LA-UR- 96-1267

CONF-960804 --18

Title: CHARACTERIZING CEMENTED TRU WASTE FOR RCRA HAZARDOUS CONSTITUENTS

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Submitted to: SPECTRUM '96
Seattle, WA
August 1996

MASTER

Los Alamos
NATIONAL LABORATORY

Form No. 836 R5
ST 2629 10/91

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ABSTRACT

Los Alamos National Laboratory (LANL) has characterized drums of solidified transuranic (TRU) waste from four major waste streams. The data will help the State of New Mexico determine whether or not to issue a no-migration variance for the Waste Isolation Pilot Plant (WIPP) so that WIPP can receive and dispose of waste. The need to characterize TRU waste stored at LANL is driven by two additional factors: 1) the *LANL RCRA Waste Analysis Plan*[i] for EPA compliant safe storage of hazardous waste; 2) the WIPP Waste Acceptance Criteria (WAC). The LANL characterization program includes headspace gas analysis, radioassay and radiography for all drums, and solids sampling on a random selection of drums from each waste stream. Data are presented showing that the only identified non-metal RCRA hazardous component of the waste is methanol.

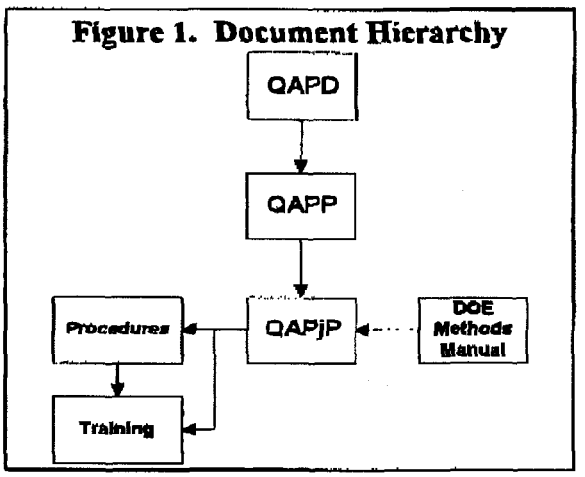
INTRODUCTION

With funding primarily from the National TRU Waste Program Office, Los Alamos National Laboratory (LANL) operated on an accelerated schedule to provide information on four of its waste streams to the DOE Carlsbad Area Office for inclusion in the WIPP no migration variance petition. Coring and sampling from 20 drums resulted in data packages that will provide additional data upon which the New Mexico Environment Department (NMED) will determine whether or not to grant a no migration variance for the opening of the WIPP repository. The variance will allow storage of mixed waste at the facility near Carlsbad, New Mexico. The characterization process included carefully identifying Resource Conservation and Recovery Act (RCRA) hazardous constituents. The program quality was guided by the DOE TRU Waste Characterization Quality Assurance Program Plan[ii] (QAPP), and also met the NMED characterization requirements for safe storage of the waste, and assured that the waste met requirements for safety and handling operations of WIPP. Department of Transportation (DOT) and Nuclear Regulatory Commission requirements for characterizing the waste prior to transporting it to WIPP are also met.

Randomly chosen drums of homogeneous, cemented TRU waste were retrieved, radiographed, radioassayed, gas sampled, core drilled, and returned to storage. Samples taken from the cores were analyzed for selected RCRA metals, volatile organic compounds (VOC's), and semi-volatile organic compounds (SVOC's). The analyses from 20 drums are presented in this report as Table 2.

QUALITY ASSURANCE PROGRAM AND DOCUMENTS

Quality of the project was specified by the DOE Carlsbad Area Office (CAO), in the Quality Assurance Program Description[iii] (QAPD) that calls for adherence to ASME NQA-1 standards. The QAPP provided guidance and regulation for the characterization activities, to assure that all waste was characterized in the same manner. Site specific quality assurance and the associated procedure development were governed by the LANL TRU Waste Characterization Quality Assurance Project Plan[iv] (QAPjP). Personnel were trained to procedures specified in the QAPjP. The document hierarchy is shown in Figure 1.



WASTE STREAMS

A waste stream is waste material generated from a single process or activity that is similar in material, physical form, isotopic make-up, and hazardous constituents. The four waste streams and typical drum contents that LANL characterized[7] are shown in Table 1. Homogeneous wastes came from two sources – radioactive waste water treatment plant operations at TA-50, and plutonium processing operations at TA-55. All waste drums contained less than 200 Fissile Gram Equivalents of ^{239}Pu, and that amount consisted mostly of ^{239}Pu, having lesser amounts of ^{241}Am, ^{238}Pu, ^{241}Pu, and other actinides. Waste drums had a contact dose rate of under 200 mR/hr, but some of the components in the waste, such as individually packaged pyrochemical salt cans containing americium, had dose rates as high as 3R/hr, contact. Waste consistency ranged from hard Portland cement to wet chalk.

The first waste analyzed was from waste water treatment plant operations consisting of caustic sludge stabilized with Portland cement to form a noncorrosive solid monolith. The sludge was a residue from the treatment of blended acidic and caustic aqueous liquid radioactive waste treated with calcium hydroxide, ferric sulfate, and a flocculation aid. The waste was solidified by agitating 55-gallon drums of sludge with vermiculite, Portland cement, and sodium silicate, yielding a smooth monolith.

The other waste types were from plutonium processing operations that produce two main categories of waste. The first is evaporator bottoms, consisting of precipitate sludge, leachates, ash, salt and other effluents, solidified as a monolith. The sludge is mixed with gypsum or Portland cement and is stirred with a drum stirrer inside a 55-gallon drum or is mixed and left to solidify in 1-gallon cans. The second waste type is cooled molten salts from pyrochemical processes such as electrorefining, molten salt extraction, salt stripping, fluoride reduction, and direct oxide reduction. The salts are melted in a crucible and allowed to cool, after which the crucible is broken away from the salt block and valuable materials are recovered. The remaining salt block and the broken crucible shards are placed into a can. The can is then placed into a bag and is radioassayed and is placed into a drum. Drums have one or more drum liner bags that are twisted and taped closed.

Table 1.
Typical TRU Waste Stream Composition
Principle isotopes are ^{241}Am, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu, ^{233}U, ^{238}U, ^{235}U

Source and TRUCON Code	Composition of Waste	Structure and Matrix	Total Curies per Drum
Waste Water Treatment LA111A	cemented aqueous waste	smooth monolith Portland cement	max. = 7 avg. =3.2
Plutonium Processing LA114A (RCRA F-listed)	vacuum-concentrated precipitates of caustic and acidic effluents	smooth monolith Gypsum cement	max. = 73 avg. = 46
Plutonium Processing LA114A (Non-mixed)	vacuum-concentrated precipitates of caustic and acidic effluents	smooth monolith Gypsum cement	max. = 31 avg. = 16
Plutonium Processing LA124A	pyrochemical salts	granular to rock-like salt blocks	max. = 70 avg. = 45

STATISTICAL SELECTION

Of the 10,000 drums of homogeneous TRU waste at LANL, a few were chosen to represent the entire waste stream. The drums were first segregated into waste streams based on existing information. Utilizing the statistical approach presented in the QAPP, drums from each waste stream were selected for coring, sample collection, and analysis. Drums that presented safety problems, or that for any other reason were not appropriate for sampling, were rejected. Finally, five drums from each of four waste streams were selected for sampling. The analytical results from the 20 drums were used to calculate upper and lower 90 percent confidence limits for the mean concentrations of RCRA-regulated constituents, and those limits were compared to the regulatory threshold limits for the constituent.

DRUM STORAGE AND HANDLING

The candidate drums for this program were selected from among 2000 drums that have been vented and have been in inspectable storage for 3 to 10 years. Personnel at TA-54, Area-G retrieved the drums from above ground storage arrays and prepared each of them for shipment to the sampling facility by checking the condition of the filter/vent, attaching a custody seal, and initiating the waste container chain of custody form. For handling safety, some drums were overpacked into 83-gallon drums. Shipment to TA-50, building 69, the Waste Characterization, Reduction, and Repackaging Facility[vi] (WCRRF) was accomplished via road closure in lieu of using TRUPACT-II packaging. At the WCRRF, personnel accepted custody of the drums and allowed them to thermalize in the facility for at least 72 hours prior to gas or solid sampling. To comply with the building Safety Analysis Report limits for radionuclide inventory, only five drums at a time were opened.

GAS SAMPLING, REAL TIME RADIOGRAPHY, AND RADIOASSAY

Headspace gas sampling, radiography, and radioassay are not required for the RCRA analysis, but are required for waste to meet the WIPP WAC. Therefore, each drum was sampled for headspace gases before core sampling. In addition to meeting the WIPP WAC, the non-destructive assay and examination adds reliability and safety to the program by verifying prior knowledge of drum contents, and by helping to assure that allowable facility limits for storing fissionable material are not exceeded.

Gas samples were collected from each drum by inserting a needle through the vent filter and pumping headspace gas into a 250 ml. SUMMA® canister to a pressure of 20 psi (2.4×10^5 Pa.). The samples were sent to Idaho National Engineering Laboratory for analysis by gas chromatography.

Real time radiography was performed using a mobile high resolution 450kV radiography system with a 12-inch (30 cm) image intensifier and a charge coupled camera. A 36-inch (91 cm) linear diode array was also available for digital radiography.

Radioassay[vii] was performed using both Passive Active Neutron (PAN) assay and Segmented/Tomographic Gamma Scanner (S/TGS) system. These instruments are mobile. The gamma assay system was needed to obtain usable assay results for many of the drums characterized because the high (alpha, n) neutron radiation signal from them (due to high americium content and the presence of low-Z waste matrix materials) made neutron counting results questionable.

CORE DRILLING AND SAMPLING AT THE WCRRF

After gas sampling, all five drums from one waste stream were introduced to the WCRRF cutting chamber, where they were core drilled and sampled. Operators, standing outside the east wall of the 20' x 30' x 16' (6.1m x 9.1m x 4.9m) WCRRF cutting chamber glovebox, controlled the position, speed, and feed rate of the drill, collecting a 30-inch (76 cm) core from a randomly chosen location in less than five minutes. The LANL-designed drill machine drove an auger that was developed jointly by DOE and hard rock drill manufacturers[viii]. Rotation was near 300 Hz with a feed rate of 0.1 inches per second (0.25 cm-s^{-1}). The drillers transferred the core, inside the glovebox, to a sampling crew standing outside the west wall of the glovebox. The samplers subdivided the core into samples and packaged them for analysis by a qualified Los Alamos chemical laboratory at the Chemical and Metallurgical Research (CMR) building. Sample sizes were 5 grams for VOC's, 5 grams for metals, 30 grams for SVOC's, and 30 grams for an archive sample to be held for further

study. Following sampling, the drums were removed from the WCRRF glovebox and were placed in overpack containers for shipment to a decontamination facility. The outsides of the drums were cleaned and the drums were returned to storage.

Because sample duplicates were required for two drums, 22 samples were required. These were taken from 20 drums in ten days, with four samples being taken on each of two days. The fast pace was enabled because the WCRRF glovebox was large enough to accommodate parallel processes. Also, four augers were used during a drilling shift, and were left dirty for a decontamination crew that cleaned them and prepared the work space for the next day's drilling and sampling.

Handling and Shipping Samples

The samples were packaged in glass jars according to the QAPjP and variances to the QAPjP. Each jar was passed out of the WCRRF glovebox into a clean plastic bag and then into a foam lined 1-pint paint-type can. There was no contamination on the outside of any cans, and the dose rates were less than 5.0 mR/hr. The cans were stored in a refrigerator near 4°C.

In order to meet preservation and holding times, the samples were kept at about 4°C until extraction and analysis, no later than 40 days after sampling in the case of VOC's and SVOC's. Normally, environmental samples are shipped in coolers with ice packs to assure that temperature requirements are met. However, the high radionuclide content of the samples required either DOT Type-B packaging or costly road closures, and LANL chose to use the Type-B packaging. Because ice is not allowed in the 6M/2R containers that LANL used, temperature control during shipment was problematic. The solution was to freeze some or all of the interior Cellotex padding of the 6M drum before loading the samples. This method provided holding times of up to 13 hours as demonstrated by testing in one of LANL's environmental test chambers. A recording thermometer and a water trip blank were shipped in a separate 6M/2R package along with each set of samples so that temperature excursions could be noted, and to verify that handling methods were not contaminating the samples.

ANALYSIS FOR VOC's, SVOC's, and METALS

Samples of the gas from the headspace of waste container must be analyzed for 30 volatile organic compounds by gas chromatograph /mass spectrometer (GC/MS) (alcohols and ketones by gas chromatograph /flame ionization detector (GC/FID)) and for hydrogen and methane by GC. The QAPjP lists over 30 gas volatile or total volatile organic compounds, 10 semivolatile organic compounds, 7 PCB's, and 14 metals to be sought by analysis of the core samples. The samples were analyzed by LANL laboratories that had been qualified in the WIPP performance demonstration program. The methods used for VOC's were based on SW846[ix] for GC/MS, or on the DOE Methods Manual Procedure 440.2[x] for GC/FID for nonhalogenated VOC's. To minimize handling of the samples in the analysis glovebox, the VOC samples were packed at the coring site into two separate vials with septum tops. At the LANL laboratories in the CMR building, water was injected into one vial, methanol into the other, and the two leachates were then analyzed for VOC's. The SVOC's were analyzed using method SW846. Approximately 25 gm. of sample was pulverized and extracted for 24 hours with methylene chloride. Analysis was performed by GC/MS; no analysis for PCB's was required for these samples.

A 5 gm split of the pulverized sample was microwave digested in HNO_3+HCl. The solution was then analyzed by ICP/MS and ICP/AES. Mercury analysis was performed by cold vapor atomic fluorescence spectroscopy.

RESULTS OF CHARACTERIZATION

A summary of the preliminary[xi] data follows as Table 2. Results from the VOC and SVOC analyses indicate that methanol is the only RCRA hazardous substance in the core samples. Trichloroethylene, known to be used in the plutonium processing stream, was not detected in the core samples. Waste water treatment sludge that is F001-listed[xii] did not contain detectable quantities of the target compounds. Metals analyses have not been completed at the date of publishing but will be supplied as an addendum to this report.

TABLE 2

AVERAGE CONCENTRATION OF RCRA HAZARDOUS CONSTITUENTS BY WASTE STREAM OF SELECTED TRU WASTE AT LOS ALAMOS NATIONAL LABORATORY

Analyte	HEADSPACE GAS Waste Stream (N) / Average concentration (ppm)	VOC's Waste Stream (N) / Average concentration (ppm)
Benzene	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Bromoform	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Carbon Disulfide	N/A	1/ u , 2/ u , 4/ u .
Carbon Tetrachloride	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Chlorobenzene	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Chloroform	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
1,4-Dichlorobenzene	N/A	1/ u , 2/ u , 4/ u .
ortho-Dichlorobenzene	N/A	1/ u , 2/ u , 4/ u .
Cyclohexane	1/ u , 2/ u , 4/ u .	N/A
1,1-Dichloroethane	1/ u , 2/ u , 4/ u .	N/A
1,2-Dichloroethane	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
1,1-Dichloroethylene	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
cis- 1,2 -Dichloroethylene	1/ u , 2/ u , 4/ u .	N/A
Ethyl Benzene	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Ethyl Ether	1/ u , 2/ u , 4/ u .	N/A
Methylene Chloride	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
1,1,2,2-Tetrachloroethane	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Tetrachloroethylene	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Toluene	1/ 4.70 , 2/ 3.65 , 4/ 4.60 .	1/ u , 2/ u , 4/ u .
1,1,1-Trichloroethane	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
1,1,2-Trichloroethane	N/A	1/ u , 2/ u , 4/ u .
Trichloroethylene	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Trichlorofluoromethane	N/A	1/ u , 2/ u , 4/ u .
1,1,2-Trichloro-1,2,2-trifluoroethane	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
1,2,4 -Trimethylbenzene	1/ u , 2/ u , 4/ u .	N/A
1,3,5-Trimethylbenzene	1/ u , 2/ u , 4/ u .	N/A
Vinyl Chloride	N/A	1/ u , 2/ u , 4/ u .
p/m-Xylene	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
o-Xylene	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Acetone	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Butanol	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Ethyl Ether	N/A	1/ u , 2/ u , 4/ u .
Formaldehyde	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Isobutanol	N/A	1/ u , 2/ u , 4/ u .
Methanol	1/ , 2/ 160 , 4/ 125	1/ ** , 2/ ** , 4/ ** .
Methyl ethyl ketone	1/ u , 2/ u , 4/ u .	1/ u , 2/ u , 4/ u .
Methyl isobutyl ketone	1/ u , 2/ u , 4/ u .	N/A
Pyridine	N/A	1/ u , 2/ u , 4/ u .
Hydrogen (Volume %)	1/ 0.15, 2/ 0.19, 3/ u, 4/ u	N/A
Methane (Volume %)	1/ u , 2/ u , 3/ u , 4/ u	N/A
Tentativily Identified Compounds (Number)	1/ 12 , 2/ 11.5 , 3/ 0.2 , 4/ 13	1/ , 2/ , 3/ , 4/ .

Analyte	SVOC's Waste Stream (N) / Average concentration (ppm)
Creosols	1/ u , 2/ u , 3/ u , 4/ u
1,4-Dichlorobenzene	1/ u , 2/ u , 3/ u , 4/ u
ortho-Dichlorobenzene	1/ u , 2/ u , 3/ u , 4/ u
2,4-Dinitrophenol	1/ u , 2/ u , 3/ u , 4/ u
2,4-Dinitrotoluene	1/ u , 2/ u , 3/ u , 4/ u
Hexachlorobenzene	1/ u , 2/ u , 3/ u , 4/ u
Hexachloroethane	1/ u , 2/ u , 3/ u , 4/ u
Nitrobenzene	1/ u , 2/ u , 3/ u , 4/ u
PCB's (not required)	N/A
Pentachlorophenol	1/ u , 2/ u , 3/ u , 4/ u
Pyridine	1/ u , 2/ u , 3/ u , 4/ u
Tentativily Identified Compounds (Number)	1/ , 2/ , 3/ , 4/ .

Analyte	METALS (Total) * Waste Stream (N) / Average concentration (ppm)
Antimony	1/ , 2/ , 3/ , 4/ .
Arsenic	1/ , 2/ , 3/ , 4/ .
Barium	1/ , 2/ , 3/ , 4/ .
Beryllium	1/ , 2/ , 3/ , 4/ .
Cadmium	1/ , 2/ , 3/ , 4/ .
Chromium	1/ , 2/ , 3/ , 4/ .
Lead	1/ , 2/ , 3/ , 4/ .
Mercury	1/ , 2/ , 3/ , 4/ .
Nickel	1/ , 2/ , 3/ , 4/ .
Selenium	1/ , 2/ , 3/ , 4/ .
Silver	1/ , 2/ , 3/ , 4/ .
Thallium	1/ , 2/ , 3/ , 4/ .
Vanadium	1/ , 2/ , 3/ , 4/ .
Zinc	1/ , 2/ , 3/ , 4/ .
Tentativily Identified Compounds (Number)	1/ , 2/ , 3/ , 4/ .

NOTES

u = Below the Project Required Quantitation Limit (PRQL)
* = Metals data not reported at publishing time.
VOC's and SVOC's are not presswent in Pyrochemical salts.
PRELIMINARY data only. Data has not been reviewed for Quality
**Analyte present above PRQL, but data is only verbally transmitted

Waste Stream

1	Waste Water Treatment LA111A
2	Plutonium Processing LA114A (RCRA F-listed)
3	Plutonium Processing LA1124A (Pyrochem salts)
4	Plutonium Processing LA114A (Not F-listed)

Headspace gas analysis indicates that toluene is present in the headspace of nearly all of the waste containers except for those in the pyrochemical salts waste stream, but that it is below the project required quantitation limit (PRQL). Trichloroethylene and other F-listed constituents were not observed in any of the headspace samples. Over 10 tentatively identified compounds have been noted in the cemented waste streams but only one from the pyrochemical salts. There is no requirement at this time to identify or quantify them. Hydrogen is present in two of the waste streams at about 0.2 volume percent.

DISCUSSION AND CONCLUSIONS

Because toluene was not detected above the PRQL in the solid samples, it is assumed that the presence of toluene in the headspace of cemented waste drums is not due to toluene in the waste matrix. Instead, the headspace toluene is thought to be due to off-gassing from tape used to seal bags inside the drums. However, pyrochemical salt waste drums also contain tape, and do not show toluene above the PRQL in the headspace. This may be because the headspace in the pyrochemical salt drums is about 10 times the headspace in the other drums, diluting any tape-evolved toluene below the PRQL.

Based on preliminary data from headspace gas, VOC, and SVOC analyses, the only reason to classify the LANL waste in this study as mixed waste is the presence of methanol. When data on the metals analyses are reported, presence of metals, if any, in the samples could also cause the waste to be classified as mixed TRU. Even the presence of metals would not require classification of the waste as mixed TRU because this study determined only the total metal content and did not measure the leachability of metals from the matrix.

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[i] *Los Alamos National Laboratory RCRA Waste Analysis Plan*, Revision 0.0. LANL WAP-TRU Mixed Waste, Los Alamos, New Mexico, LANL ESH-19 (1995).

[ii] *Transuranic Waste Characterization Quality Assurance Program Plan*, Revision 0. CAO-94-1010, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy (1995).

[iii] *Quality Assurance Program Description*. CAO-94-1012, Current Revision, Carlsbad, New Mexico, Carlsbad Area Office, U.S. Department of Energy (1994).

[iv] *Los Alamos National Laboratory Transuranic Waste Characterization Quality Assurance Project Plan*. Revision 0. CSTDO-PLAN-002,R.0, Los Alamos, New Mexico, Los Alamos National Laboratory (1995).

[v] About 10% of each waste stream must be characterized to verify its composition. More drums from the 4 waste streams will be analyzed to fully certify the entire waste stream for shipment to WIPP.

[vi] The WCRRF is a multi-purpose, category 3 non-reactor nuclear facility, originally designed for cutting up salvage gloveboxes with a plasma torch.

[vii] Radioassay means determining the radionuclide content of the drums.

[viii] Connolly, Michael J., *Idaho National Engineering Laboratory Simulated Solidified Transuranic Waste Sampling Program*, EGG-WM-1122, Bechtel National, Inc., San Francisco, (1994).

[ix] *Test Methods for Evaluating Solid Waste, Volume IA: Laboratory Manual Physical/Chemical Methods*, Current Revision, Washington, DC, U.S. Environmental Protection Agency (1986).

[x] *Transuranic Waste Characterization Sampling and Analysis Methods Manual*, Revision 0, DOE/WIPP-91-043, Carlsbad, New Mexico, U.S. DOE Carlsbad Area Office (1995).

[xi] At the time of publishing, the data have been reported but have not had the full suit of quality assurance review that is required for qualification.

[xii] Containing the following spent solvents used as degreasers -- tetrachloroethylene, trichloroethylene, methylene chloride, 1,1,1-trichloroethane, carbon tetrachloride or chlorinated fluorocarbons.