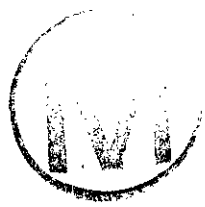
APPENDIX C8
QUALITY ASSURANCE OBJECTIVES AND DATA VALIDATION TECHNIQUES FOR WASTE CHARACTERIZATION SAMPLING AND ANALYTICAL METHODS



APPENDIX C8
QUALITY ASSURANCE OBJECTIVES FOR WASTE CHARACTERIZATION SAMPLING AND ANALYTICAL METHODS

C8-1 Validation Methods

Validation of all data (qualitative as well as quantitative) shall be performed so that data used for Waste Isolation Pilot Plant (WIPP) compliance programs will be of known and acceptable quality. Validation includes a quantitative determination of precision, accuracy, completeness, comparability, and method detection limit (as appropriate) for analytical data (headspace Volatile Organics Compounds (VOC) and total VOCs, Semivolatile Organic Compounds (SVOC), and metals data). Quantitative data validations shall be performed by the data generation level Quality Assurance (QA) officer according to the conventional methods outlined below (equations C8-1 through C8-8). These quantitative determinations will be compared to the Quality Assurance Objectives (QAOs) specified in Sections C8-2 through C8-9. A qualitative determination of representativeness will also be performed.

The qualitative data or descriptive information generated by radiography is not amenable to statistical analysis. However, radiography and visual examination are complementary techniques yielding similar data for determining the waste matrix code and waste material parameter weights of waste present in a waste container. Therefore, visual examination results shall be used to verify the waste matrix code and waste material parameter weights determined by radiography.

Representativeness of waste containers from waste streams subjected to visual examination and homogeneous solids and soil/gravel sampling and analysis will be validated, through documentation, that a true random sample was collected. Since representativeness is a quality characteristic that expresses the degree to which a sample or group of samples represents the population being studied, the random selection of waste containers ensures representativeness on a Program level. The Site Project Manager shall document that the selected waste containers from within a waste stream were randomly selected. Sampling personnel shall verify that proper procedures are followed to ensure that samples are representative of the waste contained in a particular waste container or a waste stream.

Precision

Precision is a measure of the mutual agreement among multiple measurements of a single analyte, either by the same method or by different methods. Precision is either expressed as the relative percent difference (RPD) for duplicate measurements or as the percent relative standard deviation (%RSD) for three or more replicate measurements. For duplicate measurements, the precision expressed as the RPD is calculated as follows:

$$RPD = \frac{C_1 - C_2}{\frac{(C_1 + C_2)}{2}} \times 100 \qquad \text{(C8-1)}$$

where C_1 and C_2 are the two values obtained by analyzing the duplicate samples. C_1 is the larger of the two observed values.

For three or more replicate measurements, the precision expressed as the %RSD is calculated as follows:

$$\%RSD = \frac{s}{\bar{y}} \times 100 \qquad \text{(C8-2)}$$

where s is the standard deviation and $\bar{y}$ is the mean of the replicate sample analyses.

The standard deviation, s, is calculated as follows:

$$s = \sqrt{\sum_{i=1}^{n} \frac{(y_i - \bar{y})^2}{n - 1}} \qquad \text{(C8-3)}$$

where y_i is the measured value of the ith replicate sample analysis measurement, and n equals the number of replicate analyses.

Another aspect of precision is associated with analytical equipment calibration. In these instances, the percent difference (%D) between multiple measurements of an equipment calibration standard shall be calculated as follows:

$$\%D = \left|\frac{C_1 - C_2}{C_1}\right| \times 100 \qquad \text{(C8-4)}$$

where C_1 is the initial measurement and C_2 is the second or other additional measurement.

Accuracy

Accuracy is the degree of agreement between a measured analyte concentration (or the average of replicate measurements of a single analyte concentration) and the true or known concentration. Accuracy is determined as the percent recovery (%R).

For situations where a standard reference material is used, the %R is calculated as follows:

$$\%R = \frac{C_m}{C_{srm}} \times 100 \qquad \text{(C8-5)}$$

where C_m is the measured concentration value obtained by analyzing the sample and C_{srm} is the "true" or certified concentration of the analyte in the sample.

For measurements where matrix spikes are used, the %R is calculated as follows:

$$\%R = \frac{S - U}{C_{sc}} \times 100 \qquad \text{(C8-6)}$$

where S is the measured concentration in the spiked aliquot, U is the measured concentration in the unspiked aliquot, and C_{sc} is the actual concentration of the spike added.

Method Detection Limit

The method detection limit (MDL) is the minimum concentration of an analyte that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. The MDL for all quantitative measurements (except for those using Fourier Transform Infared Spectroscopy [FTIRS]) is defined as follows:

$$MDL = t_{(n-1,1-\alpha=.99)} \times s \qquad \text{(C8-7)}$$

where $T_{(n-1,1-\alpha=.99)}$ is the t-distribution value appropriate to a 99 percent confidence level and a standard deviation estimate with n-1 degrees of freedom, n is the number of observations, and s is the standard deviation of replicate measurements.

For headspace-gas analysis using FTIRS, MDL is defined as follows:

$$\mathbf{MDL = 3s} \qquad \text{(C8-8)}$$

where s is the standard deviation. Initially, a minimum of seven samples of ambient air or seven blanks must be used to establish the MDLs. MDLs should be constantly updated using the results of the laboratory control sample or on-line control sample.

Completeness

Completeness is a measure of the amount of valid data (i.e., data that meets all Quality Assurance/Quality Control (QA/QC) requirements) obtained from the overall measurement system compared to the amount of data collected and submitted for analysis. Completeness must be expressed as the number of samples analyzed with valid results as a percent of the

total number of samples submitted for analysis. Completeness, expressed as the percent complete (%C), is calculated as follows:

$$\%C = \frac{V}{n} \times 100 \qquad \text{(C8-9)}$$

where V is the number of valid analytical results obtained and n is the number of samples submitted for analysis.

Comparability

Comparability is the degree to which one data set can be compared to another. Comparability of data generated at different sites will be assured through the use of standardized, approved testing, sampling, and analytical techniques and by meeting the QAOs specified in Sections C8-2 through C8-9. The techniques presented in Sections C8-2 through C8-9 are provided in detail in the Quality Assurance Program Plan (QAPP) and in the Transuranic Waste Characterization Sampling and Analysis Methods Manual (Methods Manual) (DOE,1995).

Representativeness

Representativeness is the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter that concerns the proper design of the sampling program.

C8-2 Headspace-Gas Sampling

Quality Assurance Objectives

Headspace-gas sampling may occur from three areas within drums of transuranic (TRU) waste (see Figure C6-1): 1) the drum headspace (i.e., the headspace directly under the drum lid), 2) the 55-gallon (gal) (208-liter [L]) polyethylene (poly) bag headspace, and 3) the headspace of the innermost layers of confinement. The precision and accuracy of the drum headspace-gas sampling operations must be assessed by analyzing field QC headspace-gas samples. These samples must include equipment blanks, field reference standards, field blanks, and field duplicates. If the QAOs described below are not met, a nonconformance report must be prepared, submitted, and resolved.

Precision

The precision of the headspace-gas sampling and analysis operation must be assessed by simultaneous collection of field duplicates for VOCs determination. Corrective actions must be taken if the RPD exceeds 25 percent.

Accuracy

A field reference standard must be collected using headspace-gas sampling equipment to assess the accuracy of the headspace-gas sampling operation. Corrective action must be taken if the %R of the field-reference standard is less than 70 or greater than 130.

Completeness

Sampling completeness shall be expressed as the number of valid samples collected as a percent of the total number of samples collected. Participating sampling facilities must achieve a minimum 90 percent completeness. The amount and type of data that may be lost during the headspace-gas sampling operation cannot be predicted in advance. The importance of any lost or contaminated headspace-gas samples must be evaluated by the Site Project QA Officer, and corrective action must be taken as appropriate.

Comparability

Consistent use and application of uniform procedures and equipment, as specified in the Methods Manual, should ensure that headspace gas sampling operations are comparable when sampling different layers of confinement and at the different sampling facilities.

Representativeness

Specific headspace-gas sampling steps to ensure samples are representative include:

- A sample canister cleaning and leak check
- Sampling equipment cleaning or disposal after use
- Sampling equipment leak check
- Use of sample canisters with passivated internal surfaces
- Use of low-internal-volume sampling equipment
- Collection of small-sample volume: low-sample volume to available headspace volume ratio
- Careful pressure regulation
- Performance audits
- Collection of equipment blanks, field reference standard, field blanks, and field duplicates

C8-3 Sampling of Homogenous Solids and Soils/Gravel

Quality Assurance Objectives

To ensure that sampling is conducted in a representative manner on a waste-stream basis for waste containers containing homogenous solids and soil/gravel, samples must be collected randomly in both the horizontal and vertical planes of each container's waste. For waste containers that contain homogenous solids and soil/gravel in smaller containers (e.g., 1 gal [4.0 L] poly bottles) within the waste container, one randomly chosen smaller container must be sampled.

Precision

Sampling precision must be determined by collecting and sampling field duplicates (e.g., co-located cores as described in Appendix C4-2.2) once per sampling batch or once per week during sampling operations, whichever is more frequent. A sampling batch is a suite of homogenous solids and soil/gravel samples collected consecutively using the same sampling equipment within a specific time period. A sampling batch can be up to 20 samples (excluding field QC samples), all of which must be collected within 14 days of the first sample in the batch. The RPD between co-located samples must be calculated and reported by the Site Project QA Officer.

The recommended method for establishing acceptance criteria for co-located cores is development of control charts for the RPD in the cores. Control charts will be developed for each constituent and for each waste matrix or waste type (e.g., pyrochemical salts or organic sludges), as needed, using historical analysis results. The historical analysis results currently do not exist, but would be collected over the course of future waste characterization activities. RPDs for at least 25 to 30 pairs of co-located cores would be used in the construction of the control charts. The limits for the control chart will be three standard deviations above or below the average RPD. Once constructed, RPDs for additional co-located pairs will be compared with the control chart to determine whether or not the co-located cores are acceptable. Periodically, the control charts will be updated using all available data.

In order to establish acceptance criteria to be used at the beginning of waste characterization activities, the variance between co-located cores will be compared to the variance measured within the waste stream (exclusive of containers with co-located core measurement) using a statistical test. The test will be performed for each constituent in each waste stream. The test is not considered sensitive and is presented as an interim method until the preferred method of control charting is established. Because of the expected difference between the co-located core variance and the waste stream variance, the test will rarely reject the hypothesis that the co-located core variance is less than the waste stream variance. However, without sufficient data to develop control charts and without established acceptance criteria for field duplicates (i.e., as specified by SW-846), the interim method is a reasonable approach for evaluating co-located cores.

The statistical test will involve calculating the variance for co-located cores by pooling the variances computed for each pair of co-located cores. The variance for the waste stream will be computed excluding any data from drums with co-located cores, because the test requires

the variance estimates to be independent. All data must be transformed to normality prior to computing variances and performing the test. The test hypothesis is evaluated using the F distribution and the method for testing the difference in variances. The method will be replaced with the control charting method once sufficient data are available.

Accuracy

Sampling accuracy shall not be measured. Because waste containers containing homogenous solids and soil/gravel with known quantities of analytes are not available, sampling accuracy cannot be determined. However, sampling methods and requirements described are designed to minimize sample degradation and hence maximize sampling accuracy.

Completeness

Sampling completeness shall be expressed as the number of valid samples collected as a percent of the total number of samples collected. Participating sampling facilities must achieve a minimum 90 percent completeness.

Comparability

Consistent use and application of uniform procedures, sampling equipment, and measurement units must ensure that sampling operations are comparable. The analysis results of field duplicates (samples taken of the same medium, under the same conditions, using the same procedures) are examined to determine the comparability. In addition, laboratories analyzing samples must participate in the Performance Demonstration Program (PDP).

Representativeness

Specific steps to ensure the representativeness of samples include the following for both waste containers and smaller containers:

- Coring tools and sampling equipment must be clean prior to sampling.
- The entire depth of the waste must be cored, and the core collected must have a length greater than or equal to 50 percent of the depth of the waste. This is called the core recovery and is calculated as follows:

$$\textit{Core recovery (percent)} = \frac{y}{x} \cdot 100 \qquad \text{(C8-10)}$$

where

x = the depth of the waste in the container
y = the length of the core collected from the waste.

- Coring operations and tool selection should be designed to minimize alteration of the in-place waste characteristics. Minimal waste disturbance must be verified by visually examining the core and describing the observation (e.g., undisturbed, cracked, or pulverized) in the field logbook.

If core recovery is less than 50 percent of the depth of the waste, a second coring location shall be randomly selected. The core from the second location shall be used for sample collection regardless of the core recovery.

$$MDC = K_1 K_2 (2.71 + 4.65 * s_b) \qquad \text{C8-11}$$

C8-4 Radiography

Quality Assurance Objectives

The QAOs for radiography are detailed in this section. If the QAOs described below are not met, then corrective action, such as additional operator training must be taken. It should be noted that radiography does not have a specific MDL because it is primarily a qualitative determination. The objective of radiography for the program is to verify the waste matrix parameters for each waste container and to estimate each waste material parameter weight (Table C8-1). All activities required to achieve these objectives must be described in the site quality assurance project plan (QAPjP) and standard operating procedures (SOP).

Data to meet these objectives must be obtained from an audio/videotaped scan provided by trained radiography operators at the sites. Results must also be recorded on a radiography data form. The precision, accuracy, completeness, and comparability objectives for radiography data are presented below.

Precision

The qualitative determinations, such as verifying the waste matrix code, made during radiography do not lend themselves to statistical evaluation of precision. However, comparison of data derived from radiography and visual examination on the same waste containers at the Rocky Flats Environmental Technology Site and the Idaho National Engineering Laboratory indicates that radiography operators can provide estimated inventories and weights of waste items in a waste container. As a measure of precision, the Site Project QA Officer shall calculate and report the RPD between the estimated waste material parameter weights as determined by radiography and these same parameters as determined by visual examination.

Accuracy

The accuracy with which the waste matrix code and waste material parameter weights can be determined must be documented through visual examination of a randomly selected statistical portion of waste containers. The percentage of waste containers that require assignment to a

different waste matrix code after visual examination must be calculated and reported by the Site Project QA Officer as a measure of radiography accuracy.

Completeness

An audio/videotape of the radiography examination and a validated radiography data form will be obtained for 100 percent of the retrievably stored waste containers in the program.

Comparability

The comparability of radiography data from different sites shall be enhanced by using standardized radiography procedures and operator qualifications.

C8-5 Gas Volatile Organic Compound Analysis

Quality Assurance Objectives

The development of data quality objectives (DQO) specifically for this program has resulted in the QAOs listed in Table C8-2. The specified QAOs represent the required quality of data necessary to draw valid conclusions regarding program objectives. Program-required limits, such as the program required quantitation limits (PRQL) associated with VOC analysis, are specified to ensure that the analytical data collected satisfy the requirements of all data users. A summary of the Quality Control Samples and the associated acceptance criteria is included in Table C8-3. Key data-quality indicators for laboratory measurements are defined below.

Precision

Precision shall be assessed by analyzing laboratory duplicates and replicate analyses of laboratory-control samples and PDP blind-audit samples. Results from measurements on these samples must be compared to the criteria listed in Table C8-2. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Accuracy

Accuracy as %R shall be assessed for the laboratory operations by analyzing PDP blind audit samples and laboratory-control samples. Results from these measurements must be compared to the criteria listed in Table C8-2. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Method Detection Limit

MDLs shall be expressed in nanograms for VOCs and must be less than or equal to those listed in Table C8-2. MDLs shall be determined based on the method described in the QAPP. The detailed procedures for MDL determination shall be included in site SOPs.

Program Required Quantitation Limit

Laboratories must demonstrate the capability to quantitate analytes at or below the PRQLs given in Table C8-2. Laboratories shall set the concentration of at least one calibration standard below the PRQL. The detailed procedures for PRQL demonstration shall be included in laboratory SOPs.

Completeness

Laboratory completeness shall be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Participating laboratories must meet the completeness specified in Table C8-2.

Comparability

For VOC analysis, data generated through analysis of samples from different sites shall be comparable. Comparability will be achieved by using standardized methods and traceable standards and by requiring all sites to participate in the PDP.

Representativeness

Representativeness for VOC analysis shall be achieved by collecting sufficient numbers of samples using clean sampling equipment that does not introduce sample bias. Samples must be collected as described in Appendix C4.

C8-6 Total Volatile Organic Compound Analysis

Quality Assurance Objectives

The development of DQOs specifically for this program has resulted in the QAOs listed in Table C8-4. The specified QAOs represent the required quality of data necessary to draw valid conclusions regarding program objectives. Program-required limits, such as the PRQL associated with VOC analysis, are specified to ensure that the analytical data collected satisfy the requirements of all data users. Key data-quality indicators for laboratory measurements are defined below.

Precision

Precision shall be assessed by analyzing laboratory duplicates, replicate analyses of laboratory-control samples, matrix-spike duplicates, and PDP blind-audit samples. Results from measurements on these samples must be compared to the criteria listed in Table C8-4. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Accuracy

Accuracy as %R shall be assessed for the laboratory operations by analyzing laboratory control samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from these

measurements must be compared to the criteria listed in Table C8-5. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Method Detection Limit

MDLs shall be expressed in milligrams per kilogram (mg/kg) for VOCs and must be less than or equal to those listed in Table C8-4. The detailed procedures for MDL determination shall be included in site SOPs.

Program Required Quantitation Limit

Laboratories must demonstrate the capability to quantitate analytes in samples at or below the PRQLs given in Table C8-4. Laboratories shall set the concentration of at least one calibration standard below the PRQL. The detailed procedures for PRQL demonstration shall be included in laboratory SOPs.

Completeness

Laboratory completeness shall be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Participating laboratories must meet the completeness specified in Table C8-4.

Comparability

For VOC analysis, data generated through analysis of samples from different sites shall be comparable. Comparability will be achieved by using standardized methods and traceable standards and by requiring all sites to participate in the PDP.

Representativeness

Representativeness for VOC analysis shall be achieved by collecting unbiased samples. Samples must be collected as described in Appendix C4.

C8-7 Total Semivolatile Organic Compound Analysis

Quality Assurance Objectives

The development of DQOs specifically for this program has resulted in the QAOs listed in Table C8-6. The specified QAOs represent the required quality of data necessary to draw valid conclusions regarding program objectives. Program-required limits, such as the PRQLs, are specified to ensure that the analytical data collected satisfy the requirements of all data users. A summary of Quality Control Samples and associated acceptance criteria for this analysis is included in Table C8-7. Key data-quality indicators for laboratory measurements are defined below.

Precision

Precision shall be assessed by analyzing laboratory duplicates, replicate analyses of laboratory control samples, matrix spike duplicates, and PDP blind-audit samples. Results from measurements on these samples must be compared to the criteria listed in Table C8-7. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Accuracy

Accuracy, as %R, shall be assessed for the laboratory operations by analyzing laboratory-control samples, matrix spikes, surrogate compounds, and PDP blind-audit samples. Results from these measurements must be compared to the criteria listed in Table C8-7. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Method Detection Limit

MDLs shall be expressed in mg/kg for SVOCs and must be less than or equal to those listed in Table C8-6. The detailed procedures for MDL determination shall be included in site SOPs.

Program Required Quantitation Limit

Laboratories must demonstrate the capability to quantitate analytes in samples at or below the PRQLs given in Table C8-6. Laboratories shall set the concentration of at least one calibration standard below the PRQL. The detailed procedures for PRQL demonstration shall be included in laboratory SOPs.

Completeness

Laboratory completeness shall be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Participating laboratories must meet the level of completeness specified in Table C8-6.

Comparability

For SVOC analysis, data generated through analysis of samples from different sites shall be comparable. Comparability will be achieved by using standardized methods and traceable standards and by requiring all sites to participate in the PDP.

Representativeness

Representativeness for SVOC analysis shall be achieved by collecting unbiased samples. Samples must be collected as described in Appendix C4.

C8-8 Total Metal Analysis

Quality Assurance Objectives

The development of DQOs for the program has resulted in the QAOs listed in Table C8-8. The specified QAOs represent the required quality of data necessary to draw valid conclusions regarding program objectives. Program-required limits, such as the PRQLs associated with metal analysis, are specified to ensure that the analytical data collected satisfy the requirements of all data users. A summary of Quality Control Samples and the associated acceptance criteria for this analysis is provided in Table C8-9. Key data-quality indicators for laboratory measurements are defined below.

Precision

Precision shall be assessed by analyzing laboratory matrix spike duplicates, replicate analyses of laboratory-control samples, and PDP blind-audit samples. Results from measurements on these samples must be compared to the criteria listed in Table C8-8. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Accuracy

Accuracy shall be assessed through the analysis of laboratory matrix spikes, PDP blind-audit samples, and laboratory-control samples. Results from these measurements must be compared to the criterion listed in Table C8-8. These QC measurements will be used to demonstrate acceptable method performance and to trigger corrective action when control limits are exceeded.

Program Required Detection Limits

PRDLs, expressed in units of micrograms per L (μg/L), are the maximum values for instrument detection limits (IDL) permissible for program support under the QAPP. IDLs must be less than or equal to the PRDL for the method used to quantitate a specific analyte. Any method listed in Table C-11 of the application may be used if the IDL meets this criteria. For high concentration samples, an exception to the above requirements may be made in cases where the sample concentration exceeds five times the IDL of the instrument being used. In this case, the analyte concentration may be reported even though the IDL may exceed the PRDL. IDLs shall be determined semiannually (i.e., every six months). Detailed procedures for IDL determination shall be included in laboratory SOPs.

Program Required Quantitation Limit

Laboratories must demonstrate the capability of analyte quantitation at or below the PRQLs in units of mg/kg dry weight (given in Table C8-8). The PRDLs are set an order of magnitude less than the PRQLs (assuming 100 percent solid sample diluted by a factor of 100 during preparation). Laboratories shall set the concentration of at least one QC or calibration standard

at or below the solution concentration equivalent of the PRQL. Detailed calibration procedures shall be included in site SOPs.

Completeness

Laboratory completeness shall be expressed as the number of samples analyzed with valid results as a percent of the total number of samples submitted for analysis. Participating laboratories must meet the completeness specified in Table C8-8.

Comparability

Data generated through analysis of samples from different sites shall be comparable. Comparability will be achieved by using standardized methods and traceable standards and by requiring all sites to participate in the PDP.

Representativeness

Representativeness for metals analysis shall be achieved by the collection of unbiased samples. Samples must be collected as described in Appendix C4.

C8-9 Acceptable Knowledge

Acceptable knowledge documentation provides primarily qualitative information that cannot be assessed according to specific data quality goals that are used for analytical techniques. QAOs for analytical results are described in terms of precision, accuracy, completeness, comparability, and representativeness. Analytical results will be used to confirm the characterization of wastes based on acceptable knowledge (Section C9-4). To ensure that the acceptable knowledge process is consistently applied, sites must comply with the following data quality requirements for acceptable knowledge documentation:

- Precision - Precision is the agreement among a set of replicate measurements without assumption of the knowledge of a true value. The qualitative determinations, such as compiling and assessing acceptable knowledge documentation, do not lend themselves to statistical evaluations of precision. However, the acceptable knowledge information will be addressed by the independent review of acceptable knowledge information during internal and external audits.

- Accuracy - Accuracy is the degree of agreement between an observed sample result and the true value. The percentage of waste containers which require reassignment to a new waste matrix code and/or designation of different hazardous waste codes based an the reevaluation of acceptable knowledge and sampling and analysis data will be reported as a measure of acceptable knowledge accuracy.

- Completeness - Completeness is an assessment of the number of waste streams or number of samples collected to the number of samples determined to be useable through the data validation process. The acceptable knowledge record

must contain 100 percent of the required information (Section C9-3). The useability of the acceptable knowledge information will be assessed for completeness during audits.

- Comparability - Data are considered comparable when one set of data can be compared to another set of data. Comparability is ensured through sites meeting the training requirements and complying with the minimum standards outlined for procedures that are used to implement the acceptable knowledge process. All sites must assign hazardous waste codes in accordance with Section C9-4 and provide this information regarding its waste to other sites who store or generate a similar waste stream.

- Representativeness - Representativeness expresses the degree to which sample data accurately and precisely represent characteristics of a population. Representativeness is a qualitative parameter that will be satisfied by ensuring that the process of obtaining, evaluating, and documenting acceptable knowledge information is performed in accordance with the minimum standards established in Section C9-4. Sites also must assess and document the limitations of the acceptable knowledge information used to assign hazardous waste codes (e.g., purpose and scope of information, date of publication, type and extent to which waste parameters are addressed).

Each site must address quality control by tracking its performance with regard to the use of acceptable knowledge by: 1) assessing the frequency of inconsistencies among information, and 2) documenting the results of acceptable knowledge confirmation through radiography, headspace-gas analyses, and solidified waste analyses. In addition, the acceptable knowledge process and waste stream documentation must be evaluated through internal assessments by quality assurance organizations and assessments by auditors external to the organization (i.e., DOE/CAO).

C8-10 Data Review, Validation, and Verification Requirements

Data review, validation, and verification requirements include procedures for the review, validation, and verification of data at the data generation level; the validation and verification of data at the project level; and the verification of data at the CAO level. Data review determines if raw data have been properly collected and ensures raw data are properly reduced. Requirements for data reduction are provided in Sections 9.0 through 15.0 of the QAPP, as appropriate, and in the Methods Manual. Data validation confirms that the data reported satisfy the requirements defined by the user and is accompanied by signature release. Data verification authenticates that data are in fact that which is claimed. The procedures presented in this section ensure that Program records furnish documentary evidence of quality.

Data Generation Level

The following are minimum requirements for raw data collection and management:

- All raw data shall be signed and dated in black ink by the person generating it.

- All data must be recorded clearly, legibly, and accurately in field and laboratory records (bench sheets, logbooks), and include applicable sample identification numbers.

- All changes to original data must be lined out, initialed, and dated by the individual making the change. A justification for changing the original data may also be included. Original data must not be obliterated or otherwise disfigured so as not to be readable.

- All data must be transferred and reduced from field and laboratory records completely and accurately.

- All field and laboratory records must be maintained in permanent files according to NEIC guidelines.

- Data must be organized into a standard format for reporting purposes (testing, sampling, analytical or on-line batch data report), as outlined in specific sampling and analytical techniques.

- All electronic and video data must be stored appropriately to ensure that waste container, sample, and associated QC data are readily retrievable.

Data review, validation, and verification at this level involves scrutiny and signature release from *qualified independent technical reviewer(s), technical supervisors(s), and a QA officer*, as specified below. Any nonconformance identified during this process shall be documented on a nonconformance report (Section C8-13). Facilities may combine the positions of independent technical reviewer and QA officer. Individuals conducting this data review, validation, and verification must use checklists that address all of the items included in this section. Checklists must contain tables showing the results of sampling, analytical or on-line batch QC samples, if applicable. Completed checklists must be forwarded with testing, sampling, analytical and on-line batch data reports to the project level.

- One hundred percent of the batch data reports must receive an independent technical review. This review shall be performed by an individual other than the data generator who is qualified to have performed the initial work. The reviewer(s) must release the data as evidenced by signature, and as a consequence ensure the following:

  - Data generation and reduction were conducted in a technically correct manner in accordance with the methods used. Data were reported in the proper units and correct number of significant figures.

  - Calculations have been verified by a valid calculation program, a spot check of verified calculation programs, and/or 100 percent check of all hand calculations.

  - All variances from an accepted method and the rationale for the variations have been documented and approved (Section C8-13).

- The data have been reviewed for transcription errors.

- The testing, sampling, or analytical data QA documentation (testing batch, sampling batch, analytical or on-line batch) is complete and includes raw data, calculation records, chain-of-custody (COC) forms, calibration records, QC sample results, and gas canister sample tags (if applicable).

- QC sample results are within established control limits, and if not, the data have been appropriately qualified.

- Reporting flags were assigned correctly.

- Sample holding time and preservation requirements were met, or exceptions documented.

- Radiography tapes have been reviewed, at a minimum for every tenth waste container, against the data reported on the radiography form to ensure that the data are correct and complete.

- Field sampling records are complete.

• One hundred percent of the batch data reports must receive technical supervisory signature release for each testing batch, sampling batch, analytical batch and on-line batch. This release must ensure the following:

  - The data are technically reasonable based on the technique used.

  - All data have received independent technical review with the exception of radiography tapes, which shall receive periodic technical review as specified above.

  - The testing, sampling, or analytical data QA documentation (testing batch, sampling batch, analytical batch or on-line batch) is complete and includes raw data, calculation records, COC forms, calibration records, QC sample results, and gas sample canister tags (if applicable).

  - Sample holding time requirements were met, or exceptions documented.

  - Field sampling records are complete.

• One hundred percent of the batch data reports must receive QA officer signature release. This release must ensure the following:

  - *Independent technical and technical supervisory reviews* have been performed as evidenced by the appropriate signature releases.

- The testing, sampling, or analytical data QA documentation (testing batch, sampling batch, analytical batch or on-line batch) is complete as appropriate for the point of data generation (i.e., radiography, RA, sampling, and analysis).

- Sampling and analytical QC checks have been properly performed. QC criteria that were not met are documented.

- QAOs have been met according to the methods outlined in Section C8-11.

Project Level

Data validation and verification at this level involves scrutiny and signature release from the Site Project Manager (or designee) and the Site Project QA Officer (or designee). This must be accomplished by meeting the following minimum requirements for each waste container. Any nonconformance identified during this process shall be documented on a nonconformance report (Section C8-13).

- One hundred percent of the testing, sampling, and analytical batch data reports must have Site Project Manager signature release. This signature release must ensure the following:

  - Data generation level independent technical, technical supervisory, and QA officer review, validation, and verification have been performed as evidenced by the appropriate signature releases.

  - Testing, sampling, analytical and on-line batch data review checklists are complete.

  - Testing, sampling, analytical and on-line batch data reports are complete and data are properly reported (e.g., data are reported in the correct units, with the correct number of significant figures, and with qualifying flags).

  - Reconciliation with the DQOs was performed (Section C8-12).

- One hundred percent of the testing, sampling, and analytical batch data reports must receive Site Project QA Officer signature release. This signature release must ensure the following:

  - Sampling batch QC checks (e.g., equipment blanks, field duplicates, field reference standards) were properly performed, and meet the established QAOs.

  - Testing batch QC checks (e.g., replicate scans, measurement system checks, replicate counts) were properly performed.

- Analytical batch QC checks (e.g., laboratory duplicates, laboratory blanks, matrix spikes, matrix spike duplicates, laboratory control samples) were properly performed and meet the established QAOs.

- On-line batch QC checks (e.g., field blanks, on-line blanks, on-line duplicates, on-line control samples) were properly performed and meet the established QAOs.

- Proper procedures were followed to ensure representative samples of headspace gas and homogenous solids and soil/gravel were taken.

- Radiography data are complete and acceptable based on the videotape review of one waste container per testing batch, at a minimum.

- RA data are complete and acceptable.

- The Site Project Manager and Site Project QA Officer shall ensure that a repeat of the data generation level review, validation, and verification is performed on the data for a minimum of one randomly chosen waste container quarterly (every three months). This exercise will document that the data generation level review, validation, and verification is being performed according to implementing procedures.

In association with the project-level validation and verification described above, the Site Project QA Officer (or designee) must prepare a Site Project QA Officer Summary and the Site Project Manager (or designee) must prepare a Data Validation Summary. The Site Project QA Officer Summary includes, on a per waste container basis, a validation checklist for each testing, sampling, analytical and on-line batch. Checklists for the Site Project QA Officer Summary must be sufficiently detailed to validate all aspects of a testing, sampling, analytical or on-line batch that affect data quality. The Data Validation Summary provides confirmation that, on a per waste container basis, all data have been validated in accordance with the site QAPjP. The Data Validation Summary must list each testing, sampling, analytical or on-line batch, describe how the validation was performed and whether or not problems were detected, and include a statement indicating that all data are acceptable.

Once the data have received project-level validation and verification, the Site Project Manager must ensure that the laboratory is notified. Samples must be retained by the laboratory until this notification is received. Gas sample canisters may then be released from storage for cleaning, recertification, and subsequent reuse. Sample tags must be removed and forwarded to the Site Project QA Officer before recycling the canisters. If the site project manager requests that samples or canisters be retained for future use (e.g., an experimental holding time study), the same sample identification and COC forms shall be used and cross-referenced to a document which specifies the purpose for sample or canister retention.

CAO Level

The third and final level of data verification occurs at CAO and must, at a minimum, consist of an inventory check of the data packages to verify completeness. The CAO Office of Regulatory Compliance manager is responsible for the verification that data packages include the following:

- Project-level signature releases
- Listing of all waste containers being reported in the package
- Listing of all testing, sampling, and analytical batch numbers associated with each waste container being reported in the package
- Data package case narrative
- Site Project QA Officer Summary
- Data Validation Summary
- Complete summarized qualitative and quantitative data for all waste containers

The CAO Office of Regulatory Compliance manager must verify that each data package is complete and notify the originating site in writing of the acceptance status of the data within two weeks of data package receipt. CAO will maintain the data as appropriate for use in the regulatory compliance programs.

C8-11 Reconciliation with Data Quality Objectives

Reconciling the results of waste testing and analysis with the DQOs provides a way to ensure that data will be of adequate quality to support the regulatory compliance programs. Reconciliation with the DQOs will take place at both the project level and the CAO level. At the project level, reconciliation will be performed by the Site Project Manager; at CAO, reconciliation will be performed by the CAO Office of Regulatory Compliance manager.

Reconciliation at the Project Level

The Site Project Manager will ensure that all data generated and used in decision making meet the DQOs provided in Section C-4d of the text of Chapter C. To do so, the Site Project Manager must assess whether data of sufficient type, quality, and quantity have been collected. The Site Project Manager must determine if the variability of the data set is small enough to provide the required confidence in the results. The Site Project Manager must also determine if, based on the desired error rates and confidence levels, a sufficient number of valid data points have been determined. In addition, the Site Project Manager must document that random sampling of containers was performed for the purposes of waste stream characterization.

For each waste stream characterized, the Site Project Manager must determine if sufficient data have been collected to determine the following Program-required waste parameters:

- Waste matrix code
- Waste material parameter weights
- Average mass and activity of each radionuclide of concern
- If each waste container of waste is TRU radioactive waste
- Average concentration of hydrogen, methane, and each VOC in the headspace gas of waste containers in the waste stream
- Total masses of VOCs, hydrogen, and methane in the headspace gas of the waste stream
- The potential flammability of TRU waste headspace gases
- Mean concentrations, UCL_{90} for the mean concentrations, standard deviations, and number of samples collected for VOCs, SVOCs, and metals in the waste stream
- Total masses of VOCs, SVOCs, and metals in the waste stream
- Whether the waste stream exhibits a toxicity characteristic (TC) under 40 CFR Part 261, Subpart C
- Whether the waste stream can be classified as hazardous or nonhazardous at the 90-percent confidence level
- Whether a sufficient number of waste containers have been visually examined to determine with a reasonable level of certainty that the UCL_{90} for the miscertification rate is less than 14 percent

If the Site Project Manager determines that insufficient data have been collected to make the determinations listed above, additional data collection efforts must be undertaken.

The statistical procedure presented in Appendix C6 shall be used by participating Site Project Managers to evaluate and report waste characterization data from the analysis of homogenous solids and soil/gravel. The procedure, which calculates UCL_{90} values, shall be used to assess compliance with the DQOs in Section 1.5 as well as with RCRA regulations. The procedure must be applied to all laboratory analytical data for total VOCs, total SVOCs, and total metals. For RCRA regulatory compliance (40 CFR § 261.24), data from the analysis of the appropriate metals and organic compounds shall be compared to the TC levels expressed as total values. These total values will be considered the regulatory threshold limit (RTL) values for the Program. RTL values are obtained by calculating the weight/weight concentration (in the solid) of a TC analyte that would give the regulatory weight/volume concentration (in the toxicity characteristic leaching procedure (TCLP) extract), assuming 100-percent analyte dissolution.

Reconciliation at the CAO Level

CAO must also ensure that data of sufficient type, quality, and quantity have been collected to meet Program DQOs. The CAO Office of Regulatory Compliance manager is responsible for determining if sufficient data have been collected to determine the following:

- The concentration of headspace gas VOCs in the total waste inventory to support a demonstration that VOCs will not migrate through the air beyond the WIPP unit boundary in concentrations greater than Environmental Protection Agency (EPA)-determined health-based limits during WIPP operations;

- The concentration of VOCs, SVOCs, and metals in the total waste inventory to support a demonstration that hazardous constituents will not migrate beyond the WIPP unit boundary in concentrations greater than EPA-determined health-based limits;

- The total curie, hydrogen, and methane concentrations in TRU waste to support revision of the thermal power restrictions for shipment of waste in the Transuranic Package Transporter (TRUPACT-II);

- An inventory of radioactive materials and physical waste forms to support an assessment of repository performance;

- Whether waste streams proposed for disposal in WIPP have been adequately characterized; and

- Whether data supports the preparation of the WIPP facility no-migration variance petition, the WIPP RCRA permit application, the WIPP facility 40 CFR Part 191 Certification Application, and a revised safety analysis report for the TRUPACT-II.

C8-12 Data Reporting Requirements

Data reporting requirements define the type of information and the method of transmittal for data transfer from the data generation level to the project level and from the project level to CAO.

Data Generation Level

Data shall be transmitted by hard copy from the data generation level to the project level. Transmitted data shall include all testing, sampling, and analytical batch data reports, and data review checklists. The report forms and checklists used must contain all of the information required by the testing, sampling, and analytical techniques described in Sections 7.0 through 15.0 of the QAPP, as well as the signature releases to document the review, validation, and verification as described in Section C8-10. All testing, sampling, and analytical batch data reports and checklists shall be on approved forms, as provided in site-specific documentation.

Testing, sampling, and analytical batch data reports shall be forwarded to the site project office. Site QAPjPs shall specify the individual at the site project office who will receive these reports. Testing batch data reports shall be forwarded to the site project office within 28 days of the

testing of the last waste container in a testing batch. Sampling batch data reports shall be forwarded to the site project office within 28 days of sample collection of the last sample in a sampling batch. Analytical batch data reports shall be forwarded to the site project office within 28 days of the VTSR of the last sample in an analytical batch. After review by the Site Project QA Officer, all batch data reports will be forwarded to the Site Project Manager. All testing, sampling, and analytical batch data reports shall be assigned serial numbers, and each page shall be numbered at the bottom. The serial number used for data reports can be the same as the testing, sampling, or analytical batch number.

QA documentation shall be maintained in either testing, sampling, and analytical facility files, or site project files for those facilities located on sites. Contract waste operation facilities shall forward testing, sampling, and analytical QA documentation along with testing, sampling, and analytical batch data reports to the site project office for inclusion in site central files.

Project Level

There are two aspects to project level reporting. First, summarized testing, sampling, and analytical data must be reported on a per-waste container basis. Second, summarized characterization information must be reported on a waste stream basis.

Summarized testing, sampling, and analytical data shall be transmitted by hard copy from the Site Project Manager to CAO when requested. Participating sites shall combine data from individual waste containers into data packages for reporting. Hard copy data packages shall consist of the following:

- Cover page with the site name, program identification, waste container numbers for containers included in the data package, and release signatures of the Site Project Manager and Site Project QA Officer
- Table of contents; and
- A concise narrative that summarizes the results of the project-level review and briefly describes any problems or other noteworthy items of interest associated with the data (i.e., nonconformance reports, operational variances). The narrative shall include separate sections which address results of duplicates/replicates and nonconformance reports associated with the waste containers being reported in the package.

For each waste container being reported in the data package, the following information shall be included:

- Cover page with the site name, program identification, waste container number, and approval/release signatures of the Site Project Manager and Site Project QA Officer
- A table that relates sample numbers (testing, sampling, and analytical) to waste container number

- Table of contents
- Site Project QA Officer Summary
- Data Validation Summary
- Radiography results
- Radioassay (RA) results
- Waste container headspace gas hydrogen, methane, and VOC analytical results
- Innermost layer of confinement headspace gas hydrogen, methane, and VOC analytical results for waste containers with inner layers of confinement (if applicable)
- Total VOC, SVOC, and metal analytical results for homogenous solids and soil/gravel (if applicable)

WIPP Waste Information System (WWIS) Data Reporting

The WWIS Data Dictionary (Appendix C13) contains all of the data fields, the field format and the limits associated with the data as established by various waste acceptance criteria. This data will be subjected to edit and limit checks that are performed automatically by the database.

WIPP will coordinate the data transmission with each generator site using the Internet and the TCP/IP transmission protocol. Actual data transmission will use DES encryption technology to ensure the integrity of the data transmissions. The sites with large waste inventories and large databases will populate a data structure provided by WIPP that contains the required data dictionary fields that are appropriate for the waste stream (or waste streams) at that site. For example, totals analysis data will not be requested from sites that do not have homogeneous solids or soil/gravel waste. WIPP will access this data via the Internet to ensure an efficient transfer of this data. Small quantity sites will be given a similar data structure that is tailored to their types of waste. Sites with very small quantities of waste will be provided with the ability to assemble the data interactively to this data structure on the WWIS.

C8-13 Nonconformances and Operational Variances

The status of work and the Program activities at participating sites shall be monitored and controlled by the Site Project Manager and Site Project QA Officer. This monitoring and control shall include: 1) nonconformance identification, documentation, and reporting; and 2) operational variance identification, documentation, and reporting.

Nonconformances

Nonconformances are uncontrolled and unapproved deviations from an approved plan, procedure, or expected result. Nonconforming items and activities are those that do not meet the Program requirements, procurement document criteria, or approved work procedures.

Nonconforming items shall be identified by marking, tagging, or segregating, and the affected organization(s) notified. Participating sites shall disposition nonconforming items as appropriate in accordance with the Quality Assurance Program Description (QAPD). Disposition of nonconforming items shall be identified and documented. The QAPjPs shall identify the person(s) responsible for evaluating and dispositioning nonconforming items and shall include referenced procedures for handling them.

Management at all levels shall foster a "no-fault" attitude to encourage the identification of nonconforming items and processes. Nonconformances may be detected and identified by anyone performing Program activities, including

- Project staff - during field operations, supervision of subcontractors, data validation and verification, and self-assessment
- Laboratory staff - during the preparation for and performance of laboratory testing; calibration of equipment; QC activities; laboratory data review, validation, and verification; and self-assessment
- QA personnel - during oversight activities or audits

A nonconformance report shall be prepared for each nonconformance identified. Each nonconformance report shall be initiated by the individual(s) identifying the nonconformance. The nonconformance report shall then be processed by knowledgeable and appropriate personnel. For this purpose, a nonconformance report including, or referencing as appropriate, results of laboratory analysis, QC tests, audit reports, internal memoranda, or letters shall be prepared. The nonconformance report must provide the following information:

- Identification of the individual(s) identifying or originating the nonconformance
- Description of the nonconformance
- Method(s) or suggestions for correcting the nonconformance (corrective action) or description of the variance granted
- Schedule for completing the corrective action
- An indication of the potential ramifications and overall useability the data, if applicable
- Any approval signatures specified in the QAPjPs

The Site Project QA Officer shall oversee the nonconformance report process and be responsible for developing a plan to identify and track all nonconformances and report this information to the DOE field office. Documentation of nonconformances shall be made available to the Site Project Manager, who in turn is responsible for notifying project personnel of the nonconformance. Completion of the corrective action for nonconformances must be verified by the Site Project QA Officer.

Operational Variances

Variances are approved and controlled changes to Program-related plans or procedures. The need for a variance is caused by the identification of improvement opportunities or unusual or nonroutine occurrences that affect operations but not the ability to achieve the performance standards or quality requirements specified in this QAPP or site QAPjPs. Each person performing Program activities is responsible for the quality of their work and adherence to the applicable requirements contained in this QAPP and site QAPjPs. When a need to deviate from established procedures is identified, it is the responsibility of the person performing the work to initiate a variance.

When a variance is required, the person identifying the need for the variation shall complete a Record of Variance and have a direct supervisor approve it. A Record of Variance must be completed and approved before initiation of the activity to document the variation from normal, approved procedures. The Site Project QA Officer shall assess the significance of the variance and determine if changes to the plans or procedures and further notifications are required.

A Record of Variance must contain at least the following information:

- Title or heading, "Record of Variance"
- Waste container or sample identification number
- Reason for the deviation from the requirements contained in the QAPjP or SOP
- A description of the variation from the accepted sampling, testing, or analytical procedure
- A description of special equipment or personnel required
- Initiator's signature and date
- Supervisor's signature and date
- Site Project Manager's signature and date
- Site Project QA Officer's signature and date

DOE/CAO Corrective Action Process

DOE/CAO initiates a corrective action process when internal nonconformances and nonconformances at the generator/storage sites are identified. Activities and processes that do not meet requirements are documented as deficiencies. All deficiencies regardless of type and origin are processed through the CAO corrective action process.

When a deficiency is identified by the CAO, the following process action steps are initiated:

- The condition is documented on a Corrective Action Report (CAR) by the individual identifying the problem.

- The CAO QA Manager and the National TRU Programs (NTP) Team Leader review the CAR, determine validity of the finding (determine that a requirement has been violated), classify the significance of the condition, assign a response due date, and issue the CAR to the responsible party.

- The responsible organization reviews the CAR, evaluates the extent and cause of the deficiency and provides a response to the CAO, indicating remedial actions and actions to preclude recurrence that will be taken.

- The CAO reviews the response from the responsible organization and, if acceptable, communicates the acceptance to the responsible organization.

- The responsible organization completes remedial actions and actions to preclude recurrence of the condition.

- After all corrective actions have been completed, the CAO schedules and performs a verification to assure that corrective actions have been completed and are effective. When all actions have been completed and verified as being effective, the CAR is closed by the CAO QA Manager and the NTP Team Leader.

- As part of the planning process for subsequent audits and surveillances, past deficiencies are reviewed and the previous deficient activity or process is subject to reassessment.

C8-14 Special Training Requirements and Certifications

Before performing activities that affect Program quality, all personnel are required to receive indoctrination into the scope, purpose, and objectives of the Program and the specific QAOs of the assigned task. Personnel assigned to perform activities for the Program shall have the education, experience, and training applicable to the functions associated with the work. Evidence of personnel proficiency and demonstration of competence in the task(s) assigned must be demonstrated and documented. All personnel designated to work on specific aspects of the Program shall maintain qualification (i.e., training and certification) throughout the duration of the work as specified in this QAPP and applicable QAPjPs. Job performance shall be evaluated and documented at periodic intervals, as specified in the QAPjPs.

Personnel involved in Program activities shall receive continuing training to ensure that job proficiency is maintained. Training includes both education in principles and enhancement of skills. Each participating site shall include in its QAPjP a description of the procedures for implementing personnel qualification and training in accordance with the QAPD and 10 CFR § 830.120. All training records that specify the scope of the training, the date of completion, and documentation of job proficiency shall be maintained in the site project file.

Analytical laboratory line management must ensure that analytical personnel are qualified to perform the analytical method(s) for which they are responsible. The minimum qualifications for certain specified positions for the Program are summarized in Table C8-10. QAPjPs, or their implementing SOPs, shall specify the site-specific titles and minimum training and qualification requirements for personnel performing Program activities. QAPjPs shall also contain the requirements for maintaining records of the qualification, training, and demonstrations of proficiency by these personnel.

An evaluation of personnel qualifications shall include comparing and evaluating the requirements specified in the job/position description and the skills, training, and experience included in the current resume of the person. This evaluation also must be performed for personnel who change positions because of a transfer or promotion as well as personnel assigned to short-term or temporary work assignments that may affect the quality of the Program. QAPjPs shall identify the responsible person(s) for ensuring that all personnel maintain proficiency in the work performed and identify any additional training that may be required.

C8-11 List of References

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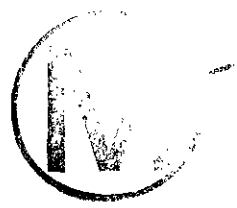

TABLES

TABLE C8-1
WASTE MATERIAL PARAMETERS AND DESCRIPTIONS

Waste Material Parameter	*Description*
Iron-based Metals/Alloys	Iron and steel alloys in the waste; does not include the waste container materials
Aluminum-based Metals/Alloys	Aluminum or aluminum-based alloys in the waste materials
Other Metals	All other metals found in the waste materials
Other Inorganic Materials	Nonmetallic inorganic waste including concrete, glass, firebrick, ceramics, sand, and inorganic sorbents
Cellulosics	Materials generally derived from high-polymer plant carbohydrates; (e.g., paper, cardboard, wood, and cloth)
Rubber	Natural or man-made elastic latex materials; (e.g., surgeons' gloves, and leaded rubber gloves)
Plastics (waste materials)	Generally man-made materials, often derived from petroleum feedstock; (e.g., polyethylene and polyvinylchloride)
Organic Matrix	Cemented organic resins, solidified organic liquids and sludges
Inorganic Matrix	Any homogeneous materials consisting of sludge or aqueous-based liquids that are solidified with cement, calcium silicate, or other solidification agents; (e.g., wastewater treatment sludge, cemented aqueous liquids, and inorganic particulates)
Soils/gravel	Generally consists of naturally occurring soils that have been contaminated with inorganic waste materials
Steel (packaging materials)	55-gal (208-L) drums
Plastics (packaging materials)	90-mil polyethylene drum liner and plastic bags

TABLE C8-2
GAS VOLATILE ORGANIC COMPOUNDS TARGET ANALYTE LIST AND QUALITY ASSURANCE OBJECTIVES

Compound	CAS Number	Precision[a] (%RSD or RPD)	Accuracy[a] (%R)	MDL[b] (ng)	FTIRS MDL[b] (ppmv)	PRQL (ppmv)	Completeness (%)
Benzene	71-43-2	≤25	70-130	10	5	10	90
Bromoform	75-25-2	≤25	70-130	10	5	10	90
Carbon tetrachloride	56-23-5	≤25	70-130	10	5	10	90
Chlorobenzene	108-90-7	≤25	70-130	10	5	10	90
Chloroform	67-66-3	≤25	70-130	10	5	10	90
1,1-Dichloroethane	75-34-3	≤25	70-130	10	5	10	90
1,2-Dichloroethane	107-06-2	≤25	70-130	10	5	10	90
1,1-Dichloroethylene	75-35-4	≤25	70-130	10	5	10	90
cis-1,2-Dichloroethylene	156-59-2	≤25	70-130	10	5	10	90
Ethyl benzene	100-41-4	≤25	70-130	10	10	10	90
Ethyl ether	60-29-7	≤25	70-130	10	5	10	90
Formaldehyde[c]	50-00-0	≤25	70-130	10		10	90
Hydrazine[d]	302-01-2	≤25	70-130	10		10	90
Methylene chloride	75-09-2	≤25	70-130	10	5	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤25	70-130	10	5	10	90
Tetrachloroethylene	127-18-4	≤25	70-130	10	5	10	90
Toluene	108-88-3	≤25	70-130	10	5	10	90
1,1,1-Trichloroethane	71-55-6	≤25	70-130	10	5	10	90
Trichloroethylene	79-01-6	≤25	70-130	10	5	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤25	70-130	10	5	10	90
m-Xylene[e]	108-38-3	≤25	70-130	10	5	10	90
o-Xylene	95-47-6	≤25	70-130	10	5	10	90
p-Xylene[e]	106-42-3	≤25	70-130	10	5	10	90
Acetone	67-64-1	≤25	70-130	150	50	100	90
Butanol	71-36-3	≤25	70-130	150	50	100	90
Methanol	67-56-1	≤25	70-130	150	50	100	90
Methyl ethyl ketone	78-93-3	≤25	70-130	150	50	100	90
Methyl isobutyl ketone	108-10-1	≤25	70-130	150	50	100	90

[a]Criteria apply to PRQL concentrations.
[b]Values based on delivering 10 mL to the analytical system.
[c]Required only for homogenous solids and soil/gravel from Los Alamos National Laboratory.
[d]Required only for homogenous solids and soil/gravel from Oak Ridge National Laboratory and the Savannah River Site.
[e]These xylene isomers cannot be resolved by GC/MS.

CAS = Chemical Abstract Service
%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
MDL = Method detection limit (maximum permissible value), for GC/MS and GC/FID; total number of nanograms delivered to the analytical system per sample (nanograms); for FTIRS based on 1m sample cell
PRQL = Program required quantitation limit (parts per million/volume basis)

TABLE C8-3
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND FREQUENCIES FOR GAS VOLATILE ORGANIC COMPOUND ANALYSIS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet method QAOs	Repeat until acceptable
Laboratory duplicates or on-line duplicates	One (1) per analytical batch for GC/MS and GC/FID. One (1) per analytical batch or on-line batch for FTIRS	RPD ≤ 25[b]	Nonconformance if RPD >25
Laboratory blanks or on-line blanks	Daily prior to sample analysis for GC/MS and GC/FID. Daily prior to sample analysis and one (1) per analytical batch or on-line batch for FTIRS.	Analyte amounts <3 x MDLs for GC/MS and GC/FID; <PRQL for FTIRS	Nonconformance if analyte amounts > 3 x MDLs for GC/MS and GC/FID; >PRQL for FTIRS
Laboratory control samples or on-line control samples	One (1) per analytical batch for GC/MS and GC/FID. One (1) per analytical batch or on-line batch for FTIRS	70-130 %R	Nonconformance is %R <70 or >130
GC/MS comparison sample (for FTIRS only)	One (1) per analytical or on-line batch	RPD ≤ 25	Nonconformance if RPD >25
Blind audit samples	Samples and frequency controlled by the Gas PDP Plan	Specified in the Gas PDP Plan	Specified in the Gas PDP Plan

[a]Corrective action per section C8-13 when final reported QC samples do not meet the acceptance criteria.
[b]Applies only to concentrations greater than the PRQLs listed in Table C8-2.

MDL = Method Detection Limit
QAO = Quality Assurance Objective
PDP = Performance Demonstration Program
PRQL = Program Required Quantitation Limit
%R = Percent Recovery
RPD = Relative Percent Difference

TABLE C8-4
TOTAL VOLATILE ORGANIC COMPOUNDS TARGET ANALYTE LIST AND QUALITY ASSURANCE OBJECTIVES

Compound	CAS Number	Precision[a] (%RSD or RPD)	Accuracy[a] (%R)	MDL (mg/kg)	PRQL (mg/kg)	Completeness (%)
Benzene	71-43-2	≤45	37-151	1	10	90
Bromoform	75-25-2	≤47	45-169	1	10	90
Carbon disulfide	75-15-0	≤50	60-150	1	10	90
Carbon tetrachloride	56-23-5	≤30	70-140	1	10	90
Chlorobenzene	108-90-7	≤38	37-160	1	10	90
Chloroform	67-66-3	≤44	51-138	1	10	90
1,4-Dichlorobenzene[b]	106-46-7	≤60	18-190	1	10	90
ortho-Dichlorobenzene[b]	95-50-1	≤60	18-190	1	10	90
1,2-Dichloroethane	107-06-2	≤42	49-155	1	10	90
1,1-Dichloroethylene	75-35-4	≤250	D-234[c]	1	10	90
Ethyl benzene	100-41-4	≤43	37-162	1	10	90
Methylene chloride	75-09-2	≤50	D-221[c]	1	10	90
1,1,2,2-Tetrachloroethane	79-34-5	≤55	46-157	1	10	90
Tetrachloroethylene	127-18-4	≤29	64-148	1	10	90
Toluene	108-88-3	≤29	47-150	1	10	90
1,1,1-Trichloroethane	71-55-6	≤33	52-162	1	10	90
1,1,2-Trichloroethane	79-00-5	≤38	52-150	1	10	90
Trichloroethylene	79-01-6	≤36	71-157	1	10	90
Trichlorofluoromethane	75-69-4	≤110	17-181	1	10	90
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	≤50	60-150	1	10	90
Vinyl chloride	75-01-4	≤200	D-251[c]	1	4	90
m-xylene	108-38-3	≤50	60-150	1	10	90
o-xylene	95-47-6	≤50	60-150	1	10	90
p-xylene	106-42-3	≤50	60-150	1	10	90
Acetone	67-64-1	≤50	60-150	10[d]	100	90
Butanol	71-36-3	≤50	60-150	10[d]	100	90
Ethyl ether	60-29-7	≤50	60-150	10[d]	100	90
Formaldehyde[e]	50-00-0	≤50	60-150	10[d]	100	90
Hydrazine[f]	302-01-2	≤50	60-150	10[d]	100	90
Isobutanol	78-83-1	≤50	60-150	10[d]	100	90
Methanol	67-56-1	≤50	60-150	10[d]	100	90
Methyl ethyl ketone	78-93-3	≤50	60-150	10[d]	100	90
Pyridine[b]	110-86-1	≤50	60-150	10[d]	100	90

[a]Criteria apply to PRQL concentrations.
[b]Can also be analyzed as a semi-volatile organic compound.
[c]Detected; result must be greater than zero.
[d]Estimate, to be determined.
[e]Required only for homogenous solids and soil/gravel from Los Alamos National Laboratory.
[f]Required only for homogenous solids and soil/gravel from Oak Ridge National Laboratory and Savannah River Site.

CAS = Chemical Abstract Service
%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
MDL = Method detection limit (maximum permissible value) (milligrams per kilogram)
PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for benzene assuming a 0.9 oz (25-gram [g]) sample, 0.1 gal (0.5 liter [L]) of extraction fluid, and 100 percent analyte extraction (milligrams per kilogram)

TABLE C8-5
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND FREQUENCIES FOR TOTAL VOLATILE ORGANIC COMPOUND ANALYSIS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet total VOC analysis QAOs	Repeat until acceptable
Laboratory duplicates[b]	One (1) per analytical batch	Meet total VOC analysis RPDs	Nonconformance if RPDs > values in Table C8-4
Laboratory blanks	One (1) per analytical batch	Analyte concentrations < 3 x MDLs	Nonconformance if analyte concentrations > 3 x MDLs
Matrix spikes	One (1) per analytical batch	Meet total VOC analysis %Rs in QAP	Nonconformance if %Rs are outside the range specified in QAPP
Matrix spike duplicates	One (1) per analytical batch	Meet total VOC analysis RPDs and %Rs	Nonconformance if RPDs and %Rs > values in Table C8-4
Laboratory control samples	One (1) per analytical batch	80 - 120 %R	Nonconformance if %R < 80 or > 120
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples for a given matrix ±3 standard deviations	Nonconformance if %R < (average %R - 3 standard deviation) or > (average %R + 3 standard deviation)
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

[a]Corrective Action per section C8-13 when final reported QC samples do not meet the acceptance criteria.
[b]May be satisfied using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PRQLs listed in Table C8-4.

MDL = Method detection limit
QAO = Quality assurance objective
PDP = Performance Demonstration Program
%R = Percent recovery
RPD = Relative percent difference

TABLE C8-6
SEMI-VOLATILE ORGANIC COMPOUND TARGET ANALYTE LIST AND QUALITY ASSURANCE OBJECTIVES

Compound	CAS Number	Precision[a] (%RSD or RPD)	Accuracy [a] (%R)	MDL (mg/kg)	PRQL (mg/kg)	Completeness (%)
Cresols	1319-77-3	≤50	60-150	5	40	90
1,4-Dichlorobenzene[b]	106-46-7	≤86	20-124	5	40	90
ortho-Dichlorobenzene[b]	95-50-1	≤64	32-129	5	40	90
2,4-Dinitrophenol	51-28-5	≤119	D-172[d]	5	40	90
2,4-Dinitrotoluene	121-14-2	≤46	39-139	0.3	2.6	90
Hexachlorobenzene	118-74-1	≤319	D-152[d]	0.3	2.6	90
Hexachloroethane	67-72-1	≤44	40-113	5	40	90
Nitrobenzene	98-95-3	≤72	35-180	5	40	90
Polychlorinated Biphenyls				5	40	90
Aroclor 1016[c]	12674-11-2	≤33	50-114	5	40	90
Aroclor 1221[c]	11104-28-2	≤110	15-178	5	40	90
Aroclor 1232[c]	11141-16-5	≤128	10-215	5	40	90
Aroclor 1242[c]	53469-21-9	≤49	39-150	5	40	90
Aroclor 1248[c]	12672-29-6	≤55	38-158	5	40	90
Aroclor 1254[c]	11097-69-1	≤62	29-131	5	40	90
Aroclor 1260[c]	11096-82-5	≤56	8-127	5	40	90
Pentachlorophenol	87-86-5	≤128	14-176	5	40	90
Pyridine[b]	110-86-1	≤50	60-150	5	40	90

CAS = Chemical Abstract Service
%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
MDL = Method detection limit (maximum permissible value) (milligrams per kilogram)
PRQL = Program required quantitation limit; calculated from the toxicity characteristic level for nitrobenzene assuming a 100-gram (g) sample, 0.5 gal (2 liter [L]) of extraction fluid, and 100 percent analyte extraction (milligrams per kilograms)

[a]Criteria apply to PRQL concentrations
[b]Can also be analyzed as a volatile organic compound
[c]Required only for waste matrix code S3220 (organic sludges)
[d]*Detected; result must be greater than zero*

TABLE C8-7
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND FREQUENCIES FOR TOTAL SEMI-VOLATILE ORGANIC COMPOUNDS ANALYSIS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table C8-7 QAOs	Repeat until acceptable
Laboratory duplicates[b]	One (1) per analytical batch	Meet Table C8-7 RPDs	Nonconformance if RPDs > Table C8-7
Laboratory blanks	One (1) per analytical batch	Analyte concentrations <3 x MDLs	Nonconformance if analyte concentrations > 3 x MDLs
Matrix spikes	One (1) per analytical batch	Meet Table C8-7 %Rs	Nonconformance if %Rs are outside the range specified in Table C8-7
Matrix spike duplicates	One (1) per analytical batch	Meet Table C8-7 RPDs and %Rs	Nonconformance if RPDs and %Rs > Table C8-7 values
Laboratory control samples	One (1) per analytical batch	80 - 120 %Rs	Nonconformance if %R < 80 or > 120
Surrogate compounds	Each analytical sample	Average %R from minimum of 30 samples from a given matrix ±3 standard deviations	Nonconformance if %R < (average %R - 3 standard deviations) or > (average %R + 3 standard deviations)
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

[a]Corrective action per section C8-13 when final reported QC samples do not meet the acceptance criteria

[b]May be satisfied by using matrix spike duplicate; acceptance criteria applies only to concentrations greater than the PQRLs listed in Table C8-6.

MDL = Method Detection Limit
QAO = Quality Assurance Objective
PDP = Performance Demonstration Program
%R = Percent Recovery
RPD = Relative Percent Difference

TABLE C8-8
TOTAL METALS TARGET ANALYTE LIST AND QUALITY ASSURANCE OBJECTIVES

Analyte	CAS Number	Precision (%RSD or RPD)[a]	Accuracy (%R)[b]	PRDL[c] (µg/L)	PRQL (mg/kg)	Completeness (%)
Antimony	7440-36-0	≤30	80-120	100	100	90
Arsenic	7440-38-2	≤30	80-120	100	100	90
Barium	7440-39-3	≤30	80-120	2000	2000	90
Beryllium	7440-41-7	≤30	80-120	100	100	90
Cadmium	7440-43-9	≤30	80-120	20	20	90
Chromium	7440-47-3	≤30	80-120	100	100	90
Lead	7439-92-1	≤30	80-120	100	100	90
Mercury	7439-97-6	≤30	80-120	4.0	4.0	90
Nickel	7440-02-0	≤30	80-120	100	100	90
Selenium	-7782-49-2	≤30	80-120	20	20	90
Silver	7440-22-4	≤30	80-120	100	100	90
Thallium	7440-28-0	≤30	80-120	100	100	90
Vanadium	7440-62-2	≤30	80-120	100	100	90
Zinc	7440-66-6	≤30	80-120	100	100	90

[a]≤ 30 percent control limits apply when sample and duplicate concentrations are ≥ 10 x IDL for ICP-AES and AA techniques, and ≥ 100 x IDL for Inductively Coupled Plasma—Mass Spectrometry (ICP-MS) techniques. If less than these limits, the absolute difference between the two values shall be less than or equal to the PRDL.
[b]Applies to laboratory control samples, laboratory matrix spikes, and PDP blind audit samples. If a solid laboratory control sample material which has established statistical control limits is used, then the established control limits for that material should be used for accuracy requirements.
[c]PRDL set such that it is a factor of 10 below the PRQL for 100 percent solid samples, assuming a 100 x dilution during digestion.

CAS = Chemical Abstract Service
%RSD = Percent relative standard deviation
RPD = Relative percent difference
%R = Percent recovery
PRDL = Program required detection limit (i.e., maximum permissible value for IDL) (milligrams per liter)
PRQL = Program required quantitation limit (milligrams pre kilogram)

TABLE C8-9
SUMMARY OF LABORATORY QUALITY CONTROL SAMPLES AND FREQUENCIES FOR TOTAL METALS ANALYSIS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action[a]
Method performance samples	Seven (7) samples initially and four (4) semiannually	Meet Table C8-9 QAOs	Repeat until acceptable
Laboratory blanks	One (1) per analytical batch	≤ 3 x IDL (≤ 5 x IDL for ICP-MS)[b]	Redigest and reanalyze any samples with analyte concentrations which are ≤10 x blank value and ≥ 0.5 x PQRL
Matrix spikes	One (1) per analytical batch	80 - 120 %Rs	Nonconformance if %Rs are <80 or >120
Matrix spike duplicates	One (1) per analytical batch	RPD ≤ 30 80-120 %R	Nonconformance if RPD > 30 or if %R < 80 or > 120
Laboratory control samples	One (1) per analytical batch	80 - 120 %Rs	Redigest and reanalyze for affected analytes
Blind audit samples	Samples and frequency controlled by the Solid PDP Plan	Specified in the Solid PDP Plan	Specified in the Solid PDP Plan

[a]Corrective action per section C8-13 when final reported QC samples do not meet the acceptance criteria

[b] Applies only to concentrations greater than the PQRLs listed in Table C8-8.

IDL = Instrument Detection Limit
PDP = Performance Demonstration Program
PQRL = Program Required Detection Limit
%R = Percent Recovery
RPD = Relative Percent Difference

TABLE C8-10
MINIMUM TRAINING AND QUALIFICATIONS REQUIREMENTS[a]

Personnel	Requirements[a]
Radiography Operators[c]	Site-specific training based on waste matrix codes and waste material parameters; requalification every 2 years
Gas Chromatography Technical Supervisors[b] Gas Chromatography Operators[c]	B.S. or equivalent experience and 6 months previous applicable experience
Gas Chromatography/Mass Spectrometry Operators[c] Mass Spectrometry Operators[c]	B.S. or equivalent experience and 1 year independent spectral interpretation or demonstrated expertise
Gas Chromatography/Mass Spectrometry Technical Supervisors[b] Mass Spectrometry Technical Supervisors[b] Atomic Absorption Spectroscopy Technical Supervisors[b] Atomic Absorption Spectroscopy Operators[c] Atomic Mass Spectrometry Operators[c] Atomic Emission Spectroscopy Operators[c]	B.S. or equivalent experience and 1 year applicable experience
Atomic Mass Spectrometry Technical Supervisors[b]	B.S. and specialized training in Atomic Mass Spectrometry and 2 years applicable experience
Atomic Emission Spectroscopy Technical Supervisors[b]	B.S. and specialized training in Atomic Emission Spectroscopy and 2 years applicable experience.

[a]Based on requirements contained in *USEPA Contract Laboratory Program Statement of Work for Organics Analysis* (Document Number OLM 01.0) and *Statement of Work for Inorganics Analysis* (Document Number ILM 03.0).
[b]Technical Supervisors are those persons responsible for the overall technical operation and development of a specific laboratory technique. QAPjPs shall include the site-specific title for this position.
[c]Operators are those persons responsible for the actual operation of analytical equipment. QAPjPs shall include the site-specific title for this position.

FIGURES

1

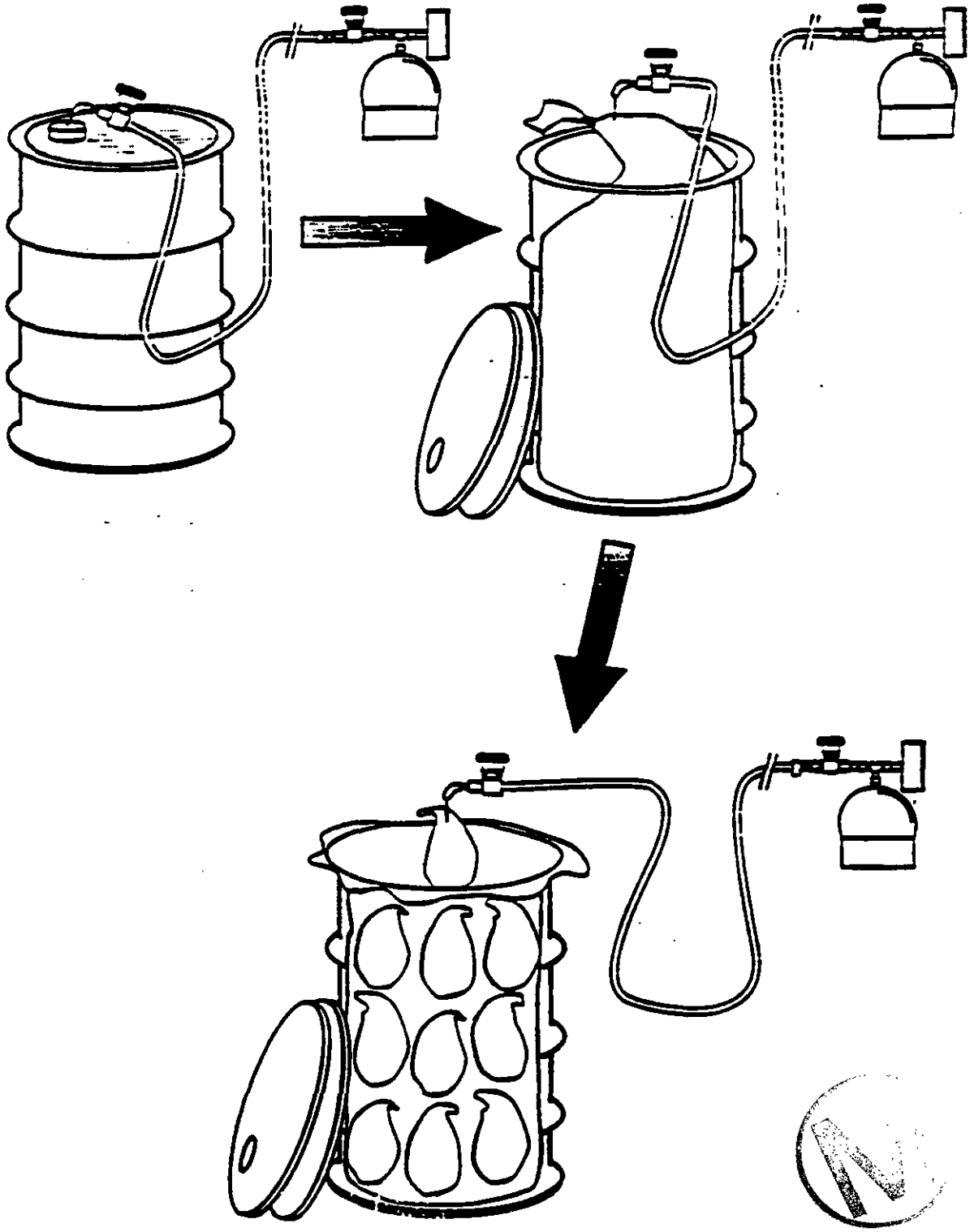

FIGURE C8-1
OVERALL HEADSPACE-GAS SAMPLING SCHEME ILLUSTRATING MANIFOLD SAMPLING