



DOE/WIPP 89 - 025  
DRAFT Revision 6.1

**Program Plan  
for the  
Pretest Characterization  
of  
WIPP Experimental Waste**

**October 1990**



**Waste Isolation Pilot Plant**



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## 1.0 INTRODUCTION

### 1.1 BACKGROUND

The Department of Energy (DOE) must show compliance with Environmental Protection Agency (EPA) regulations before waste can be disposed at the Waste Isolation Pilot Plant (WIPP). 40 CFR Part 191 (1) defines the environmental radiation protection standards for management and disposal of transuranic (TRU) radioactive wastes. This requires that the disposal system provide a reasonable expectation, based upon performance assessments (PA), that the cumulative release of radionuclides to the accessible environment for 10,000 years after disposal meets the prescribed limits.

One current unknown for WIPP is the rate of gas generation and its effect on the ability to demonstrate compliance with 40 CFR Part 191 (1). Sandia National Laboratories (SNL) has been assigned the responsibility for performing the PA and has designed multiple tests to evaluate gas generation rate and gas generation potential under simulated repository conditions using real TRU waste. These tests are described in the Bin-Scale Test Plan (2), simulating the longer term repository conditions, and the Alcove Test Plan (3), simulating post-closure and shorter term repository conditions on a room scale basis.

WIPP has submitted a No-Migration Variance Petition (NMVP) (4) to the EPA Office of Solid Waste (OSW) in order to receive a variance from Land Disposal Restrictions (5). This submittal, if approved, would allow waste to be brought to WIPP for the Bin-Scale (2) and Alcove (3) experiments. The NMVP is being reviewed by the EPA-OSW, which is in the process of granting approval. Very conservative estimates for the potential concentration of volatile organic compounds (VOCs) and/or toxic metals were made for the source term for the long-term modeling of repository performance under undisturbed conditions (4). Also, estimates of VOCs in the headspace of waste drums were used to demonstrate near-term compliance with releases of VOCs during the experimental and operations periods.

Although characterization of TRU waste mixed with hazardous constituents (TRU-mixed waste) may not be necessary to demonstrate compliance with the long-term effects of

mixed waste disposal in the WIPP repository, because of the conservative assumptions for the source term, characterization of mixed waste is necessary to:

- Verify that the assumptions made for the concentration of hazardous materials in the hydrologic modeling are indeed very conservative. The "margin of safety" stated in the NMVP is based on process knowledge, which needs to be verified.
- Comply with the Resource Conservation and Recovery Act (RCRA) requirements to characterize waste prior to shipment to a disposal facility (i.e., WIPP).
- Analyze headspace gases from additional waste containers to confirm the assumptions made about the potential releases of VOCs during the experimental and operational demonstration periods.
- Collect standardized data as part of the Pretest Waste Characterization activities in support of meeting long term compliance with 40 CFR Part 191 (1) and 40 CFR Part 268.6 (5).

The purpose of this program plan is to define the activities required to characterize the WIPP experimental waste prior to conducting the Bin-Scale (2) and Alcove (3) tests in WIPP. These activities will provide data necessary to define the inventory source terms of the waste (total amounts of material present) and, in conjunction with post-test waste characterization studies, will be used in the studies as stated above.

Details of specific actions required at the generator/storage sites, described in this program plan (Section 2.0), are based on the needs specified in the experimental Bin and Alcove Test Plans (2,3). Data generated by this characterization will also be used by other programmatic efforts, including validation of waste previously certified to the WIPP Waste Acceptance Criteria (WAC) (6), evaluation of the information on hazardous constituents in support of the No-Migration Variance Petition (4), verification of process knowledge databases, and documentation of TRUPACT-II payload compliance (7). Development of "standard" procedures for characterization and analyses of TRU and TRU-mixed wastes has been enhanced by the working interaction among DOE, the EPA Office of Radiation Programs (EPA-ORP), and the waste generator and storage sites (Section 1.5).

Overall management of the characterization program will be the responsibility of the DOE WIPP Project Office (WPO). WPO will review and approve site-specific characterization and sampling plans, and provide program surveillances and audits. The actual characterization and sampling will be performed by the contractors at waste generator/storage sites, with overview by WIPP personnel.

This program plan is a living document, and is expected to change as new requirements are identified [e.g., analytical requirements for sludge samples (Section 2.2.9) and revisions to the WAC and transportation requirements for the TRUPACT-II package]. The bin-scale wastes will be the first wastes to be received at WIPP according to current plans.

## 1.2 SCOPE

The scope of this waste characterization program plan includes the following activities:

- Determine the isotopic distribution and assay of test wastes within experimentally determined detection limits and uncertainties (Section 2.2.4).
- Identify methods for analyzing TRU mixed wastes (for the analyses identified in Section 2.0) to be applied uniformly by all DOE sites, and coordinate with the U.S. EPA and the Environmental Improvement Division (EID) of New Mexico.
- Use real-time radiography (RTR) and videotapes as records for comparison of waste content codes among the different test wastes (Section 2.2.3).
- Visually inspect Contact-Handled (CH) TRU waste (Section 2.2.6).
- Sample and analyze headspace gas for VOCs and other gases present (Section 2.2.5).
- Ensure that all experimental waste meets the WIPP WAC, Revision 3, and the TRUPACT-II Authorized Methods for Payload Control (TRAMPAC, Appendix 1.3.7 of the TRUPACT-II Safety Analysis Report, Rev. 4) before shipment to WIPP (6,7).
- Verify process knowledge (Section 1.3.1).

- **Appoint the WAC Certification Committee (WACCC) as the organization responsible to observe and oversee all pretest waste characterization activities with participation by State, Federal, and local agencies such as the EPA, the New Mexico EID, the Environmental Evaluation Group (EEG), and equivalent agencies of other affected states.**

The development of the analytical methods will be documented in a Quality Assurance Program Plan (QAPP) that will be the governing document for all analyses done in support of the pretest waste characterization. This QAPP, currently under development, is based on the analytical requirements identified in this program plan, and will be approved by DOE Headquarters. Waste characterization activities at each site shall conform to this overall QAPP, and will be documented in site-specific Quality Assurance Project Plans (QAPjPs) that will be approved by WACCC. Figure 1 shows the relationship between this program plan, the overall system QAPP, and the site-specific QAPjPs.

### 1.3 PRETEST WASTE CHARACTERIZATION

The Bin-Scale and Alcove Test Plans (2,3), developed by SNL in support of the Performance Assessment Program, have pretest waste characterization requirements specified in Sections 8.3.1 and 10.4.1, respectively. These requirements will be revised and updated as necessary to reflect the status of pretest waste characterization needs and/or developments in experimental design. This program plan addresses the present status of these pretest waste characterization needs and supersedes some of the analytical requirements in the Bin-Scale and Alcove Test Plans (2,3) for gas generation properties. The test wastes will not be selected to demonstrate representativeness for hazardous constituents (see Section 2.1).

#### 1.3.1 Waste Characterization Requirements to Demonstrate Representativeness of Waste

One goal of this program is to characterize waste used in experiments to demonstrate that the waste gas generation properties are comparable to all the CH-TRU waste in DOE's system. To achieve that goal, all sites will have to generate waste characterization data for comparison with the waste tested. The basis for comparison of TRU waste in the DOE system is process knowledge, which consists of the following:



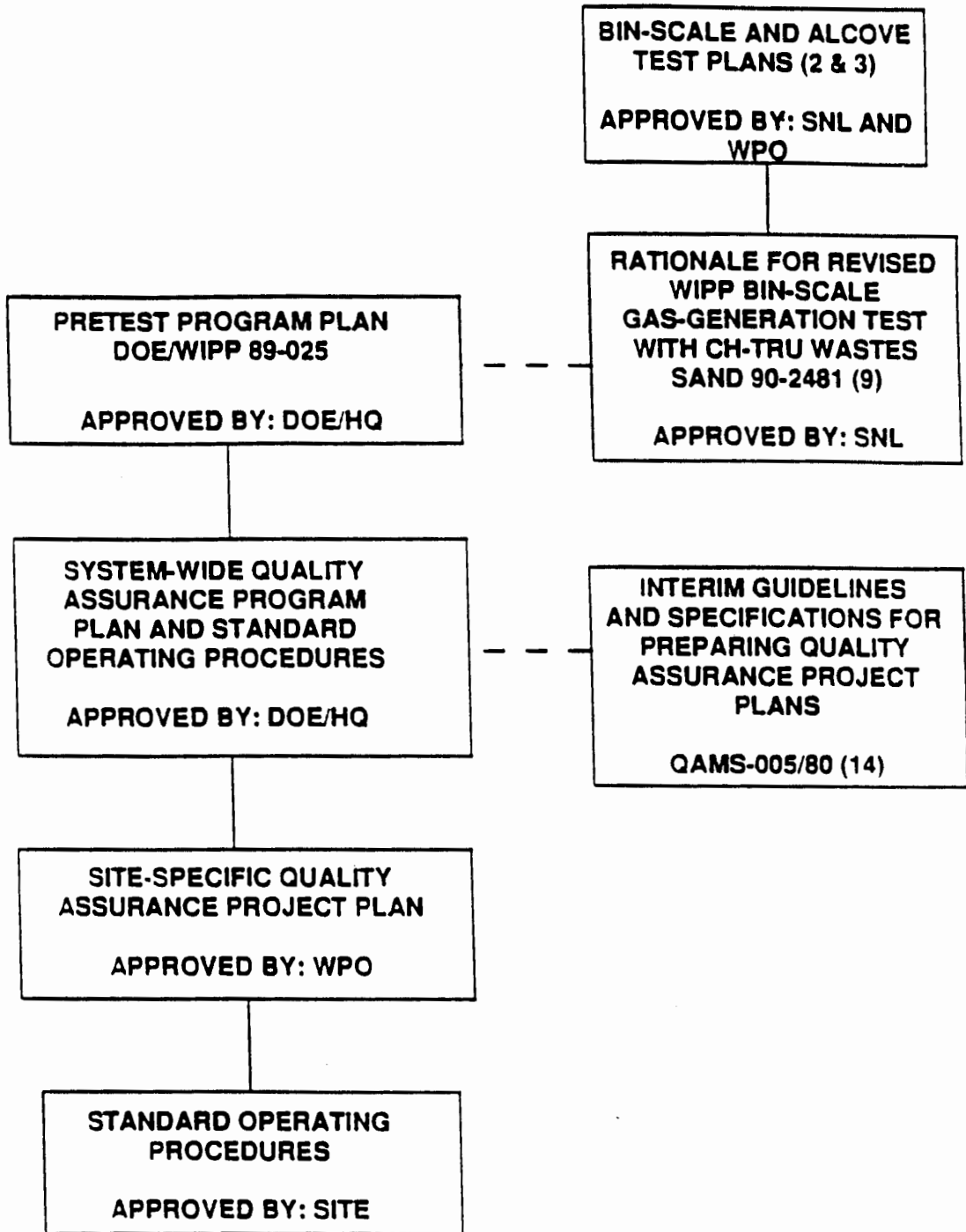


Figure 1. Documents Governing Pretest Waste Characterization

- Process flow diagrams presented in the NMVP (4)
- Information in the TRUPACT-II Content Codes (TRUCON) (8) document and chemical lists published in the NMVP
- Records and documentation from generator/storage sites.

Data on TRU waste generated as part of the present waste characterization program will be compared with the existing (process) knowledge, as outlined above, to verify that DOE has been accurate in their assessments of the physical and chemical composition of the waste (e.g. the percentages of materials listed in Table 1) or conservative in their assumptions where no previous analytical data exists.

If waste forms are identified in the system (by process knowledge) that do not appear to be comparable to the test waste, then the waste in question may have to be tested under the bin-scale test conditions (for its gas generation potential, etc.). Waste parameters governing this characterization are described in detail below.

The logic, needs, and requirements of the characterization of the WIPP waste are as follows:

- A fraction of the total inventory is being tested in the bin-scale and alcove experiments to provide input into the performance assessment studies. This fraction is chosen via a statistical methodology described in a separate Sandia National Laboratories document, SAND 90-2481 (9).
- The composition of the test wastes has been chosen such that, based on process knowledge, it represents almost the entire waste inventory across the system. This waste defines the "envelope" or the "allowable payload" for shipment to the WIPP site. The waste parameters and variables that determine this test waste envelope are listed in Table 1.

**TABLE 1**  
**CONTROLLING VARIABLES FOR WIPP PRETEST WASTE CHARACTERIZATION**

1. Cellulosics: paper, cloth, wood
2. Plastics: bags, liners, plexiglas
3. Rubber: gloves, aprons
4. Corroding metal/steels: drums, wastes
5. Corroding metal/aluminum: wastes
6. "Non-corroding" metals: lead, tantalum, copper
7. Solid inorganics: glasses, ceramics
8. Inorganic sludges
9. Cements: including additives
10. Other organics: resins, organic sludges
11. Total alpha curies

- Additional waste characterization (analytical data based on sampling) is needed for the waste at all the sites to verify the process knowledge information. This will be in the form of a sampling program across all sites that involves the examination of a fraction of the waste generated or stored at each site. These sample sizes will be based on a statistical population of the waste containers at each site as required by SAND 90-2481 (9).
- If waste is found in the system (based on the sampling) that is not represented by the test waste (that is, the gas generation properties cannot be described by the groups of waste components listed in Table 1), the following three options are available to qualify this "non-representative waste" for disposal at WIPP:
  - Reprocess the non-representative waste so that its properties fall under that of the waste being tested in the experimental program. This option is equivalent to changing the non-conforming waste such that it falls under the envelope of waste defined by the test waste.
  - Test the non-representative waste under the conditions of the experimental waste, for the same test parameters, and include these results in the performance assessment evaluations. This option is equivalent to expanding the current test waste envelope such that all of the waste in the system falls under it.
  - Show, based on conservative analysis and knowledge of the waste (without actual gas generation testing) that the waste can be included in the performance assessment studies. This is equivalent to Option 2 of expanding the test envelope, but relies on conservative analysis instead of actual gas generation testing of the waste. This option requires that sufficient information be available regarding the properties of this waste to make the conservative analysis.

The first ten parameters listed in Table 1 are groups of waste materials that affect the potential to produce gas from processes (i.e., bacterial, corrosion, and radiolytical) that might operate in the rooms of waste at WIPP.

The eleventh parameter (alpha curies) is not a waste material that can decompose to produce gas, but is energy for a mechanism (radiolysis) by which gas is potentially produced. Alpha radiation from all transuranic elements have approximately the same energy associated with an alpha decay. Therefore, equivalent alpha curies from different transuranic elements should produce the same potential amount of gas.

Representativeness of the waste with respect to the parameters in Table 1 refers only to overall properties (e.g., the gas generation potential) and not to the amount of constituents present in the waste. For example, if the maximum amount of cellulose (variable 1 in Table 1) present in a WIPP experimental drum is 50 kg, a drum of waste sampled at a site with 100 kg of cellulose can still be under the test waste envelope, as long as it does not contain material with physical or chemical properties not represented by the variables in Table 1.

The three options outlined above provide a basis for evaluating wastes which may fall outside the envelope of the variables listed in Table 1, including new unanticipated waste generation processes that may occur in the future.

#### 1.4 STATISTICAL SAMPLING REQUIREMENTS

All of the drum equivalent waste to be used in the bin-scale tests will be characterized according to the requirements described in Section 2.2. A statistical sample of the drum equivalent waste to be used in the alcove tests will be characterized in accordance with the requirements described in Section 2.3. However, RTR as described in Section 2.2.3, and assay measurements as described in Section 2.2.4, will be used to characterize all experimental waste (both bin and alcove).

The alcove test program involves two types of waste: "as received" waste and "specially prepared" waste (3). The "as received" waste doesn't require the opening of drums or boxes of wastes for test preparation. However, some specific characterization activities are required to be able to relate the "as received" waste in the alcove tests to both the waste used in the bin-scale tests and the "specially prepared" waste used in the other alcove tests.

Of the 1050 drums (or drum equivalents) to be emplaced as "as received" waste into the alcoves, a statistical sample will be randomly selected for further characterization in exactly the same manner as for the bin-scale waste. The statistical evaluations to determine these sample sizes will be conducted by SNL and made available to the DOE sites early in Fiscal Year 1991, prior to initiation of these tests. The number of drums (the sample size) in the alcove waste that will need detailed characterization (as described in Section 2.2) will depend on the results of the pretest waste characterization done in the bin-scale program. The sampling program will provide a 90% confidence level for each of the test variables (Table 1). This limited sampling will provide a database to support the RTR methodology for making comparisons between similar waste content codes.

The remainder of the drum equivalents (approximately 2800) for the "specially prepared" wastes scheduled for the alcove tests will all be opened for insertion of additional materials (3). A statistical sample of the 2800 drum population will also be characterized using the same procedures as described in Section 2.2 (for the bin-scale tests).

### 1.5 ENVIRONMENTAL PROTECTION AGENCY (EPA) INVOLVEMENT

The Office of Radiation Programs (ORP) is providing technical support on the safe and effective characterization of TRU mixed wastes under an interagency agreement (IAG) with DOE. EPA-ORP will review and comment on proposed sampling protocols, analytical protocols, and the Quality Assurance Program Plan. EPA-ORP will also coordinate an intra- and inter-laboratory performance program (including preparation and distribution of blind samples) to demonstrate the performance of the analytical methods and participant laboratories. Waste characterization activities will commence only after verification that the analytical procedures meet program objectives and laboratory performance is acceptable.

EPA-OSW will be involved in the waste characterization program throughout the development and implementation of sampling and analysis protocols. In addition to reviewing WIPP program plans, OSW will be asked to review and comment on DOE's proposed sampling and analysis methods prior to their inclusion in the QAPP. In this way, OSW comments can be incorporated into the sampling and analysis procedures before waste characterization activities are initiated.

## 2.0 WASTE CHARACTERIZATION - EXPERIMENTAL NEEDS

This section details pretest waste characterization information and the analyses needed for the different types of experimental waste. The WIPP bin-scale and alcove experiments (2,3), along with related laboratory experiments (10) are primarily intended to provide data in support of the WIPP performance assessment. The basis and presently approved details of the experiments to be placed in WIPP are described in the two test plans by Sandia National Laboratories (2,3). The experimental waste for the WIPP site can be grouped as follows:

1. Waste for the Bin-Scale Tests - The current plan involves about 800 drum volumes of waste to be repackaged into 146 bins (9).
2. Waste for the Alcove Tests - The waste for the alcove tests can be subdivided into two groups:
  - "As-received" waste
  - "Specially prepared" waste.

The waste for these experiments may come from two or more DOE waste generator and/or storage sites. Pretest waste characterization data needs for the two types of tests have been initially identified and described in the two test plans (2,3), and will be revised as appropriate to reflect updates of these requirements. Details of the pretest experimental waste characterization and site implementation needs are presented in the following sections.

As stated in Section 1.2, each site supplying waste for the test phase is required to prepare a site-specific waste characterization document (a QAPjP) detailing how the needs and requirements outlined in this program plan and the system QAPP are met by the site.

Dosimetry records will be kept by each site for all personnel involved in pretest waste characterization activities. The purpose is to be able to define the doses received for each activity. The recorded doses will, in turn, be used to evaluate the relative risks and benefits of different waste treatment and handling options. These records, without any

identities, shall be made available to the WPO. The evaluations from the records will be provided to the EPA.

## 2.1 LOGIC AND METHODOLOGY FOR PRETEST WASTE CHARACTERIZATION NEEDS

Pretest waste characterization data needs, identified in this document as applying to the governing regulations 40 CFR Part 191 (performance assessment) (1) and 40 CFR Part 268 (hazardous materials) (5), are presented in Table 2. The parameters in the table and their relevance to the two regulations are described below. The table also addresses the issue of the verification of process knowledge. Performance assessment is addressed under 40 CFR Part 191, and the No-Migration Variance Petition (4) is addressed under 40 CFR Part 268.6. It should be noted that short-term and long-term compliance with 40 CFR Part 268.6 has been documented in the NMVP, even with conservatively large estimates of the regulated compounds (VOCs and toxic metals). In other words, it is not believed that pretest waste characterization is needed to show compliance with 40 CFR Part 268.6, but waste characterization data on hazardous constituents will be collected to verify assumptions of modeling in the NMVP and to support RCRA characterization of TRU wastes at DOE sites. The WIPP experimental program and pretest waste characterization are primarily aimed at obtaining data to support evaluation of compliance with 40 CFR Part 191 and to demonstrate the representativeness of the experimental waste for their gas generation potential. The occurrence of VOCs and/or toxic metals in the wastes should not affect the gas generation rate or potential of the nonhazardous constituents. Therefore, the wastes used in the Bin-Scale and Alcove tests need not be representative of the inventory with respect to the hazardous components inventory and that is not an objective of this test program.

Table 2 lists the technical requirements for pretest characterization of the inventory. The parameters in Table 2, and the technical justification for each, are listed below:

- Real-Time Radiography (RTR): This examination provides input on the major materials in the waste (e.g., metals, paper/cloth/plastic or sludge) which provides information on waste comparability. RTR is used by the sites to determine compliance with some waste acceptance and waste transportation criteria.



**TABLE 2**  
**TECHNICAL REQUIREMENTS**  
**PRETEST WASTE CHARACTERIZATION**

| PARAMETER                          | OBJECTIVE     |               |                                      |
|------------------------------------|---------------|---------------|--------------------------------------|
|                                    | 40 CFR<br>191 | 40 CFR<br>268 | VERIFICATION OF<br>PROCESS KNOWLEDGE |
| RTR                                | YES           | YES           | YES                                  |
| ISOTOPIC DISTRIBUTION<br>AND ASSAY | YES           | NR            | YES                                  |
| HEADSPACE GASES                    | NR            | YES           | YES                                  |
| SOLID WASTE -                      |               |               |                                      |
| VISUAL INSPECTION<br>AND WEIGHING  | YES           | YES           | YES                                  |
| SLUDGE -                           |               |               |                                      |
| VOCs                               | NR            | NR            | YES                                  |
| EP-TOXIC METALS                    | NR            | NR            | YES                                  |
| MAJOR CATIONS AND<br>ANIONS, pH    | YES           | NR            | YES                                  |

NR - Characterization not required for the parameter.

Examples of parameters that can be verified by RTR are the presence of free liquids, the waste physical form, and the presence of restricted items like sealed containers (> 1 gallon in size).

- Isotopic Distribution and Assay: These results are needed as inventory information for 40 CFR Part 191 (1) compliance efforts, and will be used to determine potential radiolytic gas generation rates.
- Headspace Analysis: This is not directly used in PA analysis. It does provide an estimate of gas compositions in the waste container, which can give an indication of the dominant process occurring in a drum (e.g., production of CO<sub>2</sub> and depletion of O<sub>2</sub> due to radiolysis of paper/cloth). The presence of volatile organic compounds in the headspace of a drum can provide evidence of the presence of hazardous constituents in the waste.
- Visual Inspection and Weighing of Solid Waste: This provides data on the types and amounts of waste materials (e.g., paper towels, metal, etc., identified to the extent possible). The weight of the waste materials in a waste container will be reported for the first ten variables listed in Table 1, or noted as a material that differs from the ten categories of waste. The weights of these materials in each bin will be used to interpret gas generation data for input into PA calculations. Actual chemical analysis of the solid constituents is not practical to implement, and is not needed in support of PA or the NMVP (4).
- Sludge Analysis for Volatile Organic Compounds or Toxic Metals: This is not a concern for PA. Previous modeling in the NMVP (4) has demonstrated that even with conservatively large estimates for these compounds (VOCs and toxic metals), migration beyond the unit boundary will not occur above health-based levels. The sludges, however, will be analyzed for VOCs and toxic metals in support of waste characterization activities to comply with RCRA requirements of each site.
- Major Cations and Anions and pH of Sludges: These properties need to be quantified because of their influence on radionuclide solubilities, and are necessary inputs into the PA calculations.

The preceding text outlines the basis for the pretest characterization data needs described in Sections 2.2 and 2.3. Characterization efforts for other parameters, outside the scope of the pretest waste characterization, are described in Section 2.2.9. The characterization described in Section 2.2.9 is not a requirement for pretest activities, but will be performed as an additional check on process knowledge information and to provide data in support of RCRA characterization activities at the sites.

## 2.2 PRETEST WASTE CHARACTERIZATION FOR BIN-SCALE TESTS

### 2.2.1 Introduction

The bin-scale tests require specific amounts of four different WIPP waste test types (2). These are:

1. High-Organic Newly Generated (HONG) wastes
2. High-Organic Old wastes (HOOW)
3. Low-Organic Newly Generated (LONG) wastes\*
4. Inorganic Processed Sludge (PS)

\*LONG wastes can also include "old" waste (2,3)

A summary flowchart of the pretest waste characterization needs for this waste is presented in Figure 2. Each of the steps in the flowchart is described in detail below. Each of the drums that will be repackaged into bins for the bin-scale tests must meet the pretest waste characterization requirements.

### 2.2.2 Selection of Drums for Bin Tests

The basis and statistical justification for the number of drum equivalents of waste needed in the bin-scale tests are described in a supporting document to the test plans, SAND 90-2481 (9). Waste content codes are a subset of the four waste test types defined above, and the content codes that fit into each of the WIPP waste test types are listed in Table 3. The content codes are a waste classification system developed for transportation purposes and apply to each site (8).

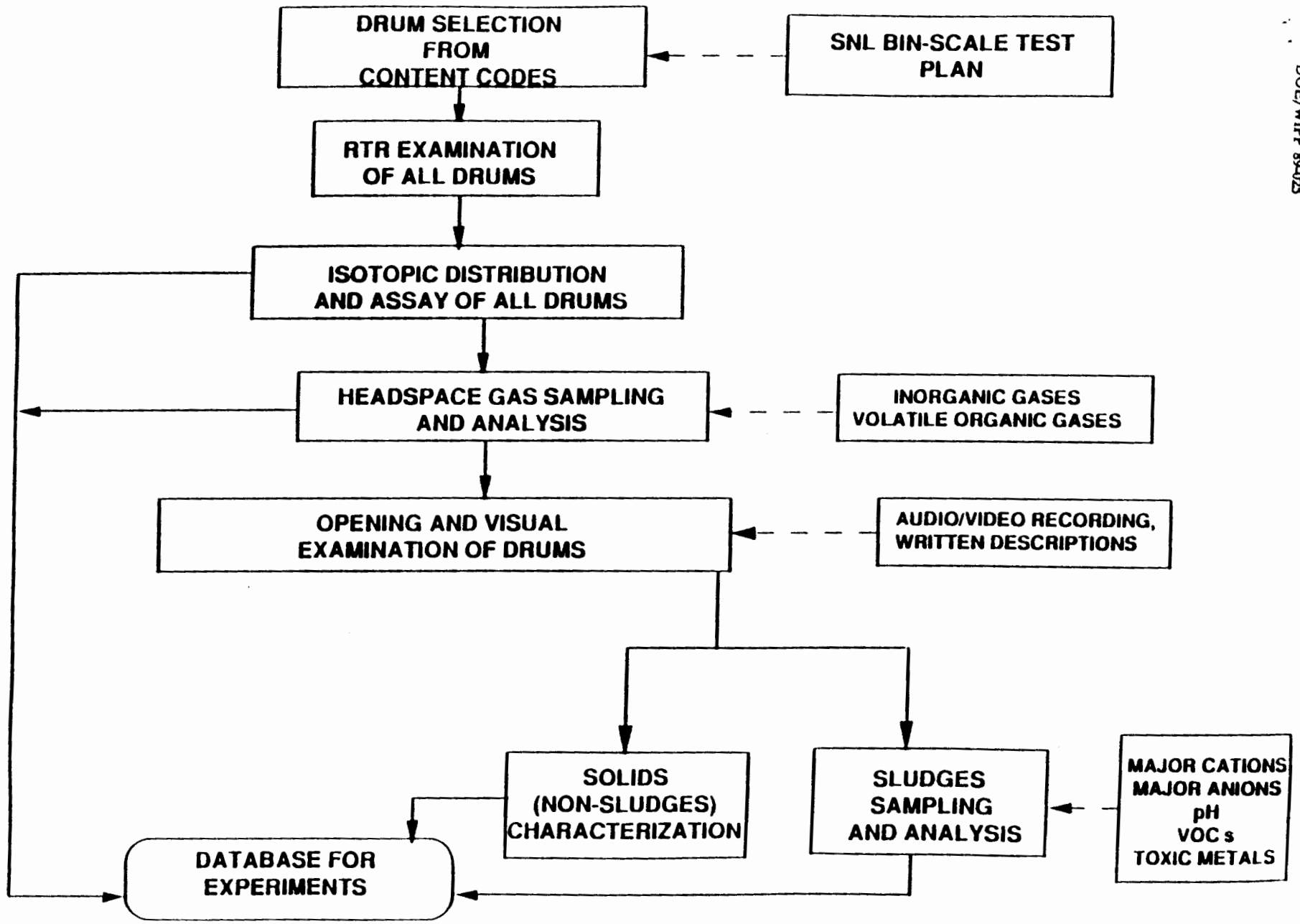


Figure 2. Pretest Characterization of Waste for Bin Scale Experiments

**TABLE 3\***  
**WIPP CH TRU WASTE TEST TYPES: TRUCON CODES CROSS-LISTING**

| <u>WIPP Waste</u><br><u>Test Type</u> | <u>TRUCON Designation:</u>                                     |   |
|---------------------------------------|--|---|
|                                       | <u>Waste Type</u>  | <u>Content Codes (description)</u>          |
| <u>HONG</u>                           | III (solid organics, newly generated)                          | 116 (paper, plastic, cloth)                 |
|                                       |  | 119 (filters; mostly organic)               |
|                                       |  | 121 (other organic solid waste)             |
|                                       |  | 123 (leaded rubber gloves)                  |
|                                       |  | 125 (solid organic and inorganic waste)     |
|                                       |  | 126 (cemented organic process solids)       |
|                                       |  | 127 (solid organic and sludge wastes)       |
| <u>HOOW</u>                           | III (solid organics, retrievably stored)                       | 216 (paper, plastic, cloth)                 |
|                                       |  | 219 (filters; mostly organic)               |
|                                       |  | 221 (other organic solid waste)             |
|                                       |  | 223 (leaded rubber gloves)                  |
|                                       |  | 225 (solid organic and inorganic waste)     |
|                                       |  | 226 (cemented organic process solids)       |
|                                       |  | 227 (solid organic and sludge wastes)       |
| <u>LONG</u>                           | II (solid inorganics, newly gen. & [old])                      | 115 [215] (graphite waste)                  |
|                                       |  | 117 [217] (metal waste)                     |
|                                       |  | 118 [218] (glass waste)                     |
|                                       |  | 122 [222] (inorganic solid waste)           |
|                                       |  | 124 [224] (pyrochemical salt waste)         |
| <u>PS</u>                             | I (solidified aqueous or homogeneous inorganic, new and [old]) | 111 [211] (cemented/solidified sludges)     |
|                                       |  | 114 [214] (cemented inorganic particulates) |

\* Adapted from Molecke, 1990 (Reference 2)

Each drum of waste to be transported to the WIPP site is assigned a specific content code. The number of drums from each waste test type, and from each content code within a waste test type, needed for the bin tests is specified by the statistical justification document (9), and all waste drums for the bin-scale tests shall be randomly selected as specified by this document. The drums that will be selected for preparation of a bin have to be chosen such that the bin meets the WIPP WAC (6) and the TRAMPAC (7). These criteria have been published and made available to the waste generator and storage sites. The methodology for selecting the drums for preparing a bin is presented in Figure 3.

### 2.2.3 RTR Examination of Waste Containers

The first step after waste drum selection is examination by real-time radiography, which will be used to make a subjective verification of the following parameters:

- Content Code of the Waste
- Presence of Free Liquids
- Contents Inventory
- Waste Packaging Configuration
- Containers That Could Contain Compressed Gases.

The purpose of this RTR examination is to utilize a non-destructive examination (NDE) technique to obtain certain waste characterization data. Correlations between the RTR examination and subsequent visual examinations (actual opening and unpacking of the waste drums documented by videotape records) can be used as verification of waste characterization in cases where only RTR examination was performed, supported by limited sampling. An example of this type of program is the pretest waste characterization of the alcove test waste, described in Section 2.3.

Results of the RTR examination shall be documented and available as records for each drum, specifically with respect to the parameters mentioned above. RTR examinations shall be performed only by trained and qualified operators, with an identifiable chain of responsibility available for each RTR record. An example of the data documentation needed from RTR examination is provided in Table 4.

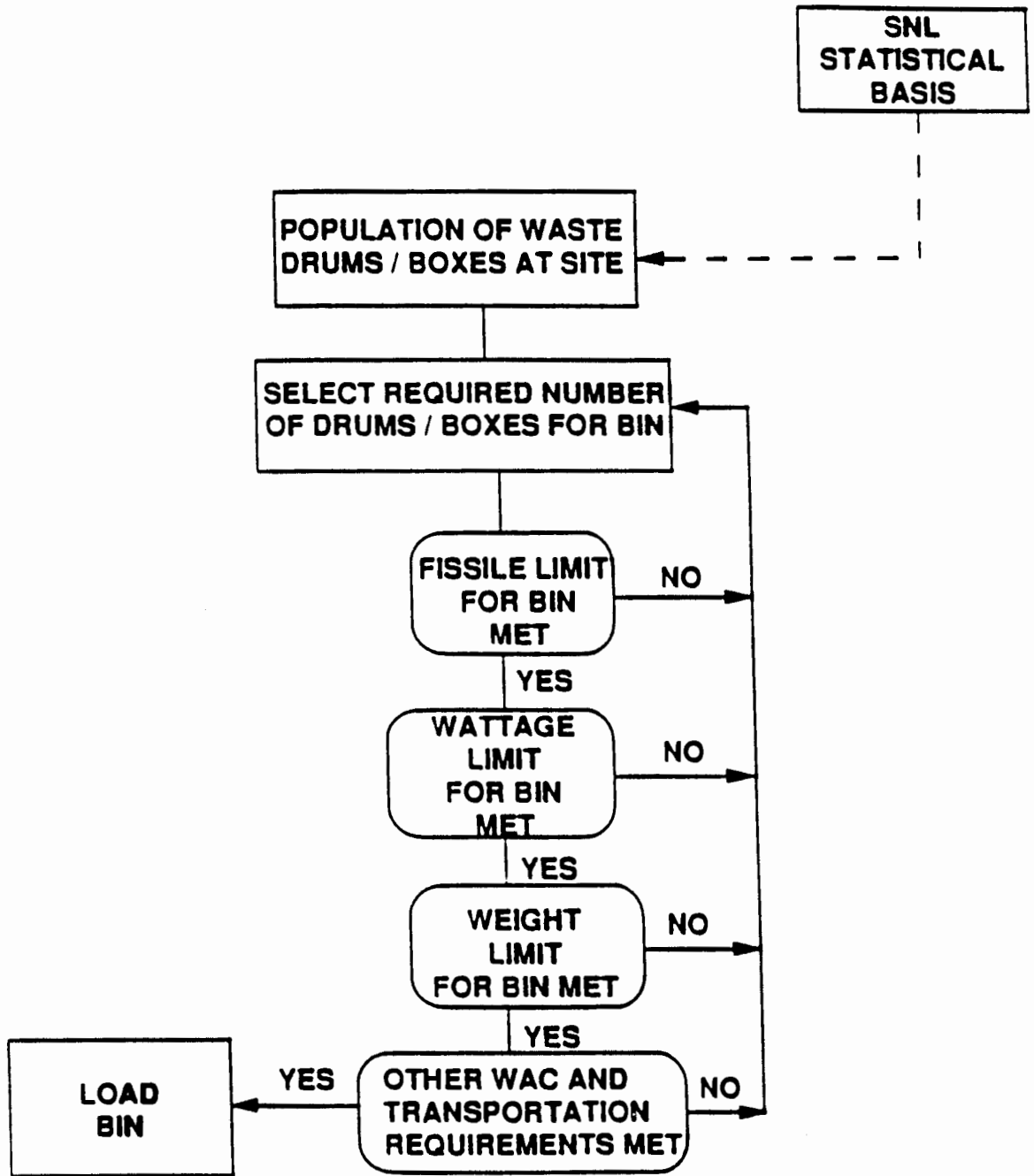


Figure 3. Criteria for Selection of Waste for Bins

**TABLE 4**  
**DATA DOCUMENTATION OF RTR**  
**EXAMINATION RESULTS**  
(Example Only)

Waste Site \_\_\_\_\_ Container I.D. \_\_\_\_\_

Waste Test Type \_\_\_\_\_

Transportation Content Code \_\_\_\_\_

RTR Examination Performed (date) \_\_\_\_\_

RTR Procedure No. \_\_\_\_\_ Rev \_\_\_\_\_

**RTR Examination Results:**

Free Liquids \_\_\_\_\_ Yes \_\_\_\_\_ No \_\_\_\_\_  
Amount \_\_\_\_\_ (estimated to the nearest 10 ml)

Compressed Gases \_\_\_\_\_

Sealed Containers Greater than 1 Gallon (TRUPACT-II limit) \_\_\_\_\_

Correct Waste Test Type \_\_\_\_\_

Brief Description of Contents Inventory \_\_\_\_\_  
\_\_\_\_\_

Waste Packaging Information \_\_\_\_\_

Waste Examination: \_\_\_ Acceptable \_\_\_ Unacceptable NCR No. \_\_\_\_\_

Operator Name (Print) \_\_\_\_\_

Operator Signature \_\_\_\_\_



#### 2.2.4 Isotopic Distribution and Assay

Every drum shall be assayed. Assay measurement techniques and error determination are described in Appendix 1.3.7 of the TRUPACT-II SAR (7). Acceptable assay techniques and lower nuclear material accountability measurement limits (it is understood that radionuclides contained in waste material are no longer accountable nuclear materials) for the different isotopes are specified in Table 5. Concentrations of some isotopes that are not measured directly are calculated, using standard isotopic compositions of the radionuclide source material used in a particular building or at a site based upon waste stream characterization (process knowledge). When bins are filled with waste unpacked from boxes, the bin will be assayed after closure.

#### 2.2.5 Requirements for Headspace Gas Samples

After a waste drum is set up in the sampling facility, a pressure measurement in the headspace shall be taken (if the drum does not have a carbon composite filter) and a gas sample obtained from the headspace. Gas sampling of the headspace shall be performed as follows:

- The drum lid and rigid drum liner lid (if present) will be removed and a gas sample shall be obtained from the headspace inside the first liner bag.
- For some drums, a sample will be taken inside the lid after puncture of the lid. This technique will provide correlation data between headspace gas inside the drum or rigid liner and head space gas sampled inside the first large liner bag.

Gas analyses shall be performed using standard gas chromatograph/mass spectrometer (GC/MS), or equivalent, testing methods. Required gas analysis and detection limits are referenced in Tables 6 and 7. Any procedural modifications from standard methods required to meet program data quality objectives for the analysis of TRU wastes must be detailed in the site-specific QAPjP. Headspace sampling for the analyses in Table 6 has been previously conducted at the Rocky Flats Plant (RFP), and may serve as the basis for development of a detailed standard sampling protocol to be applied to all DOE facilities. The RFP headspace gas sampling program is described in the report titled "Waste Drum Gas Generation Sampling Program at Rocky Flats During FY 1988" (11). Analytical deliverables and methods will be described in the system-wide pretest QAPP under development, which will be reviewed by EPA prior to initiation of sampling and analysis.

**TABLE 5**  
**ISOTOPIC CHARACTERIZATION FOR PRETEST**  
**WASTE CHARACTERIZATION**

| ISOTOPES         | ASSAY<br>TECHNIQUES                | NUCLEAR MATERIAL<br>ACCOUNTABILITY<br>MEASUREMENT LIMITS |                 |
|------------------|------------------------------------|--|-----------------|
| Pu-238           | PAN <sup>a</sup> /SGS <sup>b</sup> | 0.1 gm   | 1740 mCi        |
| Pu-239           | SGS/PAN                            | 1.0 gm   | 61 mCi          |
| Pu-240           | CNC <sup>c</sup>                   | 1.0 gm   | 239 mCi         |
| Pu-241           | --- *                              |  |                 |
| Pu-242           | W/Pu 240*                          | 1.0 gm   | 4 mCi           |
| Am-241           | SGS                                | 0.1 gm   | 324 mCi         |
| Np-237           | SGS                                | 0.5 gm   | 344 $\mu$ Ci    |
| U-233            | SGS/PAN                            | 0.5 gm   | 5 mCi           |
| U-235            | SGS/PAN                            | 1.0 gm   | 2 $\mu$ Ci      |
| U-238            | SGS/PAN                            | 1.0 kg   | 330 $\mu$ Ci    |
| Th-232           | SGS/PAN                            | 0.5 kg   | 56 mCi          |
| Cm-244           | PAN                                | 1.0 gm   | 80 Ci           |
| Cf-252           | PAN                                | 1.0 $\mu$ g  | 643 $\mu$ Ci    |
| MFP <sup>d</sup> | High Resolution<br>Gamma Detector  |  | 10-100 $\mu$ Ci |

<sup>a</sup> - Passive Active Neutron System

<sup>b</sup> - Segmented Gamma Scan System

<sup>c</sup> - Coincident Neutron Counting

<sup>d</sup> - Mixed Fission Products

\* Calculated from known initial inventory and elapsed time.

**TABLE 6**  
**HEADSPACE GAS ANALYSIS REQUIREMENTS**

| <b>ANALYTE</b>                | <b>DETECTION<br/>LIMIT</b> |
|-------------------------------|----------------------------|
| N <sub>2</sub>                | 1.0 vol%                   |
| O <sub>2</sub>                | 0.1 vol%                   |
| H <sub>2</sub>                | 0.1 vol%                   |
| CH <sub>4</sub>               | 0.1 vol%                   |
| C <sub>2</sub> H <sub>6</sub> | 0.1 vol%                   |
| C <sub>3</sub> H <sub>8</sub> | 0.1 vol%                   |
| NO <sub>x</sub>               | 0.01 vol%                  |
| CO                            | 0.01 vol%                  |
| CO <sub>2</sub>               | 0.1 vol%                   |
| Ar                            | 0.1 vol%                   |

**TABLE 7**  
**HEADSPACE GAS REQUIREMENTS**  
**VOLATILE ORGANIC COMPOUNDS (VOCs)**

| VOLATILES                                 | CAS NUMBER | LOQ<br>(ppmv) |
|---|------------|---------------|
| 1. Acetone                                | 67-64-1    | 100           |
| 2. Benzene                                | 71-43-2    | 1             |
| 3. Bromoform                              | 75-25-2    | 1             |
| 4. Butanol                                | 71-36-3    | 100           |
| 5. 2-Butanone                             | 78-93-3    | 100           |
| 6. Carbon Tetrachloride                   | 56-23-5    | 1             |
| 7. Chlorobenzene                          | 108-90-7   | 1             |
| 8. Chloroform                             | 67-66-3    | 1             |
| 9. Cyclohexane                            | 110-82-71  | 1             |
| 10. 1,1-Dichloroethane                    | 75-35-3    | 1             |
| 11. 1,2-Dichloroethane                    | 107-06-2   | 1             |
| 12. 1,1-Dichloroethene                    | 75-35-4    | 1             |
| 13. cis-1,2-Dichloroethene                | 156-59-2   | 1             |
| 14. Ethyl Benzene                         | 100-41-4   | 1             |
| 15. Ethyl Ether                           | 60-29-7    | 1             |
| 16. Methanol                              | 67-56-1    | 100           |
| 17. Methylene Chloride                    | 75-09-2    | 1             |
| 18. 4-Methyl-2-pentanone                  | 108-10-1   | 100           |
| 19. 1,1,2,2-Tetrachloroethane             | 79-34-5    | 1             |
| 20. Tetrachloroethene                     | 127-18-4   | 1             |
| 21. Toluene                               | 108-88-3   | 1             |
| 22. 1,1,1-Trichloroethane                 | 71-55-6    | 1             |
| 23. Trichloroethene                       | 79-01-6    | 1             |
| 24. 1,1,2-Trichloro-1,2,2-trifluoroethane | 76-13-1    | 1             |
| 25. 1,3,5-Trimethylbenzene                | 108-67-8   | 1             |
| 26. 1,2,4-Trimethylbenzene                | 95-63-6    | 1             |
| 27. m-Xylene                              | 108-38-3   | 1             |
| 28. o-Xylene                              | 95-47-6    | 1             |
| 29. p-Xylene                              | 106-42-3   | 1             |

### 2.2.6 Preparation of Waste Drums for Visual Examination and Weighing

Selection of proper facilities, proper protection for operators, and related safety criteria are the responsibility of the individual sites performing the waste characterization. These details shall be provided in the site-specific QAPjP and Standard Operating Procedures (SOPs). An audio video recording of the opening of the drum and the different stages of unpacking shall be performed for each drum. The video recording will focus on registering the drum contents after the drum is opened for bin preparation, details of waste packaging, and examination of the different waste packaging layers in the drum. A narration, specific to the activities of the process (e.g., "Drum Opened", "Liner Lid Removed", "Waste Bag Number XX Removed", etc.) shall accompany the video recording. In addition to the video tape recording, for each drum sampled a checklist shall be filled out detailing the findings of the visual examination. The checklist can be site specific, and shall, at a minimum, include a record of the following items:

- Site examining waste
- Date and time of examination
- Original date of closure of drum/box sampled
- Drum ID Number
- Content code of waste
- Waste test type
- Presence/absence of carbon composite filter
- Presence/absence of rigid drum liner (punctured/unpunctured)
- Gas pressure in headspace (if drum is unfiltered)
- Number of liner bags
- Configuration of inner bags
- Number of individual bags or packages
- Content descriptions and weights of individual packages, to the nearest 10 grams for solid waste and 100 grams for sludges
- Major differences with RTR examination, if any
- Date and time of bin closure
- Bin ID Number
- Name(s) of personnel performing examination

### 2.2.7 Requirements For Solid Samples

Descriptions of the solid contents of the drum shall be recorded in the checklist under "Contents of Container". The description can be brief but should clearly identify all discernable major items in the waste and classify each using the terminology of parameters listed in Table 1. Individual bags, or packages, or contents of each bag within the drum shall be weighed, with their weights recorded to the nearest 10 grams (100 grams for sludges). A brief written description of the contents of the bags shall contain a rough estimate of the amounts of each constituent in the bags (e.g., kimwipes-20%, rubber gloves-30%, etc.) based on the operator having completed the Visual Examination Training Program established by DOE. The written records shall be supplemented with the video/audio recording (Section 2.2.6).

### 2.2.8 Requirements for Sludge Samples

Drums containing sludge materials (including solidified sludges) will be sampled to characterize the contents of the sludge with respect to major cations and anions and pH. Standard protocols will be developed for the sampling and analysis of sludges to provide representative data for the analytes listed in Tables 8 and 9. All procedural modifications required to meet data quality objectives for the analyses of TRU wastes must be detailed in the site-specific QAPjP. Analytical deliverables will be described in the system-wide pretest QAPP under development. Analytical methods used in this pretest waste characterization will be reviewed by the EPA prior to their use for analyses.

### 2.2.9 Additional Characterization of Sludge Samples

Sludge samples used in the bin-scale and alcove tests are not required to be analyzed for VOCs and toxic metals as part of the pretest waste characterization (Section 2.1), in support of performance assessment, because these compounds are not expected to have an effect on gas generation rates. However, as further verification of site specific process knowledge information in support of RCRA, sludge samples from the bin-scale waste will be characterized for hazardous constituents. Sampling and analysis procedures are being developed for the sludges. The hazardous constituent data from the test waste will be used to satisfy site-specific RCRA requirements and for future comparison to data gathered by other sites as wastes continue to be generated. Method development is only in the preliminary stage; details of sludge sampling and analysis procedures are not

**TABLE 8**  
**SLUDGE ANALYSES REQUIREMENTS FOR CATIONS**

| <b>ANALYTE</b> | <b>DETECTION<br/>LIMIT</b> |
|----------------|----------------------------|
| Sodium         | 50 ppm                     |
| Calcium        | 50 ppm                     |
| Magnesium      | 50 ppm                     |
| Iron           | 50 ppm                     |
| Potassium      | 50 ppm                     |
| Aluminum       | 50 ppm                     |

**TABLE 9**  
**SLUDGE ANALYSES REQUIREMENTS FOR ANIONS**

| <b>ANALYTE</b> | <b>DETECTION<br/>LIMIT</b> |
|----------------|----------------------------|
| Nitrate        | 10 ppm                     |
| Phosphate      | 10 ppm                     |
| Chloride       | 10 ppm                     |
| Sulfate        | 20 ppm                     |
| Carbonate      | 10 ppm                     |



available, but will be documented in a future revision of this document and a future revision of the system-wide pretest QAPP, including SOPs.

### 2.3 PRETEST WASTE CHARACTERIZATION FOR ALCOVE TESTS

#### 2.3.1 Introduction

As described earlier in Section 2.0, the waste for the alcove tests is subdivided into "as received" waste and "specially prepared" waste. These wastes also fall into the same four WIPP waste test types as for the bin-scale tests (2). The number of drums belonging to each WIPP waste test type are defined in the test plan for the alcove tests (3). A summary flowchart of the pretest characterization needs for the alcove waste is presented in Figure 4. Each of the steps in the flowchart is described below. To avoid duplication, the bin-scale details presented in Section 2.2 are referenced where appropriate.

#### 2.3.2 Selection of Drums for Alcove Tests

The waste drum equivalent volumes, by WIPP waste test type, needed per alcove are listed in Table 10.4 of the test plan for the alcoves (3). Content codes that fit into the waste types are listed in Table 3 of this plan. Selection of drums or boxes within each waste test type will be done randomly, in the same way as for the bin-scale tests (Section 2.2.2). The same restrictions as for the bin-scale test drums (meeting both the WAC and the TRAMPAC) apply to these drums/boxes.

#### 2.3.3 RTR Examination of Waste Containers

RTR examination is required for all the waste drums and boxes to be used for the alcove tests. The criteria will be identical to those for the bin-scale tests (Section 2.2.3) for direct comparison with results of the bin-scale tests.

#### 2.3.4 Isotopic Distribution and Assay

All drums to be used in the alcove tests shall be assayed. Assay measurement techniques and error determination are described in Appendix 1.3.7 of the TRUPACT-II SAR (7). Acceptable assay techniques for the different isotopes are specified in Table 5 of this plan.

### 2.3.5 Requirements for Headspace Gas Samples

These requirements apply only to the statistical population of drums (as described in Section 1.4). Criteria are the same as for the waste for the bin-scale tests (Section 2.2.5). The size of the statistical population will be dependent on the results from opening and characterizing drums/boxes for loading experimental bins.

### 2.3.6 Preparation of Waste Drums for Visual Examination and Weighing

These requirements apply only to the statistical population of drums. Criteria are the same as for the waste for the bin-scale tests (Section 2.2.6).

### 2.3.7 Requirements for Solid Samples

These requirements apply only to the statistical population of drums. Criteria are the same as for the waste for the bin-scale tests (Section 2.2.7).

### 2.3.8 Requirements for Sludge Samples

These requirements apply only to the statistical population of drums. Criteria are the same as for the waste for the bin-scale tests (Section 2.2.8).

## 2.4 QUALITY ASSURANCE

The characterization program, including all the preparatory steps, samples, and analyses, must be controlled in accordance with WIPP requirements (12) and the basic criteria identified in the QAPP which corresponds to the QA requirements of EPA(13,14). The characterization program will address the EPA requirements relative to sample size, preservation techniques, chain of custody, type of containers, holding times, and any other pertinent items. EPA-approved test methods will be used, if possible, and verified. If modifications are necessary for personnel protection from the radioactivity, these modifications will be submitted by WPO through DOE Headquarters (HQ) for submittal to the EPA.

The QAPjPs developed at each participating site will provide a detailed description of the sampling and analytical functions, as well as additional quality related objectives, in accordance with the EPA Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (14) and the criteria listed in Tables 4 through 9. Characterization activities will be performed in conformance with QA requirements that will be specified in the system-wide QAPP under development. The QAPP will be submitted by the WPO through DOE-HQ for review by the EPA. If offsite laboratories are used for analyses, those laboratories must also prepare and submit a QAPjP covering their scope of work.

The participating sites will be responsible for identifying and performing the QA/QC tasks associated with the characterization and packaging activities, and must include those activities in the site-specific QAPjP submitted to the WPO for approval. The WACCC will assure that approved sampling and analytical procedures, trained personnel, specified and calibrated equipment, and adequate facilities will all be available prior to commencement of actual waste characterization activities and bin loading at all participating sites. WACCC personnel will conduct audits and/or surveillances to assure that sites are in compliance with their approved site specific QAPjPs, and affected state and federal organizations and agencies will be present as observers during these activities.

### 3.0 REFERENCES

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7. "TRUPACT-II Authorized Methods for Payload Control," Appendix 1.3.7, in Safety Analysis Report for the TRUPACT-II Shipping Package, Revision 4, Nuclear Packaging, Inc., Federal Way, Washington, August 1989.
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13. "Test Methods for Evaluating Solid Waste Physical/Chemical Methods," SW 846, 3rd edition, Office of Solid Waste and Emergency Response, Environmental Protection Agency, Washington, D.C., November, 1986.
14. "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", QAMS-005/80, Office of Monitoring Systems and Quality Assurance, Office of Research and Development, Environmental Protection Agency, Washington, D.C., February, 1983.

4-0 GLOSSARY

|            |   |
|------------|---|
| CAS        | Chemical Abstract Service   |
| CH-TRU     | Contact-Handled Transuranic                                       |
| DOE        | U.S. Department of Energy   |
| DOE-HQ     | U.S. Department of Energy-Headquarters                            |
| EEG        | Environmental Evaluation Group                                    |
| EID        | Environmental Improvement Division, in New Mexico                 |
| EPA        | U.S. Environmental Protection Agency                              |
| EPA-ORP    | U.S. Environmental Protection Agency-Office of Radiation Programs |
| EPA-OSW    | U.S. Environmental Protection Agency-Office of Solid Waste        |
| HONG       | High Organic Newly Generated Waste                                |
| HOOW       | High Organic Old Waste  |
| IAG        | Interagency Agreement   |
| LONG       | Low Organic Newly Generated Waste                                 |
| LOQ        | Limit of Quantification   |
| NMVP       | No-Migration Variance Petition                                    |
| PA         | Performance Assessment  |
| PS         | Process Sludge (TRU waste)  |
| QA         | Quality Assurance   |
| QC         | Quality Control   |
| QAPP       | Quality Assurance Program Plan                                    |
| QAPjP      | Quality Assurance Project Plan                                    |
| RCRA       | Resource Conservation and Recovery Act, 40 CFR Part 268.6         |
| RTR        | Real Time Radiography   |
| SAR        | Safety Analysis Report  |
| SNL        | Sandia National Laboratories                                      |
| TRU        | Transuranic   |
| TRAMPAC    | TRUPACT-II Authorized Methods for Payload Control                 |
| TRUPACT-II | Transuranic Package Transporter-II                                |
| VOC        | Volatile Organic Compound   |
| WAC        | Waste Acceptance Criteria   |
| WACCC      | Waste Acceptance Criteria Certification Committee                 |
| WIPP       | Waste Isolation Pilot Plant                                       |
| WPO        | WIPP Project Office   |