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VIA FACSIMILE: (202)586-4403

August 26, 1998

The Honorable Bill Richardson
Secretary of Energy
U.S. Department of Energy
Washington D.C. 20585

RE: Confirmatory Sampling and Analysis Plan for Waste Stream TA-55-43, Lot No. 1.

Dear Secretary Richardson:

The New Mexico Environment Department (NMED) has reviewed the Confirmatory Sampling and Analysis Plan (SAP) submitted by the Department of Energy (DOE) on July 27, 1998. NMED has concluded, based upon review of the SAP, that DOE has failed to adequately characterize Waste Stream TA-55-43, Lot No. 1, to demonstrate that it contains no hazardous waste under the New Mexico Hazardous Waste Act and regulations.

The SAP contains numerous inadequacies which are detailed in the attached "Review of the Los Alamos Confirmatory Sampling and Analysis Plan for Waste Stream TA-55-43, Lot No. 1". NMED has determined the following areas to be inadequate:

1. Sample representativeness, including the selection of samples, sample collection within containers, inter-material variability and analytical suites;
2. Sample weight/size;
3. Sample integrity; and
4. Sample frequency.

Please refer to the Review for further information.

If you have any questions, or would like to meet regarding this decision, please call Ed Kelley of my staff at (505)827-2855.

Sincerely,

Peter Maggiore

Peter Maggiore, Secretary
New Mexico Environment Department

cc w/att:
The Honorable Senator Domenici
The Honorable Senator Bingaman
The Honorable Congressman Skeen
The Honorable Congressman Redmond
The Honorable Congresswoman Wilson
The Honorable Governor Johnson
The Honorable Tom Udall, Attorney General, State of New Mexico
The Honorable Janet Reno, Attorney General of the United States, U.S. Department of Justice
Carol Browner, Administrator, Environmental Protection Agency
Gregg Cooke, Regional Administrator, Environmental Protection Agency
Jennifer Salisbury, Secretary of Energy, Minerals & Natural Resources Department
W. John Arthur, III, Asst. Manager, Albuquerque Area Office
Mike McFadden, Manager, Carlsbad Area Office

REVIEW OF THE LOS ALAMOS CONFIRMATORY SAMPLING AND ANALYSIS PLAN FOR WASTE STREAM TA-55-43, LOT NO. 01

1.0 Introduction

The Los Alamos National Laboratory (LANL) prepared a document entitled "Confirmatory Sampling and Analysis Plan (Plan) for Waste Stream TA-55-43, Lot No. 01", which was submitted as a strategy for assessing whether the specified waste stream is a hazardous waste. The plan was reviewed by the New Mexico Environment Department (NMED) and Techlaw Inc., consultant to NMED[1]. Review of the Plan includes reference to the applicable Resource Conservation and Recovery Act (RCRA) regulations, SW-846 and a 1992 EPA/DOE publication regarding sampling and analysis of heterogenous waste[2].

The Plan was assessed in terms of:

- Sampling Representativeness
- Sample Size
- Sample Integrity
- Sample Frequency

Each deficiency or concern identified includes a discussion of the concern and a recommended correction or alternate approach. This review sets forth methods and recommendations which, if incorporated into the Plan, would adequately address deficiencies identified by NMED.

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[1] The TechLaw team included six personnel with skills and background in mixed waste characterization at DOE sites and/or RCRA waste analysis.

[2] EPA/DOE, *Characterizing Heterogeneous Wastes: Methods and Recommendations*, EPA 600/R-92/033, 1992.

2.0 Evaluation of the Plan

Summary

The Sampling and Analysis Plan (Plan) as presented is not adequate as a confirmatory sampling plan to make a hazardous waste determination regarding Lot No. 1 of Waste Stream TA-55-43. The deficiencies or concerns identified are presented below. Recommended strategies or procedures for correcting each deficiency or concern are also provided.

2.1 Sampling Representativeness

Selection of Samples. The selection of containers from which samples will be collected and the selection of samples within those containers is poorly justified in the Plan; a random container selection strategy would be more appropriate. The Plan includes the following sample selection strategies that do not provide an adequate degree of randomness based upon the requirements found in SW-846:

- HEPA filter samples will be collected from drum 57007, which is only one out of six available drums containing HEPA filters (Plan pg. 6-7). Rags, glovebox gloves, and plastic bags will be sampled from drum 57025 because all of these items were in the same drum (Plan, pg.7).
- Repackaged drums are excluded from sampling to avoid costs incurred from opening packages (Plan, pg. 6).
- Drum 55605 is excluded from sampling because it contains a small amount of nuclear material (Plan, pg. 6).
- The Plan does not indicate that drums were selected randomly and the method of random selection was not indicated.
- There is no evidence that the material sampled in each drum was selected using a random selection process or that sampling will occur in the most likely contaminated areas within a drum.

Alternative Approach: Chapter 9 of SW-846 indicates that sampling accuracy is achieved through the collection of random samples and that each unit (drum) of the waste must have an equal chance of being sampled and measured (EPA, pg NINE-6).

A random drum selection process is required. If random drum selection results in containers which cannot be sampled, than an alternative drum selection process may be proposed. The

alternative drum selection methodology and justification must be of sufficient detail to demonstrate that unbiased samples are collected; for example, the Plan did not provide any technical justification for excluding the repackaged drums. The drum selection and sampling strategy is intended to apply only to TA-55-43, Lot No. 01. The proposed selection and sampling process is not intended to apply to the entire waste stream. However, a similar strategy could be applied, with prior approval by NMED, to additional waste stream lots on a lot by lot basis.

Containers should be selected each material type, and the number of containers statistically/randomly selected from the total population of drums containing that material type should be determined using the method presented in Section 2.4 of this report.

Random sample selection is not restricted to the selection of containers, but is also required for the selection of sample location within the containers. The Plan should document the method used to randomly select sampling locations within each drum. For example, waste units (drums) may be subdivided into a three dimensional grid and a number assigned to each grid cube. Numbered grid cubes could then be chosen using a random number table or random number generator. (EPA, pg. NINE-8). In the event that a grid does not contain the waste, another grid will be randomly chosen. The random selection process should be performed for each waste material type.

Sample Collection Within Containers. With the exception of rags and plastic, the Plan proposes the collection of material from a single drum, which are later composited into 3 samples in proportions representative of the waste stream (Plan, pg. 9). However, heterogeneous debris waste as in this waste stream lot are likely to have a wide range of contamination among individual items within a material type. Collecting samples from a single drum for each waste material is not adequate for characterizing the population of drums and for assessing the average properties of the whole waste (EPA, pg. NINE-5).

Alternative Approach. The sample collection strategy should include samples from multiple drums for each waste material. As described below, randomly selected Grab or Composite samples should be collected within each of the multiple drums selected, and for each waste material type. The sample would be taken from randomly selected sectors as discussed in the previous section of this report.

The sampling strategy should include random selection of containers for sampling (see Section 2.4 for container selection process), and then random selection of sample locations from within these containers from which grab samples would be collected from each waste material. The appropriate number of grab samples to be collected is not established in available RCRA guidance. However, a minimum of 3 grab samples for each matrix from each container, is suggested. The grab samples for each material would then be composited, resulting in a single matrix sample for each drum sampled. Enough sample should be collected to account for the sample and quality control requirements of the TCLP analysis. The selection of the proper

number of drums to be sampled, assuming one matrix composite sample/drum, is defined in section 2.4 of this report.

Based on the information provided regarding the waste matrices, the following matrices should be sampled:

- Plastics
- HEPA Filters
- Metals
- Rags and Combustible Items
- Rubber
- Rust colored powder

Matrices with limited availability such as the rust colored powder may be subject to a modified sampling approach. Compositing may be required between drums containing this material to obtain an appropriate sample size, if any one drum does not contain sufficient material to collect an appropriate sample.

Inter-material variability. The Plan does not adequately account for variability of material uses within each material type. For example, the description of possible plastic items in waste indicates that plastic bags comprised the majority of plastic waste. However, tape, plastic analysis vials, Teflon containers, plastic sheeting, and plexiglass were also included as plastic wastes (Plan, pg. 4). Each of these plastic sub-materials had different purposes and were subject to potentially different types of contamination. The Plan only accounts for the sampling of plastic bags and bottles.

Alternative Approach. The Plan should be modified to account for the representative sampling of other sub-materials within each material category. At a minimum, the random selection process for each material type must be independent of sub-material type. Justification of the material subgroup exclusion should be provided to better justify the selected material and to avoid more rigorous sampling, presumably because a purpose of confirmatory sampling could include specific sampling of materials of particular concern. However, this approach must be thoroughly justified to avoid the perception that the selected samples are not appropriate and conservative.

Analytical Suites. The proposed analytical suite within the Plan is poorly justified.

Alternative Approach. The Plan specifies a limited number of TCLP analyses designed to confirm limited toxicity characteristic chemicals only. Metals TCLP samples are collected for all waste materials. However, only HEPA filters are analyzed for organics. The sampling plan does not adequately justify the existence of VOC's found in the headspace gas. As a result, additional samples and analyses should be planned and executed for those VOC compounds detected in

headspace gas to verify that these wastes are not the result of F-listed solvent use. The sampling may be limited to those materials that would be most likely to be contaminated with F-listed waste. Alternately, a more complete and acceptable evaluation of the Radiolysis and off-gassing process that adequately demonstrates that the VOC's are not attributable to F-listed processes could preclude the collection and analysis of additional samples. Further, this justification should address whether VOC headspace analytes could be the result of laboratory contamination through evaluation of associated laboratory quality control data.

2.2 Sample Weight/Size and Determination of Sample Composition

The current Plan indicates that three composite samples of 100 grams each will be collected, with sample composition to be determined via TCLP analysis of extracts from these samples. Each composite sample will consist of a proportional weight of each material type. Waste material groups with a small composition percentage in comparison to the entire waste stream, such as the rust colored powder, would be difficult to characterize if the waste material is only included in a composite sample based upon the known composition percentage of the waste materials. However, LANL has indicated that an additional three samples will be collected for TCLP organics in HEPA filters and an additional sample will be collected for the rust colored powder to evaluate TCLP metals and qualitative evaluation, implying that sufficient quantities of these materials are available. The joint DOE/EPA guidance recommends that samples should be collected from each waste material parameter (DOE/EPA, pg. 83).

In addition, the Plan does not account for the collection of sufficient sample material to account for the performance of laboratory quality control samples such as duplicates and matrix spikes. These laboratory quality control samples are necessary to assess the quality of the analytical process. The Plan also does not account for the collection of additional sample for additional methods of analysis, such as the collection of sample for TCLP VOC's for the HEPA filters.

Alternative Approach. The composite sample for each waste material should be 100 grams to accommodate the TCLP sample preparation process. That is, the three-100 gram composite samples proposed by LANL should be revised to include one 100-gram sample for each matrix composite sample collected, with the number of composite samples to be collected (i.e. number of drums to be sampled assuming one composite/drum) determined by the method presented in Section 2.4 The Plan should indicate the need to collect additional laboratory quality control samples, such as duplicates and spiked samples, that are specified and required in the analytical methods. The Plan should also account for the additional sample that is necessary for the performance of additional sampling methods, such as the TCLP VOC analysis, for the HEPA filters.

The "composition" of a given waste would then be determined by mathematically combining the appropriate proportion of material rather than by actually mixing samples and generating a

leachate for analysis.

2.3 Sample Integrity

The Plan does not include basic information about the types of sampling equipment and containers that will be used, how sampling equipment is decontaminated between uses, and how samples are preserved. These elements are necessary to demonstrate that the integrity of the sample is maintained from collection to analysis and that cross contamination between samples or media does not occur. In addition, the Plan does not include a discussion of the collection of any potential field QC samples such as field blanks and field duplicates to assess the accuracy and precision of the sample collection process. The Plan should also include a discussion of the collection of field blanks and field duplicates. The Plan does not specify chain of custody procedures that would document the integrity of the sample through the analytical and sampling process, nor does it specify analytical quality control and calibration requirements and specifications (with the exception of method detection limits for TCLP analyses). In addition, the Plan does not specify how the quality of analytical results will be assessed and verified through validation.

Suggested Approach. The Plan should include or reference adequate procedures and specifications for the following:

- Specifications for sample container type and size
- Specifications for chemical and physical preservation requirements for each analysis type
- Specifications for sampling equipment to preclude introduction of contamination
- Procedures for sampling equipment use and decontamination to prevent cross-contamination between sampling events

(EPAb, pgs 4-5)

At a minimum, the following information must also be included or referenced in the Plan:

- Collection frequency for field quality control samples
- Procedures for collecting field quality control samples
- Acceptance criteria for field quality control samples

(EPAb, pgs 3, 5)

The Plan should present an overview of the custody requirements for the project, including the following:

- Examples of custody documentation (custody records, sample labels) to be used
- Documentation of procedures used to package and ship samples to the analytical

laboratory
- Documentation of procedures used to complete custody documentation to ensure that the sample collection location, time, sampler, and associated sample identification is documented on the chain of custody documentation

(EPAb, pg 6)

The Plan should include or reference the following information:

- Calibration frequency and procedures for all analytical instrumentation to include:
  - Method of calibration
  - Instrument specific calibration procedures and requirements
  - Frequency of calibration checks
  - Define acceptance criteria for all calibration checks
- Sample analysis and preparation methods for all analyses
- Quality Assurance objective criteria for the following:
  - Field precision
  - Laboratory precision
  - Laboratory accuracy (blanks, lab control samples, spiked samples)
  - Field accuracy (field blanks)
  - Calculations for each quality assurance indicator
  - Completeness requirements for data (to include definition of valid data)
- Corrective action taken in the event Quality Assurance objectives are not met
- Data validation plan
- Data reporting requirements to include units of measure, reporting of quality assurance results, and summary of data reduction procedures

(EPAb, pgs 7-9, 13-15)

2.4 Sample Frequency

The number of samples collected in the Plan is not based on a standard statistical approach and does not account for an evaluation of sampling precision and accuracy. SW-846 indicates that the validity of a sampling scheme is based upon the sampling precision and accuracy. Sampling precision is most commonly achieved by taking an appropriate number of samples. Alternately, precision can be achieved through increasing the sample size. (EPA, pg.9-8) Substantially increased sample size may not be appropriate due to worker safety concerns. Therefore, sample precision for this waste stream would be most readily achieved through the collection of an appropriate number of samples.

Suggested Approach. SW-846 recommends a stratified random sampling strategy that is

intended to ensure that an adequate number of samples are collected in each sample strata (waste material type) (EPA pgs NINE-14 to NINE-20). A preliminary evaluation of sample size is based on any available existing data or process engineering data.

Acceptable knowledge and process data could be used to generate estimates of the mean and deviation for each constituent in each material type; this preliminary estimated mean and deviation would then be used to establish an initial sample frequency for each constituent in each material in accordance with the following equation:

$$n=t^2_{.20}s^2/RT-x_{mean}$$

Where: n = number of samples
$t_{.20}$= student t test value
RT = Regulatory Threshold
x_{mean} = concentration mean of preliminary results
s^2 = standard deviation of preliminary results
(EPA pg. NINE-3)

Note that if the preliminary samples are based on actual sampling and analysis data, the preliminary samples may be used as part of the "n" number of samples that must be collected, if these preliminary sample collection and analysis procedures are the same as those used in this Plan. The number of samples to be collected would be reevaluated after the "n" number of containers are sampled, using revised means and deviations for each constituent derived from the sample analysis. As part of this process, the mean concentration of each analyte per waste material type would be determined. Weighted constituent averages could then be calculated, based upon the most accurate available estimate of the percentage composition of the waste materials. The weighted concentration and deviation for each analyte could then be used to determine the UCL_{95} for each analyte. Revised means and standard deviations would be calculated using the new data to assess whether the "n" number would need to be increased.

Using the above statistical analysis, the following sample selection process is required:

- Statistically determine the number of drums that must be sampled for each waste material matrix.
- Randomly select a drum and collect a composite sample for each waste material matrix that is contained in that drum.
- Additional drums are randomly selected and the sampling process is repeated until all the required samples are collected for each waste material matrix.
- When the necessary number of samples have been collected for a given waste material matrix, any additional drums selected to sample other matrices will not be sampled again for the waste matrices that have been completely sampled.

This strategy is supported by the joint 1992 DOE/EPA document "Characterizing Heterogeneous Wastes: Methods and Recommendations", which indicates that heterogeneous drum wastes should be sorted by material type and that the inherent possible contamination variation within each waste type is considered through one of the following strategies:

- Collect a large portion of each material as a sample
- Collect a number of equivalent samples for each material to allow a statistical estimate of contaminant concentrations
- Deliberately sample the most contaminated objects as determined through visual examination or field screening. (DOE/EPA, pgs. 82-83)

REFERENCES

EPA/DOE, *Characterizing Heterogeneous Wastes: Methods and Recommendations*, EPA 600/R-92/033, 1992.

EPA, *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition, 1986.

EPA(b), *Preparing Perfect Project Plans*, EPA/600/9-89/087, 1989.

bc w/out att: Ed Kelley, Director of Water and Waste Management Division, Environment Department
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