FFCO

Department of Energy
Albuquerque Operations Office
Los Alamos Area Office
Los Alamos, New Mexico 87544

APR 22 1999

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CERTIFIED MAIL - RETURN RECEIPT REQUESTED

Mr. Michael Taylor
FFCO Project Manager
Hazardous and Radioactive Materials Bureau
New Mexico Environment Department
2044 Galisteo Street, Building A
P. O. Box 26110
Santa Fe, New Mexico 87505

Dear Mr. Taylor:

Subject: Los Alamos National Laboratory (LANL), Federal Facility Compliance Order (FFCO), October 4, 1995, Section V.B

The enclosed report has been modified to address concerns raised by NMED in the letter dated March 17, 1999, and during our meeting on March 24, 1999. The report contains the results of a Department of Energy (DOE) and University of California (UC) Sort, Survey, and Decontamination (SSD) project. Waste previously identified as mixed waste has been found to be hazardous waste without a radioactive component. It is intended that the information provided meets the requirements under Section V.B of the FFCO for NMED to consider such waste a covered waste for a period of ninety days upon approval by NMED of Respondents' written determination.

DOE and UC certify that strict chain of custody requirements were adhered to during this project. All waste items included in this study are confirmed to have a treatment and disposal option as hazardous waste. Within 90 days of NMED's approval of this package, all waste items will be shipped to an off-site commercial facility for treatment and disposal.

Enclosed as Attachment A is a redline/strikeout version of the document. Enclosed as Attachment B is a clean copy version of the document. Also enclosed, as Attachment C, is a Certification Statement prepared in accordance with the requirements of Section XX, *"Documents, Information, and Reporting"* of the FFCO.

Please contact me at (505) 665-5042 or Beverly Martin at (505) 665-0714 if you have any questions.

Sincerely,

H. L. "Jody" Plum
Office of Environment

LAAME:3JP-117

Enclosures

1947

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bcc w/o enclosures:
H. Haynes, Office of Counsel, LAAO
T. Baca, EM-DO, LANL, MS-J591
K. Hargis, EM-WM, LANL, MS-J591
B. Martin, EM-WM, LANL, MS-J591
J. Kelly, EM-SWO, LANL, MS-J595
A. White, EM-SWO, LANL, MS-J595
S. Brown, LC-GEN, LANL, MS-A187
J. Rochelle, LC-GEN, LANL, MS-A187
A. Jackson, ESH-19, LANL, MS-K490
J. Grimm, WMD, AL

ENCOSURE A

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1.0 Introduction

The Sort, Survey, and Decontamination Project at Los Alamos National Laboratory (LANL), which is owned by the Department of Energy (DOE) and operated by the University of California (UC), was initiated under recommendations set forth in the Albuquerque (AL) Mixed-Waste Treatment Plan (March 1994). This plan recognized that large numbers of waste items had been classified as suspect radioactive waste solely because it had come from Radioactive Materials Management Areas (RMMA) in accordance with DOE policy. Process knowledge indicated that it was unlikely that this waste was actually radioactive. Waste items for this project were specifically selected because process knowledge and generator interviews indicated that it was, in all likelihood, not radioactive. Analytical results and field survey data were used as the basis to make the determination that waste items discussed in this report are hazardous waste without a radioactive component.

During 1995 and 1996 an effort was initiated, with the support of the DOE, to perform detailed radiological surveys of this waste to verify that much of the "suspect" waste was free of radioactive constituents. RUST Geotech, Inc. as the prime contractor at the DOE Grand Junction Project Office, began this effort (termed the Sort, Survey, and Decontamination [SSD] Project). On September 5, 1996, MACTEC Environmental Restoration Services, LLC was awarded the Technical Assistance and Remediation contract for the DOE Grand Junction Project Office (GJPO) and subsequently took over the final stage of the SSD Project.

After completion of the field surveys, DOE and UC thought that it would not be cost effective to perform any additional field surveys or to complete the review process required to prepare a data submittal for NMED review. At that time DOE and UC intended to pursue treatment and disposal of this waste as MLLW. However, a data review was required to treat and dispose of this waste as MLLW. The review was necessary to ensure that the data collected would meet the commercial facilities' waste acceptance criteria. During this review process, it became clear that many of the items were not radioactive and that defensible and valid data had been collected to demonstrate this determination.

2.0 Work Plan Summary

The project was performed under the *Site-Specific Work Plan for Sort, Survey, and Decontamination of Potential Mixed Waste at Los Alamos National Laboratory* (January 1995). DOE approved the plan and implementing procedures. The plan provided the foundation upon which data would be collected to support a DOE and UC determination that the waste items were ~~free of DOE added radioactive materials~~ hazardous waste without a radioactive component. This plan is summarized below.

2.1 Objectives

The objective of the SSD Project was to reduce the mixed-waste inventory at LANL through sorting, surveying, and sampling and analysis. This objective was accomplished through:

- Deploying a mobile field team to sort, survey and perform sampling and analysis of suspect waste materials.
- Provide sorting space, sampling and analysis, and survey equipment and instrumentation.
- Preparing documentation of the process and the mechanism that results in the certification that hazardous wastes are not contaminated with radioactivity, thereby allowing DOE and UC to release waste to a RCRA permitted treatment, storage, or disposal facility.

2.1.1 Performance Objectives

SSD performance objectives consisted of administrative controls and technical procedures that have been used for documentation and management, surveying, and sampling and analyzing the waste to ensure that hazardous wastes determined to be acceptable for release to off site facilities for treatment and disposal are not contaminated with radioactivity. The following controls and procedures were developed to meet these objectives:

Administrative Controls:

- Cradle-to-Grave Tracking System
- Quality Assurance/Quality Control
- Training Records
- Records Management

Technical Procedures:

- Release limits for water, liquids other than water, surface contamination, and volume-contaminated solids.

2.1.2 Data Quality

The data quality objectives for this project have been to provide data inputs for decision-makers that will allow waste to be categorized and managed as non-radioactive waste. The data needed to satisfy the SSD objectives have been generated through procedures covering the following subjects:

- Radiation dose rate surveys.
- Hand-Held surface contamination surveys.
- Use of liquid scintillation analysis for tritium, gross alpha, gross beta analysis.
- Sampling and analysis of volume contaminated waste.
    - Sampling equipment including augers, COLIWASAs, core borers, tube thief samplers, etc.
    - Analytical instrumentation including gamma spectrometer, liquid scintillation counter and gross alpha/beta detector.
- Document control and management

- Analytical laboratory quality control practices and protocols.

2.2 Procedures

The SSD project was fully implemented under a set of "change controlled" procedures. The procedures were written specifically to ensure that the data collected would enable defensible decisions to be made regarding waste disposition. Further, the procedures required that appropriate documentation be produced and maintained so that decision-makers could complete a review of results and necessary QA/QC requirements. A full listing of these procedures is found in Attachment II.

2.2.1 Surface Radiological Surveys

Surface radiological surveys were performed on waste items. Both direct reading and removable contamination surveys were performed. The minimum detectable activity for each of these survey methods was at or below the release limits specified in DOE Order 5400.5, "Radiation Protection of the Public and Environment" (DOE, January 7, 1993). The procedures reflected and incorporated standard industry practice for completion of these types of activities.

Instruments used to carry out these surveys included:

The Eberline E-600 Ratemeter/Scaler with an Eberline SHP-330 detector was used for direct reading surveys for total alpha and beta. The E-600 is a hand held instrument that is programmed to read out directly in disintegrations per minute (dpm). The SHP-330 is a gas flow proportional probe that is capable of detecting both alpha and beta emissions. Alpha efficiencies are approximately 19% with beta efficiencies ranging from 13% to 15%. Data is collected for alpha and beta emissions in separate channels and can be data logged. The survey technique used for the SSD project was to operate the E-600 in ratemeter mode reading in dpm. Any case in which audible clicks were detected, additional measurements were taken using the instrument as a scaler and taking stationary counts. Additional technically equivalent instruments were used for direct reading surveys.

The Ludlum Model 2929 scaler with the Ludlum 43-10-1 probe was used to count smears. The 43-10-1 is a scintillation probe that combines a Zinc Sulfide scintillator for alpha with a plastic scintillator for beta. The scaler simultaneously collects both alpha and beta data in separate channels and can log this data.

The Berthold LB 770 Proportional counter was also used for smear counting. Gas proportional counters are capable of detecting both alpha and beta radiations. The radiations ionize the P-10 counting gas. The ionized particles are counted by the system. Background is eliminated as much as possible through the use of a guard counter that is installed directly above the measuring counter. The guard counter works in anticoincidence with the measuring counter so that events occurring simultaneously will be detected and eliminated. Alpha and beta radiations are discriminated because of the greatly differing ionization potential between alpha and beta particles.

A Tricarb 2750/LL liquid scintillation counter was used for alpha, beta and tritium smear analysis. Liquid scintillation counters will detect alpha and beta as well as tritium. Sample blanks were used to calibrate the equipment and develop appropriate quench curves.

2.2.2 Volume Contamination Sampling

Potentially volume contaminated waste items required sampling and analysis to determine that no detectable radioactivity had been added through DOE activities in order to declare that the items are non-radioactive. DOE guidelines specify that a determination of "no detectable activity" should be made through reasonable sample preparation, instrumentation, counting times, and analytical techniques. Best available technology was used. This approach was adopted by the SSD project through the use of various procedures reflecting and incorporating industry standard techniques in the aforementioned areas.

Instruments used to analyze for volume contamination included:

A high purity germanium detector capable of detecting gamma ray energies from 20 KeV to 3000 KeV was used for gamma spectroscopy. Both n and p type detectors were used. The analysis program used for data output was EG&G Ortec Omnigram software. An example of the output can be found in Attachment IV.

Gross alpha, beta and tritium analyses were accomplished using liquid scintillation (LSC) and gas proportional counting systems (GPC). LSC and GPC systems are used to determine the activities of radionuclides in chemically processed samples utilizing external standardization. The LSC used by the GJPO laboratory comprised two photomultiplier tubes that recognize coincident events. The GPC systems used comprised a small detector, guard detector, and pulse height discriminating circuitry for measuring alpha and beta activity. All LSC measurements utilized external standard quench correction. GPC operating parameters such as plateau, operating voltage, and crosstalk were determined every twelve months or after the instrument had been serviced. For each system the background count contribution were determined once per batch of samples and compared to acceptance criteria. Blanks and matrix spikes were run as specified in Attachment III.

2.3 Quality Assurance

The objective of the SSD QA/QC program was to provide systematic control of all tasks, including waste segregation, sampling and surveying, process documentation, sample handling, laboratory analysis, and data management, to ensure that the precision, accuracy, completeness, and representativeness of the data were known and demonstrated. Various elements of the QA/QC program are presented below.

2.3.1 Sample Control

Sample containers were labeled with container, item, identification number, site, date collected, time collected, sampler's name, and the analysis to be performed. Chain

of Custody forms and SSD Sample Tracking Logs were used to document sample custody from the time the sample was collected until it was analyzed. Chain of custody procedures were strictly adhered to. While samples were in transit, custody seals were placed across the opening of containers to ensure the integrity of the sample had not been compromised. The receiving laboratory documented the condition of the samples.

2.3.2 Field Quality Control and Instrumentation

Quality control samples were collected in the field following the procedures outlined in the EPA Test Methods for Evaluating Solid Waste (EPA 1986). Equipment blanks, duplicate samples, and trip blanks were collected.

Radiological field instruments were calibrated every six months. In addition daily operational checks were performed in accordance with procedures. Quality control limits (control charts) were maintained for each instrument based on statistical results from previous operational checks. Instruments that performed out of tolerance were removed from service.

2.3.3 Laboratory Quality Control and Instrument Calibration

Laboratory QC requirements were based on *Test Methods for Evaluating Solid Waste* (EPA 1986). For each batch of 20 or fewer samples submitted for analysis, laboratory duplicates, blanks, and quality control samples were prepared and analyzed according to SOPs specified by the analytical method. See Attachment III for QA/QC requirements summary.

2.3.4 Document Control

During the SSD project, consistent and accurate record keeping procedures were implemented. Corrections to records were made by a single line through, then initialed and dated. Documents have been managed in accordance with prescribed retention and disposition requirements. See Attachment III for a listing of these procedures. All records were maintained in a controlled area where access was limited, in order to reinforce chain of custody requirements.

3.0 Results

This section discusses the waste segregation decision criteria, provides a detailed summary of the waste items proposed, and gives a textual description of the data presented in Attachment I.

3.1 Decision Criteria

Decision criteria to segregate waste were two fold. Surface contamination decisions were based on the limits set forth in DOE Order 5400.5. Volume contamination decisions were based on a determination of no detectable activity. No detectable activity is defined by the method detection limit (MDL). Values that are less than the MDL are defined to have "no detectable activity".

In the specific case of tritium analysis, the MDL varied. The variable MDL results were due to differences in sample matrices, causing variable quench corrections to be applied. For example, an oily matrix causes the tritium MDL in a liquid scintillation counter to go up. In all of the cases where the matrix was difficult with respect to obtaining low MDLs, other factors were considered in making the final determination. The final determination was made by augmenting the less than MDL result with an understanding of the process generating the waste, the lack of any other suspect radionuclides being present, and the lack of surface contamination.

3.1.1 Surface Contamination

MDL values were determined for direct reading instruments and for the smear counting instruments. MDL for direct reading instruments were determined a priori and are documented in Rust Geotech Procedure HS-304. MDL values for direct reading instruments were at or below the release limits specified in DOE Order 5400.5.

The MDL for an instrument used to determine removable surface contamination levels was calculated using the following equation:

MDL EQUATION:

$$\frac{2.71+3.29\sqrt{(R_b)(t_{s+b})\left(1+\frac{t_{s+b}}{t_b}\right)}}{(E)(t_{s+b})}$$

Where:

Factors "2.71" and "3.29" represent the 95% confidence level

R_b = The background count rate in cpm

t_{s+b} = The sample count time plus the background count time

t_b = The background count time

E = The instrument efficiency

Example MDL equation for removable alpha, Survey ID 47507

$$\frac{2.71+3.29\sqrt{(1)(21)\left(1+\frac{21}{20}\right)}}{(30\%)(21)}=4\text{ dpm}$$

Example MDL equation for removable beta, survey ID 47507

$$\frac{2.71+3.29\sqrt{(49)(21)\left(1+\frac{21}{20}\right)}}{(29.6\%)(21)}=25\text{dpm}$$

3.1.2 Volume Contamination

The decision criteria for volume contamination were based on instrument MDL. Only those items with non detectable radioactive material were categorized as non-radioactive. The gas proportional instrument MDL gross alpha, gross beta, and tritium is calculated using:

MDL EQUATION:

$$\frac{2.71+4.65\sqrt{S_c-N_c}}{(E)(t)(s)(2.22)}$$

Where:

Factors "2.71" and "4.65" represent the 95% confidence level

S_c = Gross sample counts

N_c = Net counts

E = Instrument efficiency

t = count time

s = Sample size

2.22 = Converstion factor from dpm to pCi

$$\frac{2.71+4.65\sqrt{7-1.4}}{(18.2\%)(100)(5ml)(2.22)}=.07\,pCi/ml$$

Example alpha data from lab sample ID 235801 survey ID 47507 using data in Attachment IV.

3.2 Summary of Items Proposed

The total number of items determined by DOE and UC to be hazardous waste without a radioactive component is 532. The total volume is 3.04 m3. Of these items, 345 (2.94 m3) are currently covered by the STP, 59 (0.02 m3) have been proposed for addition in Revision 7.0 and are now approved, 60 (0.0 m3) are "split items" and 68 (0.08 m3) are additional items that were found during the SSD project and subsequent data review. All items underwent the same radiological analysis process and are free of DOE added radioactivity. The "split items" are associated with STP covered volume and only affect item count in the STP.

Summary of Proposed Items

STP Code	EPA Codes	Volume (m3)	Item Count	Description
LA-W906	D001, D002, F002, F005, U003	.01	7	Aqueous Organic Liquids
LA-W907	U080, U228, U226, U210, U077, U044, F005, D009, U211, F003, D002, D022, D039, F001, F002, D001	.99	54	Halogenated Organic Liquids
LA-W908	U019, U057, U239, U159, U122, U140, U154, U161, U196, U213, U226, U002, U003, U220, D003, U056, D001, D002, D004, D007, D008, D011, F005, F002, F003, F004, D0043	.5	276	Non-Halogenated Organic Liquids
LA-W909	D030, D033, D034, D035, D036, F002, D043, D040, F001, D008, D039, D028, D027, D022, D019, D018, D009, D007, D006, D004, D001, F003, D032, D010, F005, D029, F004	.83	4	Bulk Oils
LA-W911	D001, U165	.001	3	Organic Contaminated Combustible Solids
LA-W912	D001, D011	.003	4	Combustible Debris
LA-W913	D010, D011, D009, D008, D007, D006, D003, D002, D005	.14	19	Aqueous Waste with Heavy Metals
LA-W914	D002, D003, D001	.008	11	Corrosive Solutions
LA-W916	D005, D003	.0004	10	Water Reactive Wastes
LA-W918	U075, D001, U226, U228	.02	19	Compressed Gasses Requiring Oxidation
LA-W919	D006, D008	.42	2	Organic Contaminated Non-Combustible Solids
LA-W923	D001, D007, D011	.007	5	Inorganic Solid Oxidizers
LA-W925	D009, D003, D002	.003	5	Mercury Wastes TBD
LA-W933	D003, D005, D006, D007, D008, D010, D011, P012, P113, U144, U190, U204, U216, U219, U131, D004	.03	45	Labpacks
TBD	D001, D002, D003, D007, F003, U056, U057, U122, U140, U154, U213, U220, U226, U227, U239	.08	68	Newly Found

3.3 Discussion of Results in Attachment I

The SSD project involved unpacking containers and surveying each item within that container. In cases where there were multiple items within a container, each item was surveyed and analyzed for volume contamination. The outside container was also

surveyed. In cases where there were overpacks, each successive container "layer" was surveyed. In all cases both removable and direct reading survey results met release limits established in DOE Order 5400.5.

Attachment I provides a summary of results for each item. In all cases volume contamination results were less than or equal to MDL. Only the MDL values are reported. Column one provides the requisition number. The requisition number is the analytical laboratory number used to track all samples submitted for a given set of containers. The final reports issued by GJPO are organized by requisition number. Column two provides the Survey/Analysis ID number. Each individual item within a container was surveyed and analyzed. The Survey/Analysis ID numbers are used to document all survey and analytical data collected for a given item. Column three is the direct reading alpha survey indication, column four is the direct reading beta survey indication, column five is the removable alpha MDL, column six is the removable beta MDL, column seven is the removable tritium MDL, column eight is the gross alpha MDL, column nine is the gross beta MDL, column ten is the gross tritium MDL, column eleven is the gamma spec indication, and column twelve is the units for volume contamination.

3.3.1 Volume Contamination Analysis Results

The data reported is the MDL. A "less than or equal to" is implied. The reporting convention is to report the MDL value, "NS", "s", or "<". "NS" means that the particular analyte was not suspected in the waste. The "s" indicates that analysis was not required, as the item was factory sealed and therefore could not be volume contaminated. 100% of the items proposed received volume contamination analysis.

The "<" is used specifically for gamma spec. This indicates that the result was less than MDL for all radioisotopes. Attachment IV is provided as an example gamma spec documentation package. Additional packages are available for review upon request.

3.3.2 Direct Reading Surveys for Alpha and Beta

The reporting convention is that "y" indicates a direct reading survey for alpha and beta was performed and the results were less than MDL values. "NR" means not required. External surveys were not required if the item was a bulk liquid. Over 98% of the proposed items received a direct reading survey.

3.3.3 Surveys for Removable Alpha/Beta and Tritium Contamination

In most cases the MDL is reported for removable alpha, beta and tritium. In cases where values exceeded MDL, the result is reported. An "NS" means the analyte was not suspected in the waste and an "NR" means that the analysis was not required. One example of a "NR" designation is a case where the MDL for a direct reading survey meets the removable release requirements for the given analyte. Uranium is one analyte where this is the case. Over 99% of the proposed items underwent removable surface radiological surveys for alpha and beta and over 96% of the items received tritium smear surveys.

3.4 Results of QA Review

As an additional check of data validity, UC staff performed an independent QA review. Separate reviews were conducted for the field survey measurements and laboratory results. The parameters examined for field instruments were to verify a valid calibration and a background and source check were performed on days the instruments were in use.

All project documentation has been archived at the GJPO in Grand Junction, CO. A review of those records, performed by UC staff in April of 1998, indicated project documentation is in order and retrievable. Instruments used were in current calibration and were function checked.

QA review of analytical results encompassed evaluation of parameters such as duplicates, matrix spikes, continuing calibration verification, preparation blank, laboratory control samples, quench indicator parameter, and current calibration. See Attachment V for a full listing of quality control requirements for analytical systems. A limited number of batches did not meet all QC requirements established in the work plan. Several factors indicate that these data are valid and should still be used to characterize the waste as hazardous only. First, of the numerous batches analyzed, only a few had any QC issues. Further, the QC criteria were set with severely restrictive limits. All QC failures ranged between ± 10% and ± 20%. The parameter that was furthest outside the established criteria was a laboratory control sample (LCS) that resulted in 113% of the known value. The +13% level is exceedingly restrictive, especially when taking into account that the continuing calibration verification (CCV) resulted in 94.3% of the known value. In other words, there was no indication of an upward trend in instrument performance.

Each item with a QC issue was evaluated. In each case other QC parameters indicating similar aspects of data quality passed. Finally, because both radioactive decay, and instrument response are statistical features, a normal distribution centered around 100% is expected. On occasion values may fall outside of certain ranges without implying the data is invalid, provided other QC measures do not fail as well, and provided that the problem is not systematic or trend indicative.

In one case an instrument was used that was one month beyond the specified calibration date. While this is a procedural violation, the subsequent calibration indicated no change from the previous calibration. Further, continuing calibration verifications (CCVs) were being performed when the instrument was in use documenting acceptable instrument performance. Thus, these data were accepted.

The only other QC issue was that two batches did not have a matrix spike for tritium, gross alpha, or gross beta. However, the continuing calibration verification and the laboratory control sample results passed in each case, thus indicating appropriate instrument performance.

4.0 Conclusions

DOE and UC are proposing that the items documented in this report be considered hazardous waste only. The waste items were believed to be non-radioactive based on process knowledge and generator interviews. The verification data presented in this report support the determination that the wastes were hazardous only.

The ultimate destination for most of the proposed waste items is the APTUS incinerator, or a technically equivalent incinerator. A few containers will need to be sent to the BDT incinerator and acid wash facility, or the Deer Park, Texas incinerator. All waste containers are confirmed to have a treatment and disposal option as hazardous waste. All waste items will be shipped for treatment and disposal within 90 days of approval by NMED of this report.

Attachment I
Data Summary

Attachment II
SSD Procedure Listing

Attachment III
QA/QC

Attachment IV
Sample Documentation Package

This attachment includes documentation for Item 45070. This attachment is included as an example of the documentation that is available and that has been reviewed for all proposed items. Included are the gross alpha, gross beta and tritium determination, showing blanks, instrument efficiencies, sample size, sample count time gross counts, net counts, etc. Further, quench and calibration curves are provided. A sample gamma spec results sheet and backup documentation are included as well. Finally, there are sheets showing a direct reading survey and smear survey.

ENCLOSURE B

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The objective of the SSD Project was to reduce the mixed-waste inventory at LANL through sorting, surveying, and sampling and analysis. This objective was accomplished through:

- Deploying a mobile field team to sort, survey and perform sampling and analysis of suspect waste materials.
- Provide sorting space, sampling and analysis, and survey equipment and instrumentation.
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- Records Management

Technical Procedures:

- Release limits for water, liquids other than water, surface contamination, and volume-contaminated solids.

2.1.2 Data Quality

The data quality objectives for this project have been to provide data inputs for decision-makers that will allow waste to be categorized and managed as non-radioactive waste. The data needed to satisfy the SSD objectives have been generated through procedures covering the following subjects:

- Radiation dose rate surveys.
- Hand-Held surface contamination surveys.
- Use of liquid scintillation analysis for tritium, gross alpha, gross beta analysis.
- Sampling and analysis of volume contaminated waste.
    - Sampling equipment including augers, COLIWASAs, core borers, tube thief samplers, etc.
    - Analytical instrumentation including gamma spectrometer, liquid scintillation counter and gross alpha/beta detector.
- Document control and management

- Analytical laboratory quality control practices and protocols.

2.2 Procedures

The SSD project was fully implemented under a set of "change controlled" procedures. The procedures were written specifically to ensure that the data collected would enable defensible decisions to be made regarding waste disposition. Further, the procedures required that appropriate documentation be produced and maintained so that decision-makers could complete a review of results and necessary QA/QC requirements. A full listing of these procedures is found in Attachment II.

2.2.1 Surface Radiological Surveys

Surface radiological surveys were performed on waste items. Both direct reading and removable contamination surveys were performed. The minimum detectable activity for each of these survey methods was at or below the release limits specified in DOE Order 5400.5, "Radiation Protection of the Public and Environment" (DOE, January 7, 1993). The procedures reflected and incorporated standard industry practice for completion of these types of activities.

Instruments used to carry out these surveys included:

The Eberline E-600 Ratemeter/Scaler with an Eberline SHP-330 detector was used for direct reading surveys for total alpha and beta. The E-600 is a hand held instrument that is programmed to read out directly in disintegrations per minute (dpm). The SHP-330 is a gas flow proportional probe that is capable of detecting both alpha and beta emissions. Alpha efficiencies are approximately 19% with beta efficiencies ranging from 13% to 15%. Data is collected for alpha and beta emissions in separate channels and can be data logged. The survey technique used for the SSD project was to operate the E-600 in ratemeter mode reading in dpm. Any case in which audible clicks were detected, additional measurements were taken using the instrument as a scaler and taking stationary counts. Additional technically equivalent instruments were used for direct reading surveys.

The Ludlum Model 2929 scaler with the Ludlum 43-10-1 probe was used to count smears. The 43-10-1 is a scintillation probe that combines a Zinc Sulfide scintillator for alpha with a plastic scintillator for beta. The scaler simultaneously collects both alpha and beta data in separate channels and can log this data.

The Berthold LB 770 Proportional counter was also used for smear counting. Gas proportional counters are capable of detecting both alpha and beta radiations. The radiations ionize the P-10 counting gas. The ionized particles are counted by the system. Background is eliminated as much as possible through the use of a guard counter that is installed directly above the measuring counter. The guard counter works in anticoincidence with the measuring counter so that events occurring simultaneously will be detected and eliminated. Alpha and beta radiations are discriminated because of the greatly differing ionization potential between alpha and beta particles.

A Tricarb 2750/LL liquid scintillation counter was used for alpha, beta and tritium smear analysis. Liquid scintillation counters will detect alpha and beta as well as tritium. Sample blanks were used to calibrate the equipment and develop appropriate quench curves.

2.2.2 Volume Contamination Sampling

Potentially volume contaminated waste items required sampling and analysis to determine that no detectable radioactivity had been added through DOE activities in order to declare that the items are non-radioactive. DOE guidelines specify that a determination of "no detectable activity" should be made through reasonable sample preparation, instrumentation, counting times, and analytical techniques. Best available technology was used. This approach was adopted by the SSD project through the use of various procedures reflecting and incorporating industry standard techniques in the aforementioned areas.

Instruments used to analyze for volume contamination included:

A high purity germanium detector capable of detecting gamma ray energies from 20 KeV to 3000 KeV was used for gamma spectroscopy. Both n and p type detectors were used. The analysis program used for data output was EG&G Ortec Omnigram software. An example of the output can be found in Attachment IV.

Gross alpha, beta and tritium analyses were accomplished using liquid scintillation (LSC) and gas proportional counting systems (GPC). LSC and GPC systems are used to determine the activities of radionuclides in chemically processed samples utilizing external standardization. The LSC used by the GJPO laboratory comprised two photomultiplier tubes that recognize coincident events. The GPC systems used comprised a small detector, guard detector, and pulse height discriminating circuitry for measuring alpha and beta activity. All LSC measurements utilized external standard quench correction. GPC operating parameters such as plateau, operating voltage, and crosstalk were determined every twelve months or after the instrument had been serviced. For each system the background count contribution were determined once per batch of samples and compared to acceptance criteria. Blanks and matrix spikes were run as specified in Attachment III.

2.3 Quality Assurance

The objective of the SSD QA/QC program was to provide systematic control of all tasks, including waste segregation, sampling and surveying, process documentation, sample handling, laboratory analysis, and data management, to ensure that the precision, accuracy, completeness, and representativeness of the data were known and demonstrated. Various elements of the QA/QC program are presented below.

2.3.1 Sample Control

Sample containers were labeled with container, item, identification number, site, date collected, time collected, sampler's name, and the analysis to be performed. Chain

of Custody forms and SSD Sample Tracking Logs were used to document sample custody from the time the sample was collected until it was analyzed. Chain of custody procedures were strictly adhered to. While samples were in transit, custody seals were placed across the opening of containers to ensure the integrity of the sample had not been compromised. The receiving laboratory documented the condition of the samples.

2.3.2 Field Quality Control and Instrumentation

Quality control samples were collected in the field following the procedures outlined in the EPA Test Methods for Evaluating Solid Waste (EPA 1986). Equipment blanks, duplicate samples, and trip blanks were collected.

Radiological field instruments were calibrated every six months. In addition daily operational checks were performed in accordance with procedures. Quality control limits (control charts) were maintained for each instrument based on statistical results from previous operational checks. Instruments that performed out of tolerance were removed from service.

2.3.3 Laboratory Quality Control and Instrument Calibration

Laboratory QC requirements were based on *Test Methods for Evaluating Solid Waste* (EPA 1986). For each batch of 20 or fewer samples submitted for analysis, laboratory duplicates, blanks, and quality control samples were prepared and analyzed according to SOPs specified by the analytical method. See Attachment III for QA/QC requirements summary.

2.3.4 Document Control

During the SSD project, consistent and accurate record keeping procedures were implemented. Corrections to records were made by a single line through, then initialed and dated. Documents have been managed in accordance with prescribed retention and disposition requirements. See Attachment III for a listing of these procedures. All records were maintained in a controlled area where access was limited, in order to reinforce chain of custody requirements.

3.0 Results

This section discusses the waste segregation decision criteria, provides a detailed summary of the waste items proposed, and gives a textual description of the data presented in Attachment I.

3.1 Decision Criteria

Decision criteria to segregate waste were two fold. Surface contamination decisions were based on the limits set forth in DOE Order 5400.5. Volume contamination decisions were based on a determination of no detectable activity. No detectable activity is defined by the method detection limit (MDL). Values that are less than the MDL are defined to have "no detectable activity".

In the specific case of tritium analysis, the MDL varied. The variable MDL results were due to differences in sample matrices, causing variable quench corrections to be applied. For example, an oily matrix causes the tritium MDL in a liquid scintillation counter to go up. In all of the cases where the matrix was difficult with respect to obtaining low MDLs, other factors were considered in making the final determination. The final determination was made by augmenting the less than MDL result with an understanding of the process generating the waste, the lack of any other suspect radionuclides being present, and the lack of surface contamination.

3.1.1 Surface Contamination

MDL values were determined for direct reading instruments and for the smear counting instruments. MDL for direct reading instruments were determined a priori and are documented in Rust Geotech Procedure HS-304. MDL values for direct reading instruments were at or below the release limits specified in DOE Order 5400.5.

The MDL for an instrument used to determine removable surface contamination levels was calculated using the following equation:

MDL EQUATION:

$$\frac{2.71+3.29\sqrt{(R_b)(t_{s+b})\left(1+\frac{t_{s+b}}{t_b}\right)}}{(E)(t_{s+b})}$$

Where:

Factors "2.71" and "3.29" represent the 95% confidence level

R_b = The background count rate in cpm

t_{s+b} = The sample count time plus the background count time

t_b = The background count time

E = The instrument efficiency

Example MDL equation for removable alpha, Survey ID 47507

$$\frac{2.71+3.29\sqrt{(1)(21)\left(1+\frac{21}{20}\right)}}{(30\%)(21)}=4\text{ dpm}$$

Example MDL equation for removable beta, survey ID 47507

$$\frac{2.71+3.29\sqrt{(49)(21)\left(1+\frac{21}{20}\right)}}{(29.6\%)(21)}=25\text{dpm}$$

3.1.2 Volume Contamination

The decision criteria for volume contamination were based on instrument MDL. Only those items with non detectable radioactive material were categorized as non-radioactive. The gas proportional instrument MDL gross alpha, gross beta, and tritium is calculated using:

MDL EQUATION:

$$\frac{2.71+4.65\sqrt{S_c-N_c}}{(E)(t)(s)(2.22)}$$

Where:

Factors "2.71" and "4.65" represent the 95% confidence level

S_c = Gross sample counts

N_c = Net counts

E = Instrument efficiency

t = count time

s = Sample size

2.22 = Converstion factor from dpm to pCi

$$\frac{2.71+4.65\sqrt{7-1.4}}{(18.2\%)(100)(5ml)(2.22)}=.07\,pCi/ml$$

Example alpha data from lab sample ID 235801 survey ID 47507 using data in Attachment IV.

3.2 Summary of Items Proposed

The total number of items determined by DOE and UC to be hazardous waste without a radioactive component is 532. The total volume is 3.04 m3. Of these items, 345 (2.94 m3) are currently covered by the STP, 59 (0.02 m3) have been proposed for addition in Revision 7.0 and are now approved, 60 (0.0 m3) are "split items" and 68 (0.08 m3) are additional items that were found during the SSD project and subsequent data review. All items underwent the same radiological analysis process and are free of DOE added radioactivity. The "split items" are associated with STP covered volume and only affect item count in the STP.

Summary of Proposed Items

STP Code	EPA Codes	Volume (m3)	Item Count	Description
LA-W906	D001, D002, F002, F005, U003	.01	7	Aqueous Organic Liquids
LA-W907	U080, U228, U226, U210, U077, U044, F005, D009, U211, F003, D002, D022, D039, F001, F002, D001	.99	54	Halogenated Organic Liquids
LA-W908	U019, U057, U239, U159, U122, U140, U154, U161, U196, U213, U226, U002, U003, U220, D003, U056, D001, D002, D004, D007, D008, D011, F005, F002, F003, F004, D0043	.5	276	Non-Halogenated Organic Liquids
LA-W909	D030, D033, D034, D035, D036, F002, D043, D040, F001, D008, D039, D028, D027, D022, D019, D018, D009, D007, D006, D004, D001, F003, D032, D010, F005, D029, F004	.83	4	Bulk Oils
LA-W911	D001, U165	.001	3	Organic Contaminated Combustible Solids
LA-W912	D001, D011	.003	4	Combustible Debris
LA-W913	D010, D011, D009, D008, D007, D006, D003, D002, D005	.14	19	Aqueous Waste with Heavy Metals
LA-W914	D002, D003, D001	.008	11	Corrosive Solutions
LA-W916	D005, D003	.0004	10	Water Reactive Wastes
LA-W918	U075, D001, U226, U228	.02	19	Compressed Gasses Requiring Oxidation
LA-W919	D006, D008	.42	2	Organic Contaminated Non-Combustible Solids
LA-W923	D001, D007, D011	.007	5	Inorganic Solid Oxidizers
LA-W925	D009, D003, D002	.003	5	Mercury Wastes TBD
LA-W933	D003, D005, D006, D007, D008, D010, D011, P012, P113, U144, U190, U204, U216, U219, U131, D004	.03	45	Labpacks
TBD	D001, D002, D003, D007, F003, U056, U057, U122, U140, U154, U213, U220, U226, U227, U239	.08	68	Newly Found

3.3 Discussion of Results in Attachment I

The SSD project involved unpacking containers and surveying each item within that container. In cases where there were multiple items within a container, each item was surveyed and analyzed for volume contamination. The outside container was also

surveyed. In cases where there were overpacks, each successive container "layer" was surveyed. In all cases both removable and direct reading survey results met release limits established in DOE Order 5400.5.

Attachment I provides a summary of results for each item. In all cases volume contamination results were less than or equal to MDL. Only the MDL values are reported. Column one provides the requisition number. The requisition number is the analytical laboratory number used to track all samples submitted for a given set of containers. The final reports issued by GJPO are organized by requisition number. Column two provides the Survey/Analysis ID number. Each individual item within a container was surveyed and analyzed. The Survey/Analysis ID numbers are used to document all survey and analytical data collected for a given item. Column three is the direct reading alpha survey indication, column four is the direct reading beta survey indication, column five is the removable alpha MDL, column six is the removable beta MDL, column seven is the removable tritium MDL, column eight is the gross alpha MDL, column nine is the gross beta MDL, column ten is the gross tritium MDL, column eleven is the gamma spec indication, and column twelve is the units for volume contamination.

3.3.1 Volume Contamination Analysis Results

The data reported is the MDL. A "less than or equal to" is implied. The reporting convention is to report the MDL value, "NS", "s", or "<". "NS" means that the particular analyte was not suspected in the waste. The "s" indicates that analysis was not required, as the item was factory sealed and therefore could not be volume contaminated. 100% of the items proposed received volume contamination analysis.

The "<" is used specifically for gamma spec. This indicates that the result was less than MDL for all radioisotopes. Attachment IV is provided as an example gamma spec documentation package. Additional packages are available for review upon request.

3.3.2 Direct Reading Surveys for Alpha and Beta

The reporting convention is that "y" indicates a direct reading survey for alpha and beta was performed and the results were less than MDL values. "NR" means not required. External surveys were not required if the item was a bulk liquid. Over 98% of the proposed items received a direct reading survey.

3.3.3 Surveys for Removable Alpha/Beta and Tritium Contamination

In most cases the MDL is reported for removable alpha, beta and tritium. In cases where values exceeded MDL, the result is reported. An "NS" means the analyte was not suspected in the waste and an "NR" means that the analysis was not required. One example of a "NR" designation is a case where the MDL for a direct reading survey meets the removable release requirements for the given analyte. Uranium is one analyte where this is the case. Over 99% of the proposed items underwent removable surface radiological surveys for alpha and beta and over 96% of the items received tritium smear surveys.

3.4 Results of QA Review

As an additional check of data validity, UC staff performed an independent QA review. Separate reviews were conducted for the field survey measurements and laboratory results. The parameters examined for field instruments were to verify a valid calibration and a background and source check were performed on days the instruments were in use.

All project documentation has been archived at the GJPO in Grand Junction, CO. A review of those records, performed by UC staff in April of 1998, indicated project documentation is in order and retrievable. Instruments used were in current calibration and were function checked.

QA review of analytical results encompassed evaluation of parameters such as duplicates, matrix spikes, continuing calibration verification, preparation blank, laboratory control samples, quench indicator parameter, and current calibration. See Attachment V for a full listing of quality control requirements for analytical systems. A limited number of batches did not meet all QC requirements established in the work plan. Several factors indicate that these data are valid and should still be used to characterize the waste as hazardous only. First, of the numerous batches analyzed, only a few had any QC issues. Further, the QC criteria were set with severely restrictive limits. All QC failures ranged between ± 10% and ± 20%. The parameter that was furthest outside the established criteria was a laboratory control sample (LCS) that resulted in 113% of the known value. The +13% level is exceedingly restrictive, especially when taking into account that the continuing calibration verification (CCV) resulted in 94.3% of the known value. In other words, there was no indication of an upward trend in instrument performance.

Each item with a QC issue was evaluated. In each case other QC parameters indicating similar aspects of data quality passed. Finally, because both radioactive decay, and instrument response are statistical features, a normal distribution centered around 100% is expected. On occasion values may fall outside of certain ranges without implying the data is invalid, provided other QC measures do not fail as well, and provided that the problem is not systematic or trend indicative.

In one case an instrument was used that was one month beyond the specified calibration date. While this is a procedural violation, the subsequent calibration indicated no change from the previous calibration. Further, continuing calibration verifications (CCVs) were being performed when the instrument was in use documenting acceptable instrument performance. Thus, these data were accepted.

The only other QC issue was that two batches did not have a matrix spike for tritium, gross alpha, or gross beta. However, the continuing calibration verification and the laboratory control sample results passed in each case, thus indicating appropriate instrument performance.

4.0 Conclusions

DOE and UC are proposing that the items documented in this report be considered hazardous waste only. The waste items were believed to be non-radioactive based on process knowledge and generator interviews. The verification data presented in this report support the determination that the wastes were hazardous only.

The ultimate destination for most of the proposed waste items is the APTUS incinerator, or a technically equivalent incinerator. A few containers will need to be sent to the BDT incinerator and acid wash facility, or the Deer Park, Texas incinerator. All waste containers are confirmed to have a treatment and disposal option as hazardous waste. All waste items will be shipped for treatment and disposal within 90 days of approval by NMED of this report.

Attachment I
Data Summary

Attachment II
SSD Procedure Listing

Attachment III
QA/QC

Attachment IV
Sample Documentation Package

This attachment includes documentation for Item 45070. This attachment is included as an example of the documentation that is available and that has been reviewed for all proposed items. Included are the gross alpha, gross beta and tritium determination, showing blanks, instrument efficiencies, sample size, sample count time gross counts, net counts, etc. Further, quench and calibration curves are provided. A sample gamma spec results sheet and backup documentation are included as well. Finally, there are sheets showing a direct reading survey and smear survey.

ATTACHMENT C

CERTIFICATION

LOS ALAMOS NATIONAL LABORATORY (LANL)
FEDERAL FACILITY COMPLIANCE ORDER (FFCO)
SECTION V.B
OCTOBER 4, 1995

I certify that I am the project manager responsible for overseeing the implementation of the Site Treatment Plan for the Los Alamos National Laboratory. To the best of my knowledge and belief, the information in this document is true, accurate, and complete.

Beverly Martin

Beverly Martin
STP Project Manager
Waste Management Program
Environmental Management Programs
Los Alamos National Laboratory
Operator

April 16, 1999
Date Signed

H. L. Plum
Regulatory Permitting and Compliance Manager
Los Alamos Area Office
U.S. Department of Energy
Albuquerque Operations
Owner/Operator

April 16, 1999
Date Signed