Department of Energy
Carlsbad Area Office
P. O. Box 3090
Carlsbad, New Mexico 88221

June 7, 1999

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ENTERED

Mr. Greg Lewis, Director
Water and Waste Management Division
New Mexico Environment Department
P. O. Box 26110
Santa Fe, NM 87502-6110

Dear Mr. Lewis:

Please find enclosed the Rocky Flats Environmental Technology Site's (RFETS) *Non-Mixed Waste Determination for TRU Graphite Molds Debris Waste - Profile No. RF003.01* (GAO-26-99) dated May 1999. This is a hard copy, with signed signature page of the June 4, 1999 e-mail. This document summarizes the effort performed at RFETS and other information relied upon to characterize Item Description Code (IDC) 300 graphite molds waste for making the hazardous waste determination. Based upon the information and data summarized in this document, the drums containing graphite molds are not hazardous waste. The 26 drums identified for the first shipment also meet the requirement for transuranic (TRU) waste as defined by the *Waste Acceptance Criteria for the Waste Isolation Pilot Plant* and the criteria for disposal as non-mixed (nonhazardous) TRU waste at the WIPP.

If you have any questions or concerns regarding this response, please call me at (505) 234-7300.

Sincerely,

E K Hunter for
Inés R. Triay
Manager

Enclosures

cc w/o enclosures:
Peter Maggiore. NMED
Richard Mertz, NMED
Susan McMichael, NMED

Refer to: CAO:OWDO:NTWP RAS 99-0939 / UFC 5822

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GAO-026-99
May 1999

Non-Mixed Waste Determination for TRU Graphite Molds Debris Waste – Profile No. RF003.01

G. A. O'Leary
E. L. D'Amico
A. C. Kercher
J. L. Harrison

GAO-026-99

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G. A. O'Leary
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J. L. Harrison

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Rocky Flats Environmental Technology Site
Rocky Mountain Remediation Services, L.L.C.
Golden, Colorado 80402-0464

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Rocky Flats Environmental Technology Site

Non-Mixed Waste Determination for

TRU Graphite Molds Debris Waste – Profile No. RF003.01

6/4/99

G. A. O'Leary, Manager, RMRS TRU/TRM Waste Projects **Date**

6/4/99

A. D. Rodgers, Div. Mgr., Kaiser Hill Waste and Remediation Operations **Date**

6/4/99

for **J. A. Legare, Asst. Manager, DOE-RFFO Environmental Compliance** **Date**

SUMMARY

This document summarizes the efforts performed at the Rocky Flats Environmental Technology Site (RFETS) and other information relied upon to characterize 26 drums of graphite debris waste assigned Item Description Code (IDC) 300 for the purpose of making a hazardous waste determination. This characterization effort included a thorough compilation and documentation of acceptable knowledge, physical characterization, and headspace gas sampling and analysis. Solid waste sampling and analyses was also conducted by the Idaho National Engineering and Environmental Laboratory (INEEL). The results of this sampling effort are relevant to graphite waste in RFETS inventory because the graphite waste at the INEEL originated from the same processes at RFETS. Based upon the information and data summarized in this document, the drums containing graphite waste discussed herein are not hazardous waste (i.e., are non-mixed waste). No hazardous waste codes are assigned as specified in Title 40 of the Code of Federal Regulations, Part 261 (40 CFR 261). The 26 drums meet the requirements for transuranic (TRU) waste as defined by the Waste Acceptance Criteria for the Waste Isolation Pilot Plant, prepared by the Department of Energy (DOE). Consequently, these 26 drums meet the criteria for disposal as non-mixed (nonhazardous) TRU waste at the DOE Waste Isolation Pilot Plant.

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ACRONYMS

ACL	Analytical Chemistry Laboratory
AK	acceptable knowledge
ANL-W	Argonne National Laboratory - West
BWR	Backlog Waste Reassessment
BWRBB	Backlog Waste Reassessment Baseline Book
CAO	Carlsbad Area Office
CFR	Code of Federal Regulations
DOE	Department of Energy
DOT	Department of Transportation
EDL	economic discard limit
EPA	Environmental Protection Agency
HWA	(New Mexico) Hazardous Waste Act
IDC	Item Description Code
INEEL	Idaho National Engineering and Environmental Laboratory
NRC	Nuclear Regulatory Commission
PRQL	program-required quantitation limit
QA	quality assurance
QAPP	Quality Assurance Program Plan
QC	quality control
RCRA	Resource Conservation and Recovery Act
RFETS	Rocky Flats Environmental Technology Site
RMRS	Rocky Mountain Remediation Services
RTR	real-time radiography
SAS	SWEPP Assay System
SME	Subject Matter Expert
SVOC	semi-volatile organic compound
SWEPP	Stored Waste Examination Pilot Plant
TCLP	Toxicity Characteristic Leaching Procedure
TIC	tentatively identified compound
TRU	transuranic
TRM	transuranic mixed
VOC	volatile organic compound
WAC	Waste Acceptance Criteria
WEMS	Waste and Environmental Management System
WIPP	Waste Isolation Pilot Plant
WSRIC	Waste Stream and Residue Identification and Characterization

Rocky Flats Environmental Technology Site Non-Mixed Waste Determination for TRU Graphite Debris Waste – Profile No. RF003.01

1. INTRODUCTION

This document describes the process and information used by the Rocky Flats Environmental Technology Site (RFETS) to determine that 26 drums of graphite debris waste assigned Item Description Code (IDC) 300 is not hazardous waste regulated by the Resource Conservation and Recovery Act (RCRA) or the New Mexico Hazardous Waste Act (HWA). To be considered a hazardous waste under these statutes, a waste must either be specifically listed as a hazardous waste or exhibit the hazardous characteristic of ignitability, corrosivity, reactivity, or toxicity.

Graphite waste has been determined by RFETS to be a non-mixed (nonhazardous) waste. The defense generated waste [Ref. 1], consisting of graphite molds (IDC 300), was selected because of extensive knowledge of this waste as non-mixed waste. Thus, RFETS has focused efforts on these drums for its first shipment to the Waste Isolation Pilot Plant (WIPP).

The RCRA regulations authorize hazardous waste determinations to be made either by using approved sampling and analysis methods or by applying knowledge of the waste in light of the materials or process used, typically referred to as 'process knowledge.' The WIPP Quality Assurance Program Plan (QAPP) [Ref. 6] refers to 'acceptable knowledge' as applying knowledge of the waste based on the materials or processes used to generate the waste. The term 'acceptable knowledge,' which is referred to throughout this document, is synonymous with 'process knowledge.' RFETS used the following methods to characterize the graphite wastes (IDC 300):

- Acceptable knowledge (AK);
- Real-time radiography (RTR) and visual examination;
- Headspace gas sampling and analysis; and
- Solid waste sampling and analysis of randomly selected containers of graphite.[1]

Initially, RFETS applied AK data to support a non-mixed waste determination for this waste stream. Subsequently, RFETS conducted additional examinations to verify this determination. Additionally, the Idaho National Engineering and Environmental Laboratory (INEEL) performed solid waste sampling and analysis to confirm the waste is non-mixed (graphite waste stored at INEEL originated from RFETS). Sections 2 through 5 of this report describe in detail the actions taken and the conclusions reached by RFETS with respect to this non-mixed determination. RFETS has demonstrated that the 26 drums containing graphite waste (IDC 300) are a non-mixed waste, in accordance with the requirements of 40 Code of Federal Regulations (CFR) 261.

[1] The sampling and analysis was conducted by INEEL on IDC 300 waste originating from RFETS in storage at INEEL.

2. WASTE GENERATION PROCESS DESCRIPTION

During plutonium weapons production at RFETS, graphite molds were used in foundry and plutonium metallurgy operations to cast plutonium parts, shapes, and ingots. The molds were bagged into the glovebox line where they were assembled, prepared, and conveyed to the casting furnaces. In the gloveboxes, plutonium metal was placed inside a tantalum crucible and heated in a furnace vessel. The molten metal was poured from the crucible into the graphite mold. After the castings cooled, the molds were separated from the castings.

Building 707 was a source of generation of spent graphite molds (IDC 300) during the casting of plutonium in production foundry operations. Building 771 was a source of generation of spent graphite crucibles and molds (IDC 300) during plutonium metallurgy operations which included alloy preparation, heat treating, rolling, metal cutting, and metallurgical testing for research and development. Building 779 was a source of generation of spent graphite molds (IDC 300) during experimental casting operations. Graphite molds (IDC 300) were also generated from emission spectroscopy in Building 559 [Ref. 2].

Graphite molds to be reused were mechanically cleaned (scarfed) in Buildings 371, 707, 771, and 777 to remove the plutonium metal from the surface of the mold. Once a mold was no longer usable, it was subjected to a discard process. Both graphite molds (IDC 300) and classified graphite shapes (IDC 301) were nondestructively assayed to determine if they contained plutonium above or below the economic-discard limit (EDL). Scarfing operations removed plutonium metal from those components contaminated with plutonium above the EDL resulting in the generation of IDCs 303 (scarfed graphite chunks), 310 (graphite scarfings & fines), and 312 (coarse graphite). Graphite molds and classified shapes (IDCs 300 and 301, respectively) found to be below the EDL were stored separately for disposal or, in the case of IDC 301, subjected to size-reduction operations to meet security requirements. Graphite wastes in inventory originating in Building 776 were not generated in Building 776 but were repackaged in the size reduction area in this building. The graphite from Building 776 originated from one of the processes described in this section [Ref. 2]. Process flow diagrams for graphite waste generation are provided in Appendix A.

Of the 26 drums of graphite debris waste, 24 were generated in Building 707, one in Building 559, and one in Building 776.

Solvents were not employed in the production of new molds or in the recovery or cleanup of used molds [Ref. 3]. Carbon tetrachloride was hard-piped into the casting furnace gloveboxes for cleaning purposes [Ref. 4]. After glovebox cleaning was completed, all liquids were removed and discarded separately. Graphite molds are relatively soft, and contact with any liquids would cause the molds to crumble, rendering them useless. Therefore, the graphite molds were removed from the glovebox before cleaning took place [Ref. 5]. Freon was used in experimental casting in Building 779 to clean samples [Ref. 2 and 20]. Organic solvents were not used in the Building 559 and 771 processes that generated graphite molds [Ref. 18 and 19]. No known contact with F-listed or other listed hazardous waste sources with graphite wastes has been documented.

3. CHARACTERIZATION PROCESS

3.1 Hazardous Waste Determination

A waste must exhibit a hazardous characteristic or be listed as a hazardous waste in the regulations to be deemed a hazardous waste under RCRA. The mere presence of particular constituents in a waste does not cause the waste to be hazardous if such constituents do not result from a prescribed use or do not exceed regulatory limits in a representative sampling. The RCRA regulations authorize hazardous waste determinations to be made either by using approved sampling and analysis methods or by applying knowledge of the waste in light of the materials or process used, also known as AK. Additionally, the U.S. Environmental Protection Agency (EPA) and the U.S. Nuclear Regulatory Commission (NRC) have recognized and encouraged the use of AK for radioactive waste ("Joint NRC/EPA Guidance on Testing Requirements for Mixed Radioactive and Hazardous Waste," 62 Fed. Reg. 62079, 1997).

As discussed before, RFETS initially used the AK characterization process to support a non-mixed waste determination for graphite debris waste (IDC 300). INEEL performed RCRA compliant sampling and analysis to verify this determination.

3.2 Acceptable Knowledge

Acceptable knowledge includes information regarding the physical form of the waste, the base materials composing the waste, the nature of the radioactivity present, and the process generating the waste. This report specifically addresses the AK documentation used to characterize graphite debris waste as a non-mixed waste. Compilation of AK and the conclusions reached following review of AK for each of the categories of hazardous waste are discussed in this section and in Section 4.

3.2.1 Key Acceptable Knowledge Documentation

In October 1989, the Waste and Residue Identification and Characterization (WSRIC) program was implemented to assess the RCRA characterization of waste streams generated at RFETS, including transuranic (TRU) and transuranic mixed (TRM) waste streams. The information collected for the initial WSRIC program was documented in a book created for each building. These books describe in detail the waste streams generated by every process conducted in every building at RFETS, including process descriptions, process flow diagrams, chemical inputs, waste outputs, and waste characterization and rationale. Files containing the histories of waste streams are also maintained. The WSRIC building books and history files are under change control and are maintained as WIPP quality assurance (QA) records [Ref. 1].

In December 1993, the Backlog Waste Reassessment (BWR) program was implemented to assess waste generated prior to approximately 1992 when the Waste/Residue Traveler (documentation that travels with each container) was implemented. Existing characterization documentation, including information from WSRIC, was compiled for the stored inventory of wastes at RFETS, and a review of the information was conducted to assess the RCRA characterization. The information collected by this program was compiled in the Backlog Waste Reassessment Baseline Book (BWRBB). The BWRBB and history files are also under change control and are maintained as WIPP QA records [Ref. 1].

The characterization information for each waste container is recorded in the Waste and Environmental Management System (WEMS) database which tracks and controls the inventory, movement, and various waste management activities for waste containers from initial storage through disposal. The waste custodians are responsible for updating WEMS to incorporate WSRIC and BWR information [Ref. 1].

There are some areas of AK not addressed by the WSRIC or BWR programs because they are not within the scope of these programs. RFETS TRU Waste Acceptable Knowledge Supplemental Information, RF/RMRS-97-018, was developed in 1997 to address these additional areas of AK pertinent to TRU and TRM waste, including defense activities, radionuclides, matrix parameter categories, and waste material parameters [Ref. 1].

3.2.2 Description of Acceptable Knowledge Compilation

In an effort to meet the AK requirements in the WIPP QAPP [Ref. 6], summaries of the TRU waste stream information from the WSRIC and BWR programs and the RFETS TRU Waste Acceptable Knowledge Supplemental Information were developed. This information is documented in RMRS-WIPP-98-100, Acceptable Knowledge TRU/TRM Waste Stream Summaries [Ref. 2]. The AK waste stream summary for TRU graphite waste is provided in Appendix B.

Certification audits were conducted at RFETS in December 1997, February 1998, and March 1999. During these audits, the Carlsbad Area Office (CAO) of the Department of Energy (DOE) performed extensive reviews of the AK documentation. Various observers, including EPA personnel, participated in one or both audits. The overall results from the audits were favorable and certification status of the program was received [Ref. 15].

3.3 Discussion of Acceptable Knowledge Determination For Graphite Debris Waste

This section discusses in detail certain key AK documentation for graphite debris waste (IDC 300) which relates to the non-mixed waste determination.

For waste to be RCRA-listed, it must meet listed waste criteria set forth in 40 CFR 261.31 – 261.33. The graphite itself is not a RCRA-listed hazardous waste. AK documentation demonstrates that no listed waste was in direct contact with the graphite during or after the production process. The AK documentation notes that carbon tetrachloride, 1,1,1-trichloroethane, and 1,1,2-trichloro-1,2,2-trifluoroethane (Freon) were used for cleaning gloveboxes, equipment, and plutonium metal in the buildings where graphite waste was generated [Ref. 4, 7, and 8]. The AK documentation [Ref. 9] states that although incidental solvent contamination of the molds could have been possible (i.e., residual carbon tetrachloride contamination within the glovebox), such contamination does not meet the regulatory definition of any RCRA-listed waste [Ref. 17]. Additionally, there is no evidence that any solvents were used on the graphite molds themselves or in the generation of the graphite as waste [Ref. 4, 7, 8, 9, 18, 19, and 20]. Moreover, because the graphite molds could be reused in casting operations, and were relatively porous, there was a strong operational driver to avoid contact with liquids, which can cause the molds to crumble. Consequently, the graphite molds were removed before glovebox cleaning [Ref. 5]. The molds themselves were cleaned with brushes, and no solvents were used. Thus, the graphite never contacted an F-listed waste source which forms the basis for not characterizing the waste as a RCRA-listed waste.

To exhibit the characteristic of toxicity, constituent levels of certain metals or organics must meet or exceed regulatory thresholds defined in 40 CFR 261.24, based on the Toxicity Characteristic Leaching Procedure (TCLP) test results. As mentioned, carbon tetrachloride, a toxic-characteristic compound, was used for cleaning gloveboxes, equipment, and plutonium metal, potentially resulting in incidental contact [Ref. 3]. However, AK documentation demonstrates that graphite waste does not exhibit the characteristic of toxicity for metal or organic compounds [Ref. 7].

Based on knowledge of the process (i.e., the chemicals used), pesticides or herbicides were not present in the processes or areas generating graphite waste. Therefore, the graphite wastes do not exhibit the characteristic of toxicity due to pesticides or herbicides. Finally, the waste does not exhibit any of the other hazardous waste characteristics (i.e., corrosivity, reactivity, or ignitability) [Ref. 4, 7, 18, 19, 20].

3.4 Relevant Data and Supporting Information

Physical and chemical characterization of the graphite debris wastes was undertaken to (a) verify the AK documented for this waste form, and (b) support the determination of the non-mixed status of the waste. Included in this section are those intrusive and non-intrusive examinations and analyses conducted to ensure compliance with the WIPP waste acceptance criteria (WAC) [Ref. 6 and 13] and to confirm the AK RCRA waste determination. These examinations and analyses include non-intrusive examination (discussed in Section 3.4.2.2); headspace gas analysis (Section 3.4.1); physical inspection of the waste (Section 3.4.2.3); and sampling and analysis conducted in accordance with RCRA requirements (Section 3.4.3). The data generated from these examinations and analyses are maintained as WIPP QA records.

3.4.1 Headspace Gas Data

The applicable WIPP QAPP requirements for headspace gas sampling and analysis and the results obtained are discussed in the following sections.

3.4.1.1 Program Requirements

TRU waste containers are characterized using headspace gas sampling and analysis to determine the quantity of VOCs, hydrogen, and methane present in the container headspace. Headspace gas sampling requirements and methods are performed in accordance with the RFETS TRU Waste Characterization Program Quality Assurance Project Plan which is the site-specific document describing the implementation of the WIPP QAPP [Ref. 6]. Section 7 of the WIPP QAPP defines sampling and analysis protocols required to meet programmatic objectives and describes the equipment required to collect headspace gas samples from within drums of TRU waste. The protocols are based in large on EPA guidelines.

Headspace gas sampling and analysis must be performed on each drum destined for shipment to and disposal at the WIPP. The headspace analysis results for spent solvents are then evaluated per the requirements in the QAPP [Ref. 6, Section 5.3]. If any constituent is determined to be above the program-required quantitation limit (PRQL), the AK information for the waste stream is re-evaluated to determine the potential source of the constituent. If the source of the constituent is identified as a spent solvent used in the process or is determined to be the result of mixing a listed waste with a solid waste, the affected drums are assigned the applicable EPA hazardous waste number and are segregated into a separate waste stream.

3.4.1.2 Analytical Results

Results of the headspace gas analyses for the 26 drums of graphite debris waste are summarized in Appendix E. Based on the results given in Appendix E, no headspace gas analysis results for F-listed volatile organic solvents exceeded the PRQL at the 90% confidence level, per the QAPP [Ref. 6, Section 5]. In addition, the occurrences of tentatively identified compounds (TICs) were also evaluated. No positively identified TICs listed in 40 CFR Part 264, Appendix IX were detected in at least 25 percent of the field samples analyzed [Ref. 6, Section 12.6]. Therefore, headspace gas analysis supports the determination that this waste is not mixed waste pursuant to the New Mexico HWA.

3.4.2 Physical Characterization

For the purposes of this document, "physical" characterization includes both intrusive (visual examination) and non-intrusive (RTR) examinations or inspections, conducted under the requirements of the WIPP QAPP [Ref. 6] and EPA SW-846 [Ref. 14].

3.4.2.1 Program Requirements for Examination of Contents

Radiography is required by the QAPP [Ref. 6] to verify the waste container contents and packing configuration, determine matrix parameter categories, establish the layers of confinement, and detect prohibited materials such as pressurized containers or free liquids. The QAPP delineates the requirements for the methods to be used: the QA objectives and QC requirements for the examination; instrument testing, inspection, calibration, and maintenance requirements; the data management requirements; and the requirements for physical (visual) confirmation of examination results. The techniques and procedures for meeting these requirements have been established at RFETS, and their use has been authorized by DOE-CAO [Ref. 15].

RTR is the approved process at RFETS for meeting non-intrusive examination requirements. The equipment consists of an X-ray source and an imaging system configured to allow the X-ray image to be displayed as the examination is taking place. As a method for monitoring the results and reliability of RTR, the QAPP states, "The results of radiography will be verified through visual examination of a statistically selected portion of retrievably stored waste containers." [Ref. 6, Section 10]. As a result of this requirement, and as part of the CAO-authorized program at RFETS, the appropriate statistical measures are utilized to select X-rayed containers for direct visual examination to verify the inspection results. Selected drums to be opened are sent to Building 776 at RFETS. There, the contents of debris drums are removed, documented, and returned to the drum. The results of the intrusive examination are compared to the X-ray inspection results, discrepancies are identified and dispositioned by a RFETS Subject Matter Expert (SME), and the AK characterization is revised, as appropriate.

3.4.2.2 Real-Time Radiography Inspection Results

The results of RTR for the 26 drums of graphite waste verified the presence of graphite waste material and plastic and fiberboard packaging materials. No RCRA-regulated, WIPP-prohibited, or other extraneous items were observed. The RTR results are documented in data packages that have been validated and verified.

In addition to looking for extraneous material, RTR inspection provides the basis for determining the number of layers of confinement and estimating the amount of absorbent material present with the waste and ensuring the absence of liquids. In these cases, the visual examination provided excellent correlation with the RTR results.

3.4.2.3 Visual Examination and Verification

At RFETS, one drum of IDC 300 has been subjected to visual inspection to confirm the RTR results. During visual inspection of the drum, the contents were removed, inspected, documented, and returned to a new drum. The visual examination of this drum verified the presence of graphite waste material only.

3.4.3 Sampling and Analysis

INEEL developed a sampling and analysis plan [Ref. 11] to confirm that the graphite molds do not exhibit the characteristic of toxicity, to ensure representative samples were taken, and to ensure TCLP Quality

Assurance (QA) and Quality Control (QC) protocols were met. Five drums of IDC 300 waste were randomly selected from a waste stream lot of 50 drums to undergo sampling for TCLP extraction and analysis [Ref. 11]. Samples from the five randomly selected drums were submitted to the INEEL Analytical Chemistry Laboratory (ACL) for TCLP extraction and analysis. The results of this sampling effort are relevant to graphite waste in RFETS inventory because the graphite waste at the INEEL originated from the same processes at RFETS.

3.4.3.1 Sampling Plan

A sampling plan was developed and approved by the INEEL to select the graphite samples to be used in conjunction with TCLP. (The sampling plan is reproduced in Appendix C.) Selection of the drums from which the graphite samples were to be taken is summarized below. The drums were numbered for inspection and sampling according to their shipment order to Argonne National Laboratory - West (ANL-W). Random digits were selected from one to fifty using Stored Waste Examination Pilot Plant (SWEPP) Assay System (SAS) software (statistical software). Ten digits were randomly selected. Drums found to correspond to the last five SAS selected numbers were the drums chosen for sampling, TCLP extraction, and analysis. Five of the chosen drums underwent sampling and totals analysis and the remaining five drums sampling, TCLP extraction, and analysis. Given the selection process, a five-drum sample size was considered adequate to achieve the precision and accuracy necessary to support a non-mixed determination at a 90% upper confidence level. Three randomly selected graphite pieces were then taken from each of the five drums, broken into smaller pieces, and composited into a single sample for TCLP analysis. Thus, there were five composite TCLP samples. To ensure representative sampling of the graphite molds from within the drum, one mold piece was selected from the top third of the drum waste, one mold from the middle third, and one mold from the bottom third. The portion of the selected mold piece was also randomly selected [Ref. 11, 12].

In addition to the sampling process, the sampling plan defines the target analyte list, the analyses required, and QA/QC parameters [Ref. 11]. Samples and associated QC samples were analyzed per SW-846 [Ref. 14]. Chain-of-custody documentation was maintained throughout, and no deficiencies in sample integrity were observed. The TCLP extractions and analyses were performed well within the required hold times. No significant problems were identified in analytical QC. (See Appendix D for discussion.) The Environmental Data Services, Inc. (Concord, NH) independently validated the results of organic sample analyses. The INEEL Sample Management Office validated the results of metal analyses [Ref. 12]. A summary of qualifying flags for both validation reviews is provided in Appendix D.

3.4.3.2 Summary of Toxicity Characteristic Leaching Procedure Data

Based on the TCLP results from sampling of IDC 300 waste, no toxicity characteristic compounds were found to be greater than the regulatory threshold limits. Refer to Appendix D for a summary of the results.

4. DETAILED RCRA ANALYSIS OF GRAPHITE

For a waste to be hazardous under RCRA or the New Mexico HWA, it must be specifically listed as a hazardous waste or exhibit a hazardous characteristic as specified in the applicable regulation. The characteristics are toxicity, corrosivity, ignitability, and reactivity. The mere presence of hazardous constituents does not necessarily cause a waste to be hazardous by definition if the constituents do not result from a prescribed use or do not exceed regulatory limits. RCRA regulations authorize a generator to determine if a waste is hazardous by either applying knowledge of the hazardous nature of the waste in light of the material or the process used or by testing the waste in accordance with approved methods.

4.1 Listed Waste

Waste is potentially subject to RCRA regulations as a hazardous waste if it is a waste listed in 40 CFR 261.31 – 261.33. Based on AK, it is known that graphite waste assigned IDC 300 is not a listed waste. The F-listing is not applicable because no listed solvents came in direct contact with the graphite during or after the production process. Moreover, as previously stated in Section 3.4.1, no constituents were detected in the headspace gas analysis at or above the PRQL.

4.2 Characteristic Waste

4.2.1 Toxicity

Based on AK and chemical analysis, it has been determined that the drums containing IDC 300 waste do not meet the definition of a toxic characteristic waste. To exhibit the toxicity characteristic, waste levels of certain metals or organics must meet or exceed certain levels in an extract generated using a test method known as the TCLP. This test determines the mobility of the contaminant from the media in which it exists. The results are then compared with levels that the EPA has identified as hazardous to projected receptors.

AK documentation demonstrates graphite debris waste (IDC 300) does not exhibit toxicity for metal or organic compounds [Ref. 2 and 7]. As discussed in Section 3.4.3, the INEEL conducted TCLP on a representative sample of graphite molds (IDC 300) to confirm that the waste does not exhibit the characteristic of toxicity. As indicated in Appendix D, the TCLP results demonstrate that no VOCs, SVOCs, or metals exceed the regulatory threshold limits.

4.2.2 Corrosivity

Based on examination and knowledge of the waste stream, it has been determined that IDC 300 waste does not meet the definition of a characteristic waste due to corrosivity. To be corrosive waste under RCRA, a material must possess either of the following properties:

- It is aqueous with a pH less than or equal to 2 or greater than or equal to 12.5. To measure the pH, the EPA prescribes the use of Method 9040 in the definition of corrosivity found at 40 CFR 261.22. This method requires that greater than 20% of the total waste volume is aqueous; or
- It is a liquid as determined by its ability to pass through a certain type of filter and will corrode steel at a rate of 0.25 inches per year.

As determined by RTR and confirmed by visual inspection, this waste is neither aqueous nor liquid. Therefore the waste cannot be corrosive per RCRA definition. This conclusion is substantiated in a letter from the EPA Policy Compendium [Ref. 16]. EPA states that the characteristic of corrosivity as defined in 40 CFR 261.22 is intended to apply only to aqueous or liquid media, "unless and until the EPA promulgates a definition for solids. The agency has no plans to do this at the present time." EPA has not promulgated a definition of solids as of this date.

4.2.3 Ignitability

Based on examination and knowledge of the waste stream, it has been determined that IDC 300 waste does not meet the definition of an ignitable waste under RCRA. To be a RCRA waste in this category, a material must possess any of the following properties:

- It is a liquid other than an aqueous solution containing less then 24 percent alcohol and flash point less than 140°F (60°C);
- It is not liquid and is capable of causing fire through friction, absorption of moisture, or spontaneous chemical changes;
- It is an ignitable compressed gas; or
- It is an oxidizer as defined by U.S. Department of Transportation (DOT) regulations.

As discussed above, the waste does not contain liquids. Nothing in the waste stream is solid that has qualities likely to ignite through friction, moisture absorption, or chemical changes. There is no compressed gas in the waste stream. Finally, the waste is not an oxidizer as defined in DOT regulations in 49 CFR 173.151. RTR and visual inspection of the drums confirmed these conclusions.

4.2.4 Reactivity

Based on knowledge of the waste stream and the generation process, it has been determined that IDC 300 waste does not meet the definition of a reactive waste under RCRA. To be a RCRA waste in this category, a material must possess any one of the following properties:

- It is unstable and can undergo violent change;
- It reacts violently with water;
- It forms potentially explosive mixtures with water;
- It reacts with water to generate toxic gases, vapors, or fumes that are harmful;
- It contains cyanide or sulfide that can generate toxic gases, vapors or fumes;
- It can detonate or explode at standard temperature and pressure; or
- It is a DOT forbidden Class A or B explosive.

Graphite is stable and does not undergo violent chemical change. Graphite does not react violently with water, form potentially explosive mixtures with water, or generate toxic gases, vapors, or fumes when mixed with water. Graphite does not contain cyanides or sulfides and is not capable of detonation or explosive reaction.

5. CONCLUSION

RFETS has established that the 26 drums of graphite debris waste (IDC 300) discussed above is not hazardous waste. The waste does not exhibit any hazardous characteristic and is not a listed waste. Although the determination of whether a waste is hazardous for RCRA purposes is allowed to be made on the basis of acceptable knowledge alone, RFETS also used additional tests and analysis, including headspace gas analysis, RTR, and visual examinations to confirm that the AK documentation is correct. The analytical results from TCLP analysis of graphite molds by the INEEL also demonstrate graphite debris waste is not a hazardous waste.

6. REFERENCES

1. RFETS TRU Waste Acceptable Knowledge Supplemental Information. RF/RMRS-97-018, Revision 4. May 10, 1999.

2. Acceptable Knowledge TRU/TRM Waste Stream Summaries. RMRS-WIPP-98-100, Revision 3. May 20, 1999.

3. Letter from M. Wheeler to A. Rodgers, *Retrieval of Graphite Waste Characterization Documentation*, MW-095-98, July 1, 1998; as transmitted by letter from A. D. Rogers (Kaiser-Hill Co.) to T. L. Clements, Jr., *Characterization of Graphite Waste*, ADR-081-98, July 6, 1998.

4. Waste Stream and Residue Identification and Characterization, Building 707, Version 3.2, 1992.

5. K. J. Peters letter to S. M. Hailey, *Assessment of Graphite Waste Matrix and Solvent Usage*, KJP/004/0398, March 4, 1998.

6. *Transuranic Waste Characterization Quality Assurance Program Plan*, Interim Change, CAO-94-1010, November 15, 1996.

7. Backlog Waste Reassessment Baseline Book, Waste Form 56 Graphite. February 17, 1998.

8. K. J. Peters letter to S. M. Hailey, *Graphite Waste*, KJP/003/0298, February 3, 1998.

9. K. J. Peters letter to S. M. Hailey, *Evaluation of Potential Sources of RCRA Compounds Found in Graphite Waste Headspace*, KJP/114/0298, February 25, 1998.

10. K. J. Peters letter to S. M. Hailey, *Assessment of Graphite Waste for Toxicity Characteristic Compounds*, KJP/105/0198, January 23, 1998.

11. *Special Characterization Sampling and Analysis of Graphite Mold Waste*, Engineering Design File EDF-RWMC-1028, Rev. 1, August 13, 1998.

12. *Nonmixed Waste Determination for IDC 300 Waste (Graphite Molds)*. INEEL/EXT-98-01137, February 1999.

13. *Waste Acceptance Criteria for the Waste Isolation Pilot Plant*, DOE/WIPP-069, Revision 5, April 1996.

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

15. G. E. Dials letter to J. Roberson, *Certification of Rocky Flats Environmental Technology Site (RFETS)*, CAO:NTP:RAS 97-0651 UFC 5822, March 26, 1998.

16. Environmental Protection Agency Policy Compendium, 9443.1987(17), August 18, 1987.

17. Environmental Protection Agency Policy Compendium, 9444. 1987 (33) August 7, 1987.

18. Waste Stream and Residue Identification and Characterization, Building 559, Version 3.2, 1991.

19. Waste Stream and Residue Identification and Characterization, Building 771, Version 3.2, 1993.

20. Waste Stream and Residue Identification and Characterization, Building 779, Version 3.2, 1991.

Appendix A

Graphite Process Flow Diagrams

- Building 559, Process 7, Figure 7.4 – Emission Spectroscopy
- Building 707, Process 1, Figure 1.1 – Foundry (Casting) Module A
- Building 771, Process 26, Figure 26.1 – Plutonium Metallurgy
- Building 779, Process 14, Figure 14.5 – Experimental Casting

559-V3.2

7-5

9/27/91

AutoCAD 11, File Name: f559-19d 9/24/91

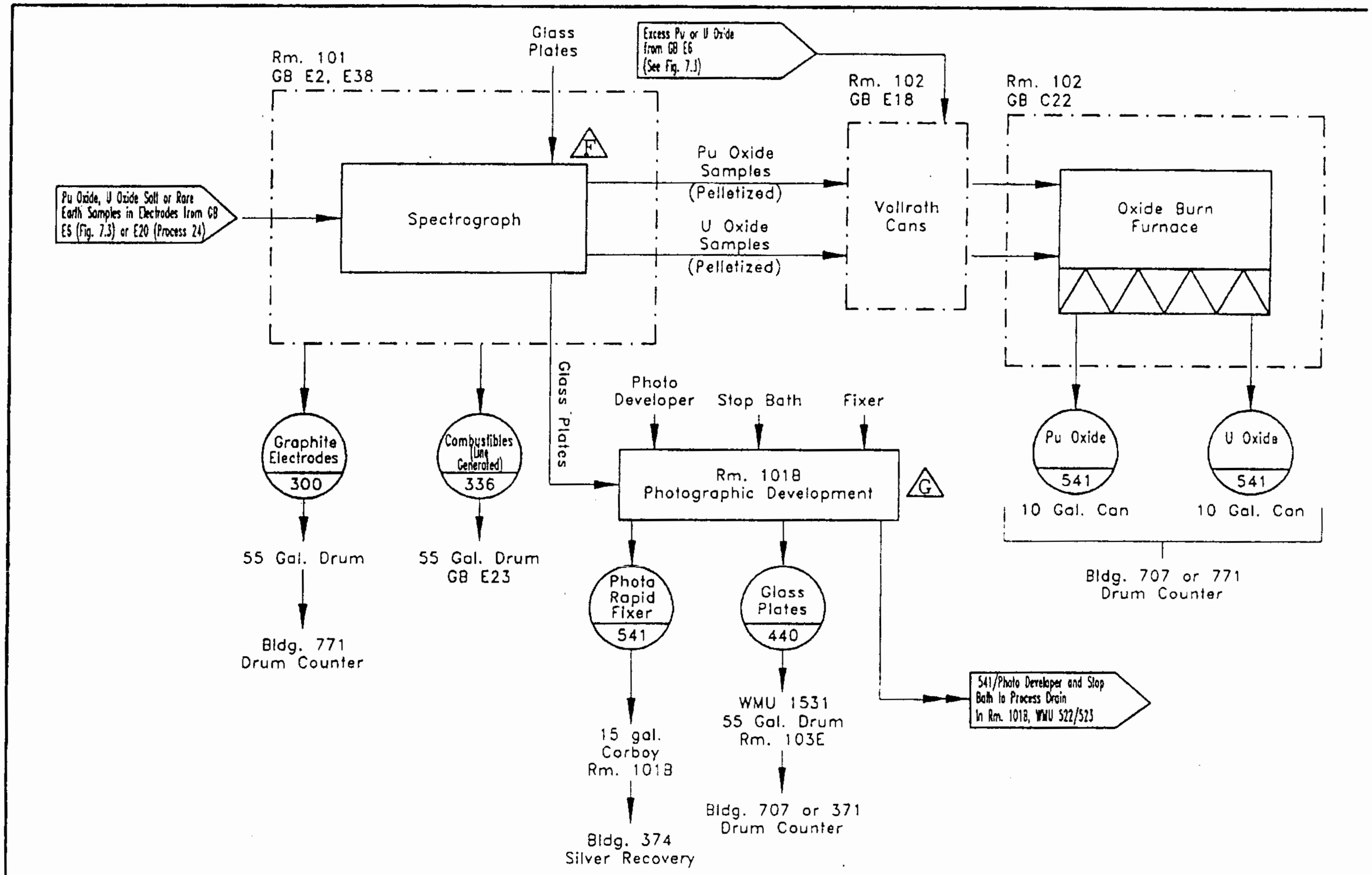


FIGURE 7.4

Rocky Flats Plant Waste Stream & Residue Identification & Characterization	BUILDING 559/PROCESS 7 EMISSION SPECTROSCOPY (DIRECT READING)	Task	By	Date
		Designed	C. Conroy	4/20/90
		Drawn	K. Tate	6/28/90
		Checked	B. Peterson	9/23/91

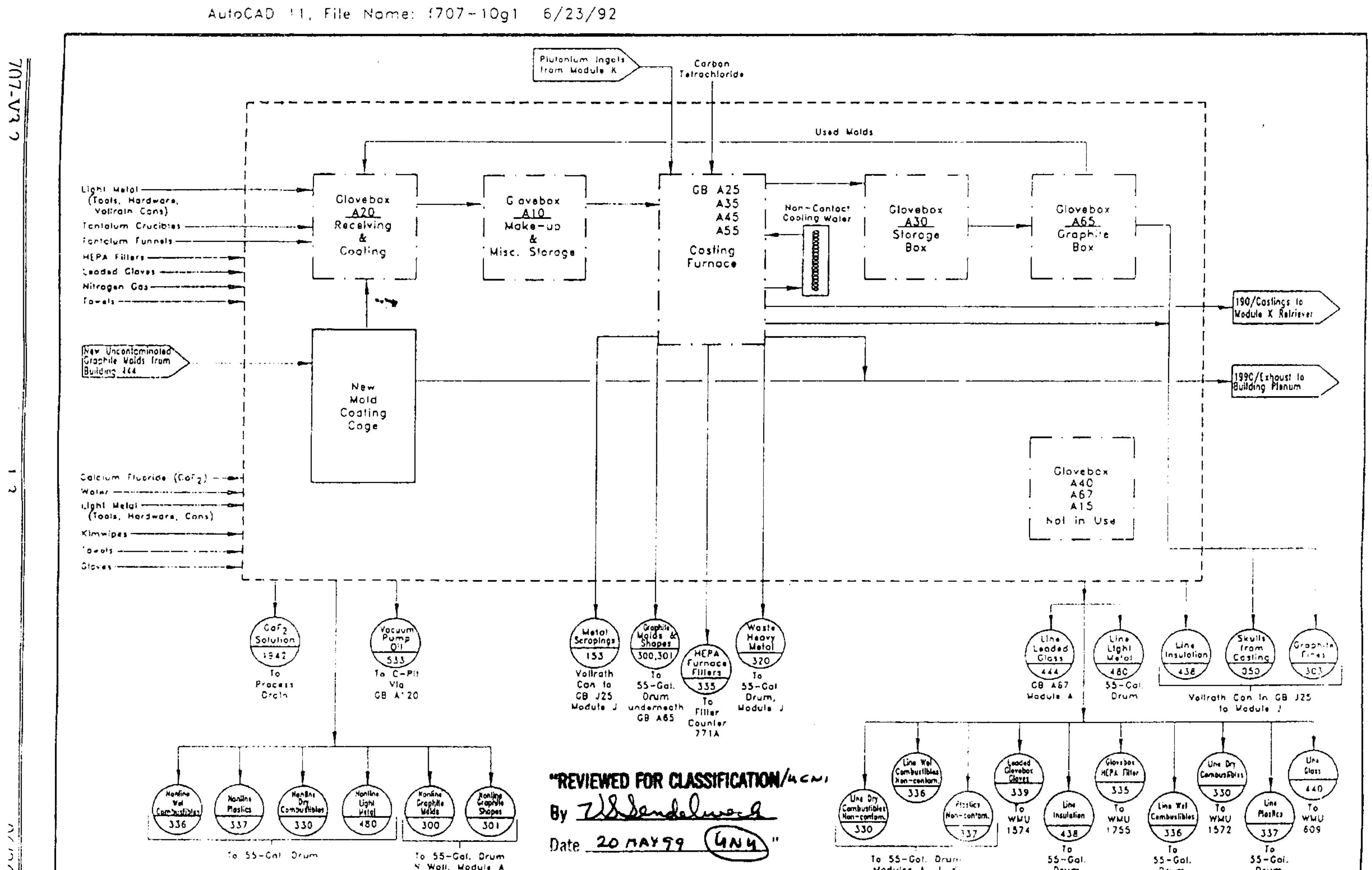


FIGURE 1.1

Rocky Flats Plant Waste Stream & Residue Identification & Characterization	BUILDING 707/PROCESS 1 FOUNDRY (CASTING) MODULE A	Task	By	Date
		Designer	K. Tilton	6/10/92
		Drawn	C. Stadt	6/15/92
		Checked	K. Tilton	6/16/92

AutoCAD 11, File Name: I771-26k 07/08/93

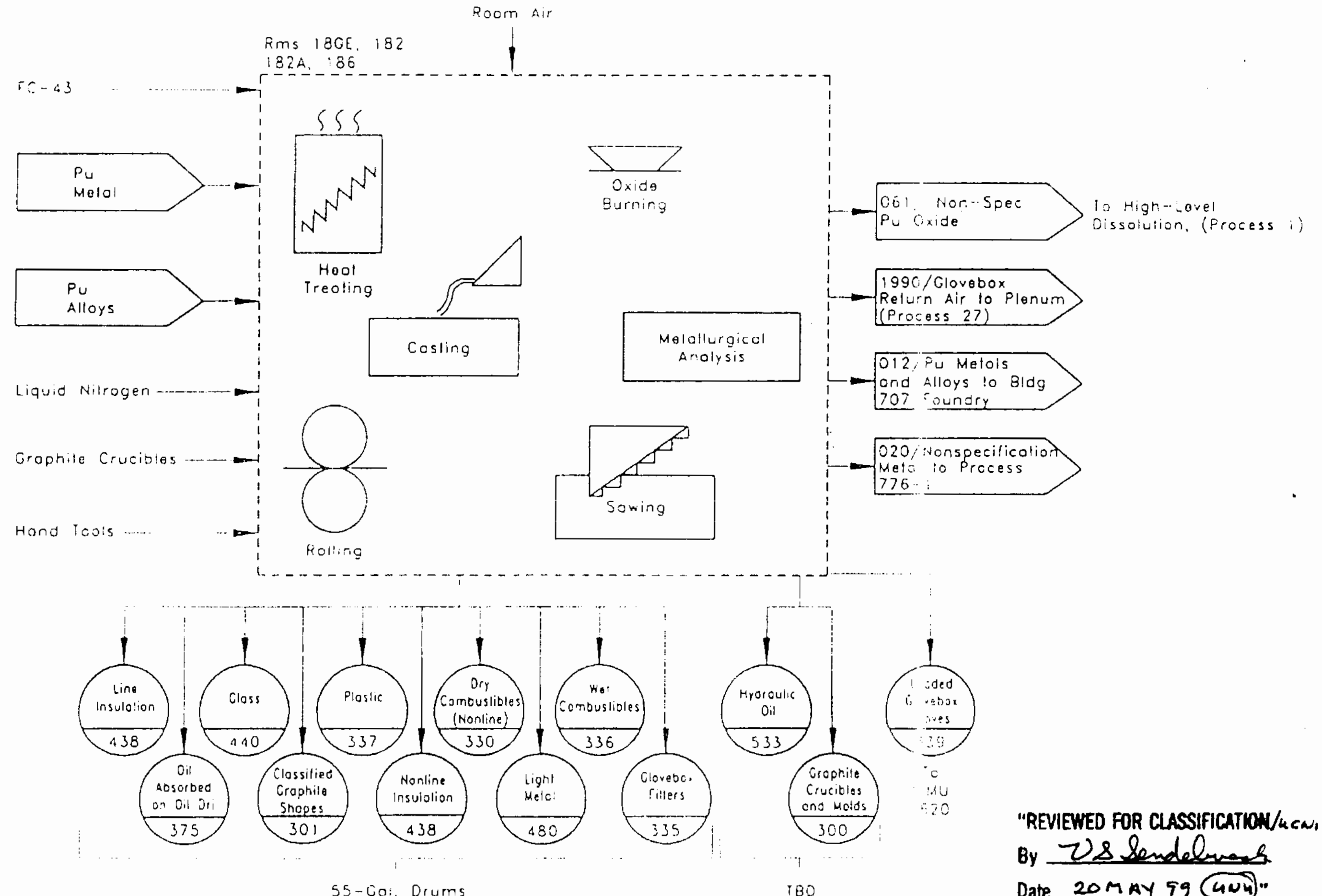


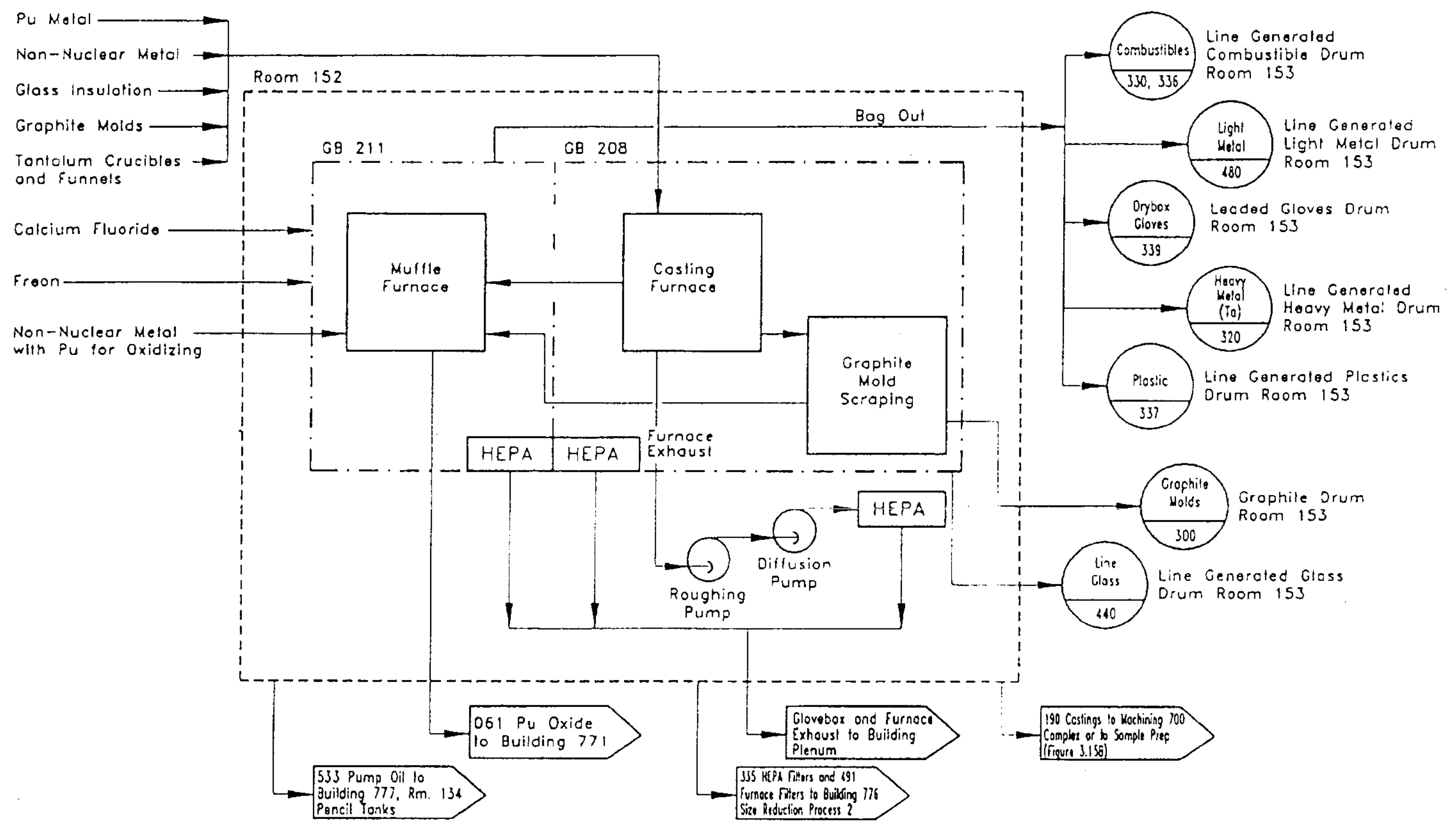
FIGURE 26.1

"REVIEWED FOR CLASSIFICATION/UCNI
By V8 Sendelweck
Date 20 MAY 99 (UNU)"

Rocky Flats Plant Waste Stream & Residue Identification & Characterization	BUILDING 771/PROCESS 26 PLUTONIUM METALLURGY	Task	By	Date
		Designer	R. Medlyn	07/11/90
		Drawn	J. Sikora	07/18/90
		Checked	J. Poynter	07/08/93

779-V3.2 Chg. 1 14-7 10/28/91

AutoCAD 11, File Name: f779-40d 9/11/91



"REVIEWED FOR CLASSIFICATION/UCNI"
By [signature]
Date 20 MAY 99 (UNU)

FIGURE 14.5

Rocky Flats Plant Waste Stream & Residue Identification & Characterization	BUILDING 779/PROCESS 14 EXPERIMENTAL CASTING	Task	By	Date
		Designed	M. Selman/B. Peters	6/21/90
		Drawn	E.B.McDonald	8/8/90
		Checked	S. Muse	8/1/91

Appendix B

TRU Graphite Debris
Acceptable Knowledge Waste Stream Summary

5.2 TRU Graphite Waste — Profile No. RF003.01

Acceptable Knowledge Waste Stream Summary

Waste Stream: TRU Graphite Waste, RF-300, RF-301U, RF-303, RF-310, RF-310P, RF-312

Generation Buildings: Buildings 371, 559, 707, 771, 776, 777, and 779 (6,10)

Waste Stream Volume (Current): 256 55-gallon drums(10,11)

Generation Dates (Current): October 1986 – April 1999(10,11)

Waste Stream Volume (Projected): 1,243 55-gallon drums (11,16)

Generation Dates (Projected): April 1999 – January 2000 (11,16)

TRUCON Content Code (1,12): RF 115A, RF 115B (pending), RF 115D (pending), RF 115E (pending), RF 115F (pending), RF 122C, RF 122D

Transuranic Waste Baseline Inventory Report Information (2)

WIPP Identification Number(s): RF-TT0300, RF-TR0300, RF-TT0301, RF-TR0301, RF-T0303, RF-TR0303, RF-TR0310, RF-TT0312, RF-TR0312

Summary Category Group: S5000 Waste Matrix Code Group: Not Identified

Waste Stream Name: Coarse Graphite/TRU

Description from the WTWBIR: Graphite chunks and coarse graphite

Waste Stream Description

TRU graphite waste consists of graphite molds (IDC 300), classified graphite shapes (IDC 301), formerly classified graphite shapes (IDC 301U), scarfed graphite chunks (IDC 303), graphite scarfings and fines (IDC 310), blended graphite scarfings and fines (IDC 310P), and coarse graphite (IDC 312). The following table presents the matrix parameter category and waste material parameters for graphite waste.(3)

IDC	IDC Description	Matrix Parameter Category	Waste Material Parameters	Weight % (Average)
300	Graphite Molds	S5126, Graphite Debris	Other Inorganic Materials	100%
301	Classified Graphite Shapes	S5126, Graphite Debris	Other Inorganic Materials	100%
301U	Formerly Classified Graphite Shapes	S3119, Unknown/Other Inorganic Particulates	Other Inorganic Materials	100%
303	Scarfed Graphite Chunks	S5126, Graphite Debris	Other Inorganic Materials	100%
310	Graphite Scarfings & Fines	S3119, Unknown/Other Inorganic Particulates	Other Inorganic Materials	100%
310P	Blended Graphite Scarfings & Fines	S3119, Unknown/Other Inorganic Particulates	Other Inorganic Materials	100%
312	Coarse Graphite	S5126, Graphite Debris	Other Inorganic Materials	100%

IDC 300, Graphite Molds: Molds used for casting plutonium metal. May also include graphite spacers or liners used in high temperature furnaces or ovens. Limited to 200 lb net weight per drum.[(4)]

IDC 301, Classified Graphite Shapes: Classified graphite shapes (primarily graphite molds and cores) or any part of a classified graphite shape. Limited to 200 lb net weight per drum. This waste will be declassified before disposal at WIPP. IDC 301 will become IDC 301U or 312 after declassification.[(4,13)]

IDC 301U, Formerly Classified Graphite Shapes: Classified graphite shapes that have been sanitized by crushing in a hammermill to a size of less than ½-inch in diameter.[(13)]

IDC 303, Scarfed Graphite Chunks: Graphite chunks and pieces remaining after scarfing are normally one inch in diameter or larger. Limited to 200 lb net weight per drum.[(4)]

IDC 310, Graphite Scarfings and Fines: Graphite scarfings and fines are finely divided graphite particles (20-25 micron average particle size) produced by mold cleaning (scarfing). This IDC also includes graphite floor sweepings. The calcium fluoride mold coating will also be present.[(4,6,13)]

IDC 310P, Blended Graphite Scarfings & Fines: A blended product of IDC 310 and IDC 301U.[(13)]

IDC 312, Coarse Graphite: Coarse graphite is that component of the graphite particles produced by mold cleaning which are generally less than one inch in diameter, but larger than fines. Limited to 200 lb net weight per drum.[(4)]

Areas of Operation

TRU graphite wastes were generated by the following defense operations: [3,6,13,14,15]

- Plutonium Production
- Plutonium Recovery
- Laboratory Operations
- Research and Development
- Decontamination and Decommissioning
- Residue Repackaging and Treatment

Generation Processes

Building 707 generated used graphite molds (IDC 300) and classified graphite shapes (IDC 301) during the casting of plutonium in production foundry operations. IDCs 300 and 301 were segregated. The working surfaces of the molds were coated with calcium fluoride prior to casting. Carbon tetrachloride was piped into the casting gloveboxes for cleaning purposes. After the plutonium casting was removed from the mold, the molds were collected in drums. Drums that contained recoverable amounts of plutonium were stored for subsequent scarfing of the molds.[5,6] See WSRIC 707-1 Figure 1.1 for a process flow diagram showing the generation of graphite molds and classified graphite shapes.[5]

After the casting of plutonium in production foundry operations, graphite molds were mechanically cleaned in Buildings 371, 707, 771, and 777 using a hand-held rotary-type sanding tool to grind off contamination, generating graphite chunks (IDC 303), graphite scarfings and fines (IDC 310), and coarse graphite (IDC 312). The mechanical cleaning (scarfing) of the mold surface removed most of the mold coating and plutonium contamination.[6] See WSRIC 707-1 Figure 1.1 for a process flow diagram showing the generation of scarfed graphite.[5]

Building 771 generated graphite crucibles and molds (IDC 300) and classified graphite shapes (IDC 301) during plutonium metallurgy operations which included alloy preparation, heat treating, rolling, metal cutting, and metallurgical testing cutting for research and development. See WSRIC 771-26 Figure 26.1 for a process flow diagram showing the generation of graphite.[6,7] Experimental casting operations in Building 779 tested metal compatibilities with various substrates. Samples were cleaned with Freon TF. Plutonium was heated in a furnace until molten and poured into graphite molds. See WSRIC 779-14 Figure 14.5 for a process flow diagram showing the generation of graphite.[6]

Graphite molds (IDC 300) were also generated from the Emission Spectroscopy process of Building 559. Plutonium oxide samples were mixed with sodium fluoride and packed into a graphite electrode used in the spectrometer during the analysis. The electrodes were replaced when their integrity was lost.[6]

Currently, Building 779 is undergoing decontamination and decommissioning. Graphite molds from historical research and development operations in the building are removed from gloveboxes for disposal. See WSRIC 779-40 Figure 40.1 for a process flow diagram showing the generation of graphite [15]

Graphite molds, chunks, scarfings and fines residues (IDCs 300, 303, and 310) are repackaged in Building 707, and coarse graphite residue (IDC 312) is repackaged in Buildings 707 and 776/777. The graphite is being repackaged to meet residue interim safe storage criteria and waste acceptance criteria (WAC) for the WIPP. Once the drums are opened, the contents examined, sorted, sized reduced if necessary, then repackaged and bagged out of the glovebox. See WSRIC 707-41 Figure 41.1, 707-42 Figure 42.1, and 776_777-6 Figure 6.4 for process flow diagrams for graphite repackaging.[13,14]

Classified graphite shapes (IDC 301) were originally generated in Buildings 707, 771, and 779 from plutonium casting and metallurgy operations that included alloy preparation, heat treating, rolling, metal cutting, and research and development. See WSRIC 707-1 Figure 1.1, WSRIC 771-26 Figure 26.1, and WSRIC 779-14 Figure 14.5 for process flow diagrams showing the generation of classified graphite shapes.[5,7,8] The classified graphite shapes are being declassified in Building 707 to meet WIPP WAC. The shapes are size reduced to less than ½-inch in diameter using a hammermill and labeled as IDCs 301U or 312. Some of the declassified graphite material will be blended with graphite scarfings and fines (IDC 310) to reduce the plutonium concentration to below ISSC limits. The blended graphite scarfings and fines are repackaged as IDC 310P. See WSRIC 707-41 Figure 41.1 for a process flow diagram showing the graphite declassification and blending processes.[13]

Graphite wastes in inventory originating in Building 776 (prefixes 19 and 25) were not generated in Building 776 but were repackaged in the size reduction area in this building. The graphite from Building 776 may have originated from any of the processes described in this section. See WSRIC 776-2 Figure 2.1 for a process flow diagram showing repackaging operations.[9]

RCRA Characterization

The following table presents the chemical constituent codes (CCC) and EPA Hazardous Waste Numbers associated with the BWR Subpopulations and WSRIC Waste Streams assigned to TRU graphite waste containers. Supporting characterization information is provided in the *BWR Baseline Book, WSRIC Building Book*, and *WSRIC archived files*.

IDC	BWR Subpopulation	WSRIC Waste Stream	RCRA CCC	Non-RCRA CCC	EPA Hazardous Waste Numbers
		Graphite Molds			
0300		707 - 41 - 2	00	1020	None
0300		779 - 40 - 130	00	00	None
0300		D&D - 3 - 92	00	00	None
0300	56A		00	1020	None
0300	56G		00	00	None
		Formerly Classified Graphite Shapes			
301U		707 - 41 - 30	00	00	None
		Scarfed Graphite Chunks			
0303		707 - 41 - 3	00	1020	None

IDC	BWR Subpopulation	WSRIC Waste Stream	RCRA CCC	Non-RCRA CCC	EPA Hazardous Waste Numbers
0303	56C		00	00	None
		Graphite Scarfings and Fines			
0310		707 - 41 - 31	00	00	None
0310		707 - 42 - 13	00	BR	None
		Blended Graphite Scarfings & Fines			
310P		707 - 41 - 32	00	00	None
310P		707 - 42 - 27	00	00	None
		Coarse Graphite			
0312		707 - 41 - 4	00	1020	None
0312		707 - 41 - 17	00	1020	None
0312		776_777 - 6 - 103	00	00	None
0312	56E		00	1020	None

Radionuclides

The following table summarizes the radionuclides present in graphite waste.[(3)]

IDC	Description	Radionuclides[1,2,3]
0300	Graphite Electrodes from Building 559	WG Pu
	Graphite Molds from Building 779 D&D	WG Pu, Am-241, DU, EU, Np-237, U-233
	Graphite Molds from all other operations	WG Pu, Am-241, EU, Np-237
0301	Classified Graphite Shapes	WG Pu, Am-241, EU, Np-237
301U	Formerly Classified Graphite Shapes	WG Pu, Am-241, EU, Np-237
0303	Scarfed Graphite Chunks	WG Pu, Am-241, EU, Np-237
310P	Blended Graphite Scarfings & Fines	WG Pu, Am-241, EU, Np-237
0312	Graphite, Coarse	WG Pu, Am-241, EU, Np-237

Key:		
	WG Pu	weapons-grade plutonium
	Am-241	americium-241
	DU	depleted uranium
	EU	enriched uranium
	Np-237	neptunium-237
	U-233	uranium-233

Notes:
1. Only waste generated before 1986 may contain Np-237 because processing of this material was discontinued at this time.
2. Only waste generated before 1983 may contain U-233 because processing of this material was discontinued at this time.
3. Am-241 (above ingrowth) and U-235 were detected by radioassay of backlog wastes from Building 707, prefix 12. These radionuclides were not anticipated based on acceptable knowledge but are being added for wastes generated before 1992 from prefix 12 because of these results.

References

1. DOE 1996. TRUPACT-II Content Codes (TRUCON), Revision 10. DOE/WIPP 89-004.
2. DOE 1995. Transuranic Waste Baseline Inventory Report, Revision 2. DOE/CAO-95-1121.

3. RMRS 1999. RFETS TRU Waste Acceptable Knowledge Supplemental Information. RF/RMRS-97-018, Revision 4.

4. RFETS 1998. Solid Radioactive Waste Packaging, 4-D99-WO-1100, Revision 1.

5. EG&G 1992. Waste Stream and Residue Identification and Characterization Building 707, Version 3.2.

6. RMRS 1998. Backlog Waste Reassessment Baseline Book, Waste Form 56, Graphite.

7. EG&G 1993. Waste Stream and Residue Identification and Characterization Building 771, Version 3.2.

8. EG&G 1993. Waste Stream and Residue Identification and Characterization Building 779, Versions 3.2.

9. EG&G 1993. Waste Stream and Residue Identification and Characterization Building 776, Version 3.2.

10. Waste and Environmental Management System (WEMS) database.

11. RMRS 1999. Interoffice Memorandum from Jeff Harrison to TWCP Records. JLH-002-1999. April 16.

12. DOE 1997. TRUPACT-II Content Codes (TRUCON), Revision 11. DOE/WIPP 89-004.

13. RFETS 1999. Waste Stream and Residue Identification and Characterization Building 707, Version 6.0.

14. RFETS 1999. Waste Stream and Residue Identification and Characterization Building 776/777, Version 6.0.

15. RFETS 1999. Waste Stream and Residues Identification and Characterization Building 779. Version 6.0.

16. K-H 1999. Kaiser-Hill Interoffice Memorandum from K. P. Ferrera to G. A. O'Leary. KPF-014-99. April 15.

Appendix C

INEEL Sampling Plan

Appendix C

Sampling Plan

IDC 300 Waste (Graphite Molds)

Note: The sampling plan was written to address contingencies (i.e., corrosivity, totals analysis, and prohibited item sampling and analysis) considered significant at the time of writing which, as it turns out, do not require addressing to demonstrate a nonmixed determination for the graphite waste. The sampling plan has been reproduced here as it was originally written and approved. The plan did not receive technical editing prior to its implementation; as a result, it contains typographical errors and errata that do not affect its technical content or validity. To allay potential concerns that a late revision date might reflect post-operational changes, no effort has been made to modify or edit the sampling plan from its original form.

SPECIAL CHARACTERIZATION SAMPLING AND ANALYSIS

OF GRAPHITE MOLD WASTE

August 13, 1998

Prepared by:

Lockheed Martin Idaho Technologies Company
Idaho National Engineering and Environmental Laboratory
PO Box 1625
Idaho Falls, Idaho 83415

Introduction

In order to meet the "Settlement Agreement with the State of Idaho" (a.k.a. the Batt agreement), the Idaho National Engineering and Environmental Laboratory (INEEL) is required to initiate shipments of transuranic (TRU) contaminated waste to the Waste Isolation Pilot Plant (WIPP) by April 30, 1999. The WIPP has not received their Resource Conservation and Recovery Act (RCRA) permit for accepting mixed waste. Therefore, the initial shipment(s) to WIPP will consist of non-mixed transuranic (TRU) waste only. To accomplish this, the INEEL must ensure that RCRA characterization is complete, retrievable, and defendable. The waste must be sampled and analyzed in a representative manner to allow accurate characterization of the waste as hazardous or nonhazardous. This Engineering Design File (EDF-RWMC-1028) describes the process used to insure that sampling is representative, defines the types of analyses required, and identifies the target analyze list. Following the sampling instructions given in this EDF will ensure RCRA compliance sampling. In addition, analytical methods from SW-846 and associated quality control (QC) protocols will be employed for analysis of the samples.

Corrosively tests per Method 9045C will also be conducted to assess if the waste has become corrosive due to potential radiolytic generation of HCl.

EDF-1028 is designed for the special needs of this initial waste shipment and is intended to compliment the INEEL's general characterization sampling plan (EDF 909 - Transuranic Sampling Plan for the INEEL). EDF-1028 does not replace or supersede the more general instruction given in EDF-909.

Because it has the greatest potential of being non-mixed TRU waste, IDC 300 (graphite molds) generated from Rocky Flats foundry and casting operations has been selected for the initial shipments to the WIPP. From the estimated 1100 IDC 300 drums, the INEEL has segregated 50 drums that real-time radiography (RTR) indicates contains only graphite mold waste. However previous IDC 300 visual examinations and the Acceptable Knowledge (AK) document provide information indicating that extraneous listed hazardous waste (i.e. rags, blotter paper, lab equipment) could be co-mingled with the graphite and not identified by RTR. As a result, all 50 drums will undergo intrusive visual examination at Argon National Laboratory West (ANL-W) to verify contents and absence of co-mingled extraneous items in the graphite waste. Drums found to contain prohibited items (rags, blotter paper, laboratory equipment, etc.) will be identified and not shipped to WIPP. Prohibited combustible items (i.e., rags, blotter paper, etc.) found will also undergo sampling and analysis. Additionally, ten drums from the population of 50 drums will be randomly selected for sampling and analysis of the graphite molds. One composite sample from each of these ten drums will be obtained. Five of the samples will undergo Toxicity Characteristic Leaching Procedure (TCLP) for metals, VOC and SVOC analyses, and the other five samples will undergo total VOC and SVOC analyses. Metals analysis is not required for the totals analysis because the intent of the totals analysis is to determine whether the graphite molds contain potentially "listed" VOCs or SVOCs.

Sampling and analysis will evaluate whether graphite itself is characteristically hazardous per the TCLP methods and whether the graphite contains any listed constituents known to have been used, or have had the potential to come into contact with the IDC 300 graphite molds. The IDC 300 mold process did not have any known or suspected changes to the process which would affect changes in the EPA code identification. Therefore, sample results from the analysis of the graphite molds described in this EDF can be used for RCRA evaluation of the remaining IDC 300 containers with similar contents.

Random Selection of 10 Drums from the Population of 50 Drums

Based on Stored Waste Examination Pilot Plant (SWEPP) RTR results, the 50 drums that make up the initial shipment population were carefully chosen to be as similar as possible in physical form, and free from any prohibited items. Therefore, the drum-to-drum variability of any contents should be very low. A random sample of 10 drums will be selected from the 50 drums. The first five of the 10 drums will be used for total VOC and SVOC analyses, and the other five drums will be used for TCLP analyses. Given the selection process a sample size of 5 drums is expected to be adequate to achieve the accuracy and precision necessary to show with high confidence (90% based on Chapter 9 in SW-846), whether this graphite mold waste is nonhazardous or hazardous. In addition to the TCLP and total VOCs/SVOCs analyses, portions of the sampled material will undergo testing to determine their corrosivity characteristics. The corrosivity testing will be described in its own section at the end of this report.

The 10 drums were randomly selected by serially numbering[2] the 50 drums and using the drums numbered below. These 10 random digits were selected from a list of 50 using SAS® software and they are given below.

Sample Drums
5
6
19
26
31
36
37
43
44
47

It should be noted that if during repackaging activities any one of the 50 drums is found to contain prohibited combustible materials (e.g. rags, blotter paper), then a representative sample of this material will be taken for total VOCs/SVOCs analyses. This drum will not be shipped to WIPP. If a drum selected for TCLP or total VOCs/SVOCs sampling does not meet the debris definition (greater than 50% by volume of the material within a drum must exceed 60 mm particle size), this drum does not represent the population being characterized and the next available drum will take its place for the intrusive sampling. Specifically the remaining drum's serial numbers will be decreased by one. For example, if drum number 12 did not meet the criteria for debris waste, it would be excluded from the population. The drum previously numbered 13 would become 12, drum number 14 would become 13, and so on.

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[2] Serially number the drums according to their barcode order or their shipment order to Argonne Laboratory (see letter from T. L. Clements to C.C. Dwight, TLC-67-98). These factors are independent of the drawn sample and thus prevent any bias.

Random Selection of Three Graphite Mold Pieces from Each of the of 10 Sample Drums

Three randomly selected graphite mold pieces will be taken from each sample drum and broken into smaller pieces and composited into a single sample for the TCLP or total VOCs/SVOCs analyses. To obtain a representative sample of the graphite molds from within a drum, one mold piece will be randomly sampled from the top 1/3 of the drum waste, one mold from the middle 1/3 of the waste, and one mold from the bottom 1/3 of the waste[3] This vertically spread sample will ensure adequate coverage of the drum waste from top to bottom in case there are any such spatial differences.

The process used to draw this random sample of graphite mold pieces follows:

1) Determine the next available number on the table of random starting numbers (Table 1 at the end of this EDF). Only one random starting number is used per sample drum and the numbers should be crossed off the table as they are used so they are not inadvertently reused.

2) The graphite mold pieces are removed from the drum in a systematic manner from the top of the drum to the bottom and counted as they removed. When the selected random number is reached, the corresponding graphite piece is placed on the sample table. Then every 25th piece after this is also placed in order on the table. For example, if the random starting number were 23, then the 23rd, 48th, 73rd, 98, etc. pieces would be laid out in order on the table. As the non-selected pieces are unpacked they will either be immediately placed in the repack drum or placed in a separate pile from the selected sample pieces. (Reminder: Only mold pieces of size 60mm or larger are considered for sampling. Also, it is assumed that at least 75 mold pieces will be available in the drum. If not, then the random starting number and interval should be halved.) This should result in 3 to 20 selected pieces of graphite molds.

3) When the drum is completely unpacked, divide the selected mold pieces on the table into thirds (representing the top, middle, and bottom thirds of the drum) while maintaining their order. One way of doing this would be to use a visual marker (e.g., string, pointer, slip of paper) to separate the thirds. For each third, roll a die to determine which mold piece to use for the sample. For example, if the number "2" is rolled, then the 2nd ordered piece is selected. If the number rolled is larger than the number of pieces available, then the die should be rolled again. (It is not expected that there will be more than six pieces on the table for any drum third, so a regular six-sided die should be adequate for this selection process.) When done with this step, the three mold pieces selected from the drum should be used for the next step.

Note: The lining/covering on the table should be changed between each set of molds to prevent potential RCRA cross-contamination.

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3 It is understood that this vertical stratification of the drums into 1/3s is approximate due to logistical/practical constraints.

Random Selection of a Portion of the Graphite Mold Piece for Use as Part of the Composite Sample

Once the particular mold pieces have been selected, the portion of the individual mold to be removed and used in the composite sample needs to be randomly chosen. One hundred-thirty to 150 g [EU1]of [EU2]composite material are required for the TCLP analysis and 30 to 50 g of composite material for the total VOC/SVOC analysis. (Collect 300 g on the first TCLP analysis to allow ACL to perform a laboratory duplicate extraction. Additional material is not needed for a total duplicate). Approximately one-third of the final amount required should be taken from each mold piece and added to the composite sample material (i.e. either 50 g for TCLP samples or 10 g for the total VOC/SVOC samples). The portion of the mold piece to use for the sample should be randomly chosen given the mold piece is larger than the required 50 g or 10 g. The following procedure will randomly select a portion of the mold piece to use as the sample.

With the piece lying before the handler, the assistant first flips a coin to determine whether the right or left half of the piece should be used (heads = right, tails = left). Then, if this piece is still too large, repeat this process until the piece has been reduced to appropriate size.

The three graphite mold portions will then be sized into pieces following ANL-W procedures. Clean/new equipment should be used between each drum. For TCLP samples the pieces should be capable of passing through a 9.5 mm sieve. [EU3]For total VOCs/SVOCs sampling, the pieces need to be approximately 5 grams each. [PC4]Once appropriately sized the pieces are mixed and a single composite sample is removed and placed in a glass sample bottle. For the purpose of preserving the organic constituents, headspace should be minimized in the sample bottles. This composited sample will be sent to the Analytical Chemistry Laboratory (ACL) for TCLP or total VOCs/SVOCs analysis[EU5][EU6][EU7][EU8].

1.0 Prohibited Item Sampling

Prohibited items (e.g., rags, blotter paper) discovered during the evaluation of the 50 IDC 300 drums, will undergo sampling and analysis. If blotter paper or rags are encountered, operators should examine the material for any signs of discoloration that might indicate liquids had been absorbed onto the material. If discoloration is apparent, samples should be collected from the discolored area. It is not expected that there will be multiple papers or rags in these containers, but if there are the operators should attempt to sample from each of the rags, acquiring approximately equal quantities of material from each of the locations to form a single composite. A total of 30 to 50 g of sample material must be collected. In the case that there is no discoloration, the same coin-toss methodology described for graphite molds should be applied to determine a sampling location on the paper or rags. Sample material should be placed into the smallest glass sample bottle that will accommodate the material. Analysis will be for total VOCs/SVOCs only[EU9][EU10]. Metals analysis is not required. Based on past intrusive characterization results, it is expected that no more than four drums will contain prohibited combustibles. Therefore, funding is available for up to four drum containing rags or blotter paper found in the 50 IDC 300 series drums of graphite waste.

Note: New/clean sampling equipment should be used for each drum.

2.0 Sample Identification and Shipping to Laboratory

All samples will be maintained under chain-of-custody (COC), starting at the time of sample collection. Samples will be cooled and stored at 4°C or less during storage and shipment. Samples must be shipped to the ACL in a timely manner to meet the 14-day TCLP and VOCs/SVOCs holding times.

Samples collected from graphite molds or prohibited items will be identified as "ID" followed by the drum barcode number, a serial number for the sample container, and a sample type descriptor. Multiple sample containers are provided in case the amount of sample material exceeds the sample container capacity, or for spare samples. These multiple samples will be prepared from the same composite material, so be essentially considered to be the same sample. The descriptors for sample types are "TOT" for a totals sample collected from graphite molds, "TCL" for a TCLP sample collected from graphite molds, and "PI" for samples collected from combustible prohibited items. For example, ID0155552TCL indicates the second sample container collected from drum barcode 015555 that will be analyzed by TCLP.

3.0 Sample Analysis for TCLP

TCLP extraction will be performed per SW-846 Method 1311 for metals, SVOCs, and VOCs. Per Method 1311, a separate zero-headspace extraction will be performed for VOC analysis. TCLP extracts will be analyzed for target analytes listed in Table 2 using the following EPA protocols:

Method 3010 – Acid Digestion of Aqueous Samples and Extracts for Total Metals

Method 6010 – Inductively Coupled Plasma-Atomic Emission Spectrometry

Method 7470 – Mercury in Liquid Waste (Manual Cold Vapor Technique)

Method 5030 – Purge-and-Trap for Aqueous Samples

Method 3510 – Separatory Funnel Liquid-Liquid Extraction

Method 8260 – VOC by GC/MS

Method 8270 – SVOC by GC/MS.

One duplicate TCLP extraction will be performed. QC specified in SW-846 methods will be followed and documented. TCLP extracts will not be analyzed for pesticides.

Sample Analysis of Graphite Samples for Total VOCs and SVOCs

Graphite samples will be analyzed for the total VOCs and SVOCs listed in Tables 3 and 4, respectively, using the following EPA protocols:

Method 5030 – Purge-and-Trap for Aqueous Samples

Method 3550 – Ultrasonic Extraction

Method 8260 – VOC by GC/MS

Method 8270 – SVOC by GC/MS.

QC specified in SW-846 methods will be followed and documented. A matrix spike duplicate (MSD) and a laboratory duplicate will be analyzed.

Analysis of Prohibited Item Samples for Total VOCs and SVOCs

Prohibited item samples will be analyzed for the total VOCs and SVOCs listed in Tables 3 and 4, respectively, using the following EPA protocols:

Method 5030/5035 – Purge and Trap for Aqueous/Soil & Waste Samples

Method 3550 – Ultrasonic Extraction

Method 8260 – VOC by GC/MS

Method 8270 – SVOC by GC/MS.

Due to the physical nature of the samples, Method 3550 (Ultrasonic Extraction) will be performed using an ultrasonic bath per Oak Ridge National Laboratory Standard Analytical Method # CASD-AM-SW846-3550. QC specified in SW-846 methods will be followed and documented.

Corrosivity Testing

Corrosivity testing will be performed per SW-846 Method 9045C on one of the composited graphite samples collected for total VOC/SVOC or for TCLP analysis. If sufficient volume of a single drum's composite sample remains after the total VOC/SVOC or TCLP testing sample is withdrawn (approximately 40 g needed), a corrosivity measurement and a laboratory duplicate will be performed on that sample. If an insufficient amount of single drum sample material remains after the total VOC/SVOC or TCLP testing sample is withdrawn, the laboratory will combine[4] and composite the sample material from multiple drums, until sufficient graphite material is obtained to perform the corrosivity test and laboratory duplicate. In either case, the laboratory will identify the samples and amounts of each sample used in the final composite sample.

Data Deliverables

The ACL will produce full data packages for TCLP and VOCs/SVOCs analyses, which will include reporting forms for sample and QC results, COC documentation, and copies of all associated raw data.

ANL-W will provide data packages for the repackaged drums according to the requirements and procedures used for data reporting for the transuranic waste characterization program, including

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[4] The drums should be combined in the serial order they were sampled in to avoid any potential bias.

videotapes. ANL-W will also need to include any information which documents the process used to randomly select materials for sampling from these drums.

Deviations from Planned Analytical Methods

Any deviations from this EDF or changes to planned analytical methods defined in this EDF will require approval by the Site Project Office (SPO) and the ACL, and will be documented either in a Program Change Notice (PCN) or a revision to this EDF.

Appendix to Sampling Plan

Table 1. Table of Random Starting Numbers

23
19
20
11
18
8
21
15
25
8
23
16
23
19
6
4
12
22
3
22
16
1
5
20
7

Appendix to Sampling Plan

Table 2. Target Analytes and Maximum Concentrations for TCLP

EPA HW No.	Target Analyte	[CAS#]	Regulatory Level (mg/L)
D004	Arsenic	[7440-38-2]	5.0
D005	Barium	[7440-39-3]	100.0
D018	Benzene	[71-43-2]	0.5
D006	Cadmium	[7440-43-9]	1.0
D019	Carbon tetrachloride	[56-23-5]	0.5
D021	Chlorobenzene	[108-90-7]	100.0
D022	Chloroform	[67-66-3]	6.0
D007	Chromium	[7440-47-3]	5.0
D023	o-Cresol	[95-48-7]	200*
D024	m-Cresol	[108-39-4]	200*
D025	p-Cresol	[106-44-5]	200*
D026	Cresol		200*
D027	1,4-Dichlorobenzene	[106-46-7]	7.5
D028	1,2-Dichloroethane	[107-06-2]	0.5
D029	1,1-Dichloroethylene	[75-35-4]	0.7
D030	2,4-Dinitrotoluene	[121-14-2]	0.13
D032	Hexachlorobenzene	[118-74-1]	0.13
D033	Hexachlorobutadiene	[87-68-3]	0.5
D034	Hexachloroethane	[67-72-1]	3.0
D008	Lead	[7439-92-1]	5.0
D009	Mercury	[7439-97-6]	0.2
D035	Methyl ethyl ketone	[78-93-3]	200.0
D036	Nitrobenzene	[98-95-3]	2.0
D037	Pentachlorophenol	[87-86-5]	100.0
D038	Pyridine	[110-86-1]	5.0
D010	Selenium	[7782-49-2]	1.0
D011	Silver	[7440-22-4]	5.0
D039	Tetrachloroethylene	[127-18-4]	0.7
D040	Trichloroethylene	[79-01-6]	0.5
D041	2,4,5-Trichlorophenol	[95-95-4]	400.0
D042	2,4,6-Trichlorophenol	[88-06-2]	2.0
D043	Vinyl chloride	[75-01-4]	0.2

* If o-, m-, and p-cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used.

Appendix to Sampling Plan

Table 3. Target Analytes for Total VOC Analysis.

Acetone	1,2-Dichloropropane
Benzene	cis-1,3-Dichloropropene
Bromodichloromethane	trans-1,3-Dichloropropene
Bromoform	Ethylbenzene
Bromomethane	2-hexanone (methyl butyl ketone)
2-butanone (methyl ethyl ketone)	4-methyl-2-pentanone (methyl isobutyl ketone)
Carbon disulfide	Methylene chloride
Carbon tetrachloride	Styrene
Chlorobenzene	1,1,2,2-Tetrachloroethane
Chloroform	Tetrachloroethene
Chloromethane	1,1,1-Trichloroethane
Dibromochloromethane	1,1,2-Trichloroethane
1,1-Dichloroethane	Trichloroethene
1,2-Dichloroethane	Toluene
1,1-Dichloroethene	Vinyl chloride
Cis-1,2-Dichloroethene	m-Xylene and p-Xylene
Trans-1,2-Dichloroethene	o-Xylene

Appendix to Sampling Plan

Table 4. Target Analytes for Total SVOC Analysis

Acenaphthene	2,4-Dinitrotoluene
Acenaphthylene	2,6-Dinitrotoluene
Anthracene	bis(2-Ethylhexyl)phthalate
Azobenzene (1,2-Diphenylhydrazine)	Fluoranthene
Benzo(a)anthracene	Fluorene
Benzo(a)pyrene	Hexachlorobenzene
Benzo (b and k) fluoranthene	Hexachlorobutadiene
Benzo(g,h,i)perylene	Hexachlorocyclopentadiene
4-Bromophenyl-phenylether	Hexachloroethane
Butylbenzylphthalate	Indeno(1,2,3-cd)pyrene
4-Chloroaniline	Isophorone
Bis(2-Chloroethoxy)methane	2-Methylnaphthalene
Bis(2-Chloroethyl)ether	2-Methylphenol (o-Cresol)
4-Chloro-3-methylphenol	3-Methylphenol and 4-Methylphenol (3&4-Cresol)
2-Chloronaphthalene	Naphthalene
2-Chlorophenol	2-Nitroaniline
4-Chlorophenyl-phenylether	3-Nitroaniline
Bis(2-chloroisopropyl)ether	4-Nitroaniline
Carbazole	Nitrobenzene
Chrysene	2-Nitrophenol
Dibenzo(a,h)anthracene	4-Nitrophenol
Dibenzofuran	N-Nitrosodiphenylamine
1,2-Dichlorobenzene	N-Nitroso-dimethylamine
1,3-Dichlorobenzene	N-Nitroso-di-n-propylamine
1,4-Dichlorobenzene	Pentachlorophenol
2,4-Dichlorophenol	Phenanthrene
Diethylphthalate	Phenol
2,4-Dimethylphenol	Pyrene
Dimethylphthalate	Pyridine
Di-n-butylphthalate	1,2,4-Trichlorobenzene
Di-n-octylphthalate	2,4,5-Trichlorophenol
4-6-Dinitro-2-methylphenol	2,4,6-Trichlorophenol
2,4-Dinitrophenol	Tri-n-butyl phosphate

Appendix D

INEEL Analytical Data Summary

Appendix D

INEEL Analytical Data Summary

IDC 300 Waste (Graphite Molds)

Five drums of IDC 300 waste (graphite molds) stored at the INEEL were randomly selected from a population of 50 drums certified to meet the requirements of WIPP WAC (Rev. 5) to undergo sampling, TCLP extraction, and analysis. TCLP extraction and analysis were conducted to determine if the waste stream contains toxic characteristic constituents in quantities greater than the regulatory threshold limits. The results of TCLP testing and analysis are given in the accompanying tables. Note that duplicate samples were taken whose values were averaged for this analysis. The majority of the laboratory measured analyte values were "U" flagged, indicating they were below the method detection limit (MDL). Chapter 9 of SW-846 does not give guidance for the use of less-than-MDL values. Therefore, the guidance given in the QAPP for the analysis was followed – replace the "U" flagged value with ½ the MDL. In addition, "B" flagged analyte sample values (indicating that the corresponding blank was contaminated with the given analyte) were used "as reported" for this analysis.

The formula used to calculate the 90% upper confidence limit (UCL90) is:

$$\mathbf{UCL}_{90} = \bar{\mathbf{x}} + \frac{\mathbf{t}_{.90,\,n-1}\,\mathbf{s}}{\sqrt{\mathbf{n}}}$$

Where s is the sample standard deviation, $\bar{x}$ is the sample mean, n is the number of non-MDL measurements for the analyte, and $t_{.90,\,n-1}$ is the 90th percentile value for a t distribution with n-1 degrees of freedom. Note that SW-846 specifies that the 80th percentile be used for the calculations, whereas the 90th percentile values were used. This more conservative approach was taken to be consistent with the statistical reduction methods used in the QAPP.

The parameters used to validate the analytical results discussed below were:

- Sample holding time before analysis
- GC/MS instrument tune
- Initial and continuing calibrations
- Method blank analysis
- Surrogate recoveries
- Matrix spike/matrix spike duplicate (MS/MSD) analysis
- Laboratory control sample (LCS) analysis
- Internal standard performance
- Compound identification
- Field duplicates

Appendix D

Summary of Results with Volatile Organic Compounds

No data qualification was necessary based on the validation parameters, with the exception of blank contamination. The independent validator added a "U" flag (undetected false positives) for 1,1-dichloroethene, carbon tetrachloride, and 2-butanone.

Summary of Results with Semi-Volatile Organic Compounds

No data qualification was necessary based on the validation parameters, with the exception of one MS sample. The independent validator added a qualifier for the pentachlorophenol analysis on one sample. This sample was an MS sample and the pentachlorophenol recovery was 0%. Since the pentachlorophenol recovery was compliant in the laboratory control sample, only the pentachlorophenol result for the spiked sample was rejected ("R" flagged). The UCL90 data reduction was performed on four sample results. This is probably overly conservative. Closer examination of the data package supports the contention that the 0% MSD recovery for this target analyte is most likely explained by the assumption that the acid surrogates and spiking compounds were not actually added to the sample. The MS recovery for pentachlorophenol is 115%, well within acceptance limits.

Summary of Results with Metal Compounds

No data qualification was necessary based on the validation parameters, with the exception of the preparation blank for mercury (Hg) and the "J" flag for silver (Ag). The preparation blank had positive detections resulting in the addition of a "U" flag (undetected false positives).

The "J" flag (estimated concentration) for silver was based on a 64.2% Ag TCLP spike recovery. Only one sample had detected silver results. As pointed out by the validator, if the sample results were corrected for the bias implied by the TCLP spike recovery, the corrected value would be two to three orders of magnitude below the TCLP regulatory limit of 5000 μg/L. Thus, the recovery and addition of a "J" qualifying flag has no impact on the usability of the data to determine these samples do not exhibit the characteristic toxicity due to Ag.

Appendix D

Table D-1: TCLP Volatile Organic Compound (VOC) Summary Data

Analyte	No. of Samples	No. of Samples above MDL	MDL (mg/L)	Mean[1] (mg/L)	SD[1] (mg/L)	UCL_{90}[2] (mg/L)	*Reg. Level* (mg/L)
1,1-Dichloroethene	5	2	0.0010	-7.289 [L]	0.4623 [L]	0.00187 [U]	*0.7*
1,2-Dichloroethane	5	0	0.0010	0.0005	0	0.00138	*0.5*
Benzene	5	0	0.0010	0.0005	0	0.00138	*0.5*
Carbon tetrachloride	5	5	0.0020	-5.621 [L]	0.1409 [L]	0.00399 [U]	*0.5*
Methyl ethyl ketone	5	5	0.0030	0.004	0.000464	0.00432	*200.0*
Chloroform	5	0	0.0010	0.0005	0	0.00138	*6.0*
Chlorobenzene	5	0	0.0020	0.001	0	0.00275	*100.0*
Tetrachoroethene	5	0	0.0020	0.001	0	0.00275	*0.7*
Trichloroethylene	5	0	0.0010	0.0005	0	0.00138	*0.5*
Vinyl chloride	5	0	0.0010	0.0005	0	0.00138	*0.2*

[1] One half the MDL was used in these calculations for all laboratory values flagged "U" (i.e., less than MDL), per the guidance in the QAPP.

[2] Upper confidence limits for analytes with all values flagged "U" were calculated assuming the maximum possible variance and mean, thereby yielding the most conservative upper confidence limit. Using benzene for example, the maximum variance would occur when two of the five measured values were at zero and the other three values were at the MDL (e.g., $x_1 = 0$, $x_2 = 0$, $x_3 = 0.001$, $x_4 = 0.001$, $x_5 = 0.001$). The maximum mean would be 0.001.

[L] Value in natural log (*ln*) units – i.e., *ln* transformation used to make data more normally distributed (as measured by Shapiro-Wilk statistic). This procedure is per the QAPP.

[U] Value in untransformed units (mg/L).

Appendix D

Table D-2: TCLP Semi-VOC Summary Data

Analyte	No. of Samples	No. of Samples above MDL	MDL (mg/L)	Mean[1] (mg/L)	SD[1] (mg/L)	UCL90[2] (mg/L)	Reg. Level (mg/L)
o-Cresols	5	0	0.013	0.0065	0	0.0179	200.0
m&p-Cresols	5	0	0.014	0.0070	0	0.0193	200.0
Hexachloroethane	5	0	0.0082	0.0041	0	0.0113	3.0
Nitrobenzene	5	0	0.011	0.0055	0	0.0151	2.0
Hexchlorobutadiene	5	0	0.011	0.0055	0	0.0151	0.5
2,4,6-Trichlorophenol	5	0	0.013	0.0065	0	0.0179	2.0
2,4,5-Trichlorophenol	5	0	0.013	0.0065	0	0.0179	400.0
2,4-Dinitrotoluene	5	0	0.010	0.0050	0	0.0138	0.13
Hexachlorobenzene	5	0	0.014	0.0070	0	0.0193	0.13
Pentachlorophenol	4 [3]	0	0.010	0.0050	0	0.01473	100.0
1,4-Dichlorobenzene	5	0	0.0071	0.00355	0	0.0098	7.5
Pyridine	5	0	0.010	0.0050	0	0.0138	5.0

1 One half the MDL was used in these calculations for all laboratory values flagged "U" (less than MDL), per the guidance in the QAPP.

2 Upper confidence limits for analytes with all values flagged "U" were calculated assuming the maximum possible variance and mean, thereby yielding the most conservative upper confidence limit. Using o-Cresols for example, the maximum variance would occur when two of the five measured values were at zero and the other three values were at the MDL (e.g. $x_1 = 0$, $x_2 = 0$, $x_3 = 0.013$, $x_4 = 0.013$, $x_5 = 0.013$). The maximum mean would be 0.013.

3 Due to a pentachlorophenol recovery problem in one sample, the independent technical review rejected the reported pentachlorophenol value for that sample. This is presented in detail in the discussion on semi-volatile organic qualifying flags.

Appendix D

Table D-3: TCLP Metals Summary Data

Analyte	No. of Samples	No. of Samples above MDL	MD[L] (mg/L)	Mean[1] (mg/L)	SD1 (mg/L)	UCL90[2] (mg/L)	Reg. Level (mg/L)
Arsenic	5	0	0.0208	0.0104	0	0.0286	5.0
Barium	5	5	0.0022	0.4182	0.0582	0.458	100
Cadmium	5	3	0.0043	0.0038	0.0017	0.0057	1.0
Chromium	5	3	0.0040	0.0106	0.0105	0.0220	5.0
Lead	5	0	0.0479	0.0240	0	0.0659	5.0
Mercury	5	3	0.00014*	0.0037	0.0077	0.0121	0.2
Selenium	5	0	0.0254	0.0127	0	0.0349	1.0
Silver	5	1	0.0071	0.0107	0.0160	0.0255	5.0

1 One half the MDL was used in these calculations for all laboratory values flagged "U" (less than MDL), per the guidance in the QAPP.

2 Upper confidence limits for analytes with all values flagged "U" were calculated assuming the maximum possible variance and mean, thereby yielding the most conservative upper confidence limit. Using arsenic for example, the maximum variance would occur when two of the five measured values were at zero and the other three values were at the MDL (e.g., $x_1 = 0$, $x_2 = 0$, $x_3 = 0.0208$, $x_4 = 0.0208$, $x_5 = 0.0208$). The maximum mean would be 0.0208. For silver which has four of five values less than the MDL, the most conservative upper confidence limit was calculated where 0 was used for the four values less than MDL, and the mean was $\bar{x} = ½(4*MDL + 0.0392)$.

* For one sample (8BP89), the MDL was higher (0.035 mg/L) because a smaller aliquot was used due to high alpha activity in the sample. The data, however, were applied in the normal manner.

Appendix E

Headspace Gas Analysis Data Summary

Appendix E

Headspace Gas Analysis Data Summary

Graphite Waste (IDC 300)

Twenty-six drums have been subjected to headspace gas sampling. The headspace gas analysis results have been statistically evaluated. The upper confidence limit was calculated as follows:

$$\mathbf{UCL}_{90} = \bar{\mathbf{x}} + \frac{\mathbf{t}_{.90,n^*-1}\,\mathbf{s}}{\sqrt{\mathbf{n}}}$$

Where s is the sample standard deviation, $\bar{x}$ is the sample mean, n is the number of samples analyzed for the analyte, $t_{.90,n^*-1}$ is the 90th percentile value for a t distribution with n*-1 degrees of freedom, and n* is the number of non-MDL measurements in the data set. This conservative approach is consistent with the statistical reduction methods used in the QAPP.

Appendix E

Table E-1: Headspace Gas Summary Data

Analyte	# Samples	# Samples above MDL	Mean[1] (ppmv)	SD[1] (ppmv)	UCL_{90} (ppmv)	PRQL (ppmv)
1,1,1-Trichloroethane	26	2	0.104	0.185	0.215	10
1,1,2-Trichloro-1,2,2-trifluoroethane	26	0	0.2	0.065	0.239[m]	10
Acetone	26	26	4.573	2.443	5.203	100
Benzene	26	8	0.153	0.200	0.209	10
Butanol	26	0	4.4	1.426	5.261[m]	100
Carbon tetrachloride	26	2	0.075	0.092	0.130	10
Chlorobenzene	26	0	0.2	0.065	0.239[m]	10
Ethyl benzene	26	0	0.2	0.065	0.239[m]	10
Ethyl ether	26	0	0.2	0.065	0.239[m]	100
m,p-Xylene	26	0	0.2	0.065	0.239[m]	10
Methanol	26	22	8.915	7.615	10.89	100
Methyl ethyl ketone	26	3	0.366	0.294	0.475	100
Methyl isobutyl ketone	26	0	2.2	0.713	2.630[m]	100
Methylene chloride	26	2	0.093	0.153	0.185	10
o-Xylene	26	0	0.2	0.065	0.239[m]	10
Tetrachloroethylene	26	0	0.2	0.065	0.239[m]	10
Toluene	26	2	0.288	0.866	0.811	10
Trichloroethylene	26	0	0.2	0.065	0.239[m]	10

1. Unless otherwise noted, one half the analysis Method Detection Limit (MDL) was used in these calculations for laboratory values flagged "U" (less than MDL), per guidance in QAPP. Note that the MDL for a given analyte may vary from sample aliquot to sample aliquot.

m. Upper confidence limits for analytes with all values flagged "U" were calculated assuming the maximum possible variance, mean and t statistic (i.e., $t_{90,1}$), thereby yielding the most conservative upper confidence limit. Using butanol for example, the maximum variance occurs where 15 of the 26 measured values are assumed to be zero and the other 11 values are at the associated MDL. To be conservative, it was assumed the maximum possible mean would be the highest MDL recorded.

All data are reported in untransformed units (ppmv).