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

July 29, 1999

RECEIVED 1999

ENTERED

Mr. Steve Zappe
RCRA Permitting
New Mexico Environment Department
P. O. Box 26110
Santa Fe, NM 87502

Dear Mr. Zappe:

Please find enclosed the Idaho National Engineering and Environmental Laboratory (INEEL) Non-hazardous Waste Determination for IDC 300 (Graphite Molds) Waste Stream (INEEL/EXT-98-01137) dated July, 1999. As we discussed at the WIPP Quarterly Review Meeting on July 28, 1999, the original package was hand delivered to New Mexico Environment Department (NMED) on July 9, 1999.

If you have any questions please call me at (505) 234-7483.

Sincerely,

Robert A. Stroud, Team Leader
National TRU Waste Programs
Carlsbad Area Office

Enclosure

CAO:OWDO:RAS 99-0982 / UFC 5822



990722

INEEL/EXT-98-01137

July 1999

# Nonhazardous Waste Determination for IDC 300 (Graphite Molds) Waste Stream

*R. E. Arbon*

INEEL/EXT-98-01137

# Nonhazardous Waste Determination for IDC 300 (Graphite Molds) Waste Stream

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Published July 1999

Idaho National Engineering and Environmental Laboratory
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Idaho Falls, Idaho 83415

Prepared for the
U.S. Department of Energy

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Contract DE-AC07-94ID13223

# SUMMARY

This document, Revision 2 of the Nonmixed Determination Report, summarizes the efforts performed at the Idaho National Engineering and Environmental Laboratory (INEEL) to make a hazardous waste determination on Item Description Code (IDC) 300 drums containing graphite waste which meet WIPP Waste Acceptance Criteria (WAC) and Trupact II Authorized Methods for Payload Control (TRAMPAC) requirements. This characterization effort included a thorough compilation and documentation of acceptable knowledge, physical characterization, waste form sampling, and chemical analyses, and additional headspace gas sampling data. Based upon the information and data summarized in this document, drums containing IDC 300 graphite molds are not hazardous waste (i.e., are nonmixed waste). No hazardous waste codes are assigned to the above IDC 300 (graphite molds) waste stream as specified in Title 40 of the Code of Federal Regulations, Part 261 (40 CFR 261). Characterization of drums containing IDC 300 waste per the Transuranic Waste Characterization Quality Assurance Program Plan (QAPP) will continue. Only drums that meet the Waste Acceptance Criteria and TRAMPAC for the Waste Isolation Pilot Plant, prepared by the Department of Energy (DOE) will be shipped.

INEEL/EXT-98-01137
Rev 2
July 1999

# Idaho National Engineering and Environmental Laboratory

## Nonhazardous Waste Determination

## for IDC 300 (Graphite Molds) Waste Stream

| | |
|---|---|
| R. E. Arbon, Manager, INEEL Site Project Office | 7/7/99 Date |
| T. L. Clements, Jr., Manager, INEEL TRU Waste Program | 7/7/99 Date |
| G. E. "Jud" Ellis, Assistant Manager, Office of Program Execution | 7/7/99 Date |
| L. L. Fritz, Director, DOE-ID Waste Management Program | 7/7/99 Date |
| Jerry L. Lyle, DOE-ID, Assistant Manager, Office of Program Execution | 7/8/99 Date |

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# ACRONYMS

| | |
|---|---|
| ACL | Analytical Chemistry Laboratory |
| AK | acceptable knowledge |
| ANL-W | Argonne National Laboratory - West |
| BWR | Backlog Waste Reassessment |
| CAO | Carlsbad Area Office |
| CFR | Code of Federal Regulations |
| COC | chain of custody |
| DOE | Department of Energy |
| EDF | Engineering Design File |
| EDL | economic discard limit |
| EPA | Environmental Protection Agency |
| GC/MS | gas chromatography/mass spectroscopy |
| IDC | Item Description Code |
| IHWMA | Idaho Hazardous Waste Management Act |
| INEEL | Idaho National Engineering and Environmental Laboratory |
| LANL | Los Alamos National Laboratory |
| LWA | Land Withdrawal Act |
| MDL | Method Detection Limit |
| MSD | matrix spike duplicate |
| NDA | nondestructive assay |
| PAN | passive-active neutron interrogation |
| PCN | Program Change Notice |
| PRQL | program-required quantitation limit |
| PVC | polyvinyl chloride |

| | |
|---|---|
| QA | quality assurance |
| QAPP | Quality Assurance Program Plan |
| QC | quality control |
| RA | radioassay |
| RCRA | Resource Conservation and Recovery Act |
| RFETS | Rocky Flats Environmental Technology Site |
| RMRS | Rocky Mountain Remediation Services |
| RTR | real-time radiography |
| SARP | Safety Analysis Report for the TRUPACT-II Shipping Package |
| SAS | SWEPP Assay System |
| SVOC | semi-volatile organic compound |
| SWEPP | Stored Waste Examination Pilot Plant |
| TCLP | Toxicity Characteristic Leaching Procedure |
| TRU | transuranic |
| TRUPACT-II | Transuranic Package Transporter - II |
| VOC | volatile organic compound |
| WAC | Waste Acceptance Criteria |
| WIPP | Waste Isolation Pilot Plant |
| WSRIC | Waste Stream and Residue Identification and Characterization |
| γ-SPEC | gamma-ray spectroscopy |

# Idaho National Engineering and Environmental Laboratory Nonhazardous Waste Determination for IDC 300 Waste (Graphite Molds) Waste Stream

## 1. INTRODUCTION

This document describes the process and information used by the Idaho National Engineering and Environmental Laboratory (INEEL) to determine that waste assigned Item Description Code (IDC) 300 (graphite molds) do not contain hazardous waste regulated by the Idaho Hazardous Waste Management Act (IHWMA), Resource Conservation and Recovery Act (RCRA) or the New Mexico Hazardous Waste Act (HWA). For purposes of this document, reference to RCRA imply either federal or state laws and regulations, or both. To be considered a hazardous waste under these statutes, a waste must either be specifically listed as hazardous or exhibit a hazardous characteristic of ignitability, corrosivity, reactivity, or toxicity.

Graphite waste, stored at the INEEL, has been identified by the INEEL as the best candidate for nonmixed waste shipments to the Waste Isolation Pilot Plant (WIPP). The defense generated waste [Ref. 1] consisting of graphite molds, bearing IDC 300, was selected because of its relatively large population, extensive acceptable knowledge (AK), and an AK determination as nonmixed. The majority of the graphite molds (~1100 drums) were generated in the Foundry and Casting Operations in Building 707 at the Rocky Flats Environmental Technology Site (RFETS) between December 1972 and June 1988. Of these 1100 drums, 432 currently meet WIPP WAC and TRAMPAC requirements. This hazardous waste determination covers these drums and any additional drums containing IDC 300 waste found to meet WIPP WAC and TRAMPAC requirements. Available documentation and chemical characterization performed to date indicates that the processes that generated graphite molds as waste changed very little over the production years. Thus, the INEEL has concluded that the IDC 300 graphite mold waste is homogeneous with respect to chemical properties.

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. Initially, the INEEL applied AK data to support a nonmixed waste determination for this waste stream. Subsequently, INEEL performed approved sampling and analysis and conducted additional examinations to verify this determination. The following sections describe in detail the actions taken and the conclusions reached by the INEEL with respect to this nonmixed determination. Based upon the information discussed below, the INEEL has demonstrated that drums containing IDC 300 (graphite molds) are a nonmixed waste, in accordance with the requirements of IHWMA (40 CFR 261). This determination is based on:

- acceptable knowledge; including required verification activities;
- original waste generator verification that the waste is not a listed waste;
- sampling and analysis of a representative sample of graphite molds to confirm compliance with toxic characteristic regulatory thresholds; and
- visual examination of a representative sample of IDC 300 graphite to verify the absence of potentially listed/characteristic waste materials;

- sampling and analysis of headspace gas to meet disposal requirements.

The results of this characterization effort conclusively demonstrate that the drums containing IDC 300 graphite are nonmixed, i.e., nonhazardous. To further verify the nonmixed determination and ensure compliance with WIPP WAC and TRAMPAC, Waste Acceptance Criteria, the INEEL will characterize the waste to be shipped in accordance with the requirements of the QAPP [Ref. 6].

## 2. WASTE GENERATION PROCESS DESCRIPTION

During plutonium weapons production at RFETS, graphite molds were used in foundry operations to cast plutonium parts, shapes, and ingots. In RFETS Building 444, molds were cut from solid blocks, logs, or slabs of graphite. The new molds were transferred to Building 707. 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.

Once a mold was no longer usable, it was subjected to a discard process, shown in Figure 1 along with the respective graphite IDCs generated by this process. Graphite molds to be reused were mechanically cleaned to remove the plutonium metal from the surface of the mold, generating IDC 310 (graphite scarfings & fines). Both graphite molds (IDC 300) and graphite cores (IDC 301) were nondestructively assayed. Scarfing operations removed plutonium metal from those components contaminated above the economic-discard limit (EDL), generating IDCs 303 (scarfed graphite chunks), 310 (graphite scarfings & fines), and 312 (coarse graphite). Graphite molds and cores (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.

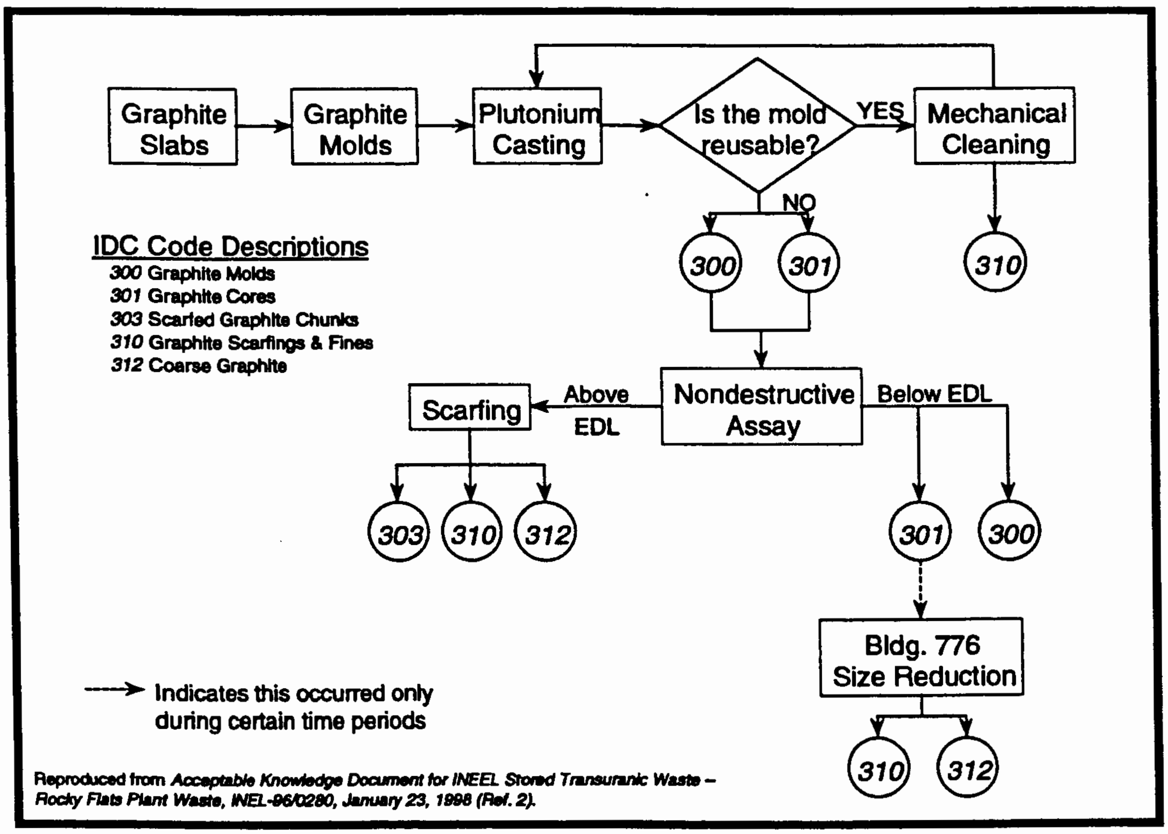


**Figure 1.** Graphite waste generation flow diagram.

At no point were solvents 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 furnace gloveboxes and used to clean the gloveboxes, but not the castings or the molds [Ref. 4]. 1,1,1-Trichloroethane was also used to clean gloveboxes [Ref. 10]. After glovebox cleaning was completed, all liquids were removed and discarded separately [Ref. 5]. Graphite molds are relatively soft, and contact with any liquids would cause the molds to crumble, rendering them useless [Ref. 5]. Therefore, the graphite molds were removed from the glovebox before cleaning took place [Ref. 5]. No known contact with "F" coded or other listed waste sources has been documented.

# 3. CHARACTERIZATION PROCESS

## 3.1 Hazardous Waste Determination

A waste must exhibit a hazardous characteristic or be listed in the regulations as hazardous to be regulated under state and federal law. Moreover, the mere presence of particular constituents in a waste does not mean a waste is 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 determination 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 Acceptable Knowledge (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, INEEL initially used the AK characterization process to support a nonmixed waste determination for the IDC 300 graphite mold waste stream. INEEL subsequently performed sampling and analysis in accordance with SW 846 criteria and conducted additional examinations to verify this determination.

## 3.2 Acceptable Knowledge

AK documents the waste-generator information on listed waste, toxicity, corrosivity, ignitability, and reactivity. AK compilation and the conclusions reached following review of AK for each of these areas are discussed in this section and in Section 4.

### 3.2.1 Description of Acceptable Knowledge Compilation

Beginning in spring 1996, AK documentation was compiled for the RFETS transuranic (TRU) waste stored at the INEEL. This documentation was developed in accordance with the AK requirements in WIPP Quality Assurance Program Plan (QAPP) [Ref. 6]. The initial compilation consisted of more than 300 references, and was based on guidance from Benchmark Environmental Corp., the EPA, and the WIPP Waste Analysis Plan [Ref. 7]. The INEEL AK document includes summary chapters for each waste form, reference excerpts for each waste form, and complete copies of all references. The AK document was revised [Ref. 2] to provide additional information on radionuclides and isotopic ratios.

### 3.2.2 Key Acceptable Knowledge Documentation for Graphite

Approximately 60 reference sources were used to develop AK bases and characterize the graphite waste form. The primary sources of AK information were the RFETS *Waste Stream and Residue Identification and Characterization* (WSRIC) books [Ref. 4 and 8] and the *Backlog Waste Reassessment* (BWR) Baseline Book [Ref. 9].

Graphite waste was first evaluated in 1990 by RFETS to address Resource Conservation and Recovery Act (RCRA) characterization requirements under the WSRIC program. The WSRIC books describe waste generated from every process at RFETS, by building, and include process descriptions, process flow diagrams, chemical inputs, waste outputs, and waste characterization and rationale. WSRIC information was developed primarily through process knowledge, including extensive interviews with cognizant personnel. Although production operations at RFETS were halted in 1989, the WSRIC program initially described plutonium production processes in anticipation that these activities would resume.

The BWR program, initiated in 1994 by RFETS, reassessed waste inventories generated at RFETS before the implementation of the WSRIC program. Waste containers were sorted into sub-populations with similar RCRA characterization. Information from WSRIC books and several other sources, including sampling and analysis data, were reviewed, and the program documented the characterization of low-level, TRU, and residue wastes in the BWR Baseline Book.

## 3.3 Discussion of Acceptable Knowledge Determination

The following section discusses in detail certain key AK documentation for IDC 300 graphite waste which relates to the nonmixed waste determination.

### 3.3.1 Graphite

For waste to be listed as hazardous it must meet waste criteria set forth in 40 CFR 261.31 - 261.33. The graphite itself is not a hazardous waste. Moreover, the AK documentation demonstrates that no listed hazardous waste came in direct contact with the graphite during or after the production process. 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 building where the graphite molds were produced [Ref. 4, 8, and 10]. The AK documentation [Ref. 11] states that although incidental solvent contamination of the molds could have been possible (i.e., residual carbon tetrachloride and/or 1,1,1,-Trichloroethane contamination within the glovebox), such contamination does not meet the regulatory definition of any listed hazardous waste [Ref. 25]. Additionally, there is no evidence that any solvents were used on the graphite molds themselves or in the production of the molds as waste [Ref. 10 and 11]. 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 molds never contacted an "F" listed waste source. These conclusions were recently substantiated by RFETS in a letter from the Rocky Mountain Remediation Services (RMRS), dated July 1, 1998 [Ref. 3]. This letter contains the following statement:

To the best of our knowledge, the IDC 300 graphite waste did not come into direct contact with listed hazardous waste, and therefore, should not be characterized as a listed hazardous waste under IHWMA or RCRA.

RMRS is the custodian of the historical waste records for the RFETS, under its contract with Kaiser Hill Company. RMRS was not at RFETS at the time the graphite waste was generated. However, as the record custodians, RMRS is in the best position to examine the available information and determine the accuracy of the earlier waste determinations for this same material. The INEEL is using this as AK confirmation from the generating facility that the IDC 300 waste is not a listed hazardous waste. The above documentation is further supported by relevant data generated by the headspace gas analysis discussed in Section 3.4.1.

To exhibit the characteristic of toxicity, waste levels of certain metals or organics must meet or exceed regulatory thresholds defined in 40 CFR 261, based on the TCLP test results. As mentioned, AK documentation states carbon tetrachloride, a toxic-characteristic compound, was used for cleaning plutonium parts in Building 707 production area, and used in cleaning gloveboxes and furnaces in the casting area, potentially resulting in incidental contact [Ref. 3].

TCLP extract analyses conducted at RFETS on graphite chunks (IDC 312) and graphite fines (IDC 310) indicate graphite does not exhibit toxicity for metal or organic compounds [Ref. 12]. Accordingly, RFETS analysis of the graphite chunks and fines provide supporting evidence that the INEEL-stored graphite molds do not exhibit a toxicity characteristic. Notwithstanding AK information that determined graphite molds are nonmixed waste, the INEEL developed a sampling and analysis plan [Ref. 13] to determine definitively if the graphite molds 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 were randomly selected to undergo sampling and TCLP extraction and analysis from the 50 drums containing IDC 300 waste chosen for visual inspection [Ref. 13] and initial shipment to WIPP. The selection of drums for sampling and the TCLP results are discussed in Section 3.4.3. TCLP results are given in Appendix B. The INEEL followed the guidance provided by the EPA in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods* (EPA) 1986) (SW-846), Chapter Nine, "Sampling Plan" to collect a representative sample of the waste stream for the characterization effort. SW-846 Chapter Nine defines a representative sample "as exhibiting average properties of the whole waste" (EPA 1996, page Nine-5). Based on extensive acceptable knowledge (AK) and discussions with RFETS, the waste generator, graphite molds generated from operations in the Foundry and Casting Building (Building 707) and the process changed very little over the period of waste generation (1972-1988). As a result the INEEL has concluded that the IDC 300 graphite mold waste stream is homogeneous with respect to chemical properties. As a result the INEEL performed TCLP and visual inspections on a representative sample of drums.

Additionally, based on process knowledge (i.e., the chemicals used), pesticides or herbicides were not present in the areas or processes generating graphite waste [Ref. 2]. Therefore, the graphite molds waste stream does not exhibit the characteristic of toxicity due to pesticides or herbicides. Finally, based on knowledge of the waste stream, the waste does not exhibit any of the other hazardous waste characteristics, i.e., corrosivity, reactivity, or ignitability.

### 3.3.2 Extraneous Material

As discussed in Section 2, process description from RFETS indicated that the IDC 300 waste contained no hazardous material. However, between 1983 and 1999, the INEEL visually inspected 35 drums of IDC 300 waste, and two of those drums were found to contain material suspected to be blotter paper [Ref 14]. The INEEL conservatively assumed that the suspected blotter paper could have been used as towels to wipe up carbon tetrachloride. Accordingly, the INEEL tentatively assigned hazardous waste number F001 for carbon tetrachloride [Ref. 5] to those two drums. When the 50 drums of IDC 300 waste selected for the initial nonmixed waste shipment were visually inspected for prohibited items in 1998, particular vigilance was given to blotter paper, since blotter paper cannot be consistently identified through other means (i.e., real-time radiography). The results of this effort are discussed in greater detail in Section 3.4.2. No blotter paper was found in the 50 drums. In addition the two drums suspected of containing blotter paper were retrieved from inventory and visually reinspected. The material suspected as blotter paper was actually determined to be cardboard liner [Ref. 15 and 16]. Therefore, visual inspection resolved any uncertainty regarding the AK information. In fact, visual inspection of approximately seven percent of the INEEL's total IDC 300 waste inventory supports the AK determination that the waste contains no hazardous waste.

## 3.4 Relevant Data and Supporting Information

Physical and chemical characterization of the graphite wastes was undertaken to (a) verify the AK documented for this waste form [Ref. 2], and (b) support the determination of the nonmixed status of the waste. Included in this section are those intrusive and nonintrusive examinations and analyses conducted to ensure that the requirements for the disposal of waste in WIPP were met [Ref. 17 and 6], and confirm the AK hazardous waste determination. These examinations and analyses include nonintrusive 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 SW 846 guidelines, (Section 3.4.3).

### 3.4.1 Headspace Gas Data

The applicable WIPP QAPP requirements for headspace gas analysis and the results obtained are as follows:

***3.4.1.1 Program Requirements.*** Section 7 of the WIPP QAPP [Ref. 6] contains an extensive discussion on Headspace Gas Sampling, based on EPA guidelines. The document 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.

Headspace gas sampling and analysis must be performed on each drum destined for shipment to WIPP. The headspace analysis results for spent solvents are then combined 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), sites must reevaluate their acceptable knowledge information and determine the source of the constituent [QAPP Section 4.2.2.2].

***3.4.1.2 Analytical Results.*** Results of the headspace gas analyses for 201 drums are summarized in Appendix C, Table 4. Based on the results given in Appendix C, Table 4, F-listed volatile organic solvents did not exceed the PRQL at the 90%-confidence level, per the QAPP [Ref. 6, Section 5]. 1,1,1-Trichloroethane was present at 6.7 ppm and the analyte closest to the PRQL. Although below the PRQL, the most likely source of 1,1,1-trichloroethane was evaluated. Reference 11 states that "polyethylene liner lids were glued in place with a Raycohesive" (tradename). It goes on to state that trichloroethane is a constituent of this adhesive. This taken in conjunction with potential incidental contamination due to glovebox cleaning [Ref. 10] is the most likely source of 1,1,1-trichloroethane. Neither source meets any listed waste regulatory definition. In addition, the occurrences of tentatively identified compounds (TICs) were evaluated. TICs listed in Appendix VIII 40 CFR Part 261and Appendix IX 40 CFR Part 264 were detected but at a frequency much less than the 25% frequency requirement and no hazardous waste codes were assigned based on TICs.

### 3.4.2 Physical Characterization

For the purposes of this document, "physical" characterization includes both intrusive and nonintrusive examinations or inspections, conducted under the requirements of References 18 and 6. Moreover, additional specific examination and analysis tasks were incorporated to verify the nonmixed waste determination made by AK (discussed in Section 3.3).

***3.4.2.1 Program Requirements for Examination of Contents.*** Radiography is required by the QAPP [Ref. 6] for verification of waste container contents and packing configuration, determination of matrix parameter categories, establishment of the layers of confinement, and detection of prohibited materials such as pressurized containers or free liquids. The QAPP further 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 the INEEL, and their use has been authorized by DOE-CAO [Ref. 19].

Real-time radiography (RTR), also known as real-time radioscopy, is the approved process at the INEEL for meeting nonintrusive 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 (i.e., in "real" time). 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 the INEEL, the appropriate statistical measures are in place to select X-rayed containers for direct visual examination to verify the inspection results. Selected drums are sent to the ANL-W Waste Characterization Area to be opened. 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, and discrepancies are identified and dispositioned by the INEEL AK expert.

***3.4.2.2 Real-Time Radiography Inspection Results.*** During RTR review, no regulated hazardous waste or WIPP-prohibited items have been observed in the WIPP WAC and TRAMPAC acceptable drums. However, several unregulated extraneous items (i.e., items not identified as hazardous or WIPP restricted) were observed, such as a tape roll and a brush. These and other unregulated items were documented during the intrusive examination. (See discussion details in Section 3.4.2.3) 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.

***3.4.2.3 Intrusive Examination and Verification.*** Fifty drums selected for the initial shipment of nonmixed waste, containing IDC 300 graphite molds were subjected to visual inspection. During visual inspection of the drum, the contents were removed, inspected, documented, and returned to a new drum. Although not required by existing regulations or requirements, direct visual confirmation of the contents of each drum was conducted. This decision was based on the sensitivity surrounding the shipment of nonmixed wastes, and on the potential presence of an extraneous material (blotter paper) potentially contaminated with a listed constituent [Ref. 14].

During the visual inspection, the retrieval of graphite pieces for TCLP analysis was also performed. The approach used for sampling, along with the tests and the results, are reported in Section 3.4.3 below.

Visual inspection of the contents of the 50 drums of IDC 300 waste yielded the following results:

- Three drums contained extra plastic material not part of the packaging (referred to as "bag cuts," these are the unused tailpieces that remain following a bag-out operation).
- One drum contained a partial roll of yellow tape.
- Four drums each contained a pair of latex rubber gloves (surgical gloves), identified in AK [Ref. 2] as miscellaneous items potentially present.

- Two drums contained paper label.
- One drum contained a metal screw-on cap, 2 inches in diameter.
- One drum contained a metal marking pen.
- One drum contained a wooden-handled brush.

None of the extraneous material given above is regulated as hazardous waste or restricted by WIPP disposal criteria [Ref. 17].

The typical drum of graphite waste consisted of intact graphite molds and broken pieces of graphite molds greater than 60 mm in at least one dimension, with a small quantity (<2 liters) of graphite particles <60 mm in diameter. Working from the outer layer, the typical 55-gal drum contains a 0.090-in. thick rigid liner, one or two drum bags, and a cardboard liner. (One drum contained three drum bags; another drum lacked a cardboard liner.) The drum bags were constructed of either polyethylene or polyvinyl chloride (PVC), and were closed by gathering the end of the bag and wrapping with yellow tape. Yellow tape was also used throughout most drums to hold the bags and cardboard liner in place while graphite was being placed into the drums. All drums contain some additional plastic packaging material described as a "drum-bag stub." It is typically bagging material with an elastic band, left over when bagging through a transfer port.

3.4.2.4
~~3.2.2.4~~ ***Conclusions from Intrusive Examination.*** As previously described, the INEEL had tentatively decided that, absent further analysis, the hazardous waste number F001 for carbon tetrachloride would be assigned to any drum containing blotter paper or towels. No blotter paper, towels, or any other suspect material were found in any of the 50 drums. In fact, the only two drums in the INEEL's IDC 300 inventory that had been suspected of containing blotter paper were re-inspected and determined to not contain blotter paper [Ref. 15 and 16]. In addition, from 1983 – 1999, 35 additional IDC 300 graphite drums have been visual examined. None of the visually examined drums contained RCRA regulated material.

None of the extraneous material identified during visual examination is regulated as hazardous waste or restricted by WIPP disposal criteria [Ref. 17]. Additional visual examination and repackaging of IDC 300 graphite mold drums to verify absence of prohibited items is not required.

### 3.4.3 Sampling and Analysis

To ensure no toxic characteristic levels of volatile, semi-volatile, and metals compounds are present, five drums were randomly chosen for TCLP extraction and analysis sampling from the 50 drums identified for visual inspection. Samples from the five randomly selected drums were submitted to the INEEL Analytical Chemistry Laboratory (ACL) for TCLP extraction and analysis. Given the process that generated the waste, these results are representative of the entire waste stream. This was recently substantiated in a letter from CAO dated June 23, 1999 [Ref. 26] who approved the waste stream with no further sampling and TCLP analysis.

***3.4.3.1 Sampling Plan.*** A sampling plan [Ref. 13] was developed and approved to select the graphite samples to be used in conjunction with TCLP. (The sampling plan is reproduced in Appendix A.) Selection of the drums from which the graphite samples were to be taken is summarized below. The drums were numbered [Ref. 20] according to their shipment order to Argonne National Laboratory - West (ANL-W) for inspection and sampling. 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 number were the drums chosen for sampling, TCLP extraction, and analysis. Five of the chosen drums were to undergo sampling and totals analysis and the remaining five drums sampling, TCLP extraction, and analysis. Drum selection details can be found in Engineering Design File (EDF) 1028 [Ref. 13]. Three randomly selected graphite pieces were then taken from each of the five drums, broken into smaller pieces, and composites 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. The process used to randomly select the graphite pieces was involved; the details are discussed in EDF 1028 [Ref. 13].

In addition to the sampling process, EDF 1028 [Ref. 13] defines the target analyte list, the analyses required, and QA/QC parameters. Samples and associated QC samples were analyzed per SW-846 [Ref. 18]. 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 B for discussion.) The results of organic sample analyses were independently validated by the Environmental Data Services, Inc. (Concord, NH) [Ref. 21]; the results of metal analyses were validated by the INEEL Sample Management Office [Ref. 22]. A summary of qualifying flags for both validation reviews is provided in Appendix B.

As described in the sampling plan [Ref. 13], any deviations or changes to planned analytical methods require SPO and ACL approval, a Program Change Notice (PCM) or a revision to the EDF. The sample identification format in this EDF was replaced by the format described in PCN SPO-98-06 [Ref. 23].

***3.4.3.2 Summary of Toxicity Characteristic Leaching Procedure Data.*** Based on the results given in Appendix B, no toxicity characteristic compounds or metals were found to be greater than the regulatory threshold limits. In fact, concentrations were orders of magnitude below regulatory threshold limits. The IDC 300 graphite mold waste stream does not exhibit a hazardous characteristic of toxicity. No further toxic characteristic analysis of this waste stream will be performed.

# 4. DETAILED RCRA ANALYSIS

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, ignitability, corrosivity, and reactivity. Moreover, as previously stated, the mere presence of hazardous constituents, concentrations, or qualities 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. These 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 meets a listing description. Based on AK, it is known that no listings apply to drums containing IDC 300 waste (graphite molds). The F listing is not applicable because no solvents fitting a listing description 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 program required quantitative limit.

## 4.2 Characteristic Waste

### 4.2.1 Toxicity

Based on AK and analysis, including the Toxicity Characteristic Leaching Procedure (TLCP) analysis, it has been determined that the drums, containing IDC 300 waste (graphite molds), 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.

As previously discussed in Section 3.3.1, documentation from TCLP analysis performed by RFETS on similar graphite waste streams indicate graphite does not exhibit toxicity for metal or organic compounds. Nevertheless as discussed in Section 3.4.3, the INEEL conducted TCLP on a representative sample of IDC-300 waste (graphite molds) to determine conclusively that the waste does not exhibit the characteristics of toxicity, to ensure representative samples were taken, and to ensure TCLP QA and QC protocols were met. As indicated in Appendix B, the results conclusively demonstrate that no volatile organic compound or metals exceed the regulatory threshold limits. In fact, concentrations were orders of magnitude below regulatory threshold limits. As a result, the IDC 300 graphite mold waste stream does not exhibit a hazardous characteristic of toxicity.

### 4.2.2 Corrosivity

Based on testing and knowledge of the waste stream, it has been determined that IDC-300 waste (graphite molds) does not meet the definition of a characteristic waste due to corrosivity. To be a regulated corrosive waste, 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 real-time radiography and confirmed by visual examination of all selected drums, this waste is neither aqueous nor liquid [Ref. 2]. Therefore the waste cannot be corrosive per RCRA definition. This conclusion is substantiated in a letter from the EPA Policy Compendium [Ref. 24]. 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 testing and knowledge of the waste stream, it has been determined that IDC-300 waste (graphite molds) 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 than 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 [Ref 2]. 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 (graphite molds) 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 mixture 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 [Ref 2].

# 5. CONCLUSION

INEEL has established that the IDC-300 (graphite molds) waste stream is not hazardous waste. The waste stream does not exhibit any hazardous characteristic or meet any hazardous waste listing. Although the determination of whether a waste is hazardous for regulatory purposes is allowed to be made on the basis of acceptable knowledge alone, the INEEL determination for the graphite mold waste uses additional tests and analysis, including headspace gas analysis, real-time radiography, visual examinations, and importantly, TCLP analysis, to confirm that the information contained in the AK documentation is correct.

# 6. REFERENCES

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* (Reference 5, are letters which were written after the indicated issue date of the AK document was ready for release in January 1998. Immediately prior to its release, additional research was determined to be required. Release of the document was postponed until the middle of March 1998. The cover page should have been changed, but was not.)

# Appendix A

# Sampling Plan

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# Appendix A

# 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:*

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**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-hazardous 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 non-hazardous. 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-hazardous 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, volatile organic compound (VOC) and semi-volatile organic compound (SVOC) analysis, 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 non-hazardous 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[1] 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

1 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.

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.

**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[2] 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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2 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 [EU2]of [EU3]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. [EU4]For total VOCs/SVOCs sampling, the pieces need to be approximately 5 grams each. [PC5]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[EU6][EU7][EU8][EU9].

## 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[EU10][EU11]. 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[3] 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

[3] 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.

**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 |

**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.

**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 |

**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 B

## TCLP Data Summary

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# Appendix B

# TCLP Data Summary

## IDC 300 Waste (Graphite Molds)

Five drums of IDC 300 waste (graphite molds) were randomly selected from a population of 50 drums (These 50 drums are representative of the entire IDC 300 waste stream) 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, we followed the guidance given in the QAPP for the analysis – 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:

$$UCL_{90} = \overline{x} + \frac{t_{.90,n-1}\, s}{\sqrt{n}}$$

Where s is the sample standard deviation, $\overline{x}$ 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 B

## 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 B

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

| Analyte | No. of Samples | No. of Samples above MDL | MDL (mg/L) | Mean (mg/L) | SD (mg/L) | UCL (mg/L) | Reg. Lvl (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 B

**Table B-2: TCLP Semi-VOC Summary Data**

| Analyte | No. of Samples | No. of Samples above MDL | MDL (mg/L) | Mean (mg/L) | SD (mg/L) | UCL90 (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 B

**Table B-3: TCLP Metals 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) |
|---|---|---|---|---|---|---|---|
| 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 $\overline{X} = ½(4*MDL + 0.0392)$.

* 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 C

## Headspace Gas Analysis Data Summary

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# Appendix C

# Headspace Gas Analysis Data Summary

### IDC 300 Waste (Graphite Molds)

A total of 201 drums have been subjected to headspace gas sampling. The headspace gas analysis results have been statistically combined. The upper confidence limit was calculated as follows:

$$UCL_{90} = \bar{x} + \frac{t_{.90,n-1}\, s}{\sqrt{n}}$$

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. This conservative approach is consistent with the statistical reduction methods used in the QAPP.

The statistical test described above is based on the assumption that the measured concentrations of each contaminant are normally distributed. As required by the QAPP, this assumption must be verified. The data were analyzed and transformed by natural logarithm, resulting in a more normal distribution as measured by the Shapiro-Wilk statistical test.

# Appendix C

**Table 4.** Headspace Gas Summary Data

| Analyte | Number of samples | Number of samples above MDL[a] | Mean[b] | Standard deviation[b] | Upper 90% Confidence Limit[c] (ppmv) | Program Required Quantitation Limit (ppmv) |
|---|---|---|---|---|---|---|
| 1,1,1-Trichloroethane | 201 | 193 | 1.660 | 2.644 | 6.716 | 10 |
| 1,1,2-Trichloro-1,2,2-Trifluoroethane | 201 | 33 | -3.067 | 1.029 | 0.059 | 10 |
| Acetone | 201 | 174 | 1.377 | 1.313 | 4.506 | 100 |
| Benzene | 201 | 4 | -2.762 | 1.126 | 0.159 | 10 |
| Butanol | 201 | 9 | -1.043 | 1.179 | 0.610 | 100 |
| Carbon tetrachloride | 201 | 86 | -2.322 | 1.152 | 0.115 | 10 |
| Chlorobenzene | 201 | 0 | 0.095[d] | [d] | [d] | 10 |
| Ethyl benzene | 201 | 12 | -2.949 | 1.236 | 0.085 | 10 |
| Ethyl ether | 201 | 0 | 0.144[d] | [d] | [d] | 100 |
| M&P-Xylene | 201 | 15 | -2.708 | 1.224 | 0.102 | 10 |
| Methanol | 201 | 34 | 1.901 | 0.641 | 7.729 | 100 |
| Methyl ethyl ketone | 201 | 19 | -1.227 | 1.106 | 0.411 | 100 |
| Methylene chloride | 201 | 71 | -2.123 | 1.714 | 0.156 | 10 |
| O-Xylene | 201 | 14 | -2.951 | 1.217 | 0.081 | 10 |
| Tetrachloroethylene | 201 | 0 | 0.090[d] | [d] | [d] | 10 |
| Toluene | 201 | 38 | -2.404 | 1.129 | 0.115 | 10 |
| Trichloroethylene | 201 | 50 | -2.387 | 1.431 | 0.120 | 10 |

a. When a measurement is reported as below detection, one-half the analysis method detection limit (MDL) is used per guidance in the QAPP. Note that the MDL for a given analyte may vary from sample aliquot to sample aliquot.

b. All analytes with at least some measurements above the MDL were found to be closer to lognormally distributed than normally distributed. Therefore the data was transformed via natural logarithm before analysis and the means and standard deviations reported are in the transformed state.

c. To allow comparison to the Program Required Quantitation Limit (PRQL), the upper confidence limits have been back transformed and are in the original units (ppmv).

d. The mean presented is simply the average of the non-transformed method detection limits (after dividing by 2) since all measurements are below detection. The standard deviation and upper 90% confidence limit can not be calculated because all measurements are below detection. A sample size of 201 is substantial and given that all 201 measurements were below detection indicates that the probability that the mean exceeds the PRQL is extremely small.