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Surrogate Testing for the Pilot VAC*TRAX™ Unit

Topical Report

Work Performed Under Contract No.: DE-AC21-92MC29105

**U.S. Department of Energy
Office of Environmental Management
Office of Technology Development
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Washington, DC 20585**

For

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February 1994

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1. INTRODUCTION

The pilot testing operations conducted under the PRDA VAC*TRAX program were divided into two phases. The first involved a series of tests on mixed waste surrogate materials. Surrogates for mixed waste were defined as either soils spiked with representative RCRA regulated contaminants or RCRA regulated waste materials that are typical of or identical to mixed wastes but do not contain a radioactive component. This test report presents the results of the surrogate waste testing. These tests were conducted under task 1.4 of the PRDA program.

The VAC*TRAX treatability test runs for Task 1.4 of the VAC*TRAX PRDA Project consisted of four runs: two runs on a spiked standard reference material (EPA's Synthetic Soil Matrix, aka "SSM") and two on contaminated PPE from the DOE Rocky Flats Site. Contaminants in all cases consisted of volatiles and semi-volatiles. Additionally, one of the reference samples was spiked with molybdenum trioxide.

Task 1.4 of the VAC*TRAX PRDA Program was designed to demonstrate the effectiveness of the VAC*TRAX process on spikes and RCRA waste prior to the testing of mixed waste. Treatment goals, as specified in the contract, are:

(1) to reduce 60% of the contaminants of concern to below regulatory levels and (2) to reduce the concentrations of 80% of the remaining compounds by 90%. An additional goal, set by CTC, was to demonstrate that metals, particularly radioactive metals such as uranium, would remain in the feed, not transferring in any significant quantity to the residuals. Molybdenum was used to simulate uranium in one of the spiked materials.

The VAC*TRAX treatability testing on the spike samples, as well as the RCRA waste tests, was completed by Michael McElwee and Todd Beaton, and the report was written by Michael McElwee. This work was conducted under the supervision of Carl Palmer. The results of the runs are presented in this report.

2. DESCRIPTION OF THE LABORATORY SCALE VAC*TRAX UNIT

VAC*TRAX treatment is a low temperature separation process that evaporates or volatilizes organic contaminants from soils, sludges, and other types of solid substances or wastes. It operates by heating the contaminated material in a nitrogen atmosphere in a jacketed vessel; hot oil is passed through the vessel jacket to supply heat to the system. To facilitate desorption, the dryer has the capability to operate at high vacuum, up to 94.6 kPa (28 in. Hg) vacuum. The nitrogen serves as an inert carrier gas for the volatilized organics, transporting them to the gas-treatment system, which consists of a condensing train followed by HEPA filter and activated carbon adsorption beds.

2.1 The Vacuum Dryer System

The function of the pilot unit is to provide a preliminary assessment of VAC*TRAX capability for treating contaminated wastes. The pilot vacuum dryer is heated by a 24 kilowatt hot oil system with a maximum temperature capability of 288°C (550°F). The dryer vessel has a total volume of 54 liters (1.9 cubic feet) and a working volume of 31 liters (1.1 cubic feet). The vessel has a heat transfer area of 1.3 square meters (14 square feet), with a soil to steel contact heat transfer area of 0.78 square meters (8.4 square feet). This configuration is designed to simulate the performance achievable by a Full Scale VAC*TRAX dryer. The pilot unit can process approximately 50 kg of soil per batch.

A block flow diagram illustrating the process is shown in Figure 1 and a process flow diagram in Figure 2. The contaminated material is fed into the vacuum dryer through a flanged aperture in the top of the dryer. The hot oil passed through the jackets in the dryer is heated as high as 288°C (550°F) depending on the type of material being treated. The nitrogen carrier gas is preheated, typically up to the oil temperature and introduced into the vessel. Volatilized contaminants and water vapor are transported with the carrier gas to the gas treatment system. Fine particles from the dry treated

solids that are entrained in the carrier gas are removed by an integral steel filter in the dryer's cupola section. These particles are blown back into the dryer at the end of the treatment cycle. When the batch has been heated sufficiently, the product may be dropped from the dryer into a gasketed stainless steel collection vessel by means of a door on the bottom of the dryer. The pilot VAC*TRAX system is shown in Figure 3.

2.2 The Gas Treatment System

The contaminated carrier gas passes through a steel filter in the bonnet of the dryer before exiting the dryer and passing to the gas handling skid. The filter serves to remove particulate matter from the gas stream prior to further treatment. The gas enters the gas treatment system and passes through a shell-and-tube heat exchanger that uses a room temperature 50/50 water/ethylene glycol mixture as the cooling fluid. Next the gas enters another shell-and-tube heat exchanger, this one chilled to 4 °C (40°F) using a 50/50 water/ethylene glycol mixture. These condensers are both before the vacuum pump and are operated at full vacuum during a portion of the drying cycle. The gas then goes through a small cartridge filter and the vacuum pump. The third and final stage of the condensing train is another shell-and-tube heat exchanger cooled with a refrigerated 50/50 glycol/water mixture at 4°C (40°F). Here the carrier gas is cooled to approximately 7°C (45°F) and most of the remaining condensible components are removed. The gas stream then passes through a HEPA filter and two activated carbon adsorption drums in series and is vented to the atmosphere. The vented gas stream is approximately 93-97% nitrogen and 0-7% oxygen, with water and a trace of non-condensable volatile organic compounds making up the balance.

2.3 The Fate of Treatment Residuals

Condensates from the primary, secondary, and tertiary condensers are collected in traps which are drained at regular intervals. Additionally, solids present in the vacuum filter, a filter in place to protect the vacuum pump from damage, will be collected at

the end of a test run for disposal purposes. Samples of the treated solids, condensed water, and condensed oil are retained for chemical analyses when available. Condensate oil is typically not recoverable unless the feed material has a significant oil content. The remaining products and residuals are properly packaged for disposal or are returned to the client depending on the clients requirements as well as federal regulations. All solids and liquids are weighed for material balance purposes.

Figure 1. Block Diagram of the VAC*TRAX Process

Figure 2. Schematic Diagram of the VAC*TRAX Process

Key to Figure 2-2

TE1	thermocouple at nitrogen inlet to dryer, sensing element for controller
TE2	thermocouple in cupola
TE3	thermocouple at gas inlet to first heat exchanger
TE4	thermocouple at gas inlet to second heat exchanger
TE5	thermocouple at gas inlet to third heat exchanger
TE6	thermocouple at gas outlet to third heat exchanger
TE7	thermocouple through bottom of dryer, measuring product temperature
TE8	thermocouple measuring oil temperature entering dryer agitator
TE9	thermocouple measuring oil temperature exiting dryer agitator
TI12	temperature indicator measuring cooling water entering hot oil unit
TI13	temperature indicator measuring cooling water entering first condenser
TI14	temperature indicator measuring cooling water entering second condenser
TI15	temperature indicator measuring cooling water entering third condenser
V1	ball valve for positive shutoff of house nitrogen supply
V2	ball valve for positive shutoff of backup nitrogen supply
V3	ball valve opened when V4 is closed to charge accumulator tank
V4	ball valve used with V3 to charge accumulator to blow back cupola
V5	ball valve used to control nitrogen flow to dryer
V6	ball valve used to control nitrogen flow to dryer
V7	valve adjusted to keep pressure at 0 when nitrogen flows to dryer
V8	gate valve on dryer at oil inlet
V9	gate valve on hot oil unit at oil discharge
V10	gate valve on hot oil unit at oil return
V11	gate valve on dryer at oil outlet
V12	ball valve at controlled cooling water return
V13	ball valve at controlled cooling water supply
V15	ball valve used to adjust flow to condenser 1
V16	ball valve used to isolate system from atmosphere during condensate collection
V17	ball valve for emptying condensate trap
V18	ball valve used to isolate system from atmosphere during condensate collection
V19	ball valve for emptying condensate trap
V20	ball valve used to isolate system from atmosphere during condensate collection
V21	ball valve for emptying condensate trap
V22	ball valve used to control flow to condenser 2
V23	ball valve used to control flow to condenser 3
V24	ball valve used to adjust vacuum in system
V25	ball valve used to adjust flow to ventilation
V30	needle valve used to control nitrogen flow to hot oil system
V31	ball valve used to control flow of cooling water to hot oil system
V32	gate valve used to bypass oil flow to dryer
V33	ball valve used to allow an air sample to be taken for analysis

V34	ball valve at chilled water subsystem supply
V35	ball valve at chilled water subsystem return
V36	ball valve used to bleed air from system
V37	ball valve used to allow an air sample to be taken for analysis
V38	ball valve opened to allow oil to be added to the hot oil system
PI1	pressure indicator at top of cupola
PI2	pressure indicator at gas entry for condenser 1
PI3	pressure indicator at gas entry for condenser 2
PI4	pressure indicator at gas entry for condenser 3
PI5	pressure indicator for nitrogen entering dryer
PI6	pressure indicator for the nitrogen accumulator
PI7	pressure indicator for the house nitrogen supply
PI8	pressure indicator for hot oil entering dryer
PT1	pressure transducer reading pressure at top of cupola
FI1	flow indicator for nitrogen entering dryer - high scale
FI2	flow indicator for nitrogen entering dryer - low scale
FI3	flow indicator for cooling water to condenser 1
FI4	flow indicator for cooling water to condenser 2
FI5	flow indicator for cooling water to condenser 2
FI6	flow indicator for nitrogen to hot oil system
FI7	flow indicator for cooling water to hot oil system
TIC1	temperature controller for nitrogen heater
PRV-1	pressure regulating valve to house nitrogen supply
PRV-2	pressure regulating valve to back-up nitrogen supply
PRV-3	pressure regulating valve to exit vent to hot oil system
PSV-1	pressure safety valve, prevents overpressurizing accumulator
PSV-2	pressure safety valve, prevents overpressurizing dryer
PSV-3	pressure safety valve, prevents overpressurizing agitator
PSE	rupture disc, prevents overpressurizing expansion tank

Figure 3. Photograph of the VAC*TRAX Process

3. STANDARD PROCEDURE FOR TREATABILITY STUDIES

There is a standard procedure for conducting VAC*TRAX treatability studies. The procedure begins with the issuance of a sample authorization number which facilitates tracking. A sample authorization number must be assigned before sample shipment to CTC. Any sample shipped to CTC without a sample authorization number will not be accepted. The received sample is weighed and logged in at the sample receiving area. Feed preparation, process operation, sampling of feed and product streams, system decontamination, and disposal of residuals are discussed below.

3.1 Feed Preparation

The treatability sample size requested is usually 70 liters (15 gallons, in three five-gallon pails). Chain-of-custody documents must accompany all samples. Upon receipt the sample is visually inspected for material larger than 25 mm (1") in size that might damage the vacuum dryer agitator. If large objects are observed in the sample, the sample will be screened to remove the oversize particles. The weights of the screened and oversize fractions are recorded as well as visual observations of the treatability sample. The screened portion is sealed in five-gallon polypropylene or stainless steel pails with gasketed lids. The required amount of feed material (usually one five-gallon pail) is stored at 4°C (39°F), any excess feed is stored at room temperature. The oversize pieces are similarly sealed and stored at room temperature.

After the feed is screened, or after screening is deemed unnecessary, the sample is homogenized by hand mixing. Wet cohesive type soils (clays) are more difficult to homogenize than drier, non-cohesive type soils and may affect the resulting homogeneity. After mixing, a sample is withdrawn for physical and chemical analyses. If the material is difficult to homogenize, the smaller analytical sample is also homogenized to provide a sample of consistent quality to the laboratory. The sample is identified as the preliminary feed and is assigned a sample ID suffix of -01 (Example: CTC-01).

The analyses requested depends on the specific nature of the sample, prior analytical information for the waste, source site history, particular client requests, and the principal contaminants of concern.

The sample is not run through the pilot VAC*TRAX unit until sufficient analytical information is available. This assures that the sample contains the contaminants of concern, allows the identification of extraordinary health and safety requirements, and assures that the appropriate operating conditions are selected.

3.2 Process Operation

To begin the test run, the sample is loaded into the vacuum dryer using a stainless steel funnel bolted to an aperture at the top of the dryer vessel. If the sample emits substantial vapors, a small vacuum hood will be set up above the funnel during sample loading. Approximately 31 liters (1.1 cubic feet) of material is loaded into the system. After loading, the funnel is removed and replaced with the flanged lid for the vacuum dryer. This is resealed securely so that maximum vacuum may be achieved.

After loading the dryer with the sample, the power to the VAC*TRAX system is turned on. At this point, if necessary, oil is added to the hot oil system such that the oil level is between one-quarter and one-half filled as viewed on the expansion tank sight glass. Then, both the dryer and the hot oil system are purged with nitrogen, and oxygen is bled out of the system. The vacuum pump is also turned on at this time, but the vacuum bypass is kept almost all the way open, thus applying only a minimal vacuum to the system. Once an acceptable oxygen concentration has been achieved in the system, less than 7%, the nitrogen flow rate is reduced, typically to around 33.3 l/min (70 scfh) and the process water and chilled water flows to the condensers are turned on.

Next, the hot oil pump is turned on, and oil is pumped through the agitator and jackets in the dryer. The oil setpoint is set at the initial setpoint, typically 148°C (298°F) and the system is allowed to heat up. During this time, water, as well as low boiling volatiles, will be removed from the sample and condensed by the heat exchangers. The rationale for this reduced drying temperature is to match the drying rate of a full-scale unit having a lower surface area to mass ratio than the pilot unit has. Condensate from heat exchangers 1,2 and 3 will be collected individually, weighed, and then composited so that an adequate amount is available for analysis. During this time, pressure and temperature data will be taken frequently to obtain as much information as possible. Data will be taken at least every fifteen minutes.

While the water and low boiling volatiles are being removed from the sample, both the product and vapor temperatures will increase much more slowly than otherwise. When the water is finally removed, the vapor and product temperatures will again begin to elevate. Additionally, the rate of liquids condensing will decrease as the water is removed, reducing to around zero when almost all of the water has been removed.

Once it is obvious that the bulk of the water has been removed from the solids, the vacuum is gradually increased to full vacuum, being careful not to overload the condensers. Then, the oil setpoint is increased to 288°C (550°F), the maximum temperature achievable for the VAC*TRAX pilot system. This will result in a maximum product temperature of approximately 232-260°C (450-500°F). Once the final solids temperature has been achieved, the temperature will be maintained for a predetermined period of time, to guarantee that all of the soil has been properly heated.

After the soil has been heated for the prescribed amount of time, the vacuum in the system is reduced to slightly negative, around 7 kPa (2 in. Hg). Then, the nitrogen accumulator tank is charged to around 465 mm Hg (9 PSIG) with nitrogen, and then

the nitrogen is immediately released into the top of the cupola. In this way, the particles trapped in the filter are blown back onto the soil. After allowing the product to mix a few more minutes, it is then dropped out of the bottom of the dryer into a gasketed stainless steel collection vessel connected to the dryer. The system is allowed to run for another 30 minutes, giving the dryer agitator sufficient time to sweep the majority of the sample into the product hopper. Then, the dryer door is reclosed, and the temperature setpoint for the oil is reduced to 0°C (32°F), and the system is run at only a slight vacuum. The system is allowed to run until the discharge temperature of the hot oil unit is below 68°C (140°F). Then, the hot-oil unit is turned off, the coolant water flows are turned off, the nitrogen is turned off, the dryer agitator is turned off, the vacuum pump is turned off, and then the power to the dryer and gas skids are turned off. The system is still hooked up to the building ventilation, however, so that any fumes or vapors leaving the gas condensing skid will pass through a HEPA filter and two activated carbon drums before being vented to the atmosphere.

3.3 Sampling of Feed and Product Streams

The feed sample is taken just prior to loading the feed into the dryer. Depending on the composition and nature of the feed, the sample will either be taken from the feed bucket or from the dryer, after loading and mixing. In either event, the feed is thoroughly homogenized prior to the taking of the sample to ensure that a representative sample has been taken. The feed sample is identified as the run feed and given the sample ID suffix -02 (i.e. CTC-02).

The solid product is collected from the stainless steel collection hopper after it has been allowed to cool. The sample is given the identifier CTC-03. Typically, duplicate product samples are collected, in which case the sample identifiers will be CTC-03A and CTC-03B, for the first and second samples respectively.

The composited condensates are filtered through Whatman 54 filter paper (nominal 25-micron retention), mixed well, then samples are drawn for chemical analyses. This liquid is identified as condensed water and given the sample ID suffix -06 (i.e. CTC-06).

In the event an oil layer is present in the condensate it is removed prior to filtration if possible. Any oil recovered is weighed, recorded and sent for analysis if a sufficient quantity is recovered. Oil recovered from the condensate is identified as condensed oil and given the sample ID suffix -10 (i.e. CTC-10).

Solid material may collect in the carryover pipe between the dryer outlet and the gas handling system and in the vacuum pump suction filter. This material is recovered, weighed, and if adequate, sent for analysis. The material recovered from the carryover line will be identified with the sample ID suffix -12 (i.e. CTC-12) and the vacuum pump suction filter solids will be identified by the suffix -13 (i.e. CTC-13).

3.4 System Decontamination and Disposal of Residuals

The product, collected in a stainless steel collection vessel, is sampled and then transferred to a 5 gallon pail for disposal purposes. This is accomplished in several steps. The process begins by overlapping the top of the product collection vessel with the opening of a high-strength plastic bag and then duct-taping the bag/vessel seam. This is done with one or two more plastic bags over the original bag, reducing the possibility of a spill due to a bag ripping. Next, the bottom of the plastic bags are placed in a 5 gallon bucket and the product hopper is tipped slightly, allowing the product to pour slowly from the collection vessel to the plastic bags. When the transfer is complete, the product hopper is turned horizontally clockwise several times, causing the top of the plastic bag to twist, sealing the bag. The bag is then cut above the twisted section, separating it from the collection vessel. Transfer is now complete, and the lid can be placed on the bucket.

The dryer vessel will still contain a small amount of solid product. This is recovered by a series of steps. First, a spray bottle containing distilled water is weighed. Next, the top flange on the dryer is removed and the remaining product in the dryer is sprayed with water to prevent dusting. Then, the dryer door is opened, and the remaining sample is removed from the dryer using gloves, brushes, spatulas, etc. as required. The water bottle is weighed at the end of this step, so that the amount of water added to the solids is known. A saranex apron with arm protection or the equivalent is required for this stage. After the solids have been removed, the inside of the dryer is cleaned with a laboratory detergent and scrub brush and rinsed with tap water three times.

The gas handling system is disassembled, and each component is washed with a laboratory detergent and scrub brush. The washed components are then rinsed with tap water three times. The wash water and water from the first two rinses are combined and labeled as waste water that is disposed of as a hazardous waste. If the sample was radioactive, the decontamination liquids will be filtered to remove any radioactive material from the liquids so that it may be disposed of as a hazardous waste.

In some instances, it may be necessary to perform a sand run after testing a sample. In this instance, 10-15 kg of sand will be placed in the vacuum dryer and allowed to agitate for several hours to remove any particles adhering to the walls of the vessel or to the agitator blades. This sand will be tested, and will be disposed of as a hazardous or radioactive waste as appropriate. If the sand is a mixed waste, it must either be returned to the client or else have its hazardous component removed, allowing it to be disposed of as radioactive waste.

All solid product, feed, and scrap is packaged, labeled, and weighed for inventory and mass balance purposes. The fate of the solid material depends on the needs of the client. It is either returned to the client or disposed of as hazardous or radioactive

waste, as appropriate. Mixed waste must be returned to the client, if its hazardous constituent cannot be removed. All remaining liquids are packaged properly and disposed of as a hazardous or radioactive waste as appropriate. If radioactive, the liquids will be filtered to remove the radioactive particles. From previous experience, this should reduce the radioactivity level of the liquids to below background. All decontamination and disposal activities are conducted in a manner consistent with all relevant federal, state, and local regulations governing hazardous and/or radioactive waste at the Clemson Technical Center (CTC) facility.

4. DESCRIPTION OF THE SURROGATE TESTING TREATABILITY TEST RUNS

A description of the treatability runs is given below. The description includes a discussion of sample preparation, deviations from the standard treatability study procedure, a chronology of significant events during the test, sample collection, and sample analysis.

4.1 Description of the Synthetic Soil Matrix Treatability Study Runs

The first two surrogate tests were performed on spiked soil samples. The soil matrix and spiking compounds that were used was the Synthetic Soil Matrix (SSM) that established by the USEPA ¹. This material was established by EPA as being representative of typical soil found at contaminated sites for the evaluation of treatment processes under Superfund. Also, representative organic and toxic metal contaminants were identified by EPA. This matrix and compounds were used so that the results of the VAC*TRAX testing would be comparable to results obtained by other process development and testing programs. Furthermore, this same matrix and spiking compounds were tested in the lab X*TRAX unit early during the X*TRAX development program. By using it in VAC*TRAX a direct performance link is established between the two technologies.

4.1.1 Feed Preparation and Description

Both the first and second VAC*TRAX performance runs, runs SRM and SAR respectively, were completed using the SSM material. Both of these samples were spiked with the same volatiles and semi-volatiles; the second run, SAR, was also spiked with molybdenum trioxide, as a uranium surrogate. The procedures for spiking the reference material are shown in Appendix A. The EPA's "high organics" spiking levels were used, and no hazardous metals were used. Anthracene was eliminated from the mixture, as it is not typical of the DOE contaminant profile.

¹ Foster Wheeler Test Report, Contract No. 68-09-0033, "Clean SSM Manufacture, Physical and Chemical Analysis," Aug. 28, 1991.

After the first sample, SRM, was prepared, it was stored in 2, 19 liter (5 gallon) buckets for several weeks. Then, water was added to one of the two buckets the day before the test run to increase the moisture in the soil. All of the bucket that had water added to it, as well as half of the bucket that didn't, were added to the vacuum dryer and allowed to mix for 10 - 15 minutes. Then, the run feed was taken from the dryer and given the identifier SRM-02.

The second sample, SAR, was prepared in the same fashion as SRM. It was only stored for two days before running, however. The day before the test, water was added to one of the buckets, as was done with the SRM sample. Then, all of the bucket with water added, and half of the other bucket was added to the vacuum dryer, the agitator was run for 10 - 15 minutes, and the feed was sampled as before, given the identifier SAR-02.

The spiking compounds used, the spiking levels, and the analytical results for the run feeds from the two test runs (SRM-02 and SAR-02) are shown in Table 1. Both the SAR-02 and SRM-02 analyses indicate significantly lower concentrations of volatiles and semi-volatiles than were added to the material. Additionally, the SRM-02 material had significantly lower volatiles concentrations than the SAR-02 sample.

Table 1. Analyses of the SRM and SAR Run Feed

Component	Component Concentration in Feed (ppm)		
	Spiking Level	SRM Level	SAR Level
Acetone	6,800	423	1,440
Carbon Tetrachloride	2,500	BQL(31)	91.7
TCE	600	BQL(31)	BQL(78)
Perc	600	31.6	103
Chlorobenzene	400	BQL(31)	BQL(78)
Ethylbenzene	3,200	580	552
Xylene	8,200	1,102	2,342
Styrene	1,000	132	404
Bis(2-ethylhexyl)phthalate	2,500	728	506
1,2 Dichlorophenol	600	BQL(31)	BQL(78)
Pentachlorophenol	1,000	541	480
Molybdenum Trioxide	400*	*	185

* Only the SAR sample was spiked with molybdenum trioxide
All values are reported on an "as received" basis

4.2 Test Execution

The SRM and SAR treatability study runs were conducted on 9/30/93 and 10/7/93 respectively. Both runs were conducted using an oil setpoint of 288°C (550°F) and using the same nitrogen and cooling water flows. Insulation of the unit in both cases was equivalent.

Summaries of the first and second tests on the surrogate spike material (SRM and SAR respectively) are shown in Tables 2 and 3. The tests were performed as expected, with no processing difficulties. Both of the runs were performed with the

controlled cooling water system incapacitated; the cooling water was pumped through HX-1 at the appropriate flow rate, but the water was not cooled as it returned to the chiller. Thus, the temperature of the water to HX-1 heated from 25°C to 40°C over the course of each test run. This would affect the amount of vapor condensed in HX-1, but since HX-2 and HX-3 were operated as designed at 4°C using the chilled water system, the overall gas system performance remained unaffected.

The air turnover in the tent was significantly different for each run. The ventilation was poor, 1-2 air changes per hour, for the SRM test, and much higher, 2-3 air changes per minute, for the SAR run. The greater ventilation was accomplished by including a vacuum hood inside the tent confines. This increased ventilation altered the heat losses from the hot oil system and consequently, slightly changed the product temperature profile for the second run.

The one selected difference between the SRM and SAR treatability studies was a reduction in retention time at temperature; the retention time was set at 90 minutes for the SRM run and at 45 minutes for the SAR run.

Table 2. Chronology Table For the First Spiked Soil Run, Retention Time of 90 Minutes (SRM)

Time	Treatability Study Activity
08:00	Arrive at Lab.
08:50	Add feed sample to dryer.
08:57	Turn on dryer agitator, let run for ten minutes.
09:07	Take run feed sample.
09:35	Remove dryer funnel, put on dryer top flange to seal system.
09:37	Turn on nitrogen to system.
09:45	Drop nitrogen rate to 9.4 l/min (20 SCFH) to dryer and 20 l/hr to hot oil system.
10:00	Turn on water flows, increase Budzar hot oil setpoint to 149°C (300°F).
10:26	Increase nitrogen setpoint to 93°C (200°F).
10:41	The first condensate appears in the heat exchangers collection traps.
10:50	Begin collecting condensate.
10:58	Chiller @ 34°C with setpoint at 18°C. This is due to inadequate ventilation as well as a broken compressor.
11:19	Set nitrogen temperature setpoint to 121°C (250°F).
11:40	Condensate from HX-02 has an oil layer observable in the sight glass. Condensate from HX-01 does not.
12:21	Condensate has stopped collecting in the condensate collection traps.
12:25	Increase hot oil setpoint to 288°C (550°F).
12:30	Increase nitrogen temperature setpoint to 149°C (300°F).
12:40	Increase nitrogen temperature setpoint to 204°C (400°F).
12:45	Increase vacuum in system to (9" Hg).
12:48	No significant increase in condensation rate.
13:18	Closed oil bypass (V32) some - this will allow more oil to pass through the dryer jacket.

Table 2 (Continued)	
13:23	Set nitrogen temperature setpoint to 232°C (450°F).
13:32	Close oil bypass (V32) some more.
13:42	Slowly increase vacuum pressure to full vacuum 94.8 kPa (28" Hg).
13:49	Close bypass (V32) some more.
13:50	Set nitrogen temperature setpoint to 274°C (525°F). Product temperature is at 232°C (450°F).
13:55	Observed creaking noise inside dryer.
13:57	Note small amount of condensate collecting in the condensate trap for HX-03.
14:24	Close oil bypass (V32) some more.
15:20	Let nitrogen charge build up in accumulator tank.
15:22	Blew back cupola filter.
15:25	Reduced hot oil setpoint to 0°C (32°F).
15:26	Reduce nitrogen temperature setpoint to 204°C (400°F).
15:52	Reduce nitrogen temperature setpoint to 93.3°C (200°F).
16:55	Hot oil system off, dryer agitator off, vacuum pump off.
16:57	Coolant flows to hot oil unit and heat exchangers off.
16:58	Nitrogen off, main breakers off.

Table 3. Chronology Table For the Second Spiked-Soil Run, Retention time of 45 minutes (SAR)

Time	Treatability Study Activity
08:00	Arrive at Lab.
08:50	Add feed sample to dryer.
08:55	Turn on dryer agitator, let run for approximately ten minutes.
09:08	Take run feed sample.
09:40	Remove dryer funnel, put on dryer top flange to seal system.
09:40	Turn on nitrogen to system.
09:55	Increase setpoint on hot oil unit to 149°C (300°F), chiller on with setpoint 10°C, chiller to HX-02 and HX-03 on with setpoint 4°C (40°F) and nitrogen temperature setpoint of 38°C (100°F).
10:20	Noted small amount of brownish/reddish condensate in the traps for condenser 2.
10:25	Increase nitrogen temperature setpoint to 93.3°C (200°F).
10:30	Condensate in condenser traps 1 and 2 evident. Condensate is purplish/brown in color.
10:30	Set nitrogen flow to dryer to 35.4 l/min (75 SCFH). Condensate is lighter, now orangish in color.
10:46	Rate of condensation is increasing.
10:50	Increase nitrogen temperature setpoint to 121°C (250°F); increase nitrogen flow to dryer to 42.5 l/min (90 SCFH).
11:05	Condensate is now clear.
11:50	Condensate rate is beginning to decrease.
12:31	Decrease setpoint of chiller (to HX-02 and HX-03) to 0°C (32°F).
12:40	Increase pressure at PI-4 to 13.8 kPa (2 psig).
12:45	Lower nitrogen flow to 35.4 l/min (75 SCFH).
13:00	Condensate is minimal; increase hot oil setpoint to 288°C (550°F). Nitrogen setpoint is increased to 204°C (400°F).
13:10	Increase nitrogen flow to dryer to 47.2 l/min (100 SCFH).
13:15	Increase nitrogen flow to dryer to 51.0 l/min (110 SCFH).

Table 3 (Continued)	
13:25	Close bypass (V32) some to increase flow of hot oil through jackets in dryer.
13:42	Increase nitrogen temperature setpoint to 260°C (500°F).
14:00	Open bypass a bit - some oil was relieving through PSV-3.
14:20	Increase vacuum to 40.6 kPa (12" Hg).
14:21	Budzar hot oil temperature is still at 260°C (500°F) (Setpoint is 288°C (550°F)).
14:22	Increase vacuum to 50.8 kPa (15" Hg).
14:30	Found inadequately tightened triclamp. Tighten.
14:35	Drop nitrogen flow to dryer to 23.6 l/min (50 SCFH).
14:45	Increase vacuum to 94.8 kPa (28" Hg).
15:00	Product Reaches 232°C (450°F).
15:46	Reduce vacuum to 16.9 kPa (5" Hg).
15:47	Build up nitrogen charge in accumulator; blow back cupola filter.
15:49	Open product door to dump product.
15:50	Reduce hot oil unit setpoint to 0°C (32°F), Nitrogen setpoint to 10°C (50°F).
17:00	Hot oil system off, dryer agitator off, vacuum pump off.
17:02	Coolant flows to hot oil unit and heat exchangers off.
17:03	Nitrogen off, main breakers off.

4.1.3 Sample Collection and Analysis

Feed, solid product and liquid samples were collected using the standard procedures described in Section 3. The physical and chemical analyses requested for this treatability study are shown in Tables 4 and 5.

Table 4. Analyses Performed for the SRM VAC*TRAX Test Run

Sample ID	Description	Analyses									
		Wt	pH	Met	Ash	O&G	TPH	TDS	TSS	VOA	SVOA
SRM-02	Run Feed	O	Y	Y	Y	Y	Y	-	-	Y	Y
SRM-03	Product	O	Y	D	D	D	D	-	-	D	D
SRM-06	Condensed Liquid	O	Y	-	-	Y	Y	Y	Y	Y	Y
SRM-10	Condensed Oil	O	-	-	Y	-	-	-	-	Y	Y

Table 5. Analyses Performed for the SAR VAC*TRAX Test Run

Sample ID	Description	Analyses										
		Wt	pH	Met	Ash	O&G	TPH	TDS	TSS	TM	VOA	SVOA
SAR-02	Run Feed	O	Y	Y	Y	Y	Y	-	-	Y	Y	Y
SAR-03	Product	O	Y	D	D	D	D	-	-	Y	D	D
SAR-06	Condensed Liquid	O	Y	-	-	Y	Y	Y	Y	Y	Y	Y
SAR-10	Condensed Oil	O	-	-	Y	-	-	-	-	-	Y	Y

Note: Data for both tests was reported at CTC QA/QC Level 1

Legends:

O	=	by operator
D	=	duplicate analysis
Y	=	test requested
-	=	test not requested
Wt	=	total weight of sample
pH	=	solution pH; method SW846-9045 (solid) or SW846-9040 (water)
Met	=	moisture content; method EPA 160.3 MOD
Ash	=	ash content; method ASTM D-482
O&G	=	oil and grease; method EPA 413.2 or equiv. (water), method EPA 413.2 or equiv. after soxlet extraction (solid)
TPH	=	total petroleum hydrocarbons; method EPA 418.1 after soxlet extraction (solid), method EPA 418.1 (water)
TDS	=	total dissolved solids; method EPA 160.1
TSS	=	total suspended solids; method EPA 160.2
VOA	=	volatile organic analysis; method SW846-8240 or SW846-8260
SVOA	=	semi-volatile organic analysis; method SW846-8270
TM	=	total metals (Molybdenum); methods in SW846

4.2 DESCRIPTION OF THE ROCKY FLATS TREATABILITY RUNS

A description of the treatability runs is given below. The description includes a discussion of sample preparation, deviations from the standard treatability study procedure, a chronology of significant events during the test, sample collection, and sample analysis.

The second pair of surrogate test runs was performed on a RCRA regulated waste material from the DOE's Rocky Flats Plant (RFP). This waste stream is called "combustible shop waste" by RFP. It consists of cloth, plastics and similar trash type components that are contaminated with solvents and industrial lubricants and oils. It is very typical of a large volume mixed waste stream that has been and continues to be generated at RFP. However, the material tested did not contain the radioactive components of the mixed waste. It was of particular interest to determine if the organic components could be removed by the VAC*TRAX unit while operating the hot oil at a temperature below the melting or degradation point of the waste material. This mode of operation specifically exploited the vacuum environment of the process as the temperature was restricted to well below the maximum thermal capability of the unit.

4.2.1 Feed Preparation and Description

The Rocky Flats sample consisted of PPE, rags, brushes, paint rollers, and plastic contaminated with volatiles. A full drum of material in its "as-generated" form was shipped to CTC for this testing. The material was a RCRA listed waste, having waste codes F001-F005 for solvents from non-specific sources. The material contained many pieces larger than the size requirements for the VAC*TRAX pilot unit. Consequently, size reduction was required. This was accomplished by obtaining a representative sample from the drum and then size reducing that portion using scissors, utility knives, and hack saws as required. The procedure for this step is included in Appendix B. Enough sample material was size reduced for three runs.

After size reducing the material, it was necessary to heat check each of the components in the waste, to determine the maximum temperature that could be used without melting the plastic components. This limiting temperature was used to determine the maximum run temperature for the VAC*TRAX pilot unit for this waste; running above that temperature would cause significant quantities of the waste to melt, adhering to the VAC*TRAX internals and making decontamination and emptying of the unit exceedingly difficult. The procedure for this test is listed in Appendix C. The maximum allowable product temperature, estimated based on the results of this test, was 124°C (255°F). Above this temperature a blue plastic garment suit melted.

4.2.2 Test Execution

The RCF and RKF treatability study runs were conducted on 10/13/93 and 10/26/93 respectively. Both runs were conducted using an oil setpoint of 124°C (255°F) and using the same nitrogen and cooling water flows. No intermediate heating temperature was required for the Rocky Flats tests, as was the case with the soil runs, since any liquid in the feed was expected to be minimal. Insulation of the unit in both cases was equivalent, as was the ventilation in the tent the unit was operated under. The first of the two runs (the RCF run) was performed with the controlled cooling water system incapacitated; the cooling water was pumped through HX-1 at the appropriate flow rate, but the water was not cooled as it returned from the chiller. Thus, the temperature of the water to HX-1 heated from 25°C to 40°C over the course of the test run. This would affect the amount of vapor condensed in HX-1, but would not affect the performance of the VAC*TRAX system on the solids. The closed cooling water system was fully functional for the second Rocky Flats test run (identified as RKF). The total heating time for the first of the tests (RCF) was 4 hours and for the second test it was 6 hours.

Table 6. Chronology Table for the first Rocky Flats Run, 4 hr. retention time (RCF)

Time	Treatability Study Activity
11:36	Test motor amps for agitator with no load.
12:00	Load dryer with Rocky Flats waste, mix up waste, and take sample for analysis.
12:10	Reseal system. Run agitator forwards and backwards several times, measuring motor amps each time.
12:12	Ammeter reads between 2.3 and 2.4 amps for all cases (less than 3.6 amps, specified as the maximum allowable to protect the agitator). It is decided to reverse agitator rotation direction every minute to prevent jamming.
12:35	System purged with nitrogen, 23.6 l/min (50 SCFH) to dryer, 50 l/hr to hot oil unit. Hot oil unit turned on.
12:37	Hot oil unit setpoint increased to 250 ° F.
12:52	Opened valve inlet to dryer to allow more oil to flow to the dryer.
12:53	Leak noticed in gas system piping; leak corrected by tightening triclamp.
12:55	Increased vacuum to 27 kPa (8" Hg).
13:00	Increased nitrogen heater setpoint to 93.3°C (200°F).
13:15	Increased vacuum to 50.8 kPa (15" Hg).
13:22	Increased vacuum to 94.8 kPa (28" Hg).
13:22	Portable ammeter found to significantly affect value read for "jacket out" temperature; problem corrected by repositioning ammeter.
13:55	Increase nitrogen flow to 47.2 l/min (100 SCFH).
13:57	Increase hot oil setpoint to 124°C (255°F).
14:14	Increase nitrogen heater setpoint to 124°C (255 ° F).
15:00	Close valve to dryer from hot oil unit slightly; some oil was passing through the pressure relief valve PRV-3.
16:29	Charge accumulator to 62 kPa (9 PSI), blow back cupola filter.
16:30	Return system to atmospheric pressure.
16:33	Decrease hot oil unit setpoint to 0°C (32°F).
18:06	Turn off system.
19:11	Empty dryer through dryer door and product door.

Table 7. Chronology Table for the second Rocky Flats Run, 6 hr. Retention Time (RKF)

Time	Treatability Study Activity
09:35	Take run feed sample prior to loading. Load dryer and reseal.
10:21	Turn on chiller, set flows for chilled water and controlled cooling water to 3.8l/min (1 gpm).
10:23	Test agitator with dryer loaded, check motor amps. Results were all between 2.4 and 2.45 amps (Less than the maximum allowable level of 3.6). Agitator turned with less noise than in the previous Rocky Flats run.
10:30	Nitrogen turned on: 47.2 l/min (100 SCFH) to dryer, 50 l/h to hot oil system.
10:31	Vacuum pump turned on.
10:35	Budzar unit will not start; broken 600 V, 1 amp fuse.
10:37	Dump feed back into bucket, hammer on gasketed lid, and place bucket in refrigerator. Shut down nitrogen and water flows.
14:00	After fuse is replaced, take new run feed sample.
14:18	Reload dryer.
14:33	Establish nitrogen and cooling water flows.
14:37	Check motor amps. All were between 2.3 and 2.4 amps.
14:37	Increase nitrogen flow to 47.2 l/min (100 SCFH) to dryer. Flow to hot oil system is 50 l/h.
14:38	Reduced nitrogen flow to 33 l/min (70 SCFH). Turned on vacuum pump.
14:40	Turned on hot oil unit, raise hot oil setpoint to 124°C (255°F).
14:41	Increased nitrogen heater setpoint to 71°C (160°F). Drop nitrogen flow to dryer to 23.6 l/min (50 SCFH).
14:50	Open valve to allow increased flow of oil to dryer jackets.
14:53	Increased vacuum to 27.1 kPa (8" Hg).

Table 7 (Continued)	
15:01	Increased vacuum to 50.8 kPa (15" Hg).
15:03	Increased nitrogen heater setpoint to 116°C (240°F).
15:05	Increase vacuum to 67.7 kPa (20" Hg).
15:16	Increase vacuum to 88 kPa (26" Hg).
15:35	Decrease nitrogen flow to dryer to 28.3 l/min (60 SCFH).
15:45	Increase nitrogen heater setpoint to 127°C (260°F).
16:15	A breaker was tripped on the chiller providing flows to HX-02 and HX-03. This was noted and corrected.
20:40	Reduce hot oil setpoint to 32°F, drop vacuum to 0 kPa (0" Hg). Reduce controlled cooling water setpoint to 12°C and nitrogen heater setpoint to 21°C (70°F).
20:45	Turned off nitrogen heater
20:46	Charge nitrogen accumulator; blow back cupola.
22:15	Open bottom product door, let most of the product drop into the product hopper.
22:21	Shut down system; gather remaining product from dryer door.

4.2.3 Sample Collection and Analysis

Feed, solid product, and liquid samples were collected using the standard procedures described in Section 3.

The physical and chemical analyses requested for this treatability study are shown in Table 8. The analyses requested in both Rocky Flats runs were identical.

Table 8. Analyses Performed for the Rocky Flats VAC*TRAX Test Run

Sample ID	Analyses									
	Wt	pH	Met	Ash	O&G	TPH	TDS	TSS	VOA	SVO A
XXX-02	O	Y	Y	Y	Y	Y	-	-	Y	Y
XXX-03	O	Y	D	D	D	D	-	-	D	D
XXX-06	O	Y	-	-	Y	Y	Y	Y	Y	Y
XXX-10	O	-	-	Y	-	-	-	-	Y	Y

XXX = RCF or RKF, respectively

Note: Data for both tests was reported at CTC QA/QC Level 1

Legends:

O = by operator
 D = Duplicate analysis
 Y = test requested
 - = test not requested
 Wt = total weight of sample
 pH = solution pH; method SW846-9045 (solid) or SW846-9040 (water)
 Mst = moisture content; method EPA 160.3 MOD
 Ash = ash content; method ASTM D-482
 O&G = oil and grease; method EPA 413.2 or equiv. (water), method EPA 413.2 or equiv. after soxlet extraction (solid)
 TPH = total petroleum hydrocarbons; method EPA 418.1 after soxlet extraction (solid), method EPA 418.1 (water)
 TDS = total dissolved solids; method EPA 160.1
 TSS = total suspended solids; method EPA 160.2
 VOA = volatile organic analysis; method SW846-8240 or SW846-8260
 SVOA = semi-volatile organic analysis; method SW846-8270
 TM = total metals (Molybdenum); methods in SW846

5. DISCUSSION OF PILOT TEST RESULTS

The pilot VAC*TRAX treatability runs were completed as expected. The treatability results are presented in this section.

5.1 Surrogate Spike Material Test Results

5.1.1 Operating Data Summary

The VAC*TRAX unit was operated at a final oil setpoint of 288°C (550°F) for both test runs. Initially, the oil temperature was set at 148°C (298°F) during the water evaporation phase. It was then increased to 288°C (550°F) when it was apparent that most of the water had been removed from the soil. A summary of the operating conditions for both runs is presented in Table 9. The change in product temperature over the course of the SRM and SAR runs are shown in Figures 4 and 5 respectively.

Table 9. Operating Condition Summary For the Synthetic Soil Matrix Test Runs

Sample ID	Feed in Dryer	Oil Temp Drying	Oil Heating Temp	Final Product Temp	Vapor Temp (T-2) Last 30 min
	% Water in Feed	Drying Time	Heating Time	Retention Time	N ₂ Flow to Dryer
SRM	37.65 kg	148°C	288°C	247°C	261°C
	6.93%	145 min	85 min	90 min	30.7 l/min
SAR	42.05 kg	148°C	288°C	239°C	253°C
	12.3%	185 min	120 min	45 min	35.7 l/min

5.1.2 Process Component and Overall Mass Balances

The overall mass balance for the SRM run is shown in Figure 6. Figure 7 shows the overall mass balance for the SAR run. Tables 10 and 11 show the component mass balance for both the SRM and SAR test runs respectively.

Figure 4. Reference Material Run 2 (SRM) Product Temperature Curve

Figure 5. Reference Material Run 2 (SAR) Product Temperature Curve

Figure 6. SRM VAC*TRAX Treatability Study Overall Mass Balance

SAMPLE	TOTAL MASS IN (g)	TOTAL MASS OUT (g)	RECOVERY %
SRM	37,650	37,710	100.2

NOTE: THE OVERALL MASS BALANCE EXCLUDES THE NITROGEN ADDED TO THE SYSTEM

Figure 7. SAR VAC*TRAX Treatability Study Overall Mass Balance

SAMPLE	TOTAL MASS IN (g)	TOTAL MASS OUT (g)	RECOVERY %
SAR	41,544	41,202	99.2

NOTE: THE OVERALL MASS BALANCE EXCLUDES THE NITROGEN ADDED TO THE SYSTEM

Table 10. Component Recoveries for the first Synthetic Soil Matrix Run (SRM)

Component	Total In	Total Out	% Recovery
Solids (g)	35,040	33,561	95.8
Liquids(g)	2,610	4,149	159.0
<u>Volatiles(g)</u>			
Acetone	15.93	3.04	19.0
Carbon Tetrachloride	x	x	x
Trichloroethene	x	x	x
Tetrachloroethylene	1.19	0.012	1.0
Ethylbenzene	21.84	3.49	16.0
Xylene	41.49	5.10	12.3
Styrene	4.95	1.98	40.1
<u>Semi-volatiles(g)</u>			
1,2 Dichloroethane	x	x	x
Pentachlorophenol	20.37	3.56	17.5
bis(2-ethylhexyl)phthalate	27.41	0	0

x = non-quantifiable amount in the feed; calculation of removal efficiency not applicable

Table 11. Component Recoveries for the second Reference Material Run (SAR)

Component	Total In	Total Out	% Recovery
Solids (g)	36,840	37,102	101
Liquids(g)	4,704	4,100	87.2
<u>Volatiles(g)</u>			
Acetone	53.1	25.0	47.0
Carbon Tetrachloride	3.38	0	0
Trichloroethene	x	x	x
Tetrachloroethylene	3.76	4.17	111
Ethylbenzene	20.3	24.2	119
Xylene	86.3	97.9	113
Styrene	14.9	15.1	101
<u>Semi-volatiles(g)</u>			
1,2 Dichloroethane	x	x	x
Pentachlorophenol	17.7	5.08	28.7
bis(2-ethylhexyl)phthalate	18.6	0.069	0.37
Molybdenum Trioxide(g)	6.82	6.68	98.0

For the mass balances, the following component definitions apply to all streams.

Solids: The fraction of the material remaining after heating at 105°C

Water: Sample weight loss at 105°C; may include organics of higher volatility. Moisture and water are considered synonymous

Oil and Grease: Oil and grease analyses, or actual liquid oil recovered.

Liquids: The total weight of water (moisture) in and condensate (water and oil) out.

Contaminant concentrations are measured in the solid and liquid streams only. Analysis of contaminants in the gas stream was not within the scope of this testing.

As seen in Figures 6 and 7, the solids and water recoveries were slightly off for both the SRM and SAR test runs. Here, it is noted that, while one of the phases (solid or liquid) has a higher than 100% recovery, the other has less than 100% recovery. This brings up the possibility that either (1) the moisture analysis for the feed was incorrect or (2) the sample provided for moisture analysis was not reflective of the average moisture concentration of the feed. In any event, the recoveries were acceptable and demonstrate material accountability during the test.

Volatile organic compound recoveries ranged from 1% to 40.1 percent for the first test run (SRM), averaging around 18%. Recoveries for the second test were significantly higher, ranging from 0% to 119%, averaging around 82%. Oil recovery is thought to be the primary difference between the two runs, since over 5½ times as much oil was collected in the second test than in the first. In both tests, the low recoveries for some of the components is misleading. Due to the presence of high quantities of volatiles in the collected oil from both runs, the detection levels for all

volatiles in the oil were elevated dramatically, as high as 17,000 ppm for acetone. Then, since components listed as "below quantitation limit" (BQL) in the oil and other matrices are considered to be zero for component mass balance purposes, the percent recoveries for some components are almost guaranteed to be low. If we instead, for example, assumed all BQL values to be at the quantitation level, the recovery of carbon tetrachloride for the SAR test run would be 84% as opposed to 0%. This most certainly affected semi-volatile results as well; pentachlorophenol recovery was 18% and 29% for the first and second test runs respectively, while bis(2-ethylhexyl)phthalate had effectively a 0% recovery for both runs. Molybdenum recovery was excellent, at 98%. Overall mass recoveries were 100.2% and 99.2% respectively for the first and second test runs (SRM and SAR). These are both excellent recoveries, well within the acceptable range.

5.1.3 Particulate Carryover

As was expected, only very minimal quantities of solids were carried into the gas system piping, due mainly to the filter in the cupola. No measurable solids were recovered, so the percent carryover was less than 0.1%. Based on the quantity of feed and the configuration of the system, a carryover percentage this low is not unusual.

5.1.4 Analytical Results

The analytical results for the SRM and SAR treatability study runs are summarized in tables 12 and 13. Tables 14 and 15 summarize the removal efficiencies for selected components in the SRM and SAR runs respectively.

Table 12: Analytical Results for the First Synthetic Soil Matrix (SRM) Run

Component		Run Feed (SRM-02)	Product (SRM-03A)	Product Dup. (SRM-03B)	Cond. Water (SRM-06)	Cond. Oil (SRM-10)
Moisture	%	6.9	0	0	NA	NA
Ash	%	83.6	94.4	87.9	NA	BQL(1)
pH		8.7	8.0	-	7.6	-
oil & grease	ppm	160	BQL(10)	BQL(10)	-	-
Dissolved Solids	ppm	NA	NA	NA	171	-
Suspended Solids	ppm	NA	NA	NA	48	-
Acetone	ppm	423	5.42	3.29	696	BQL(17,000)
Carbon Tetrachloride	ppm	BQL(31)	0.3	BQL(0.21)	BQL(100)	BQL(8,300)
1,2 Dichloroethane	ppm	BQL(31)	0.29	BQL(0.21)	BQL(50)	BQL(8,300)
Trichloroethene	ppm	BQL(31)	0.21	BQL(0.21)	BQL(50)	BQL(8,300)
Tetrachloroethylene	ppm	31.6	0.36	BQL(0.21)	BQL(50)	BQL(8,300)
Ethylbenzene	ppm	580	3.84	0.72	BQL(50)	114,000
M,P Xylene	ppm	790	4.15	0.88	BQL(50)	167,000
O Xylene	ppm	312	1.26	0.25	BQL(100)	82,800
Styrene	ppm	132	0.35	BQL(0.21)	BQL(50)	66,000
Bis(2-ethylhexyl)phthalate	ppm	728	BQL(10)	BQL(10)	BQL(1.0)	2,500
Pentachlorophenol	ppm	541	97.0	112	16.0	2,500

NOTE: Results are reported on an "as-received" basis

Table 13: Removal Efficiencies for the First Synthetic Soil Matrix (SRM) Run

Component	Concentration ppm		Removal %
	SRM-02 Run Feed	SRM-03 (Avg.) Product	
Acetone	454	4.36	99.0%
Tetrachloroethylene	34	0.18	> 99.5%
Ethylbenzene	623	2.33	> 99.6%
Xylene	1,184	5.03	> 99.6%
Styrene	142	BQL(0.21)	> 99.9%
Bis(2-ethylhexyl)phthalate	782	BQL(10)	> 98.9%
Pentachlorophenol	581	126	83.2%

NOTE: Results are converted to a "dry weight" basis

Table 14: Analytical Results for the Second Synthetic Soil Matrix (SAR) Run

Component	Run Feed (SAR-02)	Product (SAR-03A)	Product Dup. (SAR-03B)	Cond. Water (SAR-06)	Cond. Oil (SAR-10)
Moisture	12.4	0	0	NA	-
Ash	83.7	89.17	77.55	NA	BQL(1)
pH	8.7	7.5	-	6.6	-
oil & grease	240	BQL(10)	BQL(10)	19	-
Dissolved Solids	NA	NA	NA	240	-
Suspended Solids	NA	NA	NA	91	-
Acetone ppm	1,440	9.62	2.33	2,740	98,600
Carbon Tetrachloride ppm	91.7	BQL(0.6)	BQL(0.2)	BQL(83)	BQL(17,000)
1,2 Dichloroethane ppm	BQL(78)	BQL(0.6)	BQL(0.2)	BQL(83)	BQL(17,000)
Trichloroethene ppm	BQL(78)	BQL(0.6)	BQL(0.2)	BQL(83)	BQL(17,000)
Tetrachloroethylene ppm	103	BQL(0.6)	BQL(0.2)	BQL(83)	27,900
Ethylbenzene ppm	552	BQL(0.6)	BQL(0.2)	BQL(83)	161,000
M,P Xylene ppm	1,740	BQL(1.2)	BQL(0.2)	BQL(166)	492,000
O Xylene ppm	602	BQL(0.6)	BQL(0.2)	BQL(83)	157,000
Styrene ppm	404	BQL(0.6)	BQL(0.2)	BQL(83)	100,000
Bis(2-ethylhexyl)phthalate ppm	506	BQL(33)	2.10	BQL(2.5)	BQL(2,300)
Pentachlorophenol ppm	480	154.0	97.4	BQL(2.5)	BQL(2,300)
Molybdenum trioxide ppm	185	203	214	BQL(0.18)	BQL(8.49)

NOTE: Results are reported on an "as received" basis

Table 15. Removal Efficiencies For the Second Synthetic Soil Matrix (SAR) Run

Component	Concentration ppm		Removal %
	SAR-02	SAR-03 (Avg.)	
Acetone	1,644	5.98	99.6%
Carbon Tetrachloride	105	BQL(0.2)	> 99.8%
Tetrachloroethylene	118	BQL(0.2)	> 99.8%
Ethylbenzene	630	BQL(0.2)	> 99.9%
Xylene	2,672	BQL(0.2)	> 99.9%
Styrene	461	BQL(0.2)	> 99.9%
Bis(2-ethylhexyl)phthalate	577	1.05	99.8%
Pentachlorophenol	548	126	77.0%
Molybdenum Trioxide	211	209	0.9%

NOTE: Results are reported on a "dry weight" basis

The remediation goal(s) for the VAC*TRAX testing, as specified in the PRDA Contract are to (1) treat 60% of the organic contaminants to below regulatory limits and, (2) to reduce the concentration of 80% of the remaining organic contaminants by 90%. Of the 9 volatiles and 2 semi-volatiles in the feed, for a total of eleven organic constituents, 10 have been reduced to below the regulatory limits. Matrix interference prevented the quantitation of three of the volatiles in the feed, but the levels to which they were spiked, as well as their presence in very minute quantities in the product, demonstrates their existence. Codes F001-F005 for the volatiles and U028 for the bis(2-ethylhexyl)phthalate were assumed to determine the regulatory limits. Therefore 91% of the organic constituents were reduced to below the regulatory limits, exceeding the 60% requirement. Now, 60% of the constituents would be, rounding up, 7 constituents. Eighty percent of the remaining constituents then, or three of the four remaining components, should have been removed by at least 90% to meet treatment goals. This was accomplished, since, as previously stated, all but one of the components were reduced by at least 99%. Thus, contract goals were met.

5.2 Rocky Flats Test Results

5.2.1 Operating Data Summary

The VAC*TRAX unit was operated at an oil setpoint of 124°C (255°F) for both test runs. For each of these tests, a total heating time was selected because little or no water was present in the solid material; this took the place of a retention time at a temperature. A summary of the operating conditions for both runs is displayed in Table 16.

Table 16. Operating Condition Summary For the Rocky Flats Test Runs

Sample ID	Feed in Dryer	Oil Temp. Heating	Final Product Temp.	Vapor Temp (T-2) Last 30 Min
	% Water in Feed	Heating Time(min)	Retention Time	N ₂ Flow to Dryer
RCF	2.55 kg	124°C	101°C	233°C
	4.5%	240 min	*	38 l/min
RKF	3.10 kg	124°C	104°C	236°C
	3.5%	360 min	*	35 l/min

- * There was only one heating region for these tests, since water content was minimal and the total heating time was chosen, as opposed to the retention time.

5.2.2 Process Component and Overall Mass Balances

The component mass balances for both Rocky Flats runs for solids and liquids are shown in Tables 17 and 18. The overall mass balances for the first and second Rocky Flats Runs (RCF and RKF respectively) are shown in Figures 8 and 9. Due to the very low quantities of feed used, it is not practical to perform a material balance on the individual organic constituents. This is especially true since condensate, while produced, was not in sufficient quantity for organic analysis.

Figure 8. RCF VAC*TRAX Treatability Study Overall Mass Balance

SAMPLE	TOTAL MASS IN (g)	TOTAL MASS OUT (g)	RECOVERY %
SAR	2,550	2,470	96.8

NOTE: THE OVERALL MASS BALANCE EXCLUDES THE NITROGEN ADDED TO THE SYSTEM

Figure 9. RKF VAC*TRAX Treatability Study Overall Mass Balance

SAMPLE	TOTAL MASS IN (g)	TOTAL MASS OUT (g)	RECOVERY %
SAR	3,100	3,064	98.8

NOTE: THE OVERALL MASS BALANCE EXCLUDES THE NITROGEN ADDED TO THE SYSTEM

Table 17. Component Recoveries for the first Rocky Flats Run (RCF)

Component	Total In	Total Out	% Recovery
Solids (g)	2,440	2,350	96.5
Liquids(g)	115	119	103.5

Table 18. Component Recoveries for the second Rocky Flats Run (RKF)

Component	Total In	Total Out	% Recovery
Solids (g)	2,990	3,000	101
Liquids(g)	115	64	55.7

For the mass balances, the following component definitions apply to all streams.

Solids: The fraction of the material remaining after heating at 105°C

Water: Sample weight loss at 105°C; may include organics of higher volatility. Moisture and water are considered synonymous

Oil and Grease: Oil and grease analyses, or actual liquid oil recovered.

Liquids: The total weight of water (moisture) in and condensate (water and oil) out.

Contaminant concentrations are measured in the solid and liquid streams only. Analysis of contaminants in the gas stream was not within the scope of this testing.

As seen in Tables 17 and 18, the solids and water recoveries were slightly off for both of the Rocky Flats test runs. For the material balance on each run, it is noted that, while one of the phases (solid or liquid) has a higher than 100% recovery, the other has less than 100% recovery. This brings up the possibility that either (1) the moisture analysis for the feed was incorrect or (2) the sample provided for moisture analysis was not reflective of the average moisture concentration of the feed. In either event, the overall component mass balances are excellent for both of the Rocky Flats Runs.

5.2.3 Particulate Carryover

As was expected, only very minimal quantities of solids were carried into the gas system piping, due mainly to the filter in the cupola. No measurable solids were recovered, so the percent carryover was less than 0.1%. Based on the quantity of feed and the configuration of the system, a carryover percentage this low is not unusual.

5.2.4 Analytical Results for the Rocky Flats Test Runs

The analytical results for the RCF and RKF treatability study runs are summarized in tables 19 and 21. Tables 20 and 22 summarize the removal efficiencies for selected components in the RCF and RKF runs respectively.

Table 19. Analytical Results for the First Rocky Flats (RCF) VAC*TRAX Run

Component	RCF-02 Run Feed	RCF-03A Product	RCF-03B Product Duplicate	RCF-06
Moisture	4.5	0	0	6.6
pH	6.3	6.3	-	
TDS	-	-	-	
TSS	-	-	-	
Oil & Grease	-	-	-	
TPH	BQL(10)	BQL(10)	BQL(10)	
Acetone ppm	7.51	< 0.41	< 0.41	11.1
MIBK ppm	18.7	0.81	1.46	4.35
Toluene ppm	3.54	< 0.20	< 0.20	BQL(0.5)
2-Hexanone ppm	5.93	< 0.20	0.39	BQL(0.5)
Ethylbenzene ppm	9.59	< 0.20	0.42	BQL(0.5)
M,P Xylene ppm	37.6	0.68	1.25	BQL(1.0)
O Xylene ppm	12.5	0.26	0.5	BQL(0.5)
Styrene ppm	90.7	2.00	3.46	1.42

Table 20. Removal Efficiencies for the First Rocky Flats (RCF) VAC*TRAX Run

Component	RCF-02 Run Feed	RCF-03A Product	RCF-03B Product Duplicate	Avg. Product	% Removal
Acetone ppm	7.51	BQL(0.41)	BQL(0.41)	BQL(0.41)	> 94.5
MIBK ppm	18.7	0.81	1.46	1.14	93.9
Toluene ppm	3.54	BQL(0.20)	BQL(0.20)	BQL(0.20)	> 94.3
2-Hexanone ppm	5.93	BQL(0.20)	0.39	0.20	96.6
Ethylbenzene ppm	9.59	BQL(0.20)	0.42	0.21	97.8
M,P Xylene ppm	37.6	0.68	1.25	0.97	97.4
O Xylene ppm	12.5	0.26	0.5	0.38	97.0
Styrene ppm	90.7	2.00	3.46	2.73	97.0

Table 21. Analytical Results for the Second Rocky Flats (RKF) VAC*TRAX Run

Component		RKF-02 Run Feed	RKF-03A Product	RKF-03B Product Duplicate	RKF-06 Cond Water	RKF-10 Cond Oil
Moisture	%	3.7	0	0	-	-
Ash	%				-	-
pH		6.3	6.6	6.6	5.5	-
TDS	ppm	-	-	-	-	-
TSS	ppm	-	-	-	-	-
Oil & Grease	ppm	200,000	200,000	160,000	-	-
TPH	ppm	170,000	180,000	150,000	-	-
Acetone	ppm	1.0	0.053	0.088	-	BQL(50)
MIBK	ppm	15.0	0.24	0.72	-	BQL(50)
Toluene	ppm	6.7	BQL(0.025)	0.110	-	BQL(25)
Ethylbenzene	ppm	20.0	BQL(0.025)	0.150	-	27
Xylene (Total)	ppm	110.0	0.033	0.150	-	210
Styrene	ppm	230.0	0.093	0.190	-	840
Naphthalene	ppm	BQL(84)	BQL(100)	BQL(230)	-	3,800
2-Methylnaphthalene	ppm	BQL(84)	BQL(100)	BQL(230)	-	4,100

Table 22. Removal Efficiencies for the Second Rocky Flats (RKF) VAC*TRAX Run

Component		RKF-02 Run Feed	RKF-03A Product	RKF-03B Product Duplicate	Avg. Prod.	% Removal
Acetone	ppm	1.0	0.053	0.088	0.071	92.9
MIBK	ppm	15.0	0.24	0.72	0.48	96.8
Toluene	ppm	6.7	BQL(0.025)	0.110	BQL(0.055)	> 99.2
Ethylbenzene	ppm	20.0	BQL(0.025)	0.150	BQL(0.075)	> 99.6
Xylene (Total)	ppm	110.0	0.033	0.150	0.092	99.9
Styrene	ppm	230.0	0.093	0.190	0.14	99.9

The F001-F005 regulations/limits that apply, as specified by 40 CFR 268.43, are shown in Table 23.

Table 23. F001-F005 Limits: CCWE - Constituent Concentrations in Waste Extract, Non-wastewaters

Component	Limits (Constituent Concentrations in Waste Extract) (CCWE)
Acetone mg/L	0.59
MIBK mg/L	0.33
Toluene mg/L	0.33
Ethylbenzene mg/L	0.053
Xylene mg/L	0.15

The results show a significant reduction in volatile concentrations. TCLP analyses were not performed for the organic constituents. However, as per 40 CFR 261, Appendix II, 1.2:

If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

This means that, if the regulatory levels could not possibly be exceeded as determined by the TCLP method, then TCLP analyses are not required. Then, from 40 CFR 261 Appendix II, 2.2:

The solid phase is extracted with an amount of extraction fluid equal to twenty times the weight of the solid phase.

This indicates that the maximum concentration of a contaminant in the extract is 1/20 of the concentration in the solid. Therefore, if the concentrations of contaminants in the treated product are less than 20 times the regulatory level, TCLP analyses are not required. A summary of the comparison is shown in Table 24.

Table 24. Determination of the Necessity for TCLP Testing

Component	Highest Product Conc. Analyzed	20 Times Reg. Limit for CCWE
Acetone ppm	0.088	11.8

MiBK	ppm	0.72	6.6
Toluene	ppm	0.11	6.6
Ethylbenzene	ppm	0.15	1.06
Xylene (Total)	ppm	0.15	3.0

As can be seen from Table 24, the treated Rocky Flat's waste is unquestionably below the regulatory limits for the F001-F005 classification. The comparison was done on a worst case basis: the highest concentration found for each of the components in the treated waste was used, and it was assumed that 100% would leach into the extract, which is unlikely.

6. CONCLUSIONS

Based on the results obtained during the surrogate testing program, several key conclusions can be drawn about the VAC*TRAX system. These are:

- VAC*TRAX is an effective tool for the removal of volatiles and select semi-volatiles from both soils and debris-type waste.
- Treatment goals, as specified by the PRDA contract, were met or exceeded in each of the four pilot tests.
- Metals concentrations (ie. radioactivity) in the feed remains unchanged by thermal desorption. This was determined by spiking one of the standard reference matrix soils with molybdenum trioxide. Furthermore, it was also determined that the metals (ie. radioactivity) does not partition into the condensed water and organic phases. This, along with the low carrier gas flow-rate that allows for the process emissions to be run through a HEPA filter and carbon drums before release in the vent, makes the system ideal for the treatment of mixed waste, particularly soils or debris-wastes

contaminated with depleted uranium, for which the molybdenum was chosen as a surrogate.

- A full-scale VAC*TRAX system of equal or greater capability could be designed feasibly and economically. This is because the oil and solids temperatures used by the pilot VAC*TRAX unit are 100°F below the practical maximum for a hot oil unit.
- The nitrogen flow required for the pilot VAC*TRAX unit is higher than desired, and if this parameter scales directly to full scale, the nitrogen flow would be roughly 100 scfm. It is desired to have a full scale nitrogen flow of roughly 50 scfm. However, a full-scale system is likely to have a lower air in-leakage rate per unit volume than the pilot unit, therefore requiring less nitrogen per ton of waste. This is the case for the small-pilot and full-scale X*TRAX thermal desorption units. As such, nitrogen flow is not expected to scale precisely. Further optimization of this parameter will be required during the full scale system design.
- The importance of the degree of vacuum was not explored. The pilot unit was always operated at full vacuum after the majority of the water was removed from the solids. It was found that during this drying phase, that higher vacuum created the potential to overload the condensation capacity of the condensers. No significant benefit was perceived to operating at less than full vacuum during the heating and hold phases. The minor benefits of lower vacuum operation would be reduced electric power consumption and lower nitrogen use rate, neither having as much economic impact as a shorter cycle time.

- Finally, the VAC*TRAX process is both technically and economically sound. The key economic objective of having a batch cycle time of less than eight hours appears easily achievable.

Based on these above conclusions, it is recommended that testing proceed to the mixed waste runs in Task 1.5. These test runs will further confirm the results obtained during the surrogate testing. They will also confirm the value to the DOE marketplace of the VAC*TRAX technology on mixed waste.

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