APPENDIX C6
STATISTICAL METHODS USED IN SAMPLING AND ANALYSIS

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C6-1 Approach for Statistically Selecting Waste Containers for Visual Inspection

As a Quality Control check on radiography, a statistically selected portion of the certified waste containers must be opened and visually examined. The data from visual examination must be used to verify the matrix parameter category and waste material parameter weights as determined by radiography.

The data obtained from the visual examination must also be used to determine, with acceptable confidence, the percentage of miscertified waste containers. Miscertified containers are those that radiography indicates meet the Waste Isolation Pilot Plant Waste Acceptance Criteria and Transuranic Package Transporter-II Authorized Methods for Payload Control but visual examination indicates do not meet these criteria.

Experience at the Idaho National Engineering Laboratory indicates that two-percent of the radiography-certified waste containers have been miscertified when compared to the results of visual examination (EG&G 1994). Participating sites must use this historical miscertification rate to calculate the number of waste containers that must be visually examined. The miscertification rate must be determined each year based on results of certification activities over a minimum of 12 months. Table C6-1 provides the number of waste containers that must be visually examined for several miscertification rates and waste container population sizes.

Table C6-1 has been developed with the use of an EG&G Idaho, Inc. engineering design file (EG&G 1994). The number of waste containers requiring visual examination will ensure the program is 80-percent confident that if the true miscertification rate is the same as the percent column heading of Table C6-1, and if the indicated number of waste containers is examined, the Upper Confidence Level of 90 percent (UCL_{90}) of the miscertification percentage will be less than 14 percent (i.e., there is only a 10-percent chance that the miscertification rate is greater than 14 percent). If the number of containers listed in Table C6-1 are visually examined, it is simply guaranteed that the UCL_{90} of the miscertification percentage will be less than 14 percent; 14 percent is a worst case. In actuality, when UCL_{90}s have been calculated from sample data, most of them will be much smaller than 14 percent.

For the hypergeometric approach to determining the number of containers to be visually examined, the acceptable level of uncertainty is the estimate of the proportion miscertified (along with the information on the previous percentage miscertified) determines the number of containers that must be examined. The rationale and details of this methodology are discussed below.

In a population of size N, there are M miscertified containers, so the true proportion of the miscertified containers in the population is $M/N = p_{true}$. Since p_{true} (or M) is not known, we wish to estimate it by randomly sampling some of the containers. If in a sample of n containers, x are found to be miscertified, the sample estimate ($\hat{p}$) of the true population proportion p_{true} is:

$$\hat{p} = \frac{x}{n} \qquad \text{(C6-1)}$$

This value is only an estimate, and as such has some uncertainty associated with it. This uncertainty is quantified by calculating the upper one-sided $(1-\alpha)$ percent confidence limit for p, defined as p_{UCL}. This confidence limit gives the largest value the true proportion could take on and still have a "reasonable" chance (e.g., an $\alpha = 0.10$ probability) of producing x miscertified containers in a sample of n out of N. This upper confidence limit is calculated as:

$$p_{UCL} = \frac{M_{UCL}}{N} \qquad \text{(C6-2)}$$

where M_{UCL} is the largest value of M such that the probability of observing x or fewer miscertified containers in a sample of size n is less than or equal to α. That is, it is the largest value of M such that the following inequality is true:

$$\sum_{k=0}^{x} \frac{\binom{M}{k}\binom{N-M}{n-k}}{\binom{N}{n}} \leq \alpha \qquad \text{(C6-3)}$$

where each term in parentheses has the usual combinatorial interpretation. For example:

$$\binom{M}{k} = \frac{M!}{k!\,(M-k)!} \qquad \text{(C6-4)}$$

Each term in the sum in Equation C6-3 is the hypogeometric probability of observing k miscertified containers in a sample size n from a population of size N in which there are M miscertified containers (and hence the population proportion of miscertified containers is $p = M/N$). The value M_{UCL} is obtained by substituting different values for M into Equation C6-3 until the largest value satisfying the inequality is found.

Note that in Equation C6-3, the upper confidence limit is dependent on x, the number of miscertifications observed in the sample, as well as on n, the sample size. So, to obtain the required sample size, the values of x that are likely to be seen also need to be considered. Sample size is thus determined by setting a desired upper confidence limit value and then manipulating x and n in Equation C6-3.

C6-2 Approach for Statistically Selecting Waste Containers for Totals Analysis

The statistical approach for characterizing retrievably stored homogeneous solids and soil/gravel waste using sampling and analysis relies on acceptable knowledge to segregate waste containers into relatively homogeneous waste streams. In this way it is reasonable to classify as hazardous or nonhazardous the entire waste stream rather than individual waste containers. Individual waste containers serve as convenient units for characterizing the combined mass of waste from the waste stream of interest. Once segregated by waste stream, random selection and sampling of the waste containers followed by analysis of the waste samples must be performed to ensure that the resulting mean contaminant concentration provides an unbiased representation of the true mean contaminant concentration for each waste stream. The site project manager shall verify that the samples collected from within a waste stream were selected randomly.

The end use of analytical results for retrievably stored homogeneous solids and soil/gravel is for determining the Environmental Protection Agency hazardous waste D-codes that apply to each waste stream. The D-codes are indicators that the waste exhibits the toxicity characteristic for specific contaminants under the Resource Conservation and Recovery Act (RCRA). The RCRA-toxicity determination is made on the basis of sampling and analysis of waste streams and on whether or not the waste stream carries F-codes. If a waste stream carries one or more RCRA F-codes, toxicity characteristic contaminants associated with the F-codes are not included in the RCRA-toxicity characteristic determination. That is, the F-codes take precedence over RCRA-toxicity D-code. Therefore, toxicity characteristics contaminants associated with F-codes(s) for a waste stream must be omitted from all calculations for determining the number of containers to sample. In addition, each toxicity characteristic contaminant associated with the F-code(s) must be excluded from evaluation of analytical results to determine D-codes. Contaminants of interest for the sampling, analysis, and RCRA-toxicity determination of a waste stream, then, excludes contaminants associated with F-codes that have been assigned to the waste stream.

The sampling and analysis strategy is illustrated in Figure C6-1. Preliminary estimates of the mean concentration and variance of each RCRA regulated contaminant in the waste will be used to determine the number of waste containers to select for sampling and analysis. The preliminary estimates will be made by obtaining a preliminary sample from the waste stream or from previous sampling from the waste stream. The applicability of the preliminary estimates to the waste stream to be sampled must be justified and documented. The estimates will be determined in accordance with the following equations:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i \qquad \text{(C6-5)}$$

$$s^2 = \frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2 \qquad \text{(C6-6)}$$

where $\bar{x}$ is the calculated mean and s^2 is the calculated concentration variance, n is the number of samples analyzed, x_i is the concentration determined in the *ith* sample, and *I* is an index from 1 to n.

The ratio of the standard deviation, s, to the mean is called the coefficient of variation (CV); preliminary estimates for CV must be calculated for all contaminants of interest which are described above. The highest CV will be used in determining the number of samples to collect and analyze. Analysis results will then be summarized on a contaminant-specific basis. The calculations for the number of samples to collect and calculations for analysis summaries are described in subsequent paragraphs.

The preliminary estimated concentration mean and associated variance for the contamination with the highest CV among all contaminants of interest must then be used to calculate the number of samples required, n, in accordance with the procedure described in Cochran (1977). As a first approximation, take

$$n_0 = \frac{s^2}{\bar{x}^2 c} \qquad \text{(C6-7)}$$

where s^2 and $\bar{x}$ are the preliminary estimates for the variance and the mean, and

$$c = \frac{r^2}{t^2_{\alpha,n_0-1}} \qquad \text{(C6-8)}$$

where $t_{\alpha,n0-1}$ is the 90th percentile for a t distribution with n_0-1 degrees of freedom. The parameter r is taken as 1.0, which represents a relative error of 100 percent. This choice of r is made in order to obtain the Type I and Type II error rates. This reduces Equation C6-7 to

$$n_0 = \frac{t^2_{\alpha,n_0-1} s^2}{\bar{x}^2} \qquad \text{(C6-9)}$$

Because $t_{\alpha,n0-1}$ is dependent on n_0, the calculation procedure is iterative. If the ratio of n_0 to the number of containers in the waste stream, N, is appreciable, the number of samples required may be reduced to

$$n = \frac{n_0}{1 + \frac{n_0}{N}} \qquad \text{(C6-10)}$$

The effect of the ratio n_0/N on n in Equation C6-10 depends on n_0. Equation C6-10 should be used for cases where it results in a different number of samples from n_0. All calculations should be rounded up to the nearest integer. A minimum of five containers must be sampled and analyzed in each waste stream. If there are fewer than the minimum or required number of containers in a waste stream, one or more containers must be sampled more than once to obtain the samples of the waste. Otherwise any one container may be selected for sampling only once.

The calculated number of required waste containers will then be randomly sampled and analyzed. If waste container samples for the preliminary mean and variance estimates were randomly collected from the same waste stream lot being examined and were collected and analyzed in the manner required for characterization samples, then these samples may be counted toward meeting the required number. The number of waste containers that must be sampled is dependent on defined levels of acceptable error for the hazardous versus nonhazardous determination, as described below.

C6-3 Upper Confidence Limit

Upon completion of the required sampling, final mean and variance estimates and the UCL_{90} for the mean concentration for each contaminant must be determined. The UCL_{90} for the mean concentration of each contaminant will be calculated in accordance with the following equation:

$$UCL_{90} = \bar{x} + \frac{t_{\alpha,n-1}s}{\sqrt{n}} \qquad \text{(C6-11)}$$

The observed sample CV must be checked against the preliminary estimate for CV used in determining the number of samples to be collected before proceeding. If the observed sample CV is greater than the preliminary estimate for CV, the required number of samples must be recomputed using the observed CV. If the observed sample CV estimate results in greater than 20 percent more required samples, then additional sampling and analysis must occur. Once sufficient sampling and analysis has occurred, the determination of whether the waste stream is RCRA-hazardous or nonhazardous will proceed. The determination will be made with 90 percent confidence. If the UCL_{90} for the mean concentration is less than the regulatory threshold limit, the waste stream will be classified as nonhazardous for this contaminant. If the UCL_{90} is greater than or equal to the regulatory threshold limit, the waste stream will be classified as hazardous for this contaminant.

C6-4 Justification for Use of the 90 Percent Confidence Limit for RCRA Characterization of Transuranic Waste

A 90 percent confidence limit was determined to be adequate and appropriate for the following reasons:

- The hazardous waste determination is made based on "Total Analysis" of waste as opposed to the Toxicity Characteristic Leaching Procedure. This approach uses the regulatory limits that incorporate assumptions that samples are 100 percent solid (i.e., no moisture content) and contaminants are 100 percent soluble in the extraction fluid. "Total Analysis" also uses a more aggressive extraction procedure (i.e., stronger acids) which more completely solubilize contaminants. This is an inherently conservative approach and waste analyzed by "Total Analysis" methods.

- Regardless of the hazardous or non-hazardous determination associated with any particular waste stream or individual waste container, all contact-handled waste will be handled in the same manner during transportation to and emplacement a the WIPP. All waste will be handled, stored, and disposed in a way that meets the requirements for hazardous waste.

- Waste will not be segregated on the basis of a RCRA hazardous determination for the purpose of handling or disposal, so safety related issues are not a concern for "mis-designated" waste. Emergency response personnel will be adequately equipped to mitigate health and safety concerns associated with hazardous waste.

Simulated hazardous waste determinations using both the 90 percent and 95 percent confidence limits indicate an approximate 60 percent increase in sampling and analysis costs to achieve a 95-percent confidence limit. The Department of Energy considered the consequence of increasing the confidence level from 90 to 95 percent for determining whether or not waste streams exhibit the toxicity characteristic. Calculations show that the number of containers to sample increases from 57 to 70 percent with the increase in confidence.

Calculations used Equation C6-9, which pertains to sampling of retrievably stored homogeneous solids and soil/gravel waste. The factor that changes with confidence level is $t^2_{\alpha, n-1}$, which also changes with the calculated number of containers to sample (n). Therefore, this factor is used to calculate the percent increase in n, for different possible values of n. The finite population factor (Equation C6-10) is not used in these calculations; however, it is not expected to significantly affect the results.

The results are given in Table C6-2. For the values of n between 5 and 500 at 90 percent confidence, increases the range from 57 to 70 percent. The values of n for both 90 and 95 percent confidence are given in the table so the number of containers required to be sampled can be directly compared. Because of the formulation for calculating the required number of containers to sample, the tabled values do not depend on the number of containers in the waste stream. The number of containers to sample reflects variabilities of constituent concentrations observed in the waste stream; in particular, it reflects the maximum ratio of standard deviation to mean concentration, which is the maximum coefficient of variation. For example, for a waste stream of any size that has a coefficient of variation of 0.918, the required number of containers to sample increases 60 percent from 5 for 90 percent confidence to 8 for 95 percent confidence.

C6-5 Control Charting for Newly Generated Waste Stream Sampling

Significant process changes and process fluctuations can be determined using statistical process control (SPC) charting techniques; these techniques require historical data for determining limits for indicator species, and subsequent periodic sampling to assess process behavior relative to historical limits. SPC may be performed on waste prior to solidification or packaging for ease of sampling. If the limits are exceeded, the waste stream must be recharacterized, and the characterization must be performed according to procedures required in the Transuranic Waste Characterization Quality Assurance Program Plan for retrievably stored waste.

A Shewhart control chart (Gilbert, 1987) is a control chart for means that can be used for checking whether current data are consistent with past data and whether shifts or trends in means have occurred. The control chart for means is constructed of a center line and upper and lower control limits that are based on the mean and standard deviation of historical data for the process. If a current sample mean from the process lies within the limits, the process is said to be "in control", or consistent with historical data. If the current mean exceeds the limits, the process has likely changed from historical periods.

Logical sets of historical data to be used for the construction of limits in this application are the data from the initial characterization of the waste stream, if available, from characterization of a different lot of the waste stream, or from a retrievably stored waste stream of the same type from the same process. The data used for construction of the limits must be justified. The underlying assumptions for control charts are that the data are independent and normally distributed with constant mean μ and constant variance σ^2. The statistical tests for normality must be conducted and data transformation to normality performed, if necessary. Transformations must take place prior to any calculations that use the data.

Each limit will be constructed such that there is a 90 percent confidence that the true mean does not exceed a limit. One-sided control limits are used because once a waste stream has been determined to be RCRA-hazardous, the limit exceedence of interest is on the lower side; that is when the process may become nonhazardous. Likewise, once a waste stream has been determined to be RCRA-hazardous, the limit exceedence of interest is on the upper side; that is when the process may become RCRA-hazardous. Whether or not exceeding the limit would result in a change in the RCRA-hazardous nature of the waste stream depends on how close the limits are to RCRA limits.

Current process data will be collected and averaged for comparison to the control limit for the mean. The collection period and number of samples to be included in the average are dependent on the waste stream characteristics. A small number of samples will reflect more of the process variability and there will potentially be more limit exceedence. If two or three samples are collected for the mean in the required annual (or batch) sampling of a relatively homogeneous waste stream, limit exceedences may not occur. If the waste stream is more variable, it will be necessary to collect more samples to meet the required confidence limit.

Periodically it will be necessary to update the control limit for a process. An update is performed that includes all historical data if there is no evidence of a trend in the process or a shift in the mean for the process. If there has been a shift in the mean, only more recent data that reflects the shift is used. In general, control limits should be based on at least ten data points that are representative of the process and do not exhibit outliers or a trend with time.

REFERENCES

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TABLES

TABLE C6-1
NUMBER OF WASTE CONTAINERS REQUIRING VISUAL EXAMINATION

Annual Number of Waste Containers Undergoing Characterization	Number of Waste Containers Requiring Visual Examination Based on Percent of Waste Containers Miscertified to WIPP-WAC by Radiography in Previous Year(s)					
	1%	2%	3%	4%	5%	6%
50	22[a]	22	22[a]	22	29[a]	29
100	15	24	24	33	33	41
200	15	26	26	35	44	52
300	15	26	26	35	44	53
400	15	26	26	36	45	62
500	16	26	26	36	45	63

[a]Number of containers for the higher even-number percent of miscertified containers is used because an odd percent implies a noninteger number of containers are likely to be miscertified.

TABLE C6-2

PERCENT INCREASE IN NUMBER OF CONTAINERS (*n*) TO SAMPLE WITH INCREASE IN CONFIDENCE FROM 90 PERCENT TO 95 PERCENT

n for 90 Percent Confidence	n for 95 Percent Confidence	Approximate Percent Increase
5	8	60.00
6	10	66.67
7	11	57.14
8	13	62.50
9	15	66.67
10	17	70.00
25	41	64.00
100	165	65.00
500	824	64.80

Note: Finite population factor not accounted for.

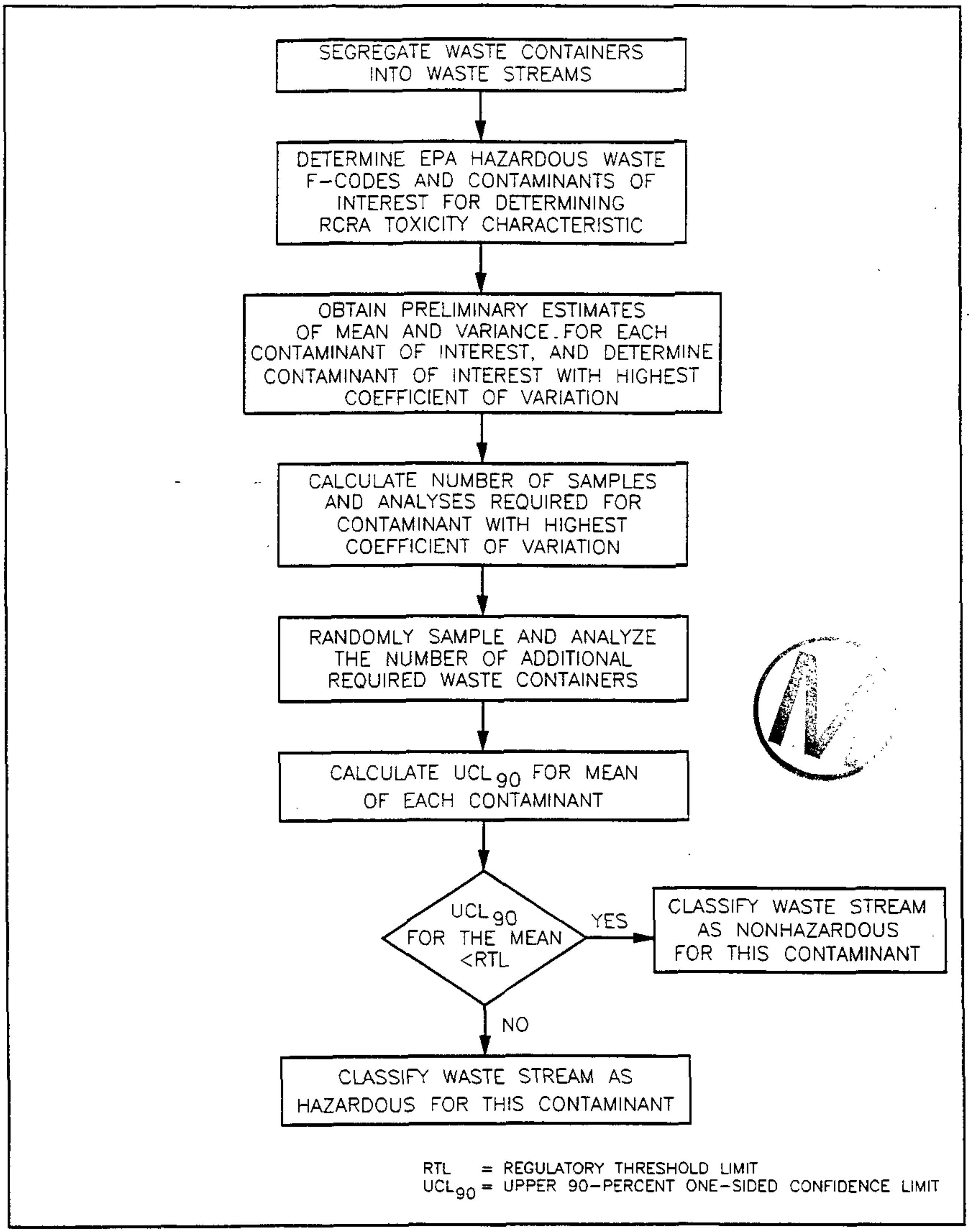

Figure C6-1

Statistical Approach to Sampling and Analysis of Waste Streams of Retrievably Stored Homogenous Solids and Soil/Gravel