Pyrolysis Autoclave Technology Demonstration Program for Treatment of DOE Solidified Organic Wastes

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ABSTRACT

In the summer of 2005, MSE Technologies Applications, Inc. (MSE) and THOR Treatment Technologies, LLC (TTT) conducted a demonstration test of the Thermal Organic Reduction (THOR^{sm}) in-drum pyrolysis autoclave system under contract to the Department of Energy. The purpose of the test was to demonstrate that the THOR^{sm} pyrolysis autoclave system could successfully treat solidified organic waste to remove organics from the waste drums. The target waste was created at Rocky Flats and currently resides at the Radioactive Waste Management Complex (RWMC) at the Idaho National Laboratory (INL). Removing the organics from these drums would allow them to be shipped to the Waste Isolation Pilot Plant for disposal.

Two drums of simulated organic setup waste were successfully treated. The simulated waste was virtually identical to the expected waste except for the absence of radioactive components. The simulated waste included carbon tetrachloride, trichloroethylene, perchloroethylene, Texaco Regal oil, and other organics mixed with calcium silicate and Portland cement stabilization agents. The two-stage process consisted of the THOR^{sm} electrically heated pyrolysis autoclave followed by the MSE off-gas treatment system.

The treatment resulted in a final waste composition that meets the requirements for WIPP transportation and disposal. There were no detectable volatile organic compounds in the treated solid residues. The destruction and removal efficiency (DRE) for total organics in the two drums ranged from >99.999% to >99.9999%. The operation of the process proved to be easily controllable using the pyrolysis autoclave heaters. Complete treatment of a fully loaded surrogate waste drum including heat-up and cooldown took place over a two-day period. This paper discusses the results of the successful pyrolysis autoclave demonstration testing.

INTRODUCTION

Over the past several years, the Idaho National Laboratory (INL) has been shipping waste contaminated with transuranic elements (TRU) to the Waste Isolation Pilot Plant (WIPP) for final disposal. The TRU was created at Rocky Flats and is stored at the INL's Radioactive Waste Management Complex (RWMC). Thousands of drums of TRU that contain high concentrations of volatile organic compounds (VOC) do not meet the WIPP waste acceptance criteria (WIPP-WAC)[1] and, therefore, cannot currently be shipped to WIPP. In addition, a portion of the TRU VOC waste contains polychlorinated biphenyls (PCB). Significant reduction of the VOC is required before shipment is possible. Reduction of the PCB is not required for shipment to WIPP but would be required for disposal of drums that do not contain enough TRU material to be classified as TRU.

Surrogate drums of the VOC/PCB TRU waste were processed in the Thermal Organic Reduction (THOR^{sm}) in-drum pyrolysis autoclave system to determine if the volatile organic compounds and polychlorinated biphenyls could be treated/removed such that the drums could be shipped to WIPP. Two drums of surrogate waste, one partially full and one completely full, were processed at the MSE facility in Butte, Montana. The objectives of the test were to: 1) determine the extent of removal/destruction of VOC and PCB, 2) to study process performance including heat-up processing and cool-down characteristics, and 3) to determine the composition and rate of release of gases from the in-drum pyrolysis autoclave.

Table I. Summary of Experimental Design Strategy

Test Objective	Data Need	Basis	Test Strategy
Determine extent of destruction/removal of VOC/PCB from surrogate waste as a result of treatment	Organic decomposition and mass loss, as determined by weighing, sampling, and analysis of pretest and posttest surrogate waste	Final waste composition meets requirements for WIPP transportation and disposal	Measure initial and final concentration of organics in surrogate waste
Characterize process during heat-up, treatment and cool-down of surrogate waste drum	Process temperature, pressure, nitrogen purge flow and cooling water flow during operation	In-drum pyrolysis system processing requirements for safety analysis and design basis	Measure process parameters while heating and cooling surrogate waste
Quantify major emissions as surrogate waste is slowly heated	Parts per million of NOx, CO, CO_2, HCl, total organic carbons (TOC), aromatics, and semivolatiles released during treatment	Offgas processing requirements for safety analysis and design basis	Measure process gas while heating surrogate waste

THOR^{sm} IN-DRUM PYROLYSIS AUTOCLAVE AND OFFGAS SYSTEM DESCRIPTION

The test system consisted of a one-drum, electrically-heated pyrolysis autoclave combined with the MSE off-gas treatment system. The MSE off-gas treatment system allowed safe processing of the pyrolysis gases from the pyrolysis autoclave for test purposes, however, the processing of

the pyrolysis gas from real waste would be conducted in a TTT steam reforming unit. Accordingly, the MSE off-gas systems are not discussed in detail.

Pyrolysis autoclave

The pyrolysis autoclave chamber (shown in Fig. 1) was installed in the A-bay area of Building 60 at the MSE Test Facility located in Butte, Montana. It consists of a 316H stainless steel chamber sized to enclose a either a 55-gal or an 85-gal overpack steel drum. Electrical resistance heaters located around the outside wall. These heaters are designed to heat the chamber and its contents up to >650° C (1,200° F), The surrogate waste material is heated to a core temperature of approximately >600° C (1,112° F). The pyrolysis autoclave chamber is designed to operate in the pressure range from a slight vacuum up to 15 psig. The system has a pressure relief valve set to actuate at 15 psig. The chamber is operated at a slight vacuum to ensure that pyrolysis gases are not forced into the work area and also to provide a minimal driving force for drawing air into the system if a leak develops. The pyrolysis chamber is purged with nitrogen to remove oxygen and to sweep pyrolysis gases out of the unit.

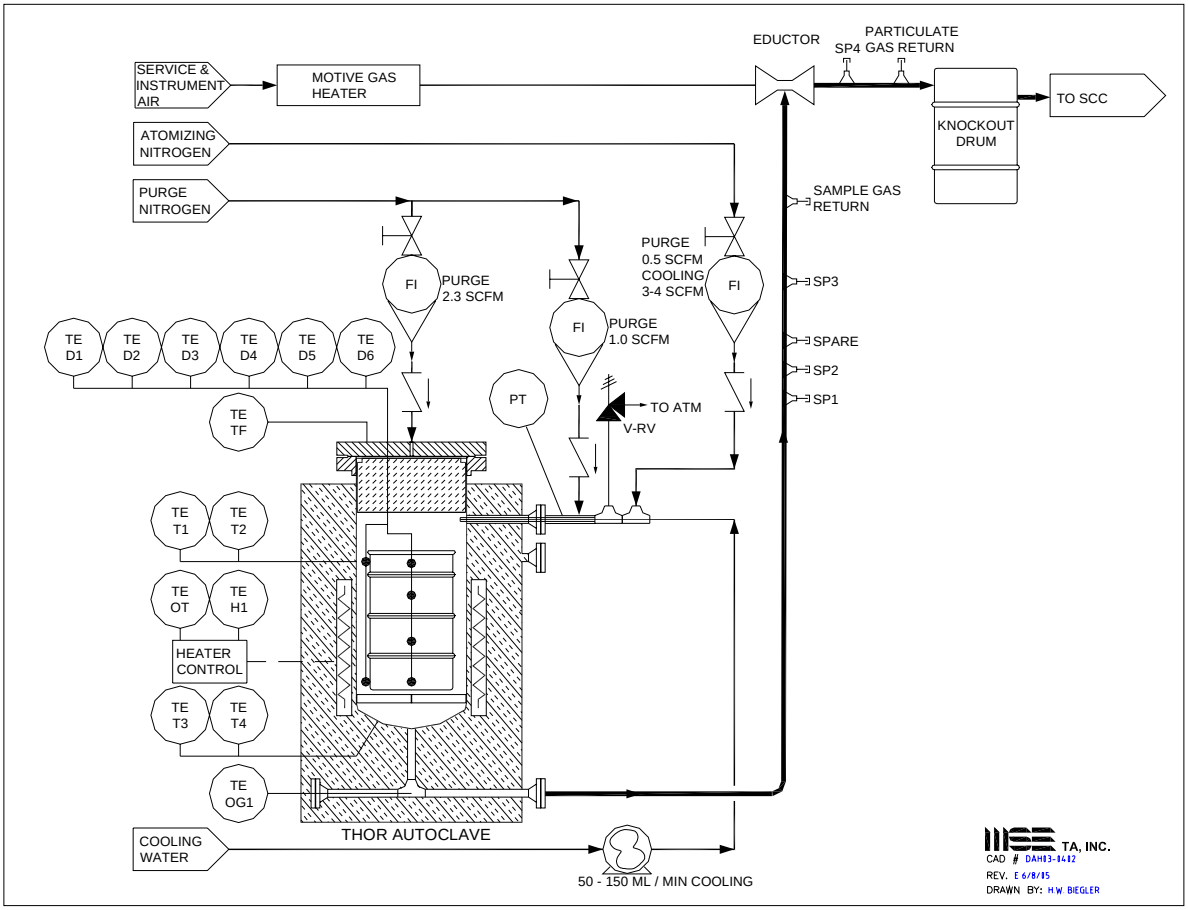

Fig. 1. TTT in-drum pyrolysis autoclave integrated to MSE test system

TE – Temperature element (thermocouple)
- TF – Pyrolysis autoclave head TE
- Dx – Drum internal TE and number
- Tx – Drum external and pyrolysis autoclave TE and number
- H1 – Heater controller TE
- OT – Heater over-temperature protection TE
- OG1 – Offgas TE

FI – Flow indication (rotameter)
PT – Pressure Transmitter
SPx – Sample Port and number
SCC – MSE offgas system
Note: Service and Instrument Air for Motive Gas Heater was supplied with nitrogen for pyrolysis test

MSE offgas system

The hot pyrolysis gases flow out of the chamber via a heated, insulated line, to an ejector pump driven by heated nitrogen, to a knock out pot, and then on to the MSE offgas system (Fig. 1.). The primary components of the MSE off-gas system are a secondary combustion chamber, a spray quencher, a caustic scrubber, various filters and a NOx reactor. Between the pyrolysis chamber and the ejector pump is a series of gas sampler ports, used to pull continuous and grab samples of the process gas so its composition, temperature, and flow rate can be measured (Fig. 1.).

SURROGATE WASTE DESCRIPTION

Two surrogate drums were treated during the test. One drum, referred to as the shakedown drum, was only about one-third full and was treated first to develop an understanding of operating characteristics under more controlled conditions. The second drum was a full surrogate drum. The composition of both drums is shown in Table II.

Table II. Composition of Surrogate Waste Drums

Organic Sludge Surrogate		Shakedown Drum Composition (wt %)	Shakedown Drum Mass (kg)	Full Surrogate Waste Drum Composition (wt %)	Full Surrogate Waste Drum Mass (kg)
Liquids					
Regal oil	Machine oil	28	16.9	28	49.4
Carbon tetrachloride	CCl_4	50	30.1	27	47.7
1,1,1-trichloroethane	$C_2H_3Cl_3$	0	0	9	15.9
Trichloroethylene	C_2HCl_3	0	0	7	12.3
Tetrachloroethylene	C_2Cl_4	0	0	7	12.3
Hexachlorobenzene	C_6Cl_6	0	0	400 ppm as PCB surrogate [a]	0.0631
Biphenyl	$C_{12}H_{10}$	0	0		0.0076
Liquids Subtotal		78	47.0	78	137.7
Solids					
Microcel E®	$CaSiO_3$	14	8.5	14	24.7
Kitty litter	Oil-dry	8	4.8	8	14.1
Solids Subtotal		22	13.4	22	38.8
Surrogate Subtotal		100	60.4	100	176.5
Other					
Absorbent (Kitty litter)		N/A[b]	4.7	N/A	8.7
90-mil HDPE liner and lid		N/A	7.3	N/A	7.3
Drum		N/A	22.9	N/A	22.9
Other Subtotal		N/A	34.9	N/A	38.9
TOTAL		N/A	95.3	N/A	215.4

[a] PCB surrogates, hexachlorobenzene and biphenyl, such that a PCB concentration of 400 ppm is simulated.

[b] Absorbent not part of surrogate, 90-mil high-density polyethylene (HDPE) liner and lid, and drum are not considered in the surrogate waste composition, therefore they are not applicable (N/A).

The surrogate composition is based on the solidified organic stream known as Idaho Disposal Code 3 (IDC 3), also known as 743-Series waste. Miller [2] describes the process that created the waste as follows:

> The large majority of organics in RFP waste streams were derived from organic "setups" known as 743-series waste. The waste is called 743-series waste because it was processed into sludge in the RFP Building 774 and was later coded at the INL as Content Code 3 organic waste to distinguish it from different types of waste from RFP Building 774 that were shipped to the INL. Processing the liquid radioactive mixed waste into sludge allowed it to be shipped to the INL for disposal.

This waste stream contained liquid organic waste generated by various plutonium and non-plutonium operations at the Rock Flats Plant. These liquid wastes were mixed with calcium silicate (Microcel-E) to form a thick grease or paste-like material. Small amounts of Oil-Dri absorbent were usually mixed with the wastes. Small amounts of Oil-Dri absorbent were also added to the drum holding the waste.

This waste stream is considered the defining stream for two main reasons: first, on a per drum basis, it contains the bulk of the organics that need to be processed; and second, it contains the overwhelming volume, mass, and number of drums to be processed (estimated at 80%).

Processing of one of these drums results in the greatest total volumetric release of gases, the most variety of released gases, and the greatest release rate of gases, principal design criteria for the sizing of the process for treating this waste. In addition, the processing of all the drums in this series represents the majority of drums to be processed, the main criterion for estimating how long it will take to treat the waste.

IN DRUM PYROLYSIS AUTOCLAVE AND OFFGAS PROCESS DESCRIPTION

To process a surrogate waste drum, the drum was first placed into the pyrolysis autoclave chamber using a lifting device. An insulating plug was placed on top of the drum. Leads from the thermocouples, associated with the drum, were connected to provide temperature indications on the process control system. The chamber lid was bolted into place and a pressure test of the system was completed. Nitrogen was used to purge air from the chamber so that oxygen was less than 1% in the autoclave. The heaters were activated and heat-up of the drum was started. During the heating process, temperature indications from the thermocouples in the pyrolysis autoclave chamber and on the outside and inside of the drum, including temperatures in the waste, were monitored. Volatilization/pyrolysis of the various organic components in the waste surrogate was identified by the temperature versus time plots for the thermocouples located inside the drum. As the temperature rose, the organics volatilized and/or pyrolyzed into short-chain hydrocarbons. The process gas from the pyrolysis autoclave chamber consisted predominately of short-chain hydrocarbons, hydrogen, CO_2, CO, water vapor, nitrogen, acid gases, such as HCl, and measurable benzene and other aromatics (0.25 to 1 wt%).

Heating was continued until the temperature at the core of the drum reached a predetermined level. This level was based on the known physical properties of the organics in the drum, typically the temperature at which all of the organics or, alternatively, just certain target organics, were volatilized and pyrolyzed.

After the heaters were turned off, the system began to cool. To accelerate the cooldown a proprietary cooling method was used. When the outside drum temperature reached ~175° C (347° F), the drum was removed from the chamber and set aside to continue cooling. The next drum was processed as soon as the first drum was removed from the unit.

A slight vacuum was maintained in the pyrolysis autoclave chamber by monitoring the chamber pressure and regulating the flow rate of the heated motive nitrogen to the ejector pump (see Fig. 2). The pyrolysis process gas passed through a hot oil knockout drum to remove the heavier, condensable organics. The process gas then flowed to the secondary combustion chamber (SCC), where it was combusted with air and natural gas at 1,093° C (2,000° F). The SCC converted hydrogen, CO, and organic vapors to water and CO_2. The process gas from the combustion chamber was rapidly quenched in the quench vessel, reducing the temperature of the gas to less than 16° C (60° F) in a fraction of a second preventing the formation or reformation of dioxins and furans. The process gas then passed into the contact condenser/scrubber, which worked in conjunction with the quench vessel to provide intimate contact between the process gas and a sodium hydroxide scrub solution removing acid gases, such as HCl. The process gas next passed through a high-energy scrubber. The high-energy scrubber worked by passing the offgas through a curtain of high-velocity, atomized scrubber liquor droplets. The liquor droplets scrubed

particulates from the gas stream. The mist in the offgas was removed as it passed through a demister and was returned to a common scrubber liquid sump. The offgas was heated and then passed through a fabric baghouse, removing any trace salt particulates and metals from the gas stream. The gas was filtered again through a series of two high-efficiency particulate air (HEPA) filters with the second HEPA filter having a sulfur-impregnated charcoal layer. A variable-speed positive displacement blower provided the motive force for the system with a bypass valve that provided additional system flow control.

The oxygen analyzer in the offgas system was used to determine burner combustion efficiency. The heatup rate of the pyrolysis autoclave chamber was used to regulate the rate of hydrocarbons released into the SCC. Supplemental air was also added at the inlet of the SCC to improve combustion and allow a faster heatup rate of the pyrolysis autoclave.

OFFGAS MONITORING

A pitot tube with a thermocouple was installed at SP-1 (see Fig. 1). The differential pressure and temperature of the pitot tube was measured and recorded using a Testo Model 350 XL emission analyzer and by hand using a digital Heise differential pressure gage.

Emission analyzer gas sampling was from SP-2 (Fig. 1). The analyzer measured concentrations of O_2, CO, CO_2, NO, NO_2, and total hydrocarbon (THC). A calculated hydrogen analysis was also provided

Tedlar bags (1 L) were used to pull process gas samples for VOCs, benzene, other aromatics, semivolatiles, and HCl/Cl_2 analyses at SP-3(Fig. 1). The VOCs of interest were carbon-tetrachloride (CCl_4); trichloroethene (C_2HCl_3, TCE, or trichloroethylene); 1,1,1-trichloroethane ($C_2H_3Cl_3$ or 1,1,1-TCA); tetrachloroethene (C_2Cl_4, PCE, tetrachloroethylene, or perchloroethylene); hexachlorobenzene (C_6Cl_6 or HCB); biphenyl ($C_{12}H_{10}$), benzene, and Total Hydrocarbons (THC).

The HCl/Cl_2 sampling and analysis procedure used was an adaptation of EPA Method 0051. Two impingers were used, one containing a sulfuric acid solution and one containing a sodium hydroxide solution.

The Tedlar bag samples were taken using battery-operated vacuum boxes. A preliminary gas sample was drawn into a sacrificial bag to purge the sampling line before taking the analysis sample. The nominal sampling rate for the bag samples was approximately 21 L/min. Samples were taken at 2-hr intervals and analyzed by MSE.

A sample port was installed at SP-4 (Fig. 1) with the inlet of the sample port facing upstream at the center of the process gas line. A Thermo Anderson Model DR-4000 aerosol monitor was used to sample and analyze for total particulate, temperature, and relative humidity of the process gas.

RESULTS

The results for the shakedown drum and the full surrogate drum were essentially the same. The discussion of results will focus on the full surrogate drum since it better represents the expected feed for the system. The discussion is divided into three sections reflecting the objectives shown in Table I.

Performance (heatup, treatment, and cooldown)

The full surrogate drum was placed in the pyrolysis autoclave on May 13, 2005. The pyrolysis autoclave was successfully pressure tested and then configured for operation. Heating started on May 13, 2005. Heater power was increased gradually over time as the pyrolysis autoclave temperature increased. The heater power was turned down when the absorber/scrubber pH went down to 2. At that time it was determined that the caustic addition pumps were not operating. As a result, heater power was further reduced. The heaters remained at this lower power level until the caustic addition pump was put back into service. At this point, the heater power was turned up, and then gradually increased over time. Heater power was reduced when the offgas eductor plugged. The plug was cleared by increasing the motive nitrogen flow rate until the plug was dislodged. The eductor plugged a second time and the same method was used to clear the plug.

Heater power was again increased after the second eductor plug was cleared. Following an extended period of heating, the absorber pH increased to 11. At this time, the pyrolysis autoclave temperature had reached >650° C so the heater power was reduced. The pyrolysis autoclave was held at >650° C by adjusting power inputs as needed until the minimum centerline temperature of the drum reached 672° C. The heater power was shut off and the drum cooling started on May 14, 2005.

The maximum temperatures in the core of the drum ranged from 701 to 715°C. The center core temperatures continued to increase for a short period of time after the heater was de-energized as the drum internal heat front continued to move to the center of the drum.

Initially the drum was cooled by the nitrogen purges. After about a short period of cooling with the purges, the water cooler was started and continued until the pyrolysis autoclave temperature (T-3) was 200°C. The drum exterior temperature at this time was 161°C and the centerline temperature was 668°C. The water cooler was shut off. As in the shakedown drum test, the temperatures in the pyrolysis autoclave and exterior of the drum rebounded slightly after the water cooler was shut off.

The pyrolysis autoclave lid was taken off and the treated drum was removed from the pyrolysis autoclave on May 15, 2005. There was no smoke or vapors observed exiting from the drum after it was removed from the pyrolysis autoclave, nor was there any odor of pyrolyzed hydrocarbons. The final weight of the drum was 93.4 kg.

Offgas emissions from the pyrolysis autoclave

Fig. 2. shows the offgas hydrogen; total acid gases and total VOCs concentration in the pyrolysis autoclave offgas along with the average temperature of the centerline drum core thermocouples as a function of time. VOCs evolution started essentially at the beginning of the test and peaked when the pyrolysis autoclave core temperature reached the normal boiling points of these compounds: 74.1° C for TCA, 76.8° C for carbon-tetrachloride, 87.2° C for TCE and 120.8° C for PCE.

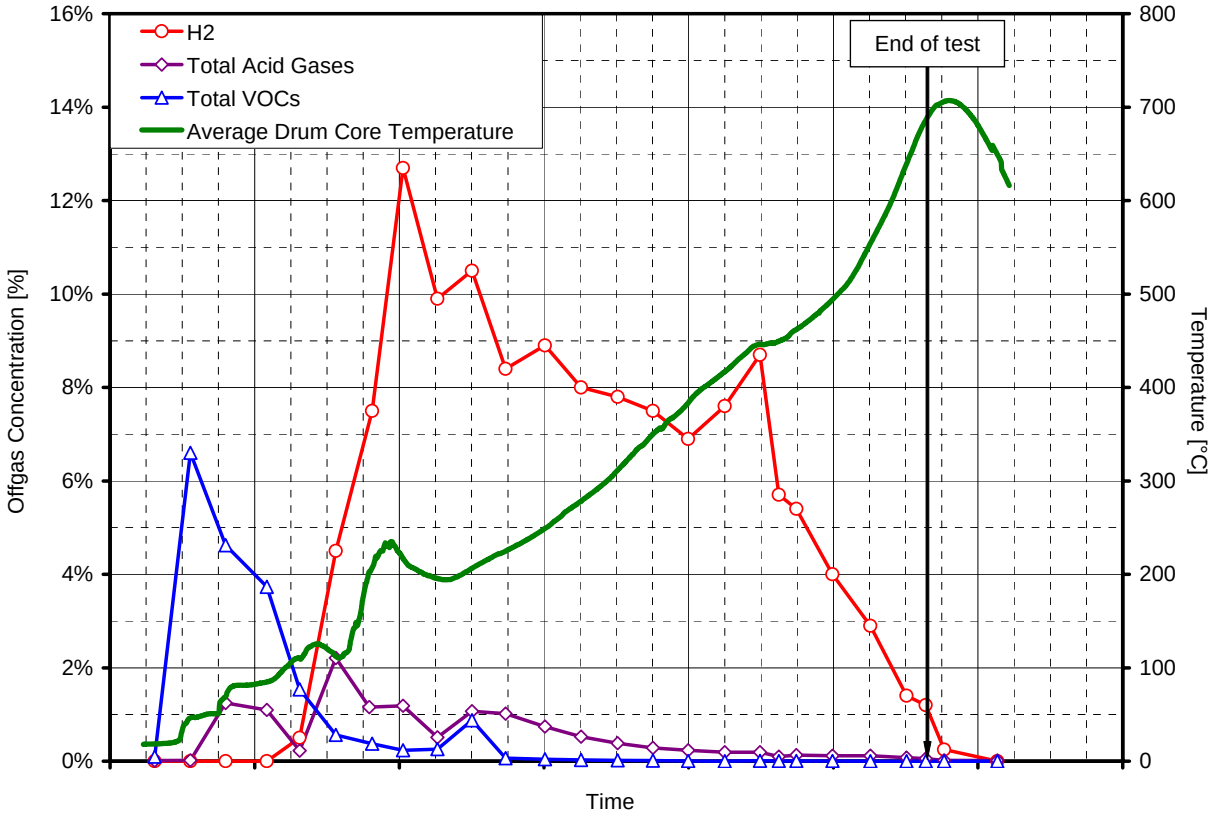

Fig. 2. Full surrogate waste drum test – offgas H_2, total acid gases, and total VOCs concentrations and average drum core temperatures as a function of time. Total Acid Gases = sum of HCl and Cl_2 concentrations. Total VOCs = sum of 1, 1, 1-trichloroethane, carbon tetrachloride, trichloroethylene, and tetrachloroethylene concentrations. Average Drum Core Temperature = Average of 4", 12" and 24" drum centerline thermocouples

Note that the drum core temperatures remained constant until each of these compounds boiled off from the simulated waste in the drum. It is probable that evaporation of the VOCs kept the drum contents below the hydrocarbon pyrolysis temperatures until the chlorinated organics were essentially gone from the drum. The VOCs evolution rate dropped off quickly and VOCs were essentially gone from the drum once the average core temperature exceeded 200° C.

Acid gas evolution started about the same time the VOCs evolution rate peaked, indicating pyrolysis of the chlorinated hydrocarbons was taking place. The peak in acid gas concentrations

occurred when the drum core temperatures had reached only ~ 100 – 150° C, in contrast to the shakedown drum which had the acid gas peak at ~ 200 – 350° C. This is probably due the different surface area/volume ratios of the drums, which would lead to a steeper temperature profile in full surrogate drum. Pyrolysis reactions would tend to occur at lower core temperatures in the full surrogate drum because of this steeper temperature profile. Acid gas pyrolysis reactions were essentially complete once the average core temperature reached ~ 450° C.

Hydrogen started to evolve from the drum when the drum core temperature was ~ 100° C, indicating that hydrocarbon pyrolysis reactions were taking place in the drum, particularly at the outer surfaces. Again, the steeper temperature profile in the full surrogate drum would cause hydrogen generation to take place at a lower core temperature than in the shakedown drum. The hydrogen concentration peaked when the core temperature reached ~ 225° C. The hydrogen concentration dropped more-or-less steadily from the peak until the end of the test. When the test was terminated, the hydrogen concentration had dropped to ~ 1% indicating that pyrolysis reactions in the simulated waste were essentially complete.

Extent of organic destruction/removal

The pretest surrogate and the solids residue remaining in the drum after treatment in the pyrolysis autoclave were chemically analyzed for various chlorinated organics, hydrocarbons, and PCB surrogates. The analyzed total weight percent of organic in the pretest surrogate was 81.2% by weight, in good agreement with the expected value of 78% by weight. As with the shakedown drum surrogate, the biphenyl concentration was higher than the makeup specification, probably as a result of traces of biphenyl contamination in the machine oil used to make up the surrogate. Specifically, the biphenyl was approximately twice that of the target.

No detectable amounts of the makeup VOCs remained in the simulated waste solid residues after processing in the pyrolysis autoclave. The only detectable residual organic compound that remained in the drum residue was chloromethane. The residual hydrocarbon concentration was very low; ranging from a minimum of 0.8 mg/kg, to an average of 1.4 mg/kg; up to a maximum of 1.8 mg/kg. Using the average value, the total organics destruction and removal efficiency (DRE) for the simulated waste in the drum was calculated to be 99.99994%. PCB surrogates were added to the simulated waste drum to give 402.1-mg/kg initial concentration. The PCB surrogate concentration in the drum after processing was only 0.1 mg/kg. The PCB surrogate DRE was calculated to be 99.992%.

The surrogate Loss on Ignition (LOI) test showed that 81.2% of the surrogate was lost during high temperature (up to 1000° C) oxidation. This is in good agreement with the target value of 78%. The LOI for the residue averaged 32.5% due to the presence of inorganic carbon char in the solids residues from the pyrolysis of the long chain organic wastes. The final drum weight was 93.4 kg, indicating a loss of 122.0 kg of surrogate, and leaving 70.5 kg of surrogate solid residues (subtracting out the weight of the steel drum). The total non-volatile portion of the surrogate fed was 47.5 kg, including the 8.7 kg of absorbent that was mixed with the surrogate. Thus, the expected remaining inorganic carbon char from the organics was 23.0 kg and the expected LOI is 32.6%. Once again, there is excellent agreement between the expected and actual values.

CONCLUSION

The THOR[sm] Treatment Technologies drum pyrolysis autoclave was used to treat two drums of simulated waste at the MSE Test Facility in Butte, MT during May 10-15, 2005. The processing resulted in a final waste composition that meets the requirements for WIPP transportation and disposal. The operation of the process proved to be easily controllable using the pyrolysis autoclave heaters. Complete treatment of a fully loaded surrogate waste drum including heat-up and cooldown took place over a 2-day period.

Processing of the shakedown drum was somewhat slowed by low oxygen levels in the secondary combustion chamber (SCC) which required operations at lower power levels until additional air was fed to the SCC to increase the oxygen concentration in the offgas. Adjusting the pyrolysis autoclave power successfully controlled the oxygen concentration in the offgas from the SCC. This demonstrated that the evolution rate of organics and hydrocarbons was controllable by occasionally adjusting the pyrolysis autoclave power input levels.

Analysis of gas samples taken during the shakedown drum test shows that the volatile chlorinated organic compound (CCl_4) evaporated from the drum very early during the heating process. Acid gas (HCl and Cl_2) evolution started when the CCl_4 offgas concentration peaked and continued until the end of the test. Hydrogen started to be evolved once the drum core temperatures reached 200 – 350° C; indicating pyrolysis reactions were taking place in the drum. Hydrogen continued to be evolved until the end of the test. The THC analyzer failed during the shakedown drum test and no THC results are available for this test.

The solids residue remaining in the shakedown drum was chemically analyzed after the test. The residue contained very low concentrations of organic compounds. The LOI for the residue was 40.4%, in excellent agreement with the LOI of 40.8% calculated based on feed composition and final drum weight. There was no detectable amount of CCl_4 in the solids residue. The DRE for total organics in the drum was 99.99993%. Although no PCB surrogates were added to the shakedown drum, sufficient biphenyl was present as a contaminant to allow the DRE for biphenyl to be calculated. The biphenyl DRE was 99.1%.

Processing of the full surrogate waste drum took 47.6 hours. Again, problems with the offgas treatment system required operation at reduced power levels for short durations of time, which demonstrated that the organics evolution rate could be controlled by adjusting the heater power level. The full simulated waste drum was processed until the drum centerline temperature reached >650° C. The hydrogen concentration in the offgas roughly correlated with the centerline temperature in the drum.

Analysis of gas samples taken during the simulated waste drum #1 test shows that the volatile chlorinated organic compounds (VOCs) evaporated from the drum very early during the heating process. The drum core temperature was relatively constant during evaporation of the VOCs. As in the shakedown drum test, acid gas (HCl and Cl_2) evolution started when the VOCs offgas concentration peaked and continued until the end of the test. Hydrogen started to be evolved once the drum core temperatures reached 100 – 150° C; indicating pyrolysis reactions were taking place in the drum.

The drum core temperatures in the full surrogate waste drum test were lower than the shakedown drum test when pyrolysis reactions started because this drum had a steeper temperature profile than the shakedown drum. The hydrogen concentration peaked when the average drum core temperature was ~ 225° C. Hydrogen continued to evolve until the end of the test; however, the H_2 offgas concentration fell steadily during the test after the peak. The H_2 offgas concentration at the end of the test was ~ 1%, indicating that pyrolysis reactions were nearly complete when the test was terminated. The THC analyzer failed during the shakedown drum test and no THC results are available for the full surrogate waste drum test.

The solid residue remaining in the full surrogate waste drum was chemically analyzed after the test. The residue contained very low concentrations of organic compounds. The LOI for the residue was 32.5%, in excellent agreement with the LOI of 32.6% calculated based on feed composition and final drum weight. There were no detectable amounts of VOCs in the solids residue. The only detected organic compound in the solids residue was chloromethane at an average concentration of 1.4 mg/kg. The DRE for total organics in the drum was 99.999994%. PCB surrogates (biphenyl and hexachlorobenzene) were added to the full surrogate waste drum to give an initial concentration of ~ 400 ppm. The PCB surrogate concentration in the solid residue at the end of the test was 0.10 mg/kg. The DRE for PCB surrogates was 99.992%.

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