--	--	6	1	3	1
		95	--	--	7	1	4	1
		99	--	--	7	3	6	1
	10	90	--	--	7	3	5	1
		95	--	--	--	--	7	1
		99	--	--	--	--	7	3
5	2	90	--	--	6	1	3	1
		95	--	--	7	1	4	1
		99	--	--	--	--	6	1
	5	90	--	--	7	2	4	1
		95	--	--	--	--	5	1
		99	--	--	--	--	7	1
	10	90	--	--	--	--	6	1
		95	--	--	--	--	6	2
		99	--	--	--	--	--	--
10	2	90	--	--	--	--	5	1
		95	--	--	--	--	7	1
		99	--	--	--	--	--	--
	5	90	--	--	--	--	6	1
		95	--	--	--	--	7	2
		99	--	--	--	--	--	--
	10	90	--	--	--	--	7	2
		95	--	--	--	--	--	--
		99	--	--	--	--	--	--

(a) Combinations of n_S and n_A having the smallest total number of analyses (n_S x n_A) that achieve the specified precision (half-width) and confidence level are displayed in the body of the table. A dash indicates no combination of $n_S \le 7$ and $n_A \le 3$ achieves the desired precision (half-width) and confidence level.

(b) $H_{1-\alpha}$ is the precision half-width with 100(1-α)% confidence.

(c) 100(1-α) is the confidence associated with the precision half-width, $H_{1-\alpha}$.

4.0 Sample Size Methods and Preliminary Calculations for Detecting Unacceptable Compositions

This section presents the statistical methods and formulas for calculating the sample sizes required to detect differences in composition from limiting values (for example, as specified in compliance specifications). Limiting values may consist of a lower limit, an upper limit, or both lower and upper limits. The limiting values may apply to individual elemental or oxide components of composition, or they may apply to linear combinations or ratios of components. Also presented are the results of preliminary calculations with sample size formulas for various combinations of input parameters (such as, the difference to be detected, confidence, and various uncertainties). The methods and results presented in this section address Objective 15 in Table 2.1.

Other detection objectives are also of interest, including Objectives 8, 10, 12, 17, 18, 19, 20, 23, and 24 in Table 2.1. Objective 19 is addressed in Section 5. However, it was not possible to address the remaining detection objectives of interest in the initial effort. The detection objectives that will be addressed as part of future work are discussed in various subsections of Section 6.

4.1 Method and Formula to Determine the Numbers of Samples and Analyses per Sample for Detecting Unacceptable Compositions

The detection objective involves deciding whether a composition is inside or outside of lower or upper limits. Hence, methodology to calculate the number of samples and the number of analyses per sample required must take into account two types of decision errors: (1) deciding that a limit is exceeded when in fact it is *not* exceeded, and (2) deciding a limit is not exceeded, when in fact it *is* exceeded.

The objective is to determine the number of samples and number of analyses per sample required to: (1) limit to α the probability of incorrectly deciding a lower limit or upper limit has been violated, and (2) detect with specified probability 1-β when the value of a composition component either exceeds its upper limit or is less than its lower limit by a specified amount D. The D (%) is called the "detectable difference", that is, the percent difference between the estimated composition and a lower or upper limit. Hence

D (%) = 100 (true composition– upper limit)/(true composition)
if the true composition is greater than the upper limit

= 100 (lower limit – true composition)/(true composition)
if the true composition is less than the lower limit.

The detectable difference $D_{\alpha/2,1-\beta}$ that satisfies the required α and 1-β for the two-sided sampling and analysis case is given by

$$D_{\alpha/2,1-\beta}(\%) = (Z_{1-\alpha/2} + Z_{1-\beta})\,[(\%RSD_S)^2 + (\%RSD_A)^2 / n_A]^{1/2} / [n_S - 0.5\,(Z_{1-\alpha/2})^2]^{1/2}, \quad (4.1)$$

where $\%RSD_S$ is the percent relative standard deviation for samples, $\%RSD_A$ is the percent relative standard deviation for analyses per sample, n_S is the number of samples collected for the detection objective, n_A is the number of analyses per sample conducted for the detection objective, $Z_{1-\alpha/2}$ is the

$(1\text{-}\alpha/2)^{th}$ quantile of the standard normal distribution, and $Z_{1-\beta}$ is the $(1\text{-}\beta)^{th}$ quantile of the standard normal distribution. Equation (4.1) was derived from Equation (4.19) in Bowen and Bennett (1987). Tabled values of the standard normal distribution (that is, the normal distribution that has mean 0 and standard deviation 1) are found in most statistics textbooks, including Bowen and Bennett (1987, Table A3, page 936). Values of the standard normal distribution are also available in many statistical software packages.

Equation (4.1) is for the two-sided case where both lower and upper limits are set on a component of composition. In practice, a quantity might violate either the lower limit or the upper limit. Hence, a two-sided approach maintains the total probability α of incorrectly deciding the quantity violates either the lower limit or the upper limit. For cases with only a lower limit or an upper limit, the detectable difference $D_{\alpha,1-\beta}$ for the one-sided sampling and analysis case is given by

$$D_{\alpha,1-\beta}(\%) = (Z_{1-\alpha} + Z_{1-\beta})\,[(\%RSD_S)^2 + (\%RSD_A)^2 / n_A\,]^{1/2} / [n_S - 0.5\,(Z_{1-\alpha})^2]^{1/2}, \qquad (4.2)$$

where the $Z_{1-\alpha/2}$ values in Equation (4.1) were replaced by the $Z_{1-\alpha}$ values in Equation (4.2). In Equation (4.2), α replaces $\alpha/2$ because there is only one opportunity to incorrectly decide a limit is violated. Hence, α need not be split in half corresponding to the two opportunities to incorrectly decide a limit is violated.

4.2 Numbers of Samples and Analyses per Sample for Detecting Unacceptable Compositions

Values of $D_{\alpha/2,1-\beta}$ (%) were calculated using Equation (4.1) for various combinations of the parameter values specified in Table 4.1. As was the case for Table 3.1 and Table 3.3, the values for $\%RSD_S$ and $\%RSD_A$ in Table 4.1 are intended to cover the range of uncertainty magnitudes expected based on past experience. The values of $\%RSD_S$ and $\%RSD_A$ for various composition detection cases applicable to the RPP-WTP will be estimated as a part of qualification activities. Calculations of $D_{\alpha/2,1-\beta}$ can be updated as needed to reflect better estimates of $\%RSD_S$ and $\%RSD_A$ for various RPP-WTP composition detection situations.

In calculating $D_{\alpha/2,1-\beta}$ values, attention was limited to between 1 and 7 samples at a given location, and 1, 2, or 3 analyses per sample, based on the desire to keep sampling and analyses at reasonable levels. However, additional calculations could be performed as desired for more than 7 samples or more than 3 analyses per sample.

The calculated values of $D_{\alpha/2,1-\beta}$ were used to construct Table 4.2, as follows. Values of $D_{\alpha/2,1-\beta}$ were calculated for 4 x 4 x 7 x 3 x 3 x 3 = 3024 combinations of parameter values shown in Table 4.1. Rather than tabulating all of these results in a very long table, the results were summarized as shown in Table 4.2. Table 4.2 displays the cases with the minimum total number of analyses (that is, the number of samples times the number of analyses per sample, n_S x n_A) needed to achieve detectable differences $D_{\alpha/2,1-\beta}$ of less than 5%, 10%, 15%, 25%, 50%, or 100% for the various combinations of $\%RSD_S$, $\%RSD_A$, $100(1 - \alpha)$, and $100(1 - \beta)$ in Table 4.1. Cases achieving the same results, but having larger values of n_S x n_A than cases achieving the minimum total number of analyses, are not displayed in Table 4.2.

Table 4.1. Parameter Values Used to Calculate Detectable Differences $D_{\alpha/2,1-\beta}$ (%) Via Equation (4.1)

Parameter [(a)]	Parameter Values Used
$\%RSD_S = 100\sigma_S/mean$	2.5% 5% 10% 20%
$\%RSD_A = 100\sigma_A/mean$	5% 10% 25% 50%
n_S	1 2 3 4 5 6 7
n_A	1 2 3
$100(1-\alpha)$ Confidence	90% 95% 99%
$100(1-\beta)$ Confidence	90% 95% 99%

(a) $\%RSD_S$ = percent relative standard deviation for *samples*
$\%RSD_A$ = percent relative standard deviation for *analyses*
n_S = number of *samples*
n_A = number of *analyses per sample*
σ_S = standard deviation for *samples*
σ_A = standard deviation for *analyses*
$100(1-\alpha)$ = percent confidence required that data will *correctly* indicate when the true value *does not* exceed the upper limit or is *not* less than the lower limit
$100(1-\beta)$ = percent confidence required that data will *correctly* indicate when the true value *does* exceed the upper limit or is *less than* the lower limit by $D_{1-\beta}$.

For several reasons, a wide range of detectable difference $D_{\alpha/2,1-\beta}$ magnitudes from <5% to <100% were considered. First, consider the objective of detecting whether certain composition components are above an upper limit or below a lower limit. Some composition components may have larger sampling and analyses variances, thus making it difficult to detect smaller differences. Second, limiting attention to $n_S \leq 7$ and $n_A \leq 3$ means that only larger differences can be detected in some cases. It should be noted that n_A must be determined separately for each analyte and then the largest n_A across analytes measured by a particular method will be used as the number of analyses per sample for that method.

Note that substantial parts of the lower left portions of Table 4.2 contain only dashes. The dashes indicate that limiting attention to $n_S \leq 7$ and $n_A \leq 3$ is insufficient to detect the specified difference with the specified probabilities of incorrect decisions for the corresponding values of $\%RSD_S$ and $\%RSD_A$. Calculations for larger values of n_S and n_A can be performed as needed if and when it is determined that combinations of uncertainty magnitudes, detectable differences, and incorrect decision probabilities showing dashes in Table 4.2 are relevant to the RPP-WTP situation.

To illustrate the results in Table 4.2, suppose that $\%RSD_S$ is 10%, $\%RSD_A$ is 5%, that we require $100(1-\alpha) = 95\%$ confidence (setting $\alpha = 0.05$) that the data correctly indicates when the true composition does not exceed the upper limit or is not less than the lower limit, and that we also require $100(1-\beta) = 95\%$ confidence (setting $\beta = 0.05$) that the data correctly indicates when the true value does exceed the upper limit or is less than the lower limit. In addition, suppose that enough samples must be collected and analyses per sample conducted so the detectable difference between the estimate and the lower or upper limit is less than 25%. From Table 4.2, the required number of samples is 5 with 1 analysis per sample, for a total of 5 analyses. If the detectable difference between the estimate and the lower or upper limit is increased from 25% to 50%, the number of samples decreases from 5 to 3, still with 1 analysis each.

Table 4.2. Best Sampling Schemes (Values of n_S and n_A)[(a)] for Detecting Specified Detectable Differences $D_{\alpha/2,1-\beta}$ (%)

				Required Detectable Differences $D_{\alpha/2,1-\beta}$											
				<5%		<10%		<15%		<25%		<50%		<100%	
%RSD$_S$	%RSD$_A$	100(1-α)[(b)]	100(1-β)[(c)]	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A
2.5	5	90	90	7	3	5	1	3	1	2	1	2	1	2	1
			95	--	--	5	1	3	1	2	1	2	1	2	1
			99	--	--	7	1	4	1	3	1	2	1	2	1
		95	90	--	--	6	1	4	1	3	1	3	1	2	1
			95	--	--	6	1	4	1	3	1	3	1	2	1
			99	--	--	6	2	5	1	3	1	3	1	2	1
		99	90	--	--	7	2	6	1	5	1	4	1	4	1
			95	--	--	7	2	6	1	5	1	4	1	4	1
			99	--	--	7	3	7	1	5	1	4	1	4	1
	10	90	90	--	--	7	2	6	1	3	1	2	1	2	1
			95	--	--	6	3	7	1	4	1	2	1	2	1
			99	--	--	--	--	6	2	5	1	3	1	2	1
		95	90	--	--	7	3	7	1	4	1	3	1	3	1
			95	--	--	--	--	6	2	5	1	3	1	3	1
			99	--	--	--	--	7	2	6	1	3	1	3	1
		99	90	--	--	--	--	6	3	6	1	4	1	4	1
			95	--	--	--	--	7	3	7	1	5	1	4	1
			99	--	--	--	--	--	--	6	2	5	1	4	1
	25	90	90	--	--	--	--	--	--	6	2	4	1	2	1
			95	--	--	--	--	--	--	7	2	5	1	3	1
			99	--	--	--	--	--	--	7	3	6	1	3	1
		95	90	--	--	--	--	--	--	6	3	5	1	3	1
			95	--	--	--	--	--	--	7	3	6	1	3	1
			99	--	--	--	--	--	--	--	--	7	1	4	1
		99	90	--	--	--	--	--	--	--	--	6	2	5	1
			95	--	--	--	--	--	--	--	--	6	2	5	1
			99	--	--	--	--	--	--	--	--	7	2	5	1
	50	90	90	--	--	--	--	--	--	--	--	6	2	4	1
			95	--	--	--	--	--	--	--	--	7	2	5	1
			99	--	--	--	--	--	--	--	--	7	3	6	1
		95	90	--	--	--	--	--	--	--	--	6	3	5	1
			95	--	--	--	--	--	--	--	--	7	3	6	1
			99	--	--	--	--	--	--	--	--	--	--	7	1
		99	90	--	--	--	--	--	--	--	--	--	--	6	2
			95	--	--	--	--	--	--	--	--	--	--	6	2
			99	--	--	--	--	--	--	--	--	--	--	7	2

Table 4.2. Best Sampling Schemes (Values of n_S and n_A)[(a)] for Detecting Specified Detectable Differences $D_{\alpha/2,1-\beta}$ (%) (cont'd)

				Required Detectable Differences $D_{\alpha/2,1-\beta}$											
				<5%		<10%		<15%		<25%		<50%		<100%	
%RSD$_S$	%RSD$_A$	100(1-α)[(b)]	100(1-β)[(c)]	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A
5	5	90	90	--	--	6	1	4	1	3	1	2	1	2	1
			95	--	--	7	1	4	1	3	1	2	1	2	1
			99	--	--	7	3	5	1	3	1	2	1	2	1
		95	90	--	--	6	2	5	1	3	1	3	1	2	1
			95	--	--	7	2	5	1	3	1	3	1	2	1
			99	--	--	--	--	7	1	4	1	3	1	3	1
		99	90	--	--	--	--	7	1	5	1	4	1	4	1
			95	--	--	--	--	7	2	5	1	4	1	4	1
			99	--	--	--	--	7	3	6	1	4	1	4	1
	10	90	90	--	--	7	3	7	1	4	1	2	1	2	1
			95	--	--	--	--	5	2	4	1	2	1	2	1
			99	--	--	--	--	7	2	5	1	3	1	2	1
		95	90	--	--	--	--	6	2	5	1	3	1	3	1
			95	--	--	--	--	7	2	5	1	3	1	3	1
			99	--	--	--	--	7	3	6	1	3	1	3	1
		99	90	--	--	--	--	--	--	7	1	5	1	4	1
			95	--	--	--	--	--	--	7	1	5	1	4	1
			99	--	--	--	--	--	--	7	2	5	1	4	1
	25	90	90	--	--	--	--	--	--	6	2	4	1	2	1
			95	--	--	--	--	--	--	6	3	5	1	3	1
			99	--	--	--	--	--	--	--	--	6	1	3	1
		95	90	--	--	--	--	--	--	6	3	5	1	3	1
			95	--	--	--	--	--	--	7	3	6	1	3	1
			99	--	--	--	--	--	--	--	--	7	1	4	1
		99	90	--	--	--	--	--	--	--	--	6	2	5	1
			95	--	--	--	--	--	--	--	--	6	2	5	1
			99	--	--	--	--	--	--	--	--	7	2	5	1
	50	90	90	--	--	--	--	--	--	--	--	6	2	4	1
			95	--	--	--	--	--	--	--	--	7	2	5	1
			99	--	--	--	--	--	--	--	--	7	3	6	1
		95	90	--	--	--	--	--	--	--	--	6	3	5	1
			95	--	--	--	--	--	--	--	--	7	3	6	1
			99	--	--	--	--	--	--	--	--	--	--	7	1
		99	90	--	--	--	--	--	--	--	--	--	--	6	2
			95	--	--	--	--	--	--	--	--	--	--	6	2
			99	--	--	--	--	--	--	--	--	--	--	7	2

Table 4.2. Best Sampling Schemes (Values of n_S and n_A)[(a)] for Detecting Specified Detectable Differences $D_{\alpha/2,1-\beta}$ (%) (cont'd)

				Required Detectable Differences $D_{\alpha/2,1-\beta}$											
				<5%		<10%		<15%		<25%		<50%		<100%	
%RSD$_S$	%RSD$_A$	100(1-α)[(b)]	100(1-β)[(c)]	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A
10	5	90	90	--	--	--	--	7	1	4	1	2	1	2	1
			95	--	--	--	--	7	2	4	1	2	1	2	1
			99	--	--	--	--	--	--	5	1	3	1	2	1
		95	90	--	--	--	--	7	3	5	1	3	1	3	1
			95	--	--	--	--	--	--	5	1	3	1	3	1
			99	--	--	--	--	--	--	6	1	3	1	3	1
		99	90	--	--	--	--	--	--	7	1	5	1	4	1
			95	--	--	--	--	--	--	7	1	5	1	4	1
			99	--	--	--	--	--	--	--	--	5	1	4	1
	10	90	90	--	--	--	--	7	3	5	1	3	1	2	1
			95	--	--	--	--	--	--	5	1	3	1	2	1
			99	--	--	--	--	--	--	7	1	3	1	2	1
		95	90	--	--	--	--	--	--	6	1	3	1	3	1
			95	--	--	--	--	--	--	7	1	3	1	3	1
			99	--	--	--	--	--	--	7	2	4	1	3	1
		99	90	--	--	--	--	--	--	7	2	5	1	4	1
			95	--	--	--	--	--	--	--	--	5	1	4	1
			99	--	--	--	--	--	--	--	--	6	1	4	1
	25	90	90	--	--	--	--	--	--	6	3	4	1	2	1
			95	--	--	--	--	--	--	7	3	5	1	3	1
			99	--	--	--	--	--	--	--	--	6	1	3	1
		95	90	--	--	--	--	--	--	--	--	5	1	3	1
			95	--	--	--	--	--	--	--	--	6	1	3	1
			99	--	--	--	--	--	--	--	--	5	2	4	1
		99	90	--	--	--	--	--	--	--	--	6	2	5	1
			95	--	--	--	--	--	--	--	--	7	2	5	1
			99	--	--	--	--	--	--	--	--	7	3	6	1
	50	90	90	--	--	--	--	--	--	--	--	6	2	4	1
			95	--	--	--	--	--	--	--	--	6	3	5	1
			99	--	--	--	--	--	--	--	--	--	--	6	1
		95	90	--	--	--	--	--	--	--	--	6	3	5	1
			95	--	--	--	--	--	--	--	--	7	3	6	1
			99	--	--	--	--	--	--	--	--	--	--	7	1
		99	90	--	--	--	--	--	--	--	--	--	--	6	2
			95	--	--	--	--	--	--	--	--	--	--	6	2
			99	--	--	--	--	--	--	--	--	--	--	7	2

Table 4.2. Best Sampling Schemes (Values of n_S and n_A)[(a)] for Detecting Specified Detectable Differences $D_{\alpha/2,1-\beta}$ (%) (cont'd)

				Required Detectable Differences $D_{\alpha/2,1-\beta}$											
				<5%		<10%		<15%		<25%		<50%		<100%	
%RSD$_S$	%RSD$_A$	100(1-α)[(b)]	100(1-β)[(c)]	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A	n_S	n_A
20	5	90	90	--	--	--	--	--	--	7	3	3	1	2	1
			95	--	--	--	--	--	--	--	--	4	1	2	1
			99	--	--	--	--	--	--	--	--	5	1	3	1
		95	90	--	--	--	--	--	--	--	--	4	1	3	1
			95	--	--	--	--	--	--	--	--	5	1	3	1
			99	--	--	--	--	--	--	--	--	6	1	3	1
		99	90	--	--	--	--	--	--	--	--	6	1	4	1
			95	--	--	--	--	--	--	--	--	7	1	5	1
			99	--	--	--	--	--	--	--	--	--	--	5	1
	10	90	90	--	--	--	--	--	--	--	--	4	1	2	1
			95	--	--	--	--	--	--	--	--	4	1	2	1
			99	--	--	--	--	--	--	--	--	5	1	3	1
		95	90	--	--	--	--	--	--	--	--	5	1	3	1
			95	--	--	--	--	--	--	--	--	5	1	3	1
			99	--	--	--	--	--	--	--	--	6	1	3	1
		99	90	--	--	--	--	--	--	--	--	7	1	5	1
			95	--	--	--	--	--	--	--	--	7	1	5	1
			99	--	--	--	--	--	--	--	--	--	--	5	1
	25	90	90	--	--	--	--	--	--	--	--	5	1	3	1
			95	--	--	--	--	--	--	--	--	6	1	3	1
			99	--	--	--	--	--	--	--	--	6	2	3	1
		95	90	--	--	--	--	--	--	--	--	7	1	3	1
			95	--	--	--	--	--	--	--	--	6	2	4	1
			99	--	--	--	--	--	--	--	--	7	3	4	1
		99	90	--	--	--	--	--	--	--	--	7	3	5	1
			95	--	--	--	--	--	--	--	--	--	--	6	1
			99	--	--	--	--	--	--	--	--	--	--	6	1
	50	90	90	--	--	--	--	--	--	--	--	6	3	4	1
			95	--	--	--	--	--	--	--	--	7	3	5	1
			99	--	--	--	--	--	--	--	--	--	--	6	1
		95	90	--	--	--	--	--	--	--	--	--	--	5	1
			95	--	--	--	--	--	--	--	--	--	--	6	1
			99	--	--	--	--	--	--	--	--	--	--	5	2
		99	90	--	--	--	--	--	--	--	--	--	--	6	2
			95	--	--	--	--	--	--	--	--	--	--	7	2
			99	--	--	--	--	--	--	--	--	--	--	7	3

(a) Combinations of n_S and n_A having the smallest total number of analyses (n_S x n_A) that achieve the specified detectable differences and confidence levels (probabilities of decision errors) are displayed in the body of the table. A dash indicates no combination of $n_S \leq 7$ and $n_A \leq 3$ achieves the desired detectable difference and confidence levels.

(b) 100 (1-α) = percent confidence required that data will *correctly* indicate when the true value *does not* exceed the upper limit or is *not* less than the lower limit.

(c) 100 (1-β) = percent confidence required that data will *correctly* indicate when the true value *does* exceed the upper limit or is *less than* the lower limit by $D_{1-\beta}$.

**Note from Table 4.2 that when restricting attention to $n_S \leq 7$ and $n_A \leq 3$, if $\%RSD_S \geq 10\%$ and $\%RSD_A \geq 10\%$ only one possibility is present for detecting a difference as small as 15%. That possibility involves 7 samples with 3 analyses per sample for the case where $\%RSD_S = 10\%$, $\%RSD_A = 10\%$,
100(1-α) = 90, and 100(1-β) = 90.**

As previously noted, Table 4.2 was based on Equation (4.1) that assumes both lower and upper limits from which differences of specified magnitudes are to be detected. Hence, Table 4.2 is applicable to two-sided detection problems. However, in practice only a lower limit or only an upper limit (a one-sided detection problem) may exist. Although a separate table could be constructed based on Equation (4.2) for this situation, it is possible to use Table 4.2 with a mental adaptation. The mental adaptation is based on Equation (4.2) being the same as Equation (4.1) except for replacing $Z_{1-\alpha/2}$ with $Z_{1-\alpha}$. Thus, when using Table 4.2 for one-sided detection situations, the values of 100(1-α) = 90, 95, and 99 should be mentally replaced with the values 95, 97.5, and 99.5. A separate table of results similar to Table 4.2, but corresponding to Equation (4.2) with 100(1-α) = 90, 95, and 99 for one-sided detection problems will be produced and included in the next revision of this report.

5.0 Sample Size Methods and Preliminary Calculations for Detecting Composition Differences Between the MFPV and MFV

One aspect of the current RPP-WTP Project strategy for qualifying the IHLW and ILAW vitrification processes is to sample and analyze the contents of the MFPV and MFV to verify that differences between the compositions in these two vessels do not exceed permissible percent differences (Objective 19 in Table 2.1). Verifying composition differences are small and are within acceptable bounds during qualification activities will eliminate the need for comparing the compositions of these tanks during production operations. During production operations (as well as during qualification activities), comparison of volume transfers between the MFPV and MFV within applicable uncertainties will demonstrate acceptable transfer of melter feeds.

This section provides information on the number of samples and analyses per sample from each of the MFPV and MFV needed to detect composition differences greater than the specified percentage.

5.1 Method and Formula Used to Determine Numbers of Samples and Analyses per Sample for Detecting Composition Differences Between the MFPV and MFV

The goal is to determine the number of samples and the number of analyses per sample for each of the MFPV and MFV required to decide, with specified confidence, whether the true composition in the MFPV differs from the true composition in the MFV by a specified percentage Δ. The specified Δ is defined as

$$\Delta\,(\%) = 100(\mu_{MFV} - \mu_{MFPV}) / \mu_{MFPV} \qquad (5.1)$$

where μ_{MFPV} **= true composition in the MFPV**
μ_{MFV} **= true composition in the MFV**

Thus, Δ is the percentage difference of the composition in the MFV relative to the composition in the MFPV. The numbers of samples and analyses per sample are to be determined so that a Δ difference in composition between the MFPV and MFV is detected with probability 1-β, and the probability of incorrectly detecting a difference is limited to α.

The detectable difference $\Delta_{\alpha/2,1-\beta}$ that satisfies the required α and 1-β for the two-sided sampling and analysis case is given by

$$\Delta_{\alpha/2,1-\beta}\,(\%) = [2^{1/2}(Z_{1-\alpha/2} + Z_{1-\beta})][(\%RSD_S)^2 + (\%RSD_A)^2 / n_A\,]^{1/2} / [\,n_S - 0.25\,(Z_{1-\alpha/2})^2\,]^{1/2}, \qquad (5.2)$$

where n_S is the number of samples taken from each of the MFPV and MFV, n_A is the number of analyses per sample from each of the MFPV and MFV, $Z_{1-\alpha/2}$ is the $(1-\alpha/2)^{th}$ quantile of the standard normal distribution, and $Z_{1-\beta}$ is the $(1-\beta)^{th}$ quantile of the standard normal distribution. The values of $\%RSD_S$ and $\%RSD_A$ are assumed to be the same for the MFPV and the MFV. Equation (5.2) was derived from Equation (4.36) in Bowen and Bennett (1987, page 164) and the

discussion on page 168. Tabled values of the standard normal distribution (that is, the normal distribution that has mean 0 and standard deviation 1) are found in most statistics textbooks, including Bowen and Bennett (1987, Table A3, page 936). Values of the standard normal distribution are also available in many statistical software packages.

The assumptions underlying Equation (5.2) are: (1) the measurements (results of samples and analyses) are normally distributed, (2) the measurements are independent and representative of the material sampled, and (3) there is no sampling or analytical bias in the data. The assumption of normality will be evaluated during qualification testing and cold commissioning. The assumptions of independence, representative samples, and lack of sampling and analytical bias will be assured by strict adherence to the specified methods for determining where and when to collect samples, mixing the vessels to be sampled, sampling, analytical measurements, and quality control procedures. Testing will also be performed to investigate and demonstrate these assumptions during qualification activities.

No need exists in this situation, as was shown in Section 4.2, to provide a one-sided version of the two-sided sample size formula [Equation (5.2) in this section]. The difference in Equation (5.1) could be negative or positive, so only the two-sided case is addressed here.

5.2 Numbers of Samples and Analyses per Sample for Detecting Differences in Composition Between the MFPV and MFV

Values of $\Delta_{\alpha/2,1-\beta}$ were calculated using Equation (5.2) for the combinations of parameter values in Table 5.1. The values of $\%RSD_S$ and $\%RSD_A$ in Table 5.1 are intended to cover the range of uncertainty magnitudes expected, based on past experience. The values of $\%RSD_S$ and $\%RSD_A$ applicable to the MFPV and MFV in the IHLW and ILAW vitrification plants will be estimated during qualification activities. Calculations of $\Delta_{\alpha/2,1-\beta}$ can be updated in the future as needed to reflect better estimates of $\%RSD_S$ and $\%RSD_A$ for the MFPV and MFV in the IHLW and ILAW vitrification processes.

Table 5.1. Parameter Values Used in Equation (5.2) for Detecting Composition Differences Greater than $\Delta_{\alpha/2,1-\beta}$ (%) in the MFPV and MFV

Parameter [a]	Parameter Values Used
$\%RSD_S = 100\sigma_S/\text{mean}$	2.5% 5% 10% 20%
$\%RSD_A = 100\sigma_A/\text{mean}$	5% 10% 25% 50%
n_S	1 2 3 4 5 6 7
n_A	1 2 3
$100(1-\alpha)$ Confidence	90% 95% 99%
$100(1-\beta)$ Confidence	90% 95% 99%

(a) $\%RSD_S$ = percent relative standard deviation for *samples* in the MFPV and MFV
$\%RSD_A$ = percent relative standard deviation for *analyses* in the MFPV and MFV
σ_S = standard deviation for *samples* from the MFPV and MFV
σ_A = standard deviation for *analyses* in samples from the MFPV and MFV
n_S = number of *samples* from the MFPV and MFV
n_A = number of *analyses* per sample from the MFPV and MFV

$100(1-\alpha)$ = percent confidence required that data correctly indicates when the true percent difference between compositions μ_{MFPV} and μ_{MFV} is less than $\Delta_{1-\beta}\%$

$100(1-\beta)$ = percent confidence required that data correctly indicates when the true percent difference between compositions μ_{MFPV} and μ_{MFV} is greater than $\Delta_{1-\beta}\%$.

In calculating $\Delta_{\alpha/2,1-\beta}$ values, attention was limited to between 1 and 7 samples at a given location, and 1, 2, or 3 analyses per sample, based on the desire to keep sampling and analyses at reasonable levels. However, additional calculations could be performed in the future as desired for more than 7 samples or more than 3 analyses per sample. It should be noted that n_A must be determined separately for each analyte and then the largest n_A across analytes measured by a particular method will be used as the number of analyses per sample for that method.

The calculated values of $\Delta_{\alpha/2,1-\beta}$ were used to construct Table 5.2, as follows. Values of $\Delta_{\alpha/2,1-\beta}$ were calculated for the 4 x 4 x 7 x 3 x 3 x 3 = 3024 combinations of parameter values shown in Table 5.1. Rather than tabulating all of these results in a very long table, the results were summarized as shown in Table 5.2. This table displays the cases with the minimum total number of analyses (that is, the number of samples times the number of analyses per sample, n_S x n_A) needed to achieve detectable differences $\Delta_{\alpha/2,1-\beta}$ of less than 10%, 15%, 25%, 50%, or 100% for the various combinations of $\%RSD_S$, $\%RSD_A$, $100(1 - \alpha)$, and $100(1-\beta)$ in Table 5.1. Cases achieving the same results, but having larger values of n_S x n_A than cases achieving the minimum total number of analyses, are not displayed in Table 5.2.

Substantial parts of the lower left portions of Table 5.2 contain only dashes. The dashes indicate that limiting attention to $n_S \leq 7$ and $n_A \leq 3$ is insufficient to detect the specified difference with the specified probabilities of incorrect decisions for the corresponding values of $\%RSD_S$ and $\%RSD_A$. Calculations for larger values of nS and n_A can be performed as needed if and when it is determined that combinations of uncertainty magnitudes, detectable differences, and incorrect decision probabilities showing dashes in Table 5.2 are relevant to the RPP-WTP situation.

To illustrate the results in Table 5.2, suppose that % RSD_S is 10%, that $\%RSD_A$ is 5%, that we require $100(1-\alpha)$ = 90% confidence (setting $\alpha = 0.10$) that data correctly indicate when the true percent difference between compositions μ_{MFPV} and μ_{MFV} is less than 25%, and that we also require $100(1-\beta)$ = 95% confidence (setting $\beta = 0.05$) that data will correctly indicate when the true percent difference between compositions μ_{MFPV} and μ_{MFV} is greater than 25%. Table 5.2 indicates that 6 samples should be taken from each MFPV and MFV and that each sample should be analyzed once. Hence, a total of 6 x 1 = 6 analyses per vessel are required. If the required $\Delta\%$ is increased from 25% to 50%, the number of samples decreases from 6 to 2 per vessel, still with one analysis per sample.

Table 5.2. Best Sampling Schemes (Values of n_S and n_A)[a] to Determine with Specified Confidence Whether the Compositions in the MFPV and the MFV Differ by a Specified Amount No Greater than $\Delta_{\alpha/2,1-\beta}$ (%)

				Required Detectable Difference, $\Delta_{\alpha/2,1-\beta}$									
				10%		15%		25%		50%		100%	
$\%RSD_S$	$\%RSD_A$	$100(1-\alpha)$[b]	$100(1-\beta)$[c]	n_S	n_A	n_S	n_a	n_s	n_A	n_S	n_A	n_S	n_A
2.5	5	90	90	7	1	4	1	2	1	1	1	1	1
			95	5	2	4	1	2	1	1	1	1	1
			99	7	2	6	1	3	1	2	1	1	1
		95	90	5	2	4	1	3	1	2	1	2	1
			95	6	2	5	1	3	1	2	1	2	1
			99	7	3	7	1	3	1	2	1	2	1
		99	90	6	3	6	1	4	1	3	1	2	1
			95	7	3	7	1	4	1	3	1	2	1
			99	--	--	6	2	5	1	3	1	2	1
	10	90	90	--	--	5	2	4	1	2	1	1	1
			95	--	--	7	2	5	1	2	1	1	1
			99	--	--	7	3	7	1	3	1	2	1
		95	90	--	--	7	2	5	1	2	1	2	1
			95	--	--	6	3	6	1	3	1	2	1
			99	--	--	--	--	5	2	3	1	2	1
		99	90	--	--	7	3	7	1	3	1	2	1
			95	--	--	--	--	5	2	4	1	3	1
			99	--	--	--	--	6	2	4	1	3	1
	25	90	90	--	--	--	--	7	3	6	1	2	1
			95	--	--	--	--	--	--	7	1	3	1
			99	--	--	--	--	--	--	5	2	3	1
		95	90	--	--	--	--	--	--	7	1	3	1
			95	--	--	--	--	--	--	5	2	3	1
			99	--	--	--	--	--	--	6	2	4	1
		99	90	--	--	--	--	--	--	6	2	4	1
			95	--	--	--	--	--	--	7	2	4	1
			99	--	--	--	--	--	--	6	3	5	1
	50	90	90	--	--	--	--	--	--	7	3	5	1
			95	--	--	--	--	--	--	--	--	7	1
			99	--	--	--	--	--	--	--	--	5	2
		95	90	--	--	--	--	--	--	--	--	7	1
			95	--	--	--	--	--	--	--	--	5	2
			99	--	--	--	--	--	--	--	--	6	2
		99	90	--	--	--	--	--	--	--	--	6	2
			95	--	--	--	--	--	--	--	--	7	2
			99	--	--	--	--	--	--	--	--	6	3

Table 5.2. Best Sampling Schemes (Values of n_S and n_A)[(a)] to Determine with Specified Confidence Whether the Compositions in the MFPV and the MFV Differ by a Specified Amount No Greater than $\Delta_{\alpha/2,1-\beta}$ (%) (cont'd)

				Required Detectable Difference, $\Delta_{\alpha/2,1-\beta}$									
				10%		15%		25%		50%		100%	
%RSD$_S$	%RSD$_A$	100(1-α)[(b)]	100(1-β)[(c)]	n_S	n_A	n_S	n_a	n_s	n_A	n_S	n_A	n_S	n_A
5	5	90	90	7	3	5	1	3	1	2	1	1	1
			95	--	--	6	1	3	1	2	1	1	1
			99	--	--	6	2	4	1	2	1	1	1
		95	90	--	--	6	1	3	1	2	1	2	1
			95	--	--	7	1	4	1	2	1	2	1
			99	--	--	7	3	4	1	2	1	2	1
		99	90	--	--	7	2	5	1	3	1	2	1
			95	--	--	7	3	5	1	3	1	2	1
			99	--	--	--	--	6	1	3	1	2	1
	10	90	90	--	--	7	2	5	1	2	1	1	1
			95	--	--	7	3	6	1	2	1	1	1
			99	--	--	--	--	7	1	3	1	2	1
		95	90	--	--	7	3	6	1	3	1	2	1
			95	--	--	--	--	7	1	3	1	2	1
			99	--	--	--	--	6	2	3	1	2	1
		99	90	--	--	--	--	6	2	4	1	3	1
			95	--	--	--	--	6	2	4	1	3	1
			99	--	--	--	--	7	3	5	1	3	1
	25	90	90	--	--	--	--	--	--	6	1	2	1
			95	--	--	--	--	--	--	7	1	3	1
			99	--	--	--	--	--	--	5	2	3	1
		95	90	--	--	--	--	--	--	7	1	3	1
			95	--	--	--	--	--	--	5	2	3	1
			99	--	--	--	--	--	--	6	2	4	1
		99	90	--	--	--	--	--	--	6	2	4	1
			95	--	--	--	--	--	--	7	2	4	1
			99	--	--	--	--	--	--	7	3	5	1
	50	90	90	--	--	--	--	--	--	7	3	6	1
			95	--	--	--	--	--	--	--	--	7	1
			99	--	--	--	--	--	--	--	--	5	2
		95	90	--	--	--	--	--	--	--	--	7	1
			95	--	--	--	--	--	--	--	--	5	2
			99	--	--	--	--	--	--	--	--	6	2
		99	90	--	--	--	--	--	--	--	--	6	2
			95	--	--	--	--	--	--	--	--	7	2
			99	--	--	--	--	--	--	--	--	6	3

Table 5.2. Best Sampling Schemes (Values of n_S and n_A)[a] to Determine with Specified Confidence Whether the Compositions in the MFPV and the MFV Differ by a Specified Amount No Greater than $\Delta_{\alpha/2,1-\beta}$ (%) (cont'd)

				Required Detectable Difference, $\Delta_{\alpha/2,1-\beta}$									
				10%		15%		25%		50%		100%	
%RSD$_S$	%RSD$_A$	100(1-α)[b]	100(1-β)[c]	n_S	n_A	n_S	n_a	n_s	n_A	n_S	n_A	n_S	n_A
10	5	90	90	--	--	--	--	5	1	2	1	1	1
			95	--	--	--	--	6	1	2	1	1	1
			99	--	--	--	--	7	1	3	1	2	1
		95	90	--	--	--	--	6	1	3	1	2	1
			95	--	--	--	--	7	1	3	1	2	1
			99	--	--	--	--	--	--	3	1	2	1
		99	90	--	--	--	--	7	3	4	1	3	1
			95	--	--	--	--	--	--	4	1	3	1
			99	--	--	--	--	--	--	5	1	3	1
	10	90	90	--	--	--	--	7	1	3	1	2	1
			95	--	--	--	--	6	2	3	1	2	1
			99	--	--	--	--	--	--	4	1	2	1
		95	90	--	--	--	--	7	2	3	1	2	1
			95	--	--	--	--	7	3	4	1	2	1
			99	--	--	--	--	--	--	4	1	2	1
		99	90	--	--	--	--	--	--	5	1	3	1
			95	--	--	--	--	--	--	5	1	3	1
			99	--	--	--	--	--	--	6	1	3	1
	25	90	90	--	--	--	--	--	--	6	1	2	1
			95	--	--	--	--	--	--	7	1	3	1
			99	--	--	--	--	--	--	6	2	3	1
		95	90	--	--	--	--	--	--	5	2	3	1
			95	--	--	--	--	--	--	6	2	3	1
			99	--	--	--	--	--	--	6	3	4	1
		99	90	--	--	--	--	--	--	7	2	4	1
			95	--	--	--	--	--	--	7	3	5	1
			99	--	--	--	--	--	--	--	--	6	1
	50	90	90	--	--	--	--	--	--	--	--	6	1
			95	--	--	--	--	--	--	--	--	7	1
			99	--	--	--	--	--	--	--	--	5	2
		95	90	--	--	--	--	--	--	--	--	7	1
			95	--	--	--	--	--	--	--	--	5	2
			99	--	--	--	--	--	--	--	--	6	2
		99	90	--	--	--	--	--	--	--	--	6	2
			95	--	--	--	--	--	--	--	--	7	2
			99	--	--	--	--	--	--	--	--	7	3

Table 5.2. Best Sampling Schemes (Values of n_S and n_A)[(a)] to Determine with Specified Confidence Whether the Compositions in the MFPV and the MFV Differ by a Specified Amount No Greater than $\Delta_{\alpha/2,1-\beta}$ (%) (cont'd)

				Required Detectable Difference, $\Delta_{\alpha/2,1-\beta}$									
				10%		15%		25%		50%		100%	
%RSD$_S$	%RSD$_A$	100(1-α)[(b)]	100(1-β)[(c)]	n_S	n_A	n_S	n_a	n_s	n_A	n_S	n_A	n_S	n_A
20	5	90	90	--	--	--	--	--	--	4	1	2	1
			95	--	--	--	--	--	--	5	1	2	1
			99	--	--	--	--	--	--	7	1	3	1
		95	90	--	--	--	--	--	--	5	1	2	1
			95	--	--	--	--	--	--	6	1	3	1
			99	--	--	--	--	--	--	7	3	3	1
		99	90	--	--	--	--	--	--	7	1	3	1
			95	--	--	--	--	--	--	--	--	4	1
			99	--	--	--	--	--	--	--	--	4	1
	10	90	90	--	--	--	--	--	--	5	1	2	1
			95	--	--	--	--	--	--	6	1	2	1
			99	--	--	--	--	--	--	7	1	3	1
		95	90	--	--	--	--	--	--	6	1	3	1
			95	--	--	--	--	--	--	7	1	3	1
			99	--	--	--	--	--	--	--	--	3	1
		99	90	--	--	--	--	--	--	7	3	4	1
			95	--	--	--	--	--	--	--	--	4	1
			99	--	--	--	--	--	--	--	--	5	1
	25	90	90	--	--	--	--	--	--	6	2	3	1
			95	--	--	--	--	--	--	7	2	3	1
			99	--	--	--	--	--	--	--	--	4	1
		95	90	--	--	--	--	--	--	7	2	4	1
			95	--	--	--	--	--	--	--	--	4	1
			99	--	--	--	--	--	--	--	--	5	1
		99	90	--	--	--	--	--	--	--	--	5	1
			95	--	--	--	--	--	--	--	--	6	1
			99	--	--	--	--	--	--	--	--	7	1
	50	90	90	--	--	--	--	--	--	--	--	6	1
			95	--	--	--	--	--	--	--	--	7	1
			99	--	--	--	--	--	--	--	--	6	2
		95	90	--	--	--	--	--	--	--	--	5	2
			95	--	--	--	--	--	--	--	--	6	2
			99	--	--	--	--	--	--	--	--	6	3
		99	90	--	--	--	--	--	--	--	--	7	2
			95	--	--	--	--	--	--	--	--	7	3
			99	--	--	--	--	--	--	--	--	--	--

(a) Combinations of n_S and n_A having the smallest total number of analyses (n_S x n_A) that achieve the specified detectable differences and confidence levels are displayed in the body of the table. A dash indicates no combination of $n_S \leq 7$ and $n_A \leq 3$ achieves the desired detectable difference and confidence levels.

(b) 100(1-α) = percent confidence required that data correctly indicates when the true percent difference between compositions μ_{MFPV} and μ_{MFV} is less than $\Delta_{1-\beta}$%.

(c) 100(1-β) = percent confidence required that data correctly indicates when the true percent difference between compositions μ_{MFPV} and μ_{MFV} is greater than $\Delta_{1-\beta}$%.

6.0 Future Work

Several estimation, detection, and process monitoring aspects of the HLW and LAW vitrification processes are not addressed in Sections 3, 4, and 5 of this report. The results contained in this report are from initial efforts to develop methods and calculate the numbers of samples, analyses per sample, and measurements of various kinds needed to control the IHLW and ILAW vitrification processes and satisfy strategies for complying with applicable specifications. Work to develop and apply sample size formulas must continue to address the aspects of vitrification processes not addressed in this initial effort. This section briefly describes sample size aspects of the IHLW and ILAW vitrification process objectives that may need to be addressed in the future. Similar to the work presented in Sections 3, 4, and 5, such future work will involve

- developing the statistical methods and corresponding sample size formulas
- performing preliminary sample size calculations for combinations of input parameters spanning the ranges of values expected to be appropriate.

Then, in future years when the values of input parameters are better determined based on qualification activities, sample sizes can be calculated for those specific parameter values.

The objectives discussed in this section may be applicable to process-product control or compliance aspects of the IHLW and ILAW vitrification facilities. Because the process-product control and compliance strategies are not finalized at this time, the objectives discussed in this section should be considered as possibilities that may need revision in the future. Subsections 6.1 to 6.13 of this section are roughly in the order of the objectives listed in Table 2.1 that were not addressed in Sections 3, 4, and 5. Subsection 6.14 briefly discusses process-monitoring objectives, where the focus is on the numbers of samples, analyses, and measurements necessary to detect undesirable trends or results in the IHLW or ILAW vitrification process.

In addition to developing and applying sample size formulas for additional aspects of IHLW and ILAW processes, future efforts must also determine appropriate values of the input parameters for the sample size formulas. Subsection 6.15 discusses this topic.

Subsection 6.16 briefly discusses that environmental and regulatory compliance strategies must also be statistically based, and that formulas for the numbers of samples, analyses, and tests will have to be developed. However, that work is being conducted and documented separately from the work covered in this report.

Finally, Subsection 6.17 mentions the need to identify the statistical data analysis methods that will be applied to data obtained by statistically determined numbers of samples, chemical analyses, and measurements.

6.1 Estimating Levels and/or Volumes of Vessel Contents

During operation of the IHLW and ILAW vitrification facilities, it will be necessary for process-product control purposes and possibly compliance purposes to estimate the levels and/or volumes of the CRV, MFPV, and MFV (Objectives 2 and 14 in Table 2.1). The specific methods to determine levels and/or volumes of the CRV, MFPV, and MFV have not yet been determined. However, one possibility may be to develop, prior to radioactive operations, a calibration

relationship to relate the level of each vessel's contents to its volume. Regardless of the methods to determine CRV, MFPV, and MFV levels and/or volumes, the results will be subject to uncertainties that will need to be considered in process-product control and possibly compliance activities. Hence, the methods to develop vessel calibration equations or otherwise estimate levels or volumes will need to utilize sufficient measurements to provide adequate confidence and precision in estimates of CRV, MFPV, and MFV levels and/or volumes.

The number of measurements needed to estimate the level and/or volume of the CRV, MFPV, and MFV with specified precision and confidence will be determined after the methods to estimate level and/or volume are determined. The relevant uncertainties (in direct measurements, plus those in any calibration equations) will be considered when determining the number of measurements of the level and/or volume that are needed. Initial sample size work will be for ranges of input parameters as in Sections 3, 4, and 5 of this report. Then later, after input parameters are determined, final sample size calculations can be performed.

6.2 Estimating Individual and Combined GFC Weights

Weights of individual and combined GFCs (Objectives 5 and 6 in Table 2.1) are envisioned to play an important role in controlling IHLW and ILAW vitrification processes. The GFC weights may also play an important compliance role, depending on the compliance strategies chosen. Methods and instruments for weighing individual and combined GFCs have not been determined. However, because weighing is a relatively straightforward process subject to less uncertainty than other processes, the objective of estimating GFC weights was not addressed in the initial sample size work summarized in this report.

The number of measurements for an individual GFC hopper or the combined GFCs hopper necessary to estimate the weight with specified precision and confidence can be determined for ranges of weighing uncertainties, precision levels, and confidence levels. Then, after the weighing systems are selected and tested to quantify uncertainties, the number of weight measurements required for given situations can be finalized. Because of the generally low uncertainty in weighing operations, the number of weight measurements per hopper is expected to be small, possibly even as low as one.

6.3 Estimating Combined GFC Compositions

Estimates of combined GFC compositions will be important for process control and may be involved in compliance strategies for the IHLW and/or ILAW vitrification processes (Objective 7 in Table 2.1). Although combined GFC compositions could be determined during production operations by sampling and analysis, it would be a time-consuming and less precise option. Rather, assuming that individual GFC compositions are homogeneous and their compositions are well characterized prior to production operations, weighing operations can be used to determine and verify transfer of correct amounts of individual GFC to the combined GFC hopper. Then, summing the proportional weights of individual GFCs multiplied by their individual compositions yields an estimate of the combined GFCs composition. This estimation process is affected by the uncertainties of the proportional weights and individual GFC compositions. However, those uncertainties are expected to be small compared to the uncertainties of sampling and analyzing combined GFC compositions.

The number of weight measurements of individual and combined GFC hoppers necessary to estimate the combined GFC composition with specified precision and confidence can be determined for ranges of weighing uncertainties, precision levels, and confidence levels. Then, after the weighing systems are selected and tested to quantify uncertainties, the numbers of weight measurements required per hopper can be finalized. Because of the generally low uncertainties in weighing operations, and the expectation that uncertainties in individual GFC compositions will be relatively larger (although still small generally), the number of weight measurements per hopper is expected to be small, possibly even as low as one.

6.4 Detecting a Difference in the Sum of Individual GFC Weights and the Combined GFC Weight

An important aspect of process control, and possibly compliance (depending on the compliance strategies for the IHLW and ILAW vitrification processes), is to detect a difference in the sum of individual GFC weights and the combined GFC weight (Objective 8 in Table 2.1). A statistically significant difference (after accounting for relevant uncertainties) would indicate a problem in transfer of individual GFC to the combined GFC hopper.

The number of weight measurements of individual and combined GFC hoppers necessary to detect a specified difference in weights with specified confidence levels of correct decisions can be determined for ranges of weighing uncertainties, detectable differences, and confidence levels. After the weighing systems are selected and tested to quantify uncertainties, the numbers of required weight measurements can be finalized. Because of the generally low uncertainties in weighing operations, the number of weight measurements per hopper is expected to be small, although it will obviously depend on the magnitude of a difference to be detected.

6.5 Estimating Glass Composition by CRV + GFC Calculation

Estimates of waste compositions in the IHLW and ILAW CRV and estimates of the combined GFC compositions will be used in a mathematical model to estimate the equivalent glass composition (Objective 9 in Table 2.1). In fact, this calculation will be related to the one that determines the amounts of individual GFCs to be added to a CRV batch in the MFPV to obtain the desired equivalent glass composition. The estimates of waste compositions in IHLW and ILAW CRVs will be based on samples and analyses that will be determined using the method and formula discussed in Sections 3.1 and 3.2. The estimates of combined GFC compositions will be based on proportional combinations of individual GFC compositions. Individual GFC compositions will be determined prior to their use, while the proportions of individual GFCs in the combined GFC batch will be determined based on weights of individual and combined GFCs (see Section 6.3). The CRV samples and analyses, and the GFC compositions and weights, will be subject to uncertainty. Other uncertainties may be associated with calculating equivalent glass compositions from slurries. Hence, the uncertainties in the calculated equivalent glass compositions will have to be determined by statistical variance propagation techniques. The magnitudes of the contributing uncertainties will impact the number of measurements (for example, samples, analyses per sample, weight measurements) necessary to estimate (via calculation) the equivalent glass composition with required levels of confidence and precision. Work necessary to determine the required levels of confidence and precision are discussed in Section 6.15.

The mathematical model to calculate the equivalent glass composition based on CRV and GFC compositions has not yet been developed. When the model has been developed, the number of measurements (samples, analyses per sample, weight measurements) required to estimate the equivalent glass composition with specified precision and confidence will be determined. Initial sample size work will be for ranges of input parameters as shown in Sections 3, 4, and 5 of this report. After input parameters are determined, final sample size calculations can be performed.

6.6 Detecting When Calculated (CRV + GFC) Glass Compositions Violate Specified Limits

It is important to detect when the glass compositions calculated from CRV + GFC information violate specified composition limits (Objective 10 in Table 2.1). Such a situation is expected to occur rarely, if at all, in IHLW and ILAW vitrification processes because quantities of individual GFCs will be determined based on CRV composition estimates to yield the desired glass composition. Uncertainties in CRV composition estimates and in GFC weighing and transfer operations will be accounted for in determining the amounts of individual GFCs needed. Then, transfers of CRV contents and GFCs will be monitored statistically to verify they occur as planned. Rarely, if ever, should the calculated glass composition violate specified limits after properly accounting for uncertainties. However, it is important to have a procedure for detecting violations in a calculated composition while there is still time to adjust the composition in the MFPV.

The numbers of CRV samples, analyses per CRV sample, and GFC weight measurements required to detect with acceptable confidence when glass composition limits are exceeded will be determined after the development of the model that will be used to calculate the CRV + GFC compositions. Initial sample size work will be for ranges of input parameters, as in Sections 3, 4, and 5 of this report. After input parameters are determined, final sample size calculations can be performed.

6.7 Estimating (Predicting) Glass Properties Based on Calculated (CRV + GFC) Glass Compositions

As part of process-product control and possibly of compliance activities during production operations, it will be necessary to estimate glass processing and glass quality properties by applying property-composition models to calculated CRV +GFC glass compositions (Objective 11 in Table 2.1). Sample size aspects of estimating glass composition based on CRV + GFC calculations were discussed in Section 6.5. Sample size formulas and calculations for predicting glass properties based on such composition estimates are affected by the uncertainties affecting the glass composition estimation, as well as the uncertainties associated with the property-composition models.

The numbers of CRV samples, analyses per CRV sample, individual and combined GFC weight measurements, and data points to develop property-composition models that are necessary to estimate (or predict) glass property values with specified levels of precision and confidence will be determined after the method for calculating CRV + GFC composition and the approach to modeling glass properties are determined. Initial sample size work will be for ranges of input parameters as in Sections 3, 4, and 5 of this report. After input parameters are determined, final sample size calculations can be performed.

6.8 Detecting When Predicted Properties of Calculated (CRV + GFC) Glass Compositions Violate Specified Limits

Property-composition models will be used with calculated CRV + GFC glass compositions to verify acceptability of glass properties (Objective 12 in Table 2.1). Property predictions will be made for processing properties such as viscosity, and product quality properties such as the Product Consistency Test (PCT) or the Vapor Hydration Test (VHT). The true properties must lie within specified lower and/or upper limits, after accounting for uncertainties. These uncertainties will include all the uncertainties affecting the CRV + GFC calculation of glass composition (see Section 6.5), as well as the uncertainty associated with a given property-composition model.

The numbers of CRV samples, analyses per CRV sample, individual and combined GFC weight measurements, and data points to develop a given property-composition model that provide for detecting or not detecting with specified confidence levels when property values lie outside the specified lower and/or upper limits will be determined after the property-composition model development approach is selected and the CRV + GFC glass composition calculation model is developed. Initial sample size work will be for ranges of input parameters as in Sections 3, 4, and 5 of this report. Then later, after input parameters are determined, final sample size calculations can be performed.

6.9 Predicting Glass Properties for Glass Composition Estimates Based on MFPV, MFV, or Glass Samples

Depending on the process-product control and compliance strategies selected by the RPP-WTP Project, it may be necessary to estimate certain glass properties with specified precision and confidence (Objectives 16 and 22 in Table 2.1). Estimates (predictions) of glass properties would be based on applying property-composition models to estimates of glass composition. Objectives 16 and 22 are similar to Objective 11, except that each one has a different basis for estimating glass composition. Thus, the natures and magnitudes of uncertainties in the estimated glass composition may differ, resulting in different required numbers of sample sizes for each objective (for example, numbers of samples and analyses per sample for this objective).

The numbers of samples and analyses per sample (from the MFPV, MFV, or produced glass), and the number of data points to develop a given property-composition model, required to estimate (predict) glass property values with specified levels of precision and confidence will be determined after the approach to modeling glass properties is decided. Initial sample size work will include ranges of input parameters as in Sections 3, 4, and 5 of this report. After input parameters are determined, final sample size calculations can be performed.

6.10 Detecting When Predicted Properties for Glass Composition Estimates Based on MFPV, MFV, or Glass Samples Violate Specified Limits

Depending on the process-product control and compliance strategies selected by the RPP-WTP Project, it may be necessary to detect or fail to detect with specified confidence levels when certain glass properties violate specified limits (Objectives 17 and 23 in Table 2.1). These objectives are similar to Objective 12 discussed in Section 6.8. However, glass composition estimates for Objective 12 are based on calculations with CRV + GFC information, whereas glass compositions for Objectives 17 and 23 are based on MFPV, MFV, or glass samples and analyses.

The numbers of samples (MFPV, MFV, or glass), analyses per sample, and data points to develop a given property-composition model that provide for detecting or not detecting, with specified confidence levels, when property values lie outside the specified lower and/or upper limits will be determined after the property-composition model development approach is selected. Initial sample size work will be for ranges of input parameters as in Sections 3, 4, and 5 of this report. After input parameters are determined, final sample size calculations can be performed.

6.11 Detecting an Important Difference Between a Measured MFPV Composition and a Calculated CRV + GFC Composition

The strategies for satisfying compliance specifications and for controlling the IHLW and ILAW processes have not been completely determined. However, it is expected qualification and/or cold commissioning activities will need to compare composition estimates based on samples and analyses from the MFPV and based on CRV + GFC calculations (Objective 18 in Table 2.1). The numbers of samples and analyses per sample from the MFPV, and weight measurements of GFCs, should be sufficient to detect with confidence when the measured MFPV composition is different to an important degree from the calculated CRV + GFC composition.

The numbers of CRV samples, analyses per CRV sample, MFPV samples, analyses per MFPV sample, and GFC weight measurements required to detect with acceptable confidence specified detectable differences between calculated CRV + GFC compositions and measured MFPV compositions will be determined after the model to be used to calculate the CRV + GFC compositions has been developed. Initial sample size work will be for ranges of input parameters, as discussed in Sections 3, 4, and 5 of this report. After input parameters are determined, final sample size calculations can be performed.

6.12 Detecting Important Differences in Volume Transfers Between the MFPV and the MFV

During operation of the IHLW and ILAW vitrification facilities, it will be necessary for process control, and possibly for specification compliance purposes, to detect differences in volumes transferred between the two tanks (Objective 20 in Table 2.1). The specific methods to determine levels, volumes, and volume transfers of the MFPV and MFV have not yet been determined, as discussed in Section 6.1. Further, the methods used to compare levels, volumes, and volume transfers in the MFPV and MFV have not yet been determined. Regardless of the methods to determine and compare MFPV and MFV levels, volumes, and volume transfers, the results will be subject to uncertainties. These uncertainties will need to be accounted for in statistically comparing the results as part of process control and possibly specification compliance activities. The methods to develop vessel calibration equations or otherwise estimate, and then compare, MFPV and MFV volume transfers will need to utilize sufficient measurements to provide adequate levels of decision errors for detecting specified differences in MFPV and MFV volume transfers.

The number of measurements that will be needed to detect specified detectable differences in volume transfers between the MFPV and MFV with specified decision error probabilities will be

determined after the methods to estimate and compare vessel levels, volumes, and volume transfers are determined. The relevant uncertainties (in direct measurements, plus those in any calibration equations) will be considered when determining the number of measurements of the level and/or volume that are needed to determine volume transfers. Initial sample size work will be for ranges of input parameters as in Sections 3, 4, and 5 of this report. After input parameters are determined, final sample size calculations can be performed.

6.13 Detecting if Measured PCT or VHT Performance is Unacceptable

Because of the time, expense, and additional personnel exposure, the RPP-WTP Project does not currently envision direct PCT (for IHLW and ILAW) and VHT (for ILAW) testing of glass samples (from the pour stream, canisters, or vitrified samples from the MFPV or MFV) as the primary strategy for meeting PCT and VHT compliance requirements during radioactive operations. Rather, the primary strategy is to obtain and chemically analyze glass or vitrified slurry samples, predict PCT and VHT performance using property-composition models, and then demonstrate compliance using the predicted PCT and VHT values (Objectives 16 and 17 or 22 and 23 in Table 2.1). However, PCT and VHT testing of glass samples may be performed as part of qualification activities and cold commissioning, where the objective would be to collect sufficient samples and perform enough PCT and VHT tests per sample to detect specified magnitudes of unacceptable PCT and VHT performance with specified probabilities of incorrect decisions (Objective 24 in Table 2.1).

The number of MFPV, MFV, or glass samples from a given vessel or canister and the number of PCT and VHT tests per vitrified sample needed to detect specified magnitudes of unacceptable PCT and VHT performance with specified decision error probabilities can be determined early in future work, if desired. Sufficient information concerning appropriate ranges of sampling and PCT and VHT testing/analysis uncertainties exists to calculate numbers of samples and PCT and VHT tests per sample for various combinations of uncertainty levels, detectable differences, and probabilities of incorrect decisions. After input parameters are determined, final sample size calculations can be performed.

6.14 Process Monitoring Objectives

As discussed briefly in Section 2.2, certain process monitoring objectives will require collecting samples, analyzing samples, and making measurements while the IHLW or ILAW process is operating. The goal in such cases is to ensure the process operates within desired limits, and to detect any departures, or trends toward departure, from desired operational limits. Sample size formulas and calculations for such situations focus on data needs over time, not at a particular point in time, as is the focus in most of this report. Future sample size work will need to address process-monitoring objectives after the statistical and other methods to monitor the IHLW and ILAW processes over time are developed.

6.15 Developing Input Parameters for Sample Size Calculations

As discussed and illustrated in Sections 3, 4, and 5 of this report, formulas to calculate the required numbers of samples, analyses per sample, and measurements of various kinds depend on various input parameters. These parameters include uncertainties (for example, sampling,

analytical, measurement, and model), the precision with which a quantity is to be estimated, the difference to be detected in a comparison (to a specified limit or between two quantities), and confidence levels.

The RPP-WTP Project has planned qualification activities to estimate sampling, analysis, and measurement uncertainties (CHG 2001a and CHG 2001b). These uncertainties may or may not depend on the location in the vitrification process (for example, FCP, CRV, MFPV, MFV, glass samples), and whether it is the IHLW or ILAW vitrification process. Qualification activities also must be performed to determine required: (i) precisions for estimation objectives, (ii) detectable differences for detection objectives, (iii) confidence levels for estimation and detection objectives, and (iv) performance characteristics for statistical process monitoring methods (for example, control charts). The requirements will depend on the objectives and the consequences of incorrect decisions or estimates. In compliance situations, confidence levels of at least 95% may be required.

6.16 Detect if Regulatory Compliance Achieved

There is also a need for environmental samples for regulatory compliance to predict plant emissions and demonstrate compliance with Land Disposal Restriction (LDR) and delisting criteria. The number of samples and chemical analyses or tests needed for these purposes will also be statistically based. However, these specific regulatory needs are not addressed in this document as those aspects of the compliance strategy are being planned, carried out, and documented separately. Still, the statistical aspects of estimation and detection objectives in the regulatory compliance arena may be the same or very similar to those objectives covered in this report. To avoid duplication of effort, the RPP-WTP Project work addressing the regulatory compliance objectives should make use of, or refer to, relevant parts of the work documented in this report.

6.17 Data Analyses After Sampling, Analyses, and Measurements

This report focuses on developing sample size formulas for various process and product steps in the RPP-WTP HLW or LAW vitrification plants, or for various qualification activities. However, other important concerns are:

A. Approaches to update (during production operations) estimates of process and product uncertainties that are inputs to sample size formulas

B. Approaches to verify that sampling, chemical analysis, measurement, and testing instruments and methods yield unbiased results during production operations, and if necessary to bias-correct the results or re-do the sampling, analysis, and measurement or testing

C. Statistical data analysis methods that will be applied to the resulting samples, chemical analyses, or measurements to control the process or demonstrate compliance for IHLW and ILAW.

Item A is important from the standpoint that process and product uncertainties may not remain the same during production operations as they were during qualification activities, cold commissioning, and hot commissioning. The uncertainties could become larger as equipment wears, while uncertainties could become smaller as better equipment or methods become available as production operations progress. Uncertainties could be smaller or larger for different waste

types that the IHLW and ILAW plants will process. It is beyond the scope covered in this report to address this topic. However, the topic is identified here as a reminder that it needs to be addressed as part of the appropriate RPP-WTP Project scope.

Item B is important because the statistical sample size formulas developed in this report assume that sampling, chemical analysis, and measurement methods will yield unbiased results. Further, any data analysis methods applied to resulting data will also need to assume that data are unbiased. The RPP-WTP Project has qualification activities planned to demonstrate that sampling methods are unbiased (CHG 2001a and CHG 2001b), but analytical and other measurement methods must also be demonstrated as unbiased during qualification activities. Still, it may be necessary to develop (as part of qualification activities) methods for detecting biases using representative, certified standards. Because a bias may be detected, methods must be developed to correct the bias, or to decide to repeat the sample, analysis, or measurement. In the case of chemical analyses of compositions, a weighted least squares method to adjust the analyzed component proportions (unbiased or bias-corrected) to sum to one is recommended for development by the RPP-WTP Project. This method is outlined by Deming (1964) and illustrated for a simple example by Mandel (1964), but requires development for application to waste glasses. This adjustment method will improve the precision of the estimated composition (Deming 1964) and avoid inducing biases in predictions made with property-composition models (which will be developed using statistical mixture experiment methods that assume component proportions of compositions sum to one). While all of these needs are important, they are beyond the scope of work contained in this report. Work addressing these needs will have to be documented in separate reports.

Item C is a very important, in that statistical methods play an important role many aspects of the RPP-WTP Project's compliance strategies for controlling the IHLW and ILAW processes and demonstrating compliance with WAPS (IHLW) and Contract (IHLW and ILAW) requirements. Statistical confidence interval and hypothesis testing methods underlie the sample size formulas presented in Sections 3, 4, and 5. However, it is somewhat premature, as well as beyond the scope of this report, to describe and illustrate the details of these statistical methods at various process and product steps of the IHLW and ILAW plants. The statistical data analysis methods to be employed will depend on the details of the process control and compliance strategies, which have not yet been fully determined by the RPP-WTP Project. When those details and statistical methods are determined, they will be documented in other reports. At that time, changes to sample size formulas may be required in a future revision of this report.

7.0 Conclusions and Recommendations

This report presents the results from initial work to develop and apply statistical sample size formulas to calculate the required numbers of samples, analyses per sample, and measurements to achieve various process-product control and compliance objectives in the IHLW and ILAW vitrification processes. The work and results are preliminary because

- **The details of the RPP-WTP Project's process control and compliance strategies for IHLW and ILAW have not been fully determined yet.**
- **Only a fraction of possible estimation and detection objectives have been addressed.**
- **No process monitoring objectives have been addressed yet.**
- **Input parameters (such as required levels of precision, confidence, and magnitudes of uncertainties) have not yet been determined.**

To compensate for the last bullet, tables of sample size calculations were developed to present results for ranges of input parameters where the actual values are expected to fall. The sample size tables are useful tools for

- **Planning for possible sampling, analysis, and measurement requirements and costs.**
- **Helping assess the advantages and disadvantages of reducing uncertainties, changing required precision levels or detectable differences, and changing required confidence levels.**

These bullets address one of the most useful (but under-utilized) roles of statistical sample size work. This role includes being aware of the costs and benefits that various options provide for making informed choices and investing effort where the benefit is the highest.

Although the sample size work and calculations summarized in this report are preliminary, several conclusions and recommendations can be made:

1. **Estimating quantities with high precision and/or high confidence will require larger numbers of samples, analyses, and measurements. Depending on the magnitudes of relevant uncertainties and desired precision and confidence levels, it may (or may not) be possible to achieve desired goals with the restrictions of $n_S \leq 7$ and $n_A \leq 3$ considered in this report.**

2. **Detecting small differences (between a quantity and applicable lower or upper limits, or between two quantities) with small probabilities of incorrect decisions will require larger numbers of samples, analyses, and measurements. Depending on the magnitudes of relevant uncertainties and desired detectable differences and probabilities of incorrect decisions, it may not be possible to achieve the desired goals with the restrictions of $n_S \leq 7$ and $n_A \leq 3$ considered in this report.**

3. **Work to estimate uncertainties must be planned and started to provide needed inputs for future sample size calculations and decisions. Estimates of uncertainties will also play important roles in process-product monitoring and control algorithms and must be well determined during qualification activities. Qualification activities need to be planned with statistical input so that data to estimate uncertainties are obtained as part of the activities.**

4. **Work to determine levels of precision and detectable differences required to meet compliance and process-product control objectives must progress to provide needed inputs for future**

sample size calculations and decisions. Generally, trade-offs must be made between required precision levels or detectable differences and associated confidence levels. Higher confidence levels are desired, but can be compensated for by less stringent requirements on precision or detectable differences to keep sample size requirements reasonable. For example, in detection situations where the quantity of interest (e.g., a regulatory constituent or the PCT boron release of a waste glass) is significantly below its specified limit, it is not necessary to specify a small detectable difference that would require a larger sample size. Rather, a larger detectable difference can be specified, which would require a smaller sample size. As a specific example, consider a case where a quantity is expected to have a value one-tenth of the limiting value, which is a 900% relative difference. Rather than specifying a detectable difference of 25% (which might be required for other quantities), a larger detectable difference of 100% or even higher could be specified, thus resulting in a smaller required sample size. Such an approach can still provide adequate protection with high confidence, and conserve resources for situations where they are more useful.

5. **Work to determine required and defensible confidence levels for estimation objectives and required probabilities of incorrect decisions (equivalently, confidence levels for correct decisions) must progress to provide needed inputs for future sample size calculations and decisions. To be defensible and publicly acceptable, confidence levels for compliance purposes probably must be at least 95%. However, for process-product control needs not related to critical quantities, lower confidence levels (such as 90%) may be sufficient.**

Pending work to finalize compliance strategies, estimate relevant uncertainties, and determine required precision levels, magnitudes of detectable differences, and confidence levels, sample size development work (similar to that presented in this report) should continue for the many objectives not yet addressed (as discussed in Section 6). As noted at the start of this section, such work will provide for making cost/benefit decisions and choosing precision levels, detectable differences, and confidence levels that are both reasonable and defensible.

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