DRUM-SCALE TESTS OF IN-SITU THERMAL DESORPTION TREATMENT OF INEEL BURIED WASTES

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ABSTRACT

The U.S. Department of Energy (DOE) is investigating in-situ thermal desorption (ISTD) as a treatment for TRU-contaminated buried waste at the Idaho National Environmental and Engineering Laboratory (INEEL) sub-surface disposal area (SDA). The SDA contains Rocky Flats Plant (RFP) wastes, including drums of potassium nitrate/sodium nitrate salts, drums of volatile organic chlorides in mineral oil adsorbed on calcium silicate-based adsorbents, and drums of combustible debris.

ISTD treatment envisioned for these wastes will place relatively low power rod heaters, rated at about 300 watts per foot of rod length, in a grid pattern throughout the SDA treatment area. The rod heaters will be enclosed in a perforated six-inch diameter steel casing. Each of these heater assemblies will be sealed at the treatment area surface, so that a slight vacuum applied at the head of individual heater assemblies will draw desorbed or thermally decomposed waste constituents, and soil air and moisture, through the heater assembly into an offgas treatment system. Temperatures within the perforated casing are expected to be approximately 1100 ^{o}F (593 ^{o}C), with temperatures in the surrounding waste/soil ranging downward from 750 ^{o}F (399 ^{o}C). In practice, heat will be applied until all the surrounding waste/soil is 220 ^{o}F (104 ^{o}C) or greater, with temperatures at six inches from the heater assembly expected to reach approximately 842 ^{o}F (450 ^{o}C).

This approach to SDA waste treatment is being tested at the drum scale at the MSE Technology Applications, Inc. (MSE-TA) test facility in Butte, Montana. The work is being conducted under the direction of Bechtel BWXT Idaho, L.L.C. (BBWI). The objective of these tests is to assess the effectiveness, at drum scale, of ISTD treatment of INEEL buried waste, particularly with regard to questions of waste reactivity, offgas composition, contaminant destruction and removal effectiveness (DRE), and actinide fixation.

In calendar year (CY) 2003, MSE-TA completed one drum-scale shakedown test and three drum-scale waste treatment tests. The waste treatment tests used surrogates for organic sludge, nitrate salts, and a mixture or organic sludge and combustible debris. The organic-sludge surrogate contained a mixture of cutting oil, halogenated solvents, and inorganic adsorbents. The nitrate-salt surrogate consisted primarily of sodium and potassium nitrate. The combustible-debris surrogate was a mixture of cotton rags, paper, and plastic beads. A mixture of water and soil, formulated to simulate the heat of vaporization of the organic-sludge halogenated solvents, was used for the shakedown test. Heat-up rates for the organic, nitrate, and combustible debris surrogates were compared to the heat-up rate for the non-combustible shakedown feed. Faster heating rates were noted for the organic sludge and combustible debris surrogates, and were assumed to be due to combustion of the surrogate waste material. Air flow and electrical power to the heater assembly were found to limit the rate of combustion.

For the organic-sludge surrogate, the constituent halogenated solvents were not detectable in the offgas, as long as electrical power was applied to the heater assembly. When the heater electrical power was turned off, constituent halogenated solvents and products of incomplete combustion were found in the offgas, even though the core of the drum continued to self-heat from combustion. The organic-sludge/combustible-debris mixture self-heated at a greater rate than did the organic sludge alone. Data from the offgas analyses for this test were not available for inclusion in this paper. Heat up for the nitrate salt surrogate was slower than for the other tests, including the water/soil shakedown test. The nitrate-salt surrogate partially melted and foamed, nearly plugging the drum outlet line, after ~65 hours of heating.

INTRODUCTION

Background

The U.S. Department of Energy (DOE) is investigating in-situ thermal desorption (ISTD) as a treatment for TRU-contaminated buried waste at the Idaho National Environmental and Engineering Laboratory (INEEL) sub-surface disposal area (SDA). The SDA contains Rocky Flats Plant (RFP) wastes, including drums of potassium nitrate/sodium nitrate salts, drums of volatile organic chlorides in mineral oil adsorbed on calcium silicate-based adsorbents, and drums of miscellaneous partially combustible debris. The carbon-steel drums were buried in the SDA thirty to fifty years ago, and are unlikely to be physically intact, thus introducing the possibility of contact mixing between the originally segregated nitrate salts, organic sludges, miscellaneous debris, added adsorbents, liners, and native soil.

ISTD treatment envisioned for these wastes will place relatively low power rod heaters in a grid pattern throughout the SDA treatment area. The rod heaters, rated at 300 watts per foot of rod length, will be enclosed in a perforated six-inch diameter steel casing. Each of these heater assemblies will be sealed at the treatment area surface, so that a slight vacuum applied at the head of individual heater assemblies will draw desorbed or thermally decomposed waste constituents, and soil air and moisture, through the heater assembly into an offgas treatment system. The heater assemblies have a maximum operating temperature of 1600 ^{o}F (871 ^{o}C). Actual temperatures within the perforated casing are expected to be approximately 1100 ^{o}F (593 ^{o}C), with temperatures in the surrounding waste/soil ranging downward from 750 ^{o}F (399 ^{o}C), depending on distance from the heater assembly and the length of time heat is applied. In practice, heat will be applied until all the surrounding waste/soil is 220 ^{o}F (104 ^{o}C) or greater, with temperatures at six inches from the heater assembly expected to reach approximately 842 ^{o}F (450 ^{o}C).

Prior to using these ISTD heater assemblies at the INEEL SDA, this approach to SDA waste treatment is being tested at the drum scale at the MSE Technology Applications, Inc. (MSE-TA) test facility in Butte, Montana. The work is being conducted under the direction of Bechtel BWXT Idaho, L.L.C. (BBWI).

Objectives

The objective of the work described in this paper was to use a drum-scale test bed to assess the effectiveness of in-situ thermal desorption (ISTD) for treatment of INEEL buried waste, particularly with regard to questions of waste reactivity, offgas composition, contaminant destruction and removal effectiveness (DRE), and actinide fixation. This work involved (1) formulating and preparing simulated nitrate sludge, organic sludge, and debris mixtures, (2) heating 55-gallon-drum quantities of various combinations of these materials and INEEL soil, (3) determining the composition of pre- and post-test feed samples, and offgases, (4) assessing the contaminant DRE of ISTD treatment, and (5) assessing offgas treatment needs for in-situ thermal desorption of INEEL SDA buried wastes.

Significance of the Work Performed

The work reported in this paper addresses the feasibility of using in-situ thermal desorption (ISTD) to treat Rocky Flats waste buried at the INEEL Subsurface Disposal Area.

BARREL THERMAL DESORPTION SYSTEM DESCRIPTION

The MSE vacuum thermal desorption system consists of a carbon steel chamber sized to completely enclose a 55-gal test drum. The test chamber was designed mechanically to withstand internal pressures from 0 to 1.5 atm (that is, to withstand an internal high vacuum or an overpressure of 0.5 atm). In the tests described in this paper, the system was operated under a slight vacuum (4 to 5 in of H_2O). The slight vacuum was primarily intended to simulate ISTD heating but also ensured that any gases released in the drum during thermal desorption were drawn into the offgas treatment system and did not leak into the work area around the test bed.

The drum and chamber vacuum is controlled by monitoring chamber pressure and regulating the motive gas through the eductor with a control valve. The eductor draws purge and process gas from the chamber and 55-gal test drum respectively to equalize the pressure between them. Process gases released during thermal desorption of the 55-gal test drum are drawn through the insertion heater assembly and past several sampling ports before mixing with the purge gas drawn from the chamber, as shown in Fig. 1.

To simulate air flow through soil into the slight vacuum of an ISTD heater assembly, air is metered by a mass flow control valve into the bottom of the 55-gal test drum, where the air is dispersed circumferentially around the drum by a distribution ring (see Fig. 1). A photograph of the test system is shown in Fig. 2. Barrel thermal desorption system components are listed in Table I.

Six thermocouples are used to monitor the temperature of the drum contents during heating. The thermocouples are installed in three stainless-steel tubes, with one thermocouple near the top and one thermocouple near the bottom of each tube. These tubes are inserted vertically into the drum feed material, with one tube inserted near the center heater assembly, one tube inserted halfway between the heater assembly and the drum wall, and one tube inserted near the drum wall. The thermocouples are identified by position as "top" or "bottom" and "inner," "middle," or "outer."

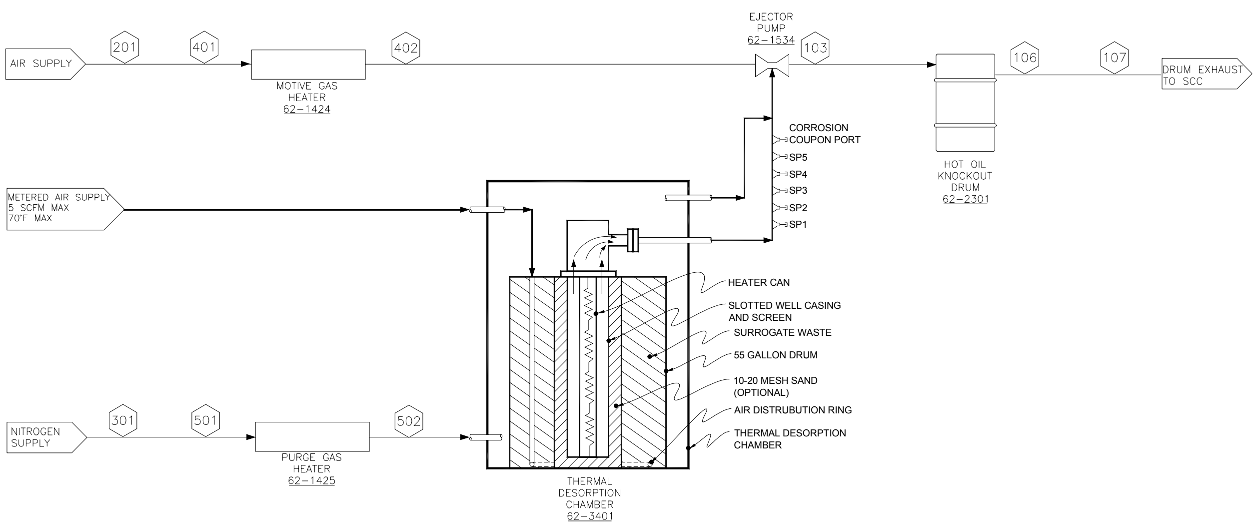

Fig. 1 Process schematic of MSE vacuum thermal desorption system. For one test, the Organic Sludge/ Combustible Debris test, the air distribution ring was removed and air was injected at a single point near the bottom of the drum. Additionally, for that test a corrosion resistant metal screen was wrapped five times around the heater can, to provide additional reactive surfaces in the void between the slotted well casing and the heater can.

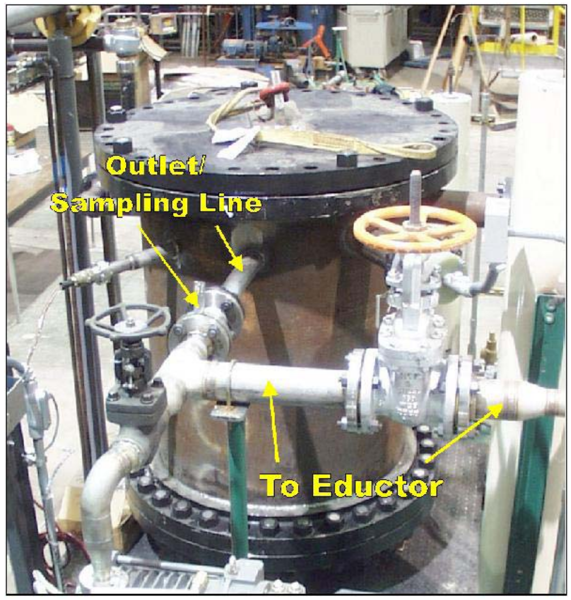

Fig. 2. Photograph of vacuum thermal desorption test chamber showing the outlet/sampling line. The individual sampling lines of Fig. 1 are not shown in this photograph. The mechanical pump partly shown in the foreground was not used, and was valved off.

Table I Vacuum Thermal Desorption System Component Descriptions (identifiers correspond to those shown in Fig. 1).

Input/Output and Components (Schematic Identification Number)	Description
Test cell (62-3401)	
Test cell tubular heater	TERRATHERM, http://www.terratherm.com, 300 W/ft, 871 °C (1,600 °F) maximum temperature.
Test cell weigh scale (62-3801)	
Motive gas heater (62-1424)	Heater for eductor motive gas. 26 kW, 399 °C (750 °F) maximum temperature, depending on flow rate.
Purge gas heater (62-1425)	Heater for test cell purge gas. 8 kW, 399 °C (750 °F) maximum temperature, depending on flow rate.
Ejector (eductor) pump (62-1534)	Venturi effect eductor pump, Croll-Reynolds Clean Air Technologies Model #2Z, http://www.croll.com/pr/vetheory.asp. Ejector can handle 59 lb/hr (13 scfm) of air at 2.9 psia and 399 °C (750 °F) while discharging to 13.3 psia using 391 lb/hr (87 scfm) of motive air at 122.8 psia and 399 °C (750 °F).
Hot oil knockout drum (62-2301)	Carbon steel construction.
Insertion point for pitot tube [Sampling Point (SP)-1]	A straight stem pitot tube was mounted, through a compression fitting, in the drum outlet line between the drum and the eductor vacuum pump. Pressure was measured with a Testo analyzer. The pitot tube was mounted upstream of the other sampling lines.
Sampling point for Testo analyzer (SP-2)	The Testo gas sampling probe was mounted, through a compression fitting, in the drum outlet line between the drum and the eductor vacuum pump. The Testo analyzer included a vacuum pump; no other motive force was required for sampling for the Testo analyzer.
Sampling line for particulate monitor (SP-3)	A 1/4-in 316 stainless steel sampling line was used to direct sample gas to a ThermoAnderson Model DR-4000 Aerosol Monitor. The sampling line was connected to the drum outlet line through a compression fitting, with the line mounted so that the opening faced upstream in the duct. Sampling was non-isokinetic. The ThermoAnderson aerosol monitor included a pump; no other motive force was needed for this sampling line.
Sampling line for Tedlar bag samples for volatile organic compounds (VOC) and HCl/Cl_2 analysis (SP-4)	A 1/4-in 316 stainless steel sampling line was mounted, through a compression fitting, in the drum outlet line between the drum and the eductor vacuum pump. This line was used to take Tedlar bag samples for VOC and HCl/Cl_2 analysis. Tedlar bag samples were taken using a "vacuum box"; no other motive force was needed for this sampling line.
Sampling line for continuous oxygen monitor (SP-5)	A 1/4-in 316 stainless steel line was mounted through a compression fitting to the drum outlet line between the drum and the ejector vacuum pump. The sampling flow was directed to an Illinois Instruments Model 6000 continuous oxygen analyzer.

TEST ACTIVITIES

Four tests were performed in FY03 and early FY04: a shakedown test, an organic-sludge surrogate test, a nitrate-salt surrogate test, and an organic-sludge/combustible-debris surrogate test. Planned test durations were five days or less. Electrical power was applied to the heater assembly on the first day of testing, as soon as the test system could be brought to readiness, and continued until a pre-selected target temperature was reached, to a maximum of four days, leaving one day for cooling down and securing the system.

Shakedown Test

The objective of the shakedown test was to check out system components and to monitor the heating profile of the drum contents. The drum contents were a mixture of INEEL soil, Kitty Litter™, Microcel E, and water, formulated to simulate the heat capacity, heat of vaporization, and total gas generation of the organic sludge. By using relatively inert constituents, comparison of the shakedown test to the later organic sludge test was expected to reveal any heating effects due to organic sludge chemical reactions. The shakedown feed formulation is shown in Table II.

Table II Shakedown Surrogate Formulation.

Shakedown Surrogate Formulation	Total Required (kg)	Percent (w/w)
INEEL soil	101.0	53.2
H_2O	16.6	8.8
Microcel E®	46.9	24.7
Kitty litter	25.3	13.3
Total	189.8	100.0

The shakedown feed material was loaded into a 55-gallon drum, which was mounted in the main test chamber of the barrel thermal desorption skid. For the Shakedown Test, electrical power was applied to the heater assembly for 65 hours. Nominal air flow into the test drum was 0.5 lb/min.

Organic Sludge Test

The Organic Sludge surrogate was a mixture of cutting oil, halogenated solvents, and adsorbents, as shown in Table III. This formulation was intended to simulate the contents of an organic sludge-containing drum buried at the RWMC. All of the organic liquids in this surrogate have fuel value, therefore this mixture had the potential to react with injected air and produce heat. This combustion process, if it occurred, was expected to take place either within the heater assembly, or in the hottest portion of feed material near the heater assembly. For the Organic Sludge Test, electrical power was applied to the heater assembly for 22 hours. Nominal air flow into the test drum was 0.5 lb/min.

Table III Organic Sludge Surrogate Formulation.

Organic Sludge Surrogate		**Req. % (w/w)**
Liquids:		
Regal Oil	Machine oil	28
Carbon tetrachloride	CCl_4	27
1,1,1-trichlorethane	$C_2H_3Cl_3$	9
trichloroethene	C_2HCl_3	7
tetrachloroethene	C_2Cl_4	7
Subtotal, Liquids		*78*
Solids:		
Microcel E	$CaSiO_3$	13
Kitty Litter	Oil-dry	7
Ceric Oxide	CeO_2	2
Subtotal, Solids		*22*
Total		100

Nitrate Salt Test

The Nitrate Salt surrogate was a mixture of 90% nitrate salts, miscellaneous other salts, 1% EDTA, and 0.5% CeO_2 (added as a plutonium surrogate), as shown in Table IV. This formulation was intended to simulate the contents of nitrate salt-containing drums buried at the RWMC. For the Nitrate Salt test, electrical power was applied to the heater assembly for 65 hours. Nominal air flow into the test drum was 0.5 lbs/min.

Table IV. Specified Nitrate Salt Surrogate Formulation. (The as-mixed formulation was normalized to a total of 100%).

Nitrate Salt Surrogate		**Req. % (w/w)**
Sodium nitrate	$NaNO_3$	60
Potassium nitrate	KNO_3	30
Sodium sulfate	Na_2SO_4	3
Sodium chloride	NaCl	3
Disodium hydrogen phosphate heptahydrate	$Na_2HPO_4.7H2O$	1
Sodium bicarbonate	$NaHCO_3$	0.5
Sodium fluoride	NaF	0.5
Sodium nitrite	$NaNO_2$	1
EDTA	$C_{10}H_{16}N_2O_8$	1
Ceric oxide	CeO_2	0.5
Total		100.5

Organic Sludge/Combustible Debris Test

A Combustible Debris surrogate consisting of 40%(w/w) cotton rags, 40%(w/w) paper towels, 10%(w/w) polyethylene beads, 5%(w/w) polyvinyl chloride beads, and 5%(w/w) ABS plastic beads was prepared for this test. The Combustible Debris surrogate was intended to simulate combustible laboratory trash from Rocky Flats. The feed formulation for this test consisted of 35%(w/w) Organic Sludge, 55%(w/w) Combustible Debris, and 10%(w/w) water. This formulation was intended to simulate the contents of drums, buried at RWMC, containing cutting oil, halogenated solvents, adsorbents, paper, cloth, and plastic scrap. For the Organic Sludge/Combustible Debris Test, electrical power was applied to the heater assembly for 50.1 hours. Nominal air flow to the test drum was 0.5 lb/min.

RESULTS AND DISCUSSION

The primary variable of interest for the ISTD drum tests was the temperature of the drum contents as a function of time. Temperatures for the drum contents for the Shakedown, Organic Sludge, Nitrate Salt, and Organic/Combustibles tests are shown in Fig. 3. The legend for Fig. 3 indicates the test and thermocouple locations as follows: The first two or three characters indicate the test, Shakedown (Shk), Organic Sludge (Org), Nitrate Salt (Nit), or Organic/Combustibles (D3); the middle character indicates the thermocouple identifier; the last two characters indicate the thermocouple locations, which were inner bottom (IB), inner top (IT), mid-bottom (MB), mid-top (MT), outer bottom (OB), and outer top (OT). As shown in the legend for Fig. 3, each line color in the graph indicates a particular test; for example, green indicates the Nitrate Salt test.

Air was injected near the bottom of the drum. The injected air was expected to flow (1) through the feed material near the heater assembly, (2) through the perforated casing surrounding the heater assembly, (3) into the heater assembly, (4) out of the top of the heater assembly, and (5) into the outlet/sampling lines leading to the eductor and, ultimately, the offgas treatment system. Temperatures were expected to rise gradually, and from the central heater assembly outward to the walls of the drum.

As shown in Fig. 3, heating rates and the maximum temperatures achieved varied substantially from test to test. Factors affecting heating rate were assumed to be power input to the heater, water content in the feed material, and fuel value of material volatilizing from the feed. In principle, heater power input would have been held constant at 900 watts. In practice, the heater target temperature was set and the heater "thermostat" then automatically varied the power input, between zero and 900 watts, to eventually reach and then maintain the target heater temperature. For the Shakedown test, power input was generally at the maximum, 900 watts, and even then the temperature of the drum contents rose relatively slowly. For the Organic Sludge and Organic/Combustibles test, after a period of initial heat up, the drum contents began self-heating, and the heater controller automatically reduced the electrical power input, sometimes to zero.

These and other factors affecting heating rates for specific tests are discussed below.

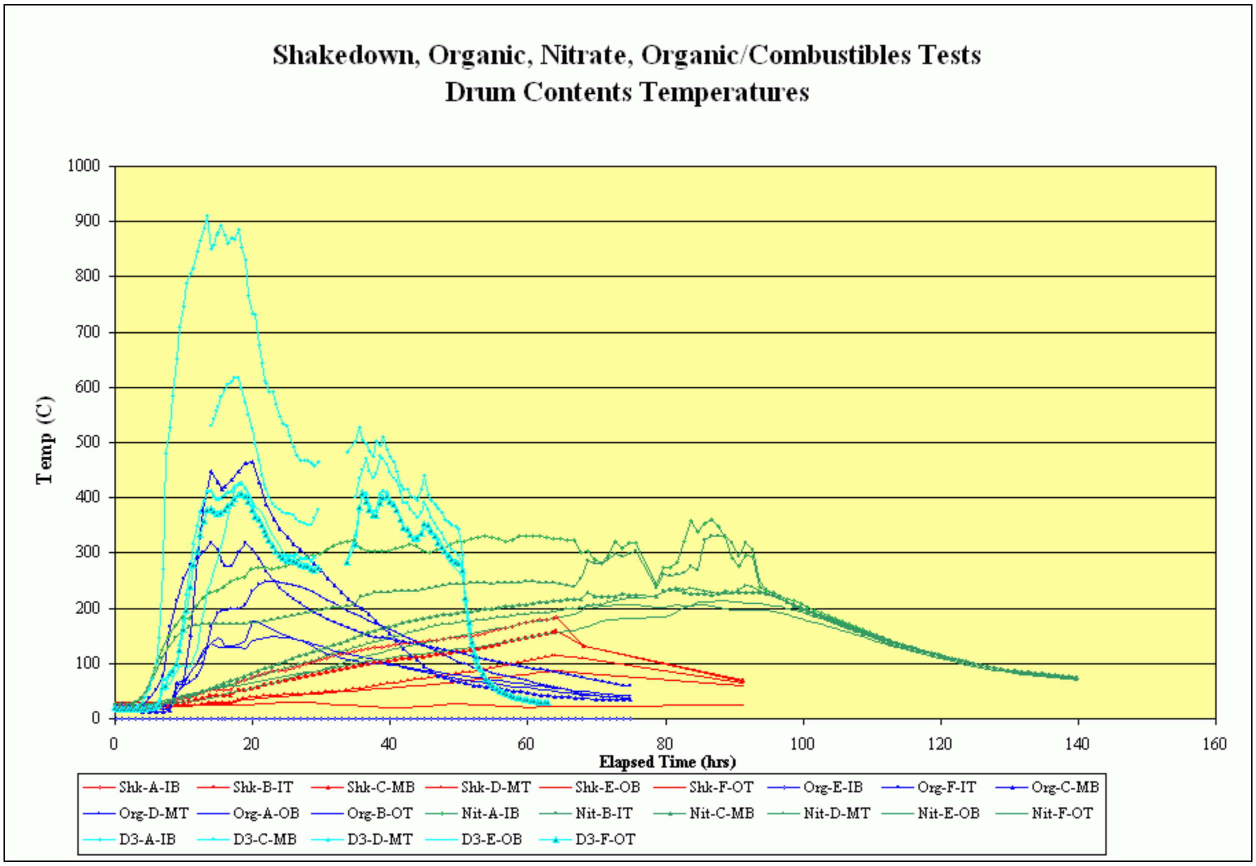

Fig. 3. Temperatures of test drum contents for the Shakedown (Shk), Organic Sludge (Org), Nitrate Salt (Nit), and Organic/Combustibles (D3) tests. Thermocouple locations were inner bottom (IB), inner top (IT), mid-bottom (MB), mid-top (MT), outer bottom (OT), and outer top (OT). See system description for a discussion of thermocouple locations.

Shakedown and Organic Sludge Tests

The Shakedown test feed material was formulated to simulate the heat capacity and heat of vaporization of the Organic Sludge feed. As shown in Fig. 3, the peak temperature achieved for the Shakedown test was approximately 180 ºC, after 65 hours of heating. The Organic Sludge feed was expected to give the same temperature profile, unless combustion of the organics, or some other effect, altered the heating rate. However, during the Organic Sludge test, the drum contents reached a peak temperature of approximately 875 ^{o}F, after 20 hours of heating, indicating a significantly accelerated heating rate relative to the Shakedown feed, apparently due to combustion of volatile organics in the Organic Sludge feed.

Offgas from the Organic Sludge test was sampled and analyzed for hydrogen chloride and chlorine gas (HCl/Cl_2) and the four volatile organic compounds (VOCs) included in the drum contents. As shown in Fig. 4, the peak HCl concentration, ~33 %(v/v), was reached after 22 hours of heating. Up to that time, there were no detectable feed VOCs in the offgas. At 22 hours, when the drum contents near the drum wall exceeded the target temperature of 105 ^{o}C, the heater assembly was powered down. At that point, the temperature of the drum contents began to decline, offgas HCl concentrations abruptly dropped off, and offgas VOC concentrations abruptly increased, with VOC concentrations peaking at about 32 hours from the start of the test.

The "crossover" of HCl and VOC concentrations was apparently due to incomplete combustion of VOCs being volatilized from the feed material. While power was supplied to the heater, the VOCs, which were the only source of chlorine, were evidently fully oxidized to HCl, carbon dioxide, and water. When the heater power was turned off, the VOCs continued to volatilize from the drum, but the presumed combustion zone near or within the heater assembly was apparently no longer hot enough to oxidize the VOCs. A qualitative organic compound "scan" of an offgas sample taken during this cool-down period showed numerous partially dechlorinated products of the feed organic VOCs, further suggesting incomplete oxidation of organics after the heater power was turned off.

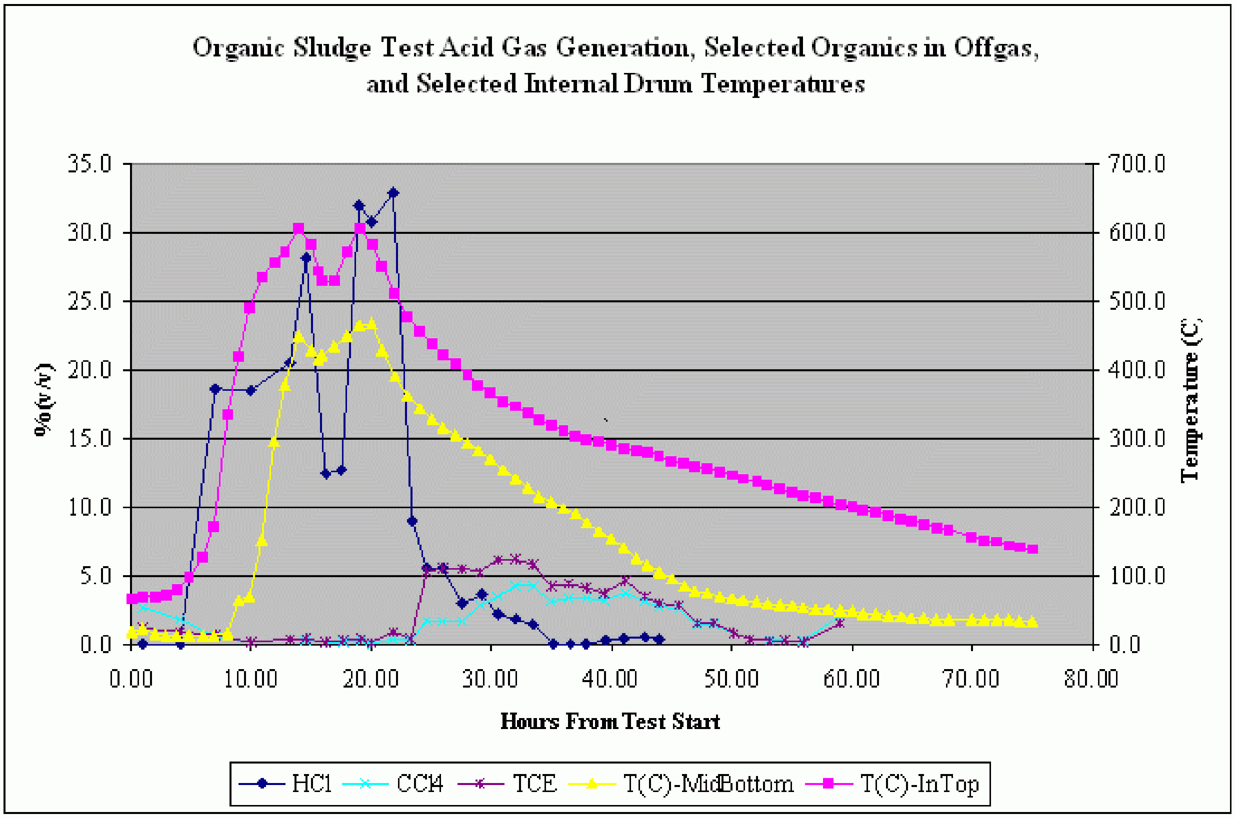

Fig. 4. Organic Sludge test acid gas generation, selected offgas volatile organics, and selected internal drum temperatures.

We concluded that the heater assembly performed as expected, that is, when power was applied, VOCs in the drum were gradually volatilized and oxidized, with generation of heat. When the heater power was turned off, the VOCs continued to volatilize as the drum cooled, but were no longer being fully oxidized. Volatilization of VOCs gradually dropped off as the drum cooled. An additional result, as expected from the pre-test safety analysis, was that a feed containing combustible organics, but no oxidizers, could be safely processed in the drum test system.

Analysis of post-test drum solids showed residual concentrations, up to 0.2% (w/w), of the four feed VOCs, indicating that the VOCs had been mostly, but not completely, volatilized.

Nitrate Salt Test

The objective of the Nitrate Salt test was to simulate, at drum-scale, ISTD treatment of a nitrate salt surrogate mixture, while monitoring heating rates, apparent reactivity, and offgas generation for the mixture.

As shown in Table IV, the Nitrate Salt feed consisted of a mixture of inorganic salts, primarily sodium nitrate, 60%(w/w), and potassium nitrate, 30%(w/w), with lesser concentrations of sodium sulfate, sodium chloride, and traces of other salts. While the nitrate compounds are strong oxidizers, particularly when heated, the only combustible compound in the mixture was EDTA, at 1%(w/w), which provided only a trace of "fuel" for the nitrate oxidizers. Accordingly, heating this mixture was expected to result in only minimal exothermic reactions. As shown in Fig. 3, the heating rate for the Nitrate Salt drum contents was slightly faster than for the Shakedown test, and much slower than for the Organic Sludge test. For the Nitrate Salt test heater power was applied for approximately 65 hours, at which time the temperature of the drum contents peaked at approximately 185 ^{o}C. The offgas was sampled and analyzed for the four VOC compounds of the Organic Sludge surrogate, and for HCl/Cl_2. As expected, since the feed did not contain VOCs, VOCs were not detected in the offgas. The feed material contained a relatively minor source of chlorine, 3%(w/w) sodium chloride, however HCl/Cl_2 also was not detectable in the offgas, probably because the temperatures reached were not sufficient to decompose sodium chloride.

Post-test examination of the drum contents and heater assembly showed that the Nitrate Salt feed material had foamed, nearly plugging the test drum offgas outlet, and clearly indicating some degree of thermal decomposition of the drum contents. Laboratory analysis of pre- and post-test drum solids indicated that the nitrate concentration of the feed was essentially unchanged by processing, suggesting relatively minor decomposition or other removal of the nitrate salts. As shown in Table IV, the feed material contained 1%(w/w) $Na_2HPO_4 \cdot 7H_2O$. This compound, disodium hydrogen phosphate heptahydrate, undergoes loss of five waters of hydration at 48.1 ^oC. Therefore, the foaming may have been principally due to loss of water vapor from the $Na_2HPO_4 \cdot 7H_2O$.

We concluded that during the Nitrate Salt test the feed material partially melted and foamed, but based on nitrate analysis of the pre- and post-test solids, there was little or no decomposition of the nitrate salts, which is consistent with the drum temperatures observed and the published decomposition temperatures for sodium nitrate and potassium nitrate (380 ^oC and 400 ^oC, respectively). An additional conclusion, as expected from the pre-test safety analysis, was that an oxidizing feed material, in the absence of an organic fuel, could be safely processed in the drum test system.

Organic Sludge/Combustible Debris Test

The objective of the Organic Sludge/Combustible Debris test was to simulate, at drum-scale, ISTD treatment of a mixture of organic sludge surrogate, combustible debris surrogate, and water, while monitoring heating rates, apparent reactivity, and offgas generation for the mixture.

The feed material, called Organic-Combustible Surrogate, was a mixture of Organic Sludge Surrogate, a Combustible Debris surrogate, and water. The Combustible Debris surrogate was prepared from cotton rags, 40%(w/w), paper towels, 40%(w/w), polyethylene beads, 10%(w/w), ABS plastic beads, 5%(w/w), and PVC plastic beads, 5%(w/w). The overall Organic Sludge/Combustible Debris feed formulation was 35%(w/w) Organic Sludge, 55%(w/w) Combustible Debris, and 10%(w/w) water.

As shown in Fig. 3, above, the peak temperature for the Organic Sludge/Combustible Debris drum contents was 910 ^oC, observed at 13.5 hours from the start of heating, at the inner bottom thermocouple. Peak temperatures/times for the middle and outer thermocouples were 617 ^oC at 18.0 hrs, and 423 ^oC at 18.5 hrs, respectively.

As shown in Fig. 3, both the rate of temperature rise and the peak temperatures reached were substantially greater than for the Shakedown, Organic Sludge, or Nitrate Salt tests. Similarly to the Organic Sludge test, the rapid rise in temperature was apparently due to combustion of the drum contents. In this case, combustible materials included rags, paper, and plastic, as well as the oil and chlorinated solvents contained in the Organic Sludge feed. Although the Organic Sludge/Combustible Debris feed included 10% water, the heat required to evaporate water apparently did not offset the additional thermal energy available from the Combustible Debris constituents.

As of this writing, offgas data and pre- and post-test drum content analyses were not available for the Organic Sludge/Combustible Debris test. The offgas data and drum content analyses will be presented in the final project report.

SUMMARY AND CONCLUSIONS

In CY03, MSE-TA completed one drum-scale shakedown test and three drum-scale waste-treatment tests. The waste-treatment tests used surrogates for organic sludge, nitrate salts, and a mixture or organic sludge and combustible debris. The organic sludge surrogate contained a mixture of cutting oil, halogenated solvents, and inorganic adsorbents. The nitrate salt surrogate consisted primarily of sodium and potassium nitrate. The combustible debris surrogate was a mixture of cotton rags, paper, and plastic beads. A mixture of water and soil, formulated to simulate the heat of vaporization of the organic sludge halogenated solvents, was used for the shakedown test. Heat-up rates for the organic, nitrate, and combustible debris surrogates were compared to the heat-up rate for the non-combustible shakedown feed. Faster heating rates were noted for the organic sludge and combustible debris surrogates, and were assumed to be due to combustion of the surrogate waste material. Air flow and electrical power to the heater assembly were found to limit the rate of combustion.

For the organic sludge surrogate, the constituent halogenated solvents were not detectable in the offgas, as long as electrical power was applied to the heater assembly. When the heater electrical power was turned off, constituent halogenated solvents and products of incomplete combustion were found in the offgas, even though the core of the drum continued to self-heat from combustion. The organic sludge/combustible debris mixture self-heated at a greater rate than did the organic sludge alone. Offgas analyses for this test were not available at the writing of this paper. Heat up for the nitrate salt surrogate was slower than for the other tests, including the water/soil shakedown test. The nitrate salt surrogate partially melted and foamed, nearly plugging the drum outlet line.

We concluded that the heater assembly performed as expected, gradually heating surrogates of buried organic sludge, nitrate salts, and organic sludge/combustible debris. Combustion of volatilized organics was essentially complete, as long as electrical power was applied to the heater assembly. A secondary conclusion was that organic sludge, nitrate salts, and organic sludge/combustible debris could be safely tested in the drum-scale test bed. Mixtures of organic sludge or combustible debris with nitrate salts, a combination of fuel and oxidizer, were not tested. Pending a continuing safety review, fuel/oxidizer mixtures of this type will be tested in FY04, either at an appropriate facility in New Mexico, or at the MSE-TA test facility in Butte, Montana.

In addition to the four drum-scale tests reported here (one shakedown and three waste treatment tests), an additional drum-scale test is planned for February or March 2004. The planned feed for this test is 40%(w/w) organic sludge, 30%(w/w) combustible debris, and 30%(w/w) soil.

RECOMMENDATIONS

MSE TA, Inc. recommends drum-scale testing of organic sludge/combustible debris/nitrate salt mixtures, pending safety review. Additionally, tests of realistically heterogeneous surrogate wastes, at the single and multiple drum scale, could be conducted at the MSE-TA test facilities, and could be used to support design efforts at INEEL. Realistically heterogeneous surrogates could, for example, be prepared by placing organic sludge, nitrate salts, and/or combustible debris, without mixing, into separate zones in the drum. Testing multiple buried drums would realistically simulate treatment of a section of a larger waste disposal site.

ACKNOWLEDGEMENTS

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