### ON THE ROAD TO WIPP: IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY EXPERIENCES

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### ABSTRACT

Initiation of Idaho National Engineering and Environmental Laboratory (INEEL) transuranic (TRU) waste shipments to the Waste Isolation Pilot Plant (WIPP) in April 1999 represented the successful culmination of almost twenty years of effort to achieve final disposal of waste generated by defense programs conducted by the U. S. Department of Energy (DOE). More importantly, initiation of waste shipments completed an historic regulatory milestone established with the State of Idaho to begin removing stored TRU waste from the State. This allows the continued receipt of DOE-owned research reactor fuel at INEEL for storage and eventual preparation for disposal at Yucca Mountain.

As of September 30, 1999, four shipments of INEEL-stored TRU waste have been shipped to WIPP for disposal. All waste shipped to date has been required to be nonmixed waste. Requirements were only recently (October 1999) finalized by the State of New Mexico Environment Department (NMED) for characterizing TRU wastes also contaminated with hazardous chemical constituents. A program was initiated in 1998 to identify a population of stored waste that could be demonstrated as not containing hazardous chemical constituents. This was especially challenging since the majority of INEEL-stored TRU waste was generated by other DOE defense facilities within the time period of 1970-1989. Development of acceptable knowledge and sampling of the waste was performed to allow determination that no regulated hazardous constituents existed in the waste stream selected for shipment to WIPP.

Prior to shipping waste to WIPP, each generator/storage site must demonstrate successful implementation and compliance with disposal and transportation criteria. Data of known quality must be generated, validated, reported and approved by WIPP on each waste container prior to shipment. Processes used to generate these data include the use of: (a) acceptable knowledge documentation, (b) nondestructive examination and assay performed at the Radioactive Waste Management Complex (RWMC) Stored Waste Examination Pilot Plant (SWEPP), (c) headspace gas sampling and analysis, and (d) intrusive examination and sampling of selected waste containers.

Information on each waste container is accumulated at each certification process step. It is then reduced, analyzed, reconciled, and compared against appropriate WIPP operational and safety, environmental, and transportation requirements. This paper provides a summary description of the activities performed to certify the INEEL's first TRU waste for shipment to WIPP and lessons learned from making these first shipments.

Final waste analysis requirements for mixed waste to be shipped to WIPP were issued by the NMED on October 27, 1999. These requirements, delineated in the WIPP Part B Permit, result in changes to previous waste characterization processes and methods. Generator/storage sites are currently implementing new requirements to allow resumption of waste shipments to WIPP. Some changes will require requests for modification or change to the existing WIPP Part B Permit requirements to allow use of alternative technology or improved waste characterization methods not reflected in the permit. Implementation of these new requirements, for debris waste, is expected to be completed by June 2000. This will allow resumption of waste shipments to ensure 3100 m<sup>3</sup> of stored TRU waste will be shipped to WIPP by December 31, 2002, to complete the next established regulatory milestone with the State of Idaho.

# INTRODUCTION

Since 1970, the INEEL has provided interim storage capacity for TRU-contaminated wastes generated by activities supporting the United States national defense needs. Approximately 60% of the nation's current inventory of TRU-contaminated waste is stored at the INEEL. This waste has been awaiting the opening of WIPP, the designated federal repository for permanent disposal of defense-generated TRU waste. The WIPP was officially ready to receive nonmixed TRU waste in May 1998, after receiving certification from the U.S. Environmental Protection Agency (EPA). Waste shipments to WIPP were delayed until March 1999, as legal challenges to the opening of WIPP were resolved. The Los Alamos National Laboratory sent the first shipment of waste to WIPP on March 25, 1999. The first INEEL shipment of TRU waste was shipped from Idaho on April 27, 1999.

Management of TRU waste in interim storage at INEEL is governed by the requirements delineated in the October 1995, Settlement Agreement<sup>1</sup> between the State of Idaho, U.S. Navy Department, and the U.S. Department of Energy. This agreement established milestones for a variety of cleanup activities at the INEEL, including the removal of stored TRU waste from the State of Idaho, to allow continued receipt of spent nuclear fuel for management at INEEL facilities. The following excerpts from the Settlement Agreement<sup>1</sup> describe some milestones for TRU waste leaving the State of Idaho, and describe the consequences if the milestones are not achieved:

DOE shall ship all transuranic waste now located at the Idaho National Engineering Laboratory (INEL), currently estimated at 65,000 cubic meters in volume, to the Waste Isolation Pilot Plant (WIPP) or other such facility designated by DOE, by a target date of December 31, 2015, and in no event later than December 31, 2018. DOE shall meet the following interim deadlines:

- The first shipments of TRU waste from INEL to WIPP or other such facility designated by DOE shall begin by April 30, 1999
- By December 31, 2002, no fewer than 3100 cubic meters (15,000 drum-equivalents) of transuranic waste shall have been shipped out of the State of Idaho.

Consequences of missing milestones described in the Settlement Agreement include ... the sole remedy for DOE's failure to meet any deadlines or requirements ... shall be the suspension of DOE spent fuel shipments to INEL...

The INEEL completed a series of upgrades to facilities and characterization systems, used to qualify stored TRU waste for shipment to WIPP, in 1997. Production characterization operations were initiated in September 1997 to begin qualifying waste for shipment to WIPP to meet the requirements of the Settlement Agreement.

### TRU WASTE CHARACTERIZATION AND CERTIFICATION

The TRU waste destined for transportation and disposal at WIPP must be in conformance with requirements identified in the WIPP Waste Acceptance Criteria (WAC). Each site participating in the characterization, certification, and transportation of TRU waste to WIPP must receive certification authority. Shipments of INEEL-stored nonmixed TRU waste to WIPP were based on certification authority received from WIPP and the EPA for debris waste. Certification authority was granted in the Spring of 1998. Certification was based on successful demonstration of implementing the WIPP-WAC requirements and performing the following functions:

- Perform TRU waste certification program activities as specified in the WIPP Quality Assurance Program Description (QAPD)
- Perform TRU waste characterization program activities as specified in the WIPP Quality Assurance Program Plan for TRU Waste Characterization and certify the WIPP Waste Stream Profile Form
- Document and certify that all TRU waste payload containers prepared for shipment to WIPP meet all specified Transuranic Package Transporter-II (TRUPACT-II) Authorized Methods for Payload Control (TRAMPAC) and WIPP-WAC requirements

- Document site approval of the review, validation, and approval of characterization data generated on each waste container to verify conformance with transportation and disposal criteria

Characterization of the nonmixed waste drums selected for shipment to WIPP was performed to determine the physical, radiological, and chemical characteristics of the waste in conformance with the WIPP-WAC requirements and RCRA as discussed in the following section of this paper. Processes used to supply this characterization data included use of acceptable knowledge documentation; weighing; radiological surveys for both dose and surface contamination levels; real-time radiography (RTR) to determine physical contents and compliance with disposal and transportation requirements (absence of free liquids and compressed gases, level of packaging, etc.); measuring radiological properties by passive-active neutron (PAN) assay and gamma spectroscopy; container integrity inspection; headspace gas sampling to determine volatile organic compound and flammable gas concentrations; and intrusive examination and sampling of a selected population of drums to verify absence of regulated or prohibited items in the waste drums. Figure 1 provides a summary description of the INEEL waste certification process.

Validation and verification of data generated to determine individual waste container compliance with WIPP transportation and disposal requirements was performed by three independent levels of review and approval. Level I validation and verification occurs at the level of data generation, such as RTR or headspace gas analysis. The data was independently reviewed by a technical reviewer qualified to have performed the initial work and the data verified as being technically correct, complete, and within established control limits. Validated data was then reviewed by a technical supervisor as being technically reasonable, complete, and verified by independent technical review. Quality personnel then verify that appropriate reviews are complete, appropriate quality assurance documentation is complete, and that the program quality assurance objectives have been met. Level I data is submitted to the Site Project Office for Level II validation, reconciliation of data, and preparation of quality records documenting the attributes of the waste drums to be shipped to WIPP. Level III validation occurs at WIPP and results in approval of characterization information for each waste drum intended for shipment to WIPP. Individual drums are then assembled into payloads and approved by WIPP prior to shipment.

### NONMIXED WASTE DETERMINATION

In March 1998, a program was implemented to determine if a nonmixed waste stream could be identified and verified as not containing hazardous chemical constituents. The majority of INEEL-stored TRU-contaminated waste is suspected of being contaminated with chemical hazardous constituents. These regulated constituents, such as carbon tetrachloride and 1,1,1-trichloroethane, were commonly used in nuclear weapon fabrication and recovery processes. Absence of regulated chemical constituents would need to meet Resource Conservation and Recovery Act (RCRA) Title 40 Code of Federal Regulations (CFR) Part 261 (40 CFR 261) and New Mexico Hazardous Waste Act requirements. Delays were expected in obtaining the final RCRA Part B Permit for WIPP for performing characterization of mixed waste and would affect the ability to comply with the first Settlement Agreement milestone to ship TRU waste out of the State of Idaho by April 30, 1999.

This effort was especially challenging since the waste intended for shipment to WIPP was not generated at the INEEL and was originally generated prior to 1989 by the Rocky Flats Environmental Technology Site (RFETS). Records concerning the chemical characteristics of the waste were limited. Specific activities were implemented to obtain the necessary information to allow a determination if a population of waste existed that could be verified as not containing regulated hazardous chemical constituents.

For a waste to be considered a hazardous waste, it must either be specifically listed as hazardous in 40 CFR 261 or exhibit a hazardous characteristic of ignitability, corrosivity, reactivity, or toxicity. Previously developed acceptable knowledge (AK) documentation, which describes the process and materials used that resulted in generation of specific waste, formed the basis for identifying a population of INEEL-stored TRU waste that could be considered. Graphite mold waste was selected. It was generated by plutonium Foundry and Casting Operations conducted at the RFETS during December 1972 and June 1988. The selection was based on the extensive acceptable knowledge documentation that existed and the relatively large population of waste (approximately 1100 drums) that existed in storage that could be used for the initial waste shipments to WIPP.

Graphite molds were used to cast plutonium components for the nuclear weapons program. The molds used in foundry operations were produced from solid blocks, slabs, or logs of graphite. During casting operations, plutonium metal was melted in a tantalum crucible and poured from the crucible into the mold. After cooling, the mold was separated from the casting. Molds were reused if possible. Unusable molds were assayed, mechanically cleaned if exceeding the economic discard limit, and bagged out of the glovebox as waste. New molds were bagged into gloveboxes and assembled for use. Item Description Code (IDC)- 300 graphite mold waste that was below the economic discard limit was bagged separately and placed into drums.

Activities performed to make a nonmixed waste determination for the graphite mold waste were based on: (a) extensive reviews of existing AK; (b) obtaining original waste generator verification that the waste was not a listed waste; (c) performing sampling and chemical analysis of randomly selected graphite molds to confirm compliance with toxic characteristic regulatory thresholds; and (d) opening and performing visual examination of selected waste drums to confirm the absence of prohibited items.

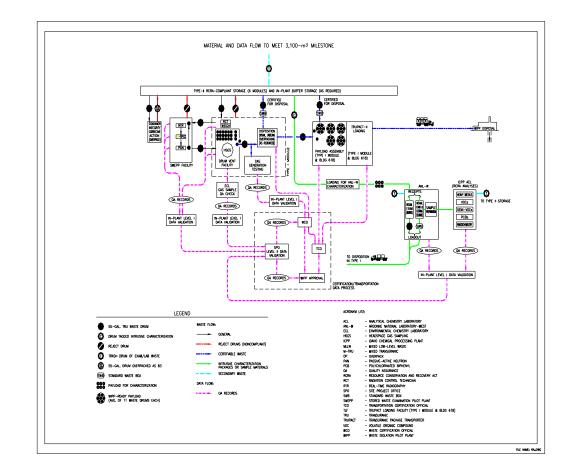
Use of AK is an EPA-authorized method<sup>2</sup> for making a hazardous waste determination in lieu of sampling and analyzing the waste. Additionally, the EPA and the U.S. Nuclear Regulatory Commission (NRC) have recognized and recommended<sup>2</sup> the use of AK for making hazardous waste determinations on radioactive waste. The AK for graphite molds produced from casting plutonium components for nuclear weapons indicated no RCRA-regulated chemicals were used in the production of new molds or in the recovery or cleanup of the molds after being used for casting plutonium parts. While regulated compounds, such as carbon tetrachloride, 1,1,1-trichloroethane, and 1,1,2-trichloro-1,2,2-trifluoroethane, were used for cleaning gloveboxes, equipment, and plutonium metal, no evidence existed that these regulated solvents came into contact with the graphite molds. Although incidental contamination of the molds was possible, there was a strong operational driver to ensure that the molds did not come into contact with liquids, as this would render them unusable.

In June 1998, efforts to obtain original waste generator verification on the regulatory status of the graphite mold waste were initiated. The RFETS, as the original waste generator, could best examine the available records and AK to verify the accuracy of earlier waste determinations and confirm that no regulated listed compounds came into contact with the molds. Extensive review of available AK was performed and additional information compiled, including interviews with individuals involved with, or familiar with, the waste generating process. The results of this effort confirmed the graphite mold waste was not a listed waste.

Sampling and analysis of randomly selected graphite mold pieces from selected drums were also performed. Expected legal challenges were behind the decision to perform these activities, in addition to the AK information that already existed, driven by the need to absolutely ensure that no regulated compounds were present above regulatory thresholds. Fifty drums of graphite mold waste were selected to support the first shipment of waste to WIPP. Each drum was nondestructively examined, assayed, and headspace gas sampled to demonstrate compliance with transportation and disposal criteria. A sampling plan was prepared in May 1998. The sampling plan defined the sampling and selection process, target analyte list, required analysis, and quality assurance/quality control parameters. Five of the 50 drums were randomly selected to provide samples of graphite mold waste to determine if the waste exhibited a toxic characteristic. Five drums were determined to be an adequate number to support a nonmixed waste determination at a 90% upper confidence level. Three graphite mold pieces, originating from the top, middle, and bottom one-third of each drum, were then randomly selected from within each of the selected drums. Once individual mold pieces were selected, the portion of the mold used to form a composite sample was also randomly chosen. These selected mold pieces were reduced in size to make a composite sample of 150 grams. Opening and sampling of the graphite mold waste drums were performed at the Argonne National Laboratory-West (ANL-W) Waste Characterization Area glovebox. Analysis of the graphite mold samples was performed at the Bechtel BWXT Idaho (BBWI) Analytical Chemistry Laboratory (ACL).

All 50 drums of waste selected as candidates for the first shipment to WIPP were sent to ANL-W for intrusive examination to verify no prohibited items existed in the waste. Although the AK and confirmation from RFETS indicated no hazardous waste items were expected, examination of waste performed between 1985 and 1996 indicated two of 27 examined drums contained blotter paper. The INEEL had conservatively assumed the blotter paper could have been used to absorb carbon tetrachloride. Each drum was opened, the molds removed and inspected, and repackaged into a new 55-gallon drum. Blotter paper or other absorbing items such as rags discovered in the drum were to be sampled and analyzed later for RCRA-regulated compounds.

Figure 1



# WASTE CHARACTERIZATION RESULTS

Graphite mold waste drums were first nondestructively examined and assayed using real-time radiography, passiveactive neutron assay, and gamma spectroscopy to determine compliance with transportation and WIPP disposal criteria. Drums containing noncompliant items such as pressurized containers, or exceeding allowable thermal wattage limits were rejected from further consideration. A population of drums passing this initial qualification process was then sampled for headspace gas. Samples were collected in Summa (tradename) canisters and sent to the Environmental Chemistry Laboratory for analysis. Analysis was performed using gas chromatography/mass spectrometry techniques to determine the concentration of volatile chemical compounds, hydrogen, and methane in the headspace gas of each drum. The analytical results were statistically combined and the upper confidence limit calculated for each analyte at the 90% confidence level. These conservative results were then compared to the program required quantitation limits to determine compliance with requirements for assigning hazardous waste numbers to a waste stream. Table 1 summarizes the results for the initial 50 drums of graphite mold waste evaluated for shipment to WIPP<sup>2</sup>. No drums exceeded any of the concentration limits for regulated F-listed compounds.

Analyte	# Samples	# Samples	Mean <sup>1</sup>	SD <sup>1</sup>	UCL <sub>90</sub>	PRQL
-	1	above MDL	(ppmv)	(ppmv)	(ppmv)	(ppmv)
1,1,1-Trichloroethane	50	48	0.510 <sup>L</sup>	2.683 <sup>L</sup>	2.755 <sup>Ú</sup>	10
1,1,2-Trichloro-1,2,2-Trifluoroethane	50	10	-3.521 <sup>L</sup>	0.841 <sup>L</sup>	$0.043^{\circ}$	10
Acetone	50	44	1.153 <sup>L</sup>	1.383 <sup>L</sup>	4.155 <sup>0</sup>	100
Benzene	50	0	0.065	0.100	0.601 <sup>m</sup>	10
Butanol	50	1	0.378	0.577	6.115 <sup>m</sup>	100
Carbon tetrachloride	50	33	-2.306 <sup>L</sup>	1.240 <sup>L</sup>	$0.132^{\circ}$	10
Chlorobenzene	50	0	0.057	0.089	0.535 <sup>m</sup>	10
Ethyl benzene	50	3	-3.473 <sup>L</sup>	1.079 <sup>L</sup>	$0.100^{\circ}$	10
Ethyl ether	50	0	0.087	0.135	$0.820^{m}$	100
M&p-Xylene	50	4	-3.215 <sup>L</sup>	$1.077^{L}$	$0.097^{\circ}$	10
Methanol	50	4	5.65	3.670	8.655	100
Methyl ethyl ketone	50	3	-1.768 <sup>L</sup>	0.964 <sup>L</sup>	$0.487^{\cup}$	100
Methyl isobutyl ketone	50	0	0.193	0.300	1.803 <sup>m</sup>	100
Methylene chloride	50	18	-2.430 <sup>L</sup>	1.752 <sup>L</sup>	0.153 <sup>U</sup>	10
o-Xylene	50	3	-3.476 <sup>L</sup>	$1.070^{L}$	0.099 <sup>U</sup>	10
Tetrachloroethylene	50	0	0.054	0.084	0.508 <sup>m</sup>	10
Toluene	50	12	-2.920 <sup>L</sup>	0.939 <sup>L</sup>	$0.078^{\circ}$	10
Trichloroethylene	50	9	-2.991 <sup>L</sup>	1.260 <sup>L</sup>	0.090 <sup>U</sup>	10

#### Table 1: Headspace Gas Summary Data

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 and mean, thereby yielding the most conservative upper confidence limit. Using benzene for example, the maximum variance occurs where 25 of the 50 measured values are assumed to be zero and the other 25 values are at the highest MDL (e.g. x1=0, x2=0,..., x24=0, x26=0.55, x26=0.55,...,x50=0.55). To be conservative, it was assumed the maximum possible mean would be 0.55. For butanol, 49 of 50 laboratory values are flagged as less than the MDL. Therefore, the most conservative upper confidence limit was calculated based on: (a) the largest MDL of 5.9 was used as the mean, and (b) the maximum variance was where the largest MDL values were used for seven of the 50 MDL values and a value of zero was used for the other 42 values.

L. Value in Ln units – that is, Ln transformation used to make data more normally distributed (as measured by Shapiro-Wilk statistic). This is per the QAPP.

U. Value in untransformed units (ppmv).

All 50 drums were then prepared for transportation to the ANL-W Waste Characterization Area for visual examination and sampling of the five randomly selected drums to obtain graphite mold samples for detailed chemical analysis using Toxicity Characteristic Leaching Procedure (TCLP) methods. Each drum was bagged into the glovebox, the contents removed, inspected, samples collected if necessary, results documented, and the contents of the drum placed into a new 55-gallon drum. Direct visual examination, although not required by existing

regulations or requirements, was used to verify the absence of any prohibited item to ensure the shipment contained only nonmixed TRU waste. In summary, a small number of extraneous materials such as a roll of tape, woodhandle brush, marking pen, and latex gloves were found in the drums. However, none of these items were a regulated hazardous waste. Additionally, the two drums of graphite mold waste suspected of containing blotter paper were retrieved from storage and shipped to ANL-W to be re-inspected. Re-examination of the waste determined that the suspect material was actually a cardboard liner used for packaging the waste. The liner was not in the glovebox and would not have been in contact with any regulated solvents.

During visual examination operations, graphite mold samples were obtained from five randomly selected drums. The samples obtained from each drum were also randomly selected. Three samples from each drum were combined to make a composite sample. The samples were then transported to the INEEL ACL for extraction using the TCLP method. Analysis of the extractant for volatile organic compound, semi-volatile organic compound, and metals was performed using EPA-approved methods. Results from these analyses were statistically evaluated using a 90% upper confidence limit. These results were then compared to the regulatory levels for toxic characteristic compounds. Tables 2, 3, and 4 provide a summary of the results<sup>2</sup>. The results show that regulated toxic characteristics were generally orders of magnitude below a threshold value and, therefore, the waste did not demonstrate a toxic characteristic.

Analyte	No. of Samples	No. of Samples above MDL	MDL (mg/L)	<b>Mean<sup>1</sup></b> (mg/L)	SD <sup>1</sup> (mg/L)	UCL <sub>90</sub> <sup>2</sup> (mg/L)	<b>Reg.</b> Level (mg/L)
1,1-Dichloroethene	5	2	0.0010	-7.289 <sup>L</sup>	0.4623 <sup>L</sup>	0.00187 <sup>U</sup>	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 <sup>L</sup>	0.1409 <sup>L</sup>	0.00399 <sup>U</sup>	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

Table 2: TCLP Volatile Organic Compound (VOC) Summary Data

<sup>1</sup> One half the MDL was used in these calculations for all laboratory values flagged "U" (that is, 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 (for example,  $x_1 = 0$ ,  $x_2 = 0$ ,  $x_3 = 0.001$ ,  $x_4 = 0.001$ ,  $x_5 = 0.001$ ). The maximum mean would be 0.001.

<sup>L</sup> Value in natural log (*ln*) units – that is, *ln* transformation used to make data more normally distributed (as measured by Shapiro-Wilk statistic). This procedure is per the QAPP.

<sup>U</sup> Value in untransformed units (mg/L).

Analyte	No. of Samples	No. of Samples above MDL	MDL (mg/L)	Mean <sup>1</sup> (mg/L)	SD <sup>1</sup> (mg/L)	UCL90 <sup>2</sup> (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 <sup>3</sup>	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

Table 3: TCLP Semi-VOC Summary Data

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

<sup>2</sup> 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 (for example,  $x_1 = 0$ ,  $x_2 = 0$ ,  $x_3 = 0.013$ ,  $x_4 = 0.013$ ,  $x_5 = 0.013$ ). 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.

Analyte	No. of Samples	No. of Samples above MDL	MD <sup>L</sup> (mg/L)	Mean <sup>1</sup> (mg/L)	SD1 (mg/L)	UCL90 <sup>2</sup> (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

Table 4: TCLP Metals Summary Data

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

- <sup>2</sup> 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 (for example,  $x_1 = 0$ ,  $x_2 = 0$ ,  $x_3 = 0.0208$ ,  $x_4 = 0.0208$ ,  $x_5 = 0.0208$ ). 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  $\overline{\mathbf{X}} = \frac{1}{2}(4*\text{MDL} + 0.0392)$ .
- \* 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.

Based on chemical analysis and AK of the graphite waste stream, it was determined that the graphite molds did not meet the definition of characteristic waste for corrosivity, reactivity, or ignitability.

The results from all characterization activities were validated and reported. A report<sup>2</sup>, summarizing the results from characterizing the graphite molds waste, was prepared and subjected to extensive technical, regulatory, and legal reviews prior to approval. The results from these reviews clearly showed the graphite mold waste was not regulated by RCRA and could be disposed of at WIPP prior to receipt of the RCRA Part B Permit at WIPP. The results of the detailed sampling and analysis of the 50 drums were then applied to the entire waste stream as being nonmixed and could be characterized and certified for disposal at WIPP.

## SHIPMENT PREPARATION

Data generated by the characterization process were submitted through three cycles of WIPP review and approval prior to shipment. Characterization data on the radiological, physical, and chemical attributes of each waste drum were manually entered and submitted to the WIPP Waste Information System (WWIS). Once the individual drum approval was received, final certification of all individual drum data was performed and submitted for approval via WWIS. Forty-two (42) of the 50 characterized drums were selected for loading into the TRUPACT-II shipping container. The TRUPACT-II is an NRC-licensed Type B container specifically designed for the transportation of

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TRU waste. Final payload compliance determinations for the shipment were made and submitted to WIPP via WWIS for review and approval. The approved payloads were then shrink-wrapped and loaded into each TRUPACT-II shipping container. Final inspections of the truck and shipping containers were performed by the Idaho State Police. At 0645 hours on April 27, 1999, the first shipment of TRU waste from the State of Idaho was on the road to WIPP for permanent disposal. As of September 30, 1999, the INEEL has sent a total of four shipments (26 cubic meters) of nonmixed graphite mold TRU waste to WIPP.

# KEY EXPERIENCES AND LESSONS LEARNED

The initial shipments of waste produced some valuable experiences and lessons that have been factored into future waste characterization, certification, and transportation activities:

- The approach to implementing requirements must be verbatim compliance as opposed to a compliance-based approach. Although there may be more efficient or cost-effective approaches to performing a specific activity, the current regulatory environment does not allow for these changes to be implemented without multiple regulatory agency review and approval. Implementing any program change without approvals can result in loss of certification authority to ship waste.
- Adequate independent oversight for all aspects of the characterization and certification process is essential for ensuring adherence to program requirements and procedures. Shipment and disposal of waste at WIPP entails implementation of thousands of requirements. A rigorous oversight program provides the necessary assurance that these requirements are being met.
- A rigorous and disciplined approach to conduct of operations must be instilled in each individual participating in the characterization, certification, and shipment of waste to WIPP. Effective training programs that not only delineate program requirements, but also teach individual responsibility and accountability for the quality of work produced, are essential to achieving an error-free or zero-defect operational capability.
- The plethora of data that must be generated, reviewed, validated, and reconciled requires a well-defined process for information management. For sites shipping hundreds of drums per year, a paper-based process with some electronic interface with WWIS might be sufficient. For sites planning on shipping thousands of drums a year to WIPP, a more robust electronic information management system should be explored. This system might not only include data gathering, but also perform automatic calculations, data quality checks, move the data between multiple approval levels, and prepare the reports necessary to obtain approval to ship. The INEEL has developed the Transuranic Reporting, Inventory, and Processing System (TRIPS) to accommodate high production throughput data management needs.

### CONCLUSION

Initiating waste shipments to WIPP marked the successful culmination of many years of effort across the DOE complex. For INEEL, this allowed the historic fulfillment of commitments made by the federal government and the State of Idaho over the past 25 years. Many challenges remain, including making timely permit modifications, but meeting these challenges is essential to ensure accomplishment of the Settlement Agreement milestone of removing 15,000 drums of waste by December 31, 2002. The recently issued WIPP Part B Permit will require modification to current characterization practices and permit modifications will be required to allow use of improved methods to characterize the waste. The valuable lessons and experiences gained by making shipments to WIPP will improve ongoing efforts to obtain certification authority for shipping waste under the new WIPP Part B requirements. The INEEL expects to re-initiate shipments to WIPP no later that June 2000.

### REFERENCES

- 1. Spent Fuel Settlement Agreement; U.S. Department of Energy, U.S. Navy Department, and State of Idaho; October 16, 1995
- Dr. R. E. Arbon, Nonmixed Waste Determination for IDC 300 Waste (Graphite Molds), INEEL/EXT-98-01137, dated February 1999