# THOR<sup>SM</sup> DEMONSTRATION TESTING RESULTS—THE THOR<sup>SM</sup> AUTOCLAVE SYSTEM DESTROYS ORGANICS, REDUCES VOLUME, AND REMOVES PROHIBITED ITEMS FROM DRUMS OF SIMULATED TRU WASTE

M. Cowen, J. B. Mason, D. Schmoker, P. Bacala, K. Ryan THOR Treatment Technologies, LLC 106 Newberry Street SW, Aiken, SC 29801

# ABSTRACT

In the first quarter of 2004, THOR Treatment Technologies, LLC (TTT) conducted a demonstration test of its THOR<sup>SM</sup> in-drum autoclave system under contract to Washington TRU Solutions, LLC. The purpose of the test was to demonstrate that the THOR<sup>SM</sup> system provides a solution for many transuranic (TRU) waste shipping and disposal problems found throughout the Department of Energy (DOE) complex. The specific test objectives were to demonstrate that the THOR<sup>SM</sup> in-drum autoclave treatment system 1) removed prohibited items (liquids, aerosol cans, and sealed containers greater than 4 liters) from drums of simulated TRU waste, 2) provided organic volume reduction, 3) provided efficient processing times, and 4) provided a preliminary qualitative demonstration of containment control. This paper discusses the results of the demonstration testing.

# INTRODUCTION

Up to 40% of TRU waste in the DOE complex does not meet shipping or disposal requirements because it contains prohibited items, such as free liquids, aerosol cans, or sealed containers greater than 4 liters. Removing these prohibited items by sorting and repackaging is costly, time consuming, and increases worker radiation exposure risk. This demonstration was conducted to determine the effectiveness of the THOR<sup>SM</sup> autoclave system in providing an alternate means of removing prohibited items.

In addition, the demonstration test was also conducted to determine the extent to which the THOR<sup>SM</sup> autoclave system would destroy the organic content of the drums, which would provide several benefits. Organic destruction would solve the problem of gas generation caused by alpha radiolysis of organics in certain waste streams, whose shipment is currently prevented by gas generation above acceptable limits. Organic destruction would also provide a mass and volume reduction. The mass reduction could provide an alternate means for disposal of waste with Curie concentrations less than 100 nCi/g because the radionuclide concentration would be higher in the processed residue. The volume reduction could also provide shipping and disposal cost savings.

# THE THOR<sup>SM</sup> AUTOCLAVE DEMONSTRATION UNIT

The test system consisted of a one-drum, electrically-heated autoclave and a simplified off-gas system consisting of a condenser, water seal pot, and exhaust blower. The condenser served to condense water and most organics to a liquid that could be shipped for disposal. The water seal pot was used to prevent oxygen ingress into the autoclave. It should be noted that an operational THOR<sup>SM</sup> system utilizes a steam reforming system for off-gas treatment, and that this test system cannot be used to obtain representative emissions data. A picture of the system, without insulation blankets, is shown in Figure 1. The key hardware items are:

### Autoclave

This is the core piece of equipment, designed to heat the drums and contents to  $>923^{\circ}$ K (650°C). It is a 168.9 cm high by 81.3 cm outside diameter vessel designed to hold a 208-liter (55-gallon) drum or a 208-liter drum overpacked inside an 321.8-liter (85-gallon) drum. It is equipped with ports for off-gas removal, gas purges, pressure measurement, temperature measurement, and is fitted with external electrical resistance heaters.

### Condenser

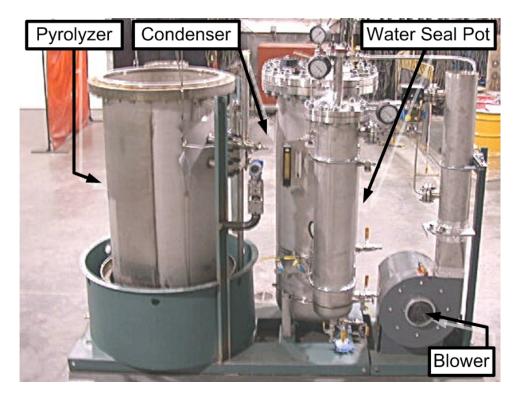
The process gases from the autoclave flow to the condenser, which is designed to remove water vapor and condensable tars and oils from the inert purge gas. To accomplish this, cooling water is recirculated by a centrifugal pump through a spray nozzle in the top of the vessel to provide intimate gas-liquid contact. It is equipped with a level gage and other ports for pressure and temperature measurement. Heat rejection, as required, is accomplished by cooling water flowing through a coil in the bottom of the vessel.

#### Water Seal Pot

The process gases from the condenser pass through the water seal pot, which is designed to isolate the oxygen-deficient autoclave system from the ambient gas atmosphere.

#### **Off-gas Blower**

System and area ventilation are provided by a 1,000 cfm blower. The blower draws in room air and discharges it into the autoclave skid's off-gas stack. In the stack, the ambient air is mixed with the cooled purge gases passing through the pressure control valve, forming the off-gas stream that is released to the environment.



# Fig. 1. The THOR<sup>SM</sup> Drum Autoclave Test System Shown Without Insulation Blankets

The autoclave test system is operated via two controllers. The first is the heater controller, located on the heater control panel, which controls the heat input rate to the simulated waste drum in the autoclave. This control panel features a breaker switch, power switches, and a digital controller, which controls the percent of maximum power output of the autoclave heaters, as set by operator input.

The second controller is a digital control unit used to open and close the system's pressure control valve, as determined by the system operator. In addition to controlling the pressure within the system, the controller also displays the system pressure for easy monitoring by the test operators.

The operator control station is the primary location from which the system operator monitors and controls the autoclave system. Located at the control station is a total hydrocarbon (THC)/oxygen analyzer, used to monitor oxygen levels and autoclave gas evolution in the autoclave during system operation. A thermocouple digital data logger is also located at the control station to monitor temperatures.

### SIMULATED WASTE DRUM ASSEMBLY

A total of ten 208-liter drums of simulated TRU debris waste were tested in the THOR<sup>SM</sup> autoclave test unit. Each drum contained a standard 90 mil high density polyethylene (HDPE) plastic liner. The drums ranged in weight from 35.4 to 161.9 kilograms. The simulated waste contained prohibited items and typical debris wastes, such as paper, paint brushes, plastic hose, electrical wiring, mop heads, concrete, gypsum wall board, etc. The prohibited items in the waste consisted of free water, aerosol cans, and sealed paint cans (both 3.8-liter [1-gallon] and 18.9-liter [5-gallon] paint cans). The 3.8-liter and 18.9-liter paint cans were assembled with typical waste sealed inside (water, paint, plastic, scrap metal). Aerosol paint cans and cans of WD-40, ranging from half-full to empty were placed in the waste. All of the simulated waste was sealed inside 3 layers of plastic and taped. The specific contents of each drum are listed in Table I.

In order to provide a preliminary qualitative demonstration that the system was capable of containment control, a fluorescent mineral powder (Willemite) was added to three drums that were then placed inside a THOR<sup>SM</sup> overpack container with an integral, high-temperature filter designed to retain fine particulates in the overpack while allowing gases to pass in and out freely. Willemite was selected because its melting point was higher than the processing temperature of the autoclave. Black light inspection was conducted post-processing to determine if the Willemite had been retained inside the overpack container.

Drum	Contents of Drum				
No.	(All 208-liter drums contained a drum liner)				
1	7.6 liters of water and 5 milliliters of Willemite were placed in the bottom of a 208-liter drum. The 208-				
	liter drum was then placed inside the 321.8-liter overpack having an integral, high-temperature filter.				
2	208-liter drum with four 3.8-liter paint cans:				
	• 0.473 liters of water was sealed inside the first 3.8-liter paint can.				
	• 0.237 liters of paint was sealed inside the second 3.8-liter paint can.				
	<ul> <li>Scrap metal was sealed inside three layers of plastic bags, then the bags were sealed inside the third 3.8-liter paint can.</li> </ul>				
	• Scrap metal was sealed inside the fourth 3.8-liter paint can (no liquids, no plastic).				
3	A 208-liter drum having the following contents:				
	<ul> <li>Scrap metal was sealed inside three layers of plastic bags, then the bags were sealed inside an 18.9-liter paint can.</li> </ul>				
	• Five cans of spray paint emptied of their contents.				
	• Five WD-40 cans emptied of their contents.				

### Table I. Drum Contents

4	A 208-liter drum having the following contents:			
	<ul> <li>Scrap was metal sealed inside three layers of plastic bags, then the bags were sealed inside liter paint can.</li> </ul>			
	• One can of WD-40, containing approximately 25% of its original contents.			
	• A 3.8-liter paint can, filled 75% full with anion exchange resin.			
	<ul> <li>Two sets of heavy latex shoe covers.</li> </ul>			
	• One set of lighter latex shoe covers.			
5	A 208-liter drum having the following contents:			
	• 4.6 meters of plastic hose sealed inside three layers of plastic bags.			
6	A 208-liter drum having the following contents:			
	<ul> <li>0.237 liters of paint was sealed inside an 18.9-liter paint can, then the paint can was sealed inside three layers of plastic.</li> </ul>			
	<ul> <li>Concrete blocks as needed to bring the total drum weight to 158.8 kilograms. The concrete blocks were sealed in three layers of plastic.</li> </ul>			
7	A 208-liter drum, having the following contents:			
	<ul> <li>Forty absorbent wipes were moistened with water, and approximately .042 cubic meters of craft paper was loosely bunched together, then the wipes and craft paper were sealed in three layers of plastic.</li> </ul>			
	<ul> <li>Scrap metal was sealed inside three layers of plastic bags, then the bags were sealed inside a 3.8- liter paint can.</li> </ul>			
	<ul> <li>0.473 liters of paint was sealed inside an 18.9-liter paint can, then the paint was can sealed inside three layers of plastic.</li> </ul>			
	<ul> <li>One aerosol can of WD-40 emptied of its contents, then the aerosol can was sealed in three layers of plastic.</li> </ul>			
	<ul> <li>Two cellulose mop heads were saturated with WD-40, then the mop heads were sealed in three layers of plastic.</li> </ul>			

8	A 208-liter drum having the contents listed below. The 208-liter drum was placed inside the 321.8-liter overpack having an integral, high-temperature filter.
	• Approximately 3.8 liters of free water was placed in the bottom of the drum.
	<ul> <li>Approximately 3.8 liters of scrap metal was sealed in three layers of plastic. Approximately 5 milliliters of Willemite powder was placed in the third plastic bag before sealing. The bags were placed in an 18.9-liter paint can and sealed.</li> </ul>
	<ul> <li>0.237 liters of water sealed in a 3.8-liter paint can and sealed, then the can was sealed in three layers of plastic.</li> </ul>
	<ul> <li>One aerosol can of WD-40 emptied of its contents, then the aerosol can was sealed in three layers of plastic.</li> </ul>
	<ul> <li>Forty absorbent wipes moistened with water, and approximately .042 cubic meters of craft paper loosely bunched together. The wipes and craft paper were then sealed in three layers of plastic.</li> </ul>
	• 30.5 meters of 12 gage insulated electrical wiring was sealed in three layers of plastic.
	<ul> <li>Five 0.3-meter square pieces of 12.7-millimeter thick dry gypsum sheet rock sealed in three layers of plastic.</li> </ul>
	<ul> <li>Concrete blocks as needed to bring the total drum weight to 68 kilograms. The concrete blocks were sealed in three layers of plastic.</li> </ul>
9	A 208-liter drum having the contents listed below. The 208-liter drum was then placed inside the 321.8-liter overpack having an integral, high-temperature filter:
	<ul> <li>Approximately 3.8 liters of scrap metal was sealed in three layers of plastic. Approximately 5 milliliters of Willemite powder was placed in the third plastic bag before sealing. The bags were placed in an 18.9-liter paint can and sealed.</li> </ul>
	<ul> <li>0.473 liters of paint was sealed in a 3.8-liter paint can, then the paint can was sealed in three layers of plastic.</li> </ul>
	• One aerosol can of WD-40 emptied of its contents, then sealed in three layers of plastic.
	• 0.426 meters of plastic hose was sealed in three layers of plastic.
	<ul> <li>Dried Bartlett Stripcoat TLC (strippable coating) that was sprayed on a flat metal surface then stripped off and bunched together (approximately 3.8 liters in volume when loosely bunched together), cellulose filter cartridges (approximately 0.42 cubic meters in volume). All of this material was sealed in three layers of plastic.</li> </ul>
	<ul> <li>Concrete blocks as needed to bring the total drum weight to 113.4 kilograms. The concrete blocks were sealed in three layers of plastic.</li> </ul>

10	A 208-liter drum, having the following contents:	
	• Approximately 3.8 liters of scrap metal sealed in three layers of plastic. Approximately 5 milliliters of Willemite powder was placed in the third plastic bag before sealing. The bags were placed in an 18.9-liter paint can and sealed.	
	<ul> <li>Approximately 0.473 liters of paint sealed inside a 3.8-liter paint can, then the can was sealed inside three layers of plastic.</li> </ul>	
	• One aerosol can of WD-40 emptied of its contents, then sealed in three layers of plastic.	
	• Two cellulose mop heads saturated with WD-40, then sealed in three layers of plastic.	
	• Three new 101.6-millimeter paintbrushes with plastic handles sealed in three layers of plastic.	
	<ul> <li>Concrete blocks, as needed to bring the total drum weight to 158.8 kilograms. The concrete blocks were sealed in three layers of plastic.</li> </ul>	

Six thermocouples were placed within the drum contents and on the drum surfaces in order to gather temperature data. The plastic drum liner lid was then put in place, followed by the metal drum lid. The thermocouple wires were routed through ~25.4 millimeter holes punched through each lid. The closure ring was then installed and bolted in place on the 208-liter drum.

Drums 2, 3, 4, 5, 6, 7, and 10 were 208-liter drums that were directly loaded into the autoclave. The assembled 208-liter drum is shown in Figure 2. Note the thermocouple wires emerging from the top of the drum and the banding on the drum for external thermocouple attachment.

Drums 1, 8, and 9 were 208-liter drums placed into an 321.8-liter overpack having an integral, high-temperature filter, also shown in Figure 2. The thermocouple wires from the 208-liter drum were routed through special fittings on the side of the overpack drum, as shown in figure 2. Willemite was added to a specific item inside the inner 208-liter drum, as listed in Table I. A black light inspection was performed post-testing to qualitatively determine the extent to which the powder was retained inside the overpack during autoclave treatment.



Fig. 2. Assembled 55-gal. drum (left) and assembled 85-gal. overpack containing a 55-gal. drum (right)

# AUTOCLAVE TEST OPERATIONS

The drum to be tested was placed in the autoclave, the insulation plug was then put into place, and the autoclave lid was secured. The thermocouple wires were connected into the data logger system and final operational checks were completed to ensure the system was ready for heating.

The heaters were initially set to 50% power and then increased to 90–100% power after about ten minutes. Heater power was sequentially lowered as the system heated up to the target temperature of 923°K. Process gas samples were periodically drawn off the bottom of the autoclave and run through the THC/oxygen analyzer to monitor the percent total hydrocarbons and oxygen. The autoclave pressure was also monitored and recorded during heat-up.

After all the drum thermocouples reached 923°K, the heaters were shut off and the cool-down was started. The test operators recorded system and drum temperatures every 15 minutes during the entirety of the heat-up and cool-down periods.

At the end of the autoclave cool-down period, the lid and insulation plug were removed, the drum thermocouple wires were disconnected, and the drum was removed from the autoclave.

### **POST-TEST INSPECTIONS**

After an additional cooling period, the drum was disassembled and inspected in compliance with procedures. The results of the inspection were recorded on the Post-Treatment Drum Observation Record. The inspection consisted of:

- Observing condition of the exterior of the drum (or overpack), including lid and closure ring
- Weighing the entire package
- If fluorescent Willemite was added to the package, inspecting all areas of the package and the interior of the autoclave with a black light for the spread of Willemite
- If overpacked:
  - $\checkmark$  Removing the overpack lid and inspecting the filter
  - ✓ Removing the 208-liter drum from the overpack
  - $\checkmark$  Observing the condition of exterior of the 208-liter drum
  - ✓ Weighing the 208-liter drum and empty overpack
- Removing the 208-liter drum lid and observing contents
  - ✓ Observing the condition of drum lid gasket
- Removing and inspecting the 208-liter drum contents:
  - ✓ Checking for free liquids
  - ✓ Inspecting aerosol cans and checking for indications of breach
  - ✓ Inspecting exteriors of 3.8-liter and 18.9-liter paint cans and checking for indications of breach
  - ✓ Performing bubble leak tests on 3.8-liter paint cans, if necessary to determine breach
  - ✓ Inspecting interiors of 3.8-liter and 18.9-liter paint cans
  - ✓ Observing the condition of 18.9-liter paint can gaskets
- Inspecting the balance of the contents of the drum—e.g., insulated wire, craft paper, absorbent wipes, gypsum sheet rock, concrete blocks, scrap metal, plastic hose, free water, dried strippable coating paint, mop heads, cellulose filters, and paint brushes.

In addition to making a written record of inspection result, digital pictures were taken of many of the items inspected. Videos were taken of representative inspections. The complete Test Report (containing many pictures), as well as a video of post-test inspections can be found on TTT's web site: www.thortt.com.

### TEST RESULTS

The demonstration testing was completely successful, as all test objectives were met:

- All prohibited items were removed from the drums by evaporation of free liquids and breaching of sealed containers
- Mass and volume reduction were achieved for all drums tested
- The test provided a preliminary qualitative demonstration that the system was capable of containment control
- No pressure surges were observed from breaching of sealed containers
- Efficient processing times were demonstrated

### **Removal of prohibited items**

The autoclave process removed all prohibited items placed in all 10 drums. Post-test inspection showed that all free liquids had been removed. All aerosol cans were breached, as shown in Figure 3. All sealed 3.8-liter and 18.9-liter containers were breached, as shown in Figure 4.



Fig. 3. All aerosol cans were breached.



Fig. 4. All sealed containers were breached

### **Mass and Volume Reduction**

The THOR<sup>SM</sup> autoclave process yielded mass reduction for all drums tested. Each assembled 208-liter drum was weighed before and after testing, and the weight reduction for each drum is shown in the Table II. The weight reductions are proportional to the mass of water and organics present in each drum.

Based on visual inspections, the organic volume of drum content was reduced by 70-90%, depending on the organic content. For example, if the original volume of waste were 10 cubic meters, and treatment produced a 90% reduction, then the post-treatment volume would only be 1 cubic meter. If the 208-liter drum had been compacted to remove the void volume in the drum, the estimated volume reduction attainable is also shown in the Table II. It should be noted that Drums 6, 8, 9, and 10 have a lower volume reduction because these drums contained non-compactable scrap metal and concrete.

Drum No.	Content Weight Reduction (Contents Weight Before Testing Less Contents	Weight Reduction (Total Drum Weight Before Testing Less Total Drum Weight	Estimated Drum Volume Reduction if Drum Were to Be
	Weight After Testing)	After Testing)	Compacted
1	99.8%	35.1 %	90 %
2	41.9%	17.3 %	80 %
3	46.4%	17.3 %	80 %
4	48.3%	20.4 %	80 %
5	99.7%	20.3 %	90 %
6	14.3%	8.6 %	40 %
7	52.7%	23.5 %	80 %
8	37.7%	22.4 %	60 %
9	27.7%	20.9 %	50%
10	19.2%	10.3 %	40 %

#### Table II. Weight and Volume Reduction

### **Containment Control**

The THOR<sup>SM</sup> 321.8-liter overpack with its integral, high-temperature filter provided containment, as airborne particles were retained within the overpack. Fluorescent Willemite added to the waste packages prior to testing was not spread outside the overpack, as indicated by black light inspection following testing. A few Willemite particles were noted on the outside of the 208-liter drum, on the inner surface of the filter, and on the inside walls of the 321.8-liter overpack. Essentially all the particles were retained in the bottom of the 208-liter drum with the waste residues and were not airborne by the treatment. No particles were noted in the autoclave or on the outside of the 321.8-liter drum. This test was intended to provide only a qualitative demonstration of the system's ability to provide containment control. Rigorous qualification of the THOR<sup>SM</sup> overpack containment system with its integral, high-temperature filter will be performed in the future.

### **System Pressure Control**

System pressure was carefully monitored during autoclave operations to observe the relative level of autoclave gas evolution and to detect any significant expansion surges resulting from the breach of sealed containers. No system pressure surges were observed from the breach of sealed containers, as the pressure effect of a can breach appeared to be well damped by the dynamics of the system. No expulsion of solids from a drum or bulging of a drum was apparent from post-treatment inspections.

### **Heat-Up Rates**

The system provided efficient heat-up times. As defined for this test, heat-up time is the time elapsed from energizing the system to the time when the temperature at the centerline of the drum reaches 923°K. As seen in this test, it would take no more than 4 hours to heat a typical 68-kilogram drum of debris waste to the processing temperature in order to remove prohibited items and destroy organics.

### CONCLUSIONS

This test demonstrated that the THOR<sup>SM</sup> autoclave system has broad potential application for treatment of problematic TRU waste streams:

- Because it successfully removes prohibited items, such as free liquids, aerosol cans, and sealed containers greater than 4 liters, the THOR<sup>SM</sup> autoclave treatment system provides an alternative to sorting and segregation operations for waste containing prohibited items. Up to 40% of the TRU waste in the DOE complex contains prohibited items.
- Because it destroys organics, the THORSM autoclave treatment system solves the problem of gas generation caused by alpha radiolysis of organics in certain waste streams, whose shipment is currently prevented by gas generation above acceptable limits.

- Because it yields mass reductions, the THORSM autoclave treatment system provides an alternate means for disposal of waste with Curie concentrations less than 100 nCi/g because the radionuclide concentration would be higher in the processed residue.
- Because it yields volume reductions, the THORSM autoclave treatment system could also provide shipping and disposal cost savings.
- Because this test provided a quantative demonstration of containment control, the THORSM in-drum autoclave treatment system demonstrated its potential for treatment of TRU waste, which requires contamination and criticality control.

Certain information addressed within this Article pertains to the performance of Contract No. 107206 between Washington TRU Solutions LLC and THOR Treatment Technologies, LLC, and in furtherance of Contract No. DE-AC04-01AL66444 between the U.S. Department of Energy and Washington TRU Solutions, LLC.

The views and opinions of the authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.