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Testing of Organic Waste Surrogate Materials in Support of the Hanford Organic Tank Program - Final Supplementary Report

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SUMMARY

To address safety issues regarding effective waste management efforts of underground organic waste storage tanks at the Hanford Site, the Bureau of Mines conducted a series of supplemental tests, at the request of the Westinghouse Hanford Company. In this series of supplemental tests, the thermal characteristics of surrogate materials, chosen by Hanford, were determined. The surrogate materials were mixtures of organic and inorganic sodium salts, representing fuels and oxidants. The oxidants were sodium nitrate and sodium nitrite. The fuels were sodium salts of acetate, stearate, and oxalate. Sodium hydroxide was also an ingredient of the mixtures, used to maintain basic conditions.

The fuels and all the mixtures shown in table 1 of this report were tested by differential scanning calorimetry and by thermogravimetric analysis. All the mixtures displayed exothermic behavior. In general, onset of exothermic behavior was not strongly dependent on heating rate or sample size. Onset temperatures for exothermic behavior for the acetate mixtures varied between 213° and 235° C in the DSC and TGA tests. Onset temperatures for exothermic behavior for the stearate mixtures varied between 210° and 270° C in the DSC tests. However, indications for slow exothermic reactions of low energy were noted in some of the tests at temperatures as low as 140° to 165° C. Onset temperatures for the same mixtures were higher in the TGA tests, and were of the order of 300° C; in a few of the tests onset temperatures were lower, as low as 240° C, but even these lower temperatures were all higher than the onset temperatures in the DSC tests. Slow gas evolution from viscous mixtures may explain this difference in results. Then again, some reactions take place without weight loss. Finally, for the

oxalate mixtures; onset temperatures varied from 230° to 270° C, and from 255° to 322° C, for the DSC and TGA tests, respectively. The only exception was found for mixture S-13, which showed exothermic behavior at the low temperatures of 105° C to 130° C.

Another concern in the case of these mixtures is the release of flammable gases such as methane and acetone, and the amount of gases released. The flammable range of methane is between 5 and 15 pct while that of acetone is between 2.6 and 13 pct. Their autoignition temperatures are 605° and 465° C, respectively.

INTRODUCTION

A series of tests was conducted to determine the thermal characteristics of surrogate materials, selected by Hanford Westinghouse Company. The surrogate materials were mixtures of inorganic and organic sodium salts, representing oxidants and fuels. The oxidants were sodium nitrate and sodium nitrite. The fuels were sodium acetate, sodium stearate and sodium oxalate. In addition, sodium hydroxide was an ingredient in all but one of the mixtures.

Two thermal analyzers, a differential scanning calorimeter (DSC) and a thermogravimetric analyzer (TGA) were used in the tests. Onset temperatures for exothermic behavior were derived from the generated thermograms of all the tests.

TEST DETAILS

MATERIALS

Mixtures of organic and inorganic sodium compounds were chosen to represent contents of the waste tanks at the Hanford Site. The organic salts, representing fuels, included sodium acetate ($\text{NaC}_2\text{H}_3\text{O}_2$), sodium stearate ($\text{NaC}_{18}\text{H}_{35}\text{O}_2$) and sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$). Two inorganic salts, sodium nitrate (NaNO_3) and sodium nitrite (NaNO_2) were the oxidants, while sodium hydroxide (NaOH) was used to render the mixtures basic. In the waste tanks, constructed of carbon steel, high alkalinity is maintained to prevent corrosion.

TESTS

Tests were conducted in the differential scanning calorimeter (DSC) and in the thermogravimetric analyzer (TGA). Test equipment and procedures, which are described in detail in the final report, were utilized in these supplementary tests, as well. Preliminary tests in the DSC were conducted in aluminum pans and lids, which are commonly used for tests in the DSC. However, since mixtures containing sodium hydroxide can react with aluminum, a platinum pan and lid were used in the tests. A platinum pan and lid, containing reference glass beads, were used in the reference side of the DSC.

According to the literature (1)¹, oxygen-free silver is only slightly attacked by molten sodium hydroxide at high temperatures. However, silver pans and lids are not available commercially. Platinum resists attack by NaOH quite well. Most of the tests were conducted in a low flow of air, and the prevalent heating rate was 10° C/min; however, heating rates of 5° and 20° C/min were also used in a few of the tests.

TEST MATRIX

Table 1 lists the chemical composition of the mixtures subjected to the tests.

SAMPLE PREPARATION

Reagent grade sodium nitrate, sodium nitrite and sodium oxalate were available from the first series of tests as fine powders of -100 mesh (-150 μ m). A sample of sodium stearate was obtained from Witco Corp. It was a fine powder of food grade quality. The powder was sieved and the -100 mesh fraction was collected and used. Reagent grade sodium acetate was ground and sieved and the -100 mesh fraction was collected and used. Reagent grade sodium hydroxide was available as hard electrolytic pellets. Small quantities of the pellets were ground at a time in a mortar and pestle, and then dried in an oven. The dried powder was sieved and the -100 mesh fraction was collected. Sodium hydroxide was the last ingredient to be weighed for each mixture. It was weighed, and added to the other ingredients, in a flow of

¹Italic numbers in parentheses refer to items in the list of references at the end of this report.

Table 1.- Composition matrix for the DSC/TGA supplemental tests

Sample	Fuel weight, g	Oxidant weight, g		Base weight, g
		NaNO ₃	NaNO ₂	NaOH
	Sodium acetate			
S-1	1.025	6.780	1.695	0.500
S-2	1.708	5.968	1.492	0.833
S-3	2.391	5.155	1.289	1.166
S-4	3.074	4.342	1.085	1.499
	Sodium stearate			
S-5	0.284	7.546	1.887	0.284
S-6	0.567	7.093	1.773	0.567
S-7	0.851	6.639	1.660	0.851
S-8	1.134	6.185	1.546	1.134
S-9	0.851	7.319	1.830	-
S-10	0.851	4.597	1.149	3.403
	Sodium oxalate			
S-11	2.232	5.344	1.336	1.088
S-12	3.905	3.352	0.838	1.905
S-13	6.137	0.696	0.174	2.993

nitrogen, inside a glove box. The glass jars containing the mixtures were then sealed and the contents were mixed by extensive tumbling. All the samples were kept in a desiccator, and were taken out only when a sample was needed. It must be noted, however, that even in the short time necessary to place a sample in a preweighed pan, weigh it, place the preweighed lid in the pan, and then set it in the DSC, moisture was adsorbed. It is believed that moisture was also adsorbed from the desiccant (calcium sulfate, CaSO_4), since the samples tested last contained the largest amount of moisture.

EXPERIMENTAL RESULTS AND DISCUSSION

THERMAL ANALYSIS TESTS

Calibration of the DSC

Pure indium and zinc metal standards were used to check the calibration of the DSC. Tests were conducted in a flow of air, at 10°C/min . Transition temperature for indium was 156.7°C , while for zinc it was 417.7°C . A one-point temperature calibration for zinc was then conducted, for fine tuning. A pure cadmium metal was then tested in the DSC, at the same experimental conditions. The resultant thermogram is shown in figure 1. Onset temperature for the melting phase was 320.0°C ; literature value is 320.9°C . Thus, calibration linearity is good.

Calibration of the TGA

Weight and temperature calibrations of the TGA were conducted. Weight calibration was performed with a 100-mg calibration weight following the

recommended procedure. A temperature calibration check was done with a fresh sample of pure nickel. The resultant thermogram is shown in figure 2. Two calculations of the onset temperature for the magnetic transition were done, and the results are included in the figure. The calculated values are 353.4° and 354.3° C. The literature value for the magnetic transition (Curie temperature) of nickel is 354° C. Thus, no additional fine tuning was necessary.

Sodium Acetate and Acetate Mixtures

Sodium acetate was heated in the DSC in gently flowing air, at 10° and 5° C/min, and respective thermograms are presented in figures 3 and 4. The main features in both thermograms include the melting endotherm and exothermic decomposition. Although anhydrous sodium acetate (certified; Fisher Scientific) was used in the preparation of the samples, there is evidence in the thermograms of moisture loss, at low temperature. Sodium acetate trihydrate, $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$, loses two of its water of hydration molecules at 58° and 78° C, while the third one is lost at 120° C. The thermograms therefore indicate the presence of some dihydrate or trihydrate in the anhydrous salt, probably from the manufacturing process. It is also known that acetic acid is sometimes adsorbed on the acetate during the process. The melting point of sodium acetate is 324° C. Onset of exothermic decomposition is at 426° and 435° C, for the tests at 5° and 10° C/min, and the shapes of the two exotherms are essentially identical, although the peak is shifted to a higher temperature at the higher heating rate, as expected. A somewhat higher onset temperature of 445° C, was found for a smaller sample. A sample of sodium

acetate was also heated in nitrogen, at 5° C/min. The thermogram, in figure 5, shows the endotherms due to water loss and melting followed by the main endotherm due to pyrolysis, with onset at about 420° C. These results indicate that the decomposition of acetate starts at the same temperature, regardless of the atmosphere, and that exothermic behavior is due mainly to the oxidation of the decomposition products in air.

The first mixture containing sodium acetate that was tested was S-1; it contained the smallest amount of sodium acetate and sodium hydroxide, and the largest amount of the nitrate and nitrite of the four mixtures, S-1 to S-4. Two thermograms obtained at 10° C/min and one at 5° C/min, all in a flow of air, are shown in figures 6 to 8. The sample used in the test depicted in figure 6 weighed 5.5 mg, and at 350° C it leaked out of the sample pan. This is seen by the sudden jump in the thermogram at that temperature. Smaller samples were used in subsequent tests. Loss of moisture from the sample is seen in figure 6 in the first, extended endotherm, starting at ambient temperature. This endotherm is followed by two other endotherms; one a very small one with peak at 164° C due to the solid phase transition of sodium nitrite, and the other a large endotherm with onset temperature at 179° C, and a peak temperature at 193° C. This latter endotherm is probably due to a combined melting of the nitrate, nitrite and hydroxide. The same three endotherms are also present in figures 7 and 8, and possess similar transition temperatures within $\pm 2^\circ$ C. Exothermic behavior then appears in the three thermograms, with onset temperature at about 215° to 220° C. The transition from the third endotherm to the exothermic phase is not sharp. One way to pinpoint this temperature more closely is to draw extended baselines, as shown in figures 7A and 8A, and to assume that the exothermic onsets start where the

thermograms depart from the baseline. Another procedure would be the drawing of tangents to find the inflection point. Accuracy will not be improved by more than a few degrees by the use of either procedure. The test, with resultant thermogram seen in figure 6, was one of the preliminary tests in which aluminum pans were used. The exothermic portion of the thermogram, between 220° and 300° C, is different from the analogous portion in figures 7 and 8, possibly due to reaction(s) with the aluminum.

Another test with a sample from the same mixture, heated at 10° C/min, resulted in a thermogram with the same features seen in figures 7 and 8. It is shown in figure 9. The residue from this test was cooled in the sample pan and then reheated at 20° C/min, with the resultant thermogram in figure 10. Only endothermic behavior is seen in this latter thermogram. Since mixture S-1 is rich in nitrate and nitrite, there is abundant oxidant for a complete reaction with the fuel when the sample is heated the first time. With the four initial ingredients, other reactions are also possible. The residue contains the unreacted nitrate and nitrite, the carbonate product of the reaction with acetate, other possible products and unreacted sodium hydroxide (all the residues were highly basic in aqueous solution). These various compounds can melt individually or together, as one or as numerous eutectics, depending on the relative proportions of the compounds. All these melting events are endothermic.

Two thermograms for samples from the S-2 mixture, both obtained at 10° C/min, are illustrated in figures 11 and 12. Thermograms for samples from the S-3 mixture, at 5°, 10° and 20° C/min, are seen in figures 13 to 15, respectively, and a thermogram for sample S-4 is presented in figure 16. All these thermograms have similar features, starting with the endotherm due to

loss of moisture, followed by the endotherm due to the combined fusion of portions of the initial ingredients, in turn undergoing exothermic reaction. The complete exothermic range in all the thermograms for the mixtures with sodium acetate consists of a series of reactions, usually three consecutive ones, and is not a simple process. The proportional sizes of the various exothermic steps depend on the proportions of the four ingredients in the mixture, the heating rate, and probably the sample size. The viscosity of the melt will also affect the rate of the reaction(s), especially since gas evolution is involved. Onset temperatures for exothermic behavior for the four mixtures containing sodium acetate are given in table 2. In addition, the temperature of the small peak due to the solid phase transition of sodium nitrite is also provided, as an internal standard, assuming that it is not influenced by the presence of the other three components. The variation in this temperature can be used to assess the precision of the results. In this case, peak temperature is $165.0^{\circ} \pm 0.8^{\circ} \text{ C}$.

Sodium acetate and the four mixtures with acetate were tested in the TGA. The thermogram for sodium acetate is shown in figure 17. Weight loss due to moisture is 0.2 pct, as determined from the thermogram at 264.4° C . Onset of weight loss due to decomposition is at 331° C , but weight loss proceeds at a slow rate; at 397° C , weight loss is only 1.4 pct by weight of initial sample. Total weight loss at 548° C is 35.6 pct. Complete decomposition of sodium acetate is as follows:

Table 2.- DSC thermal analysis results for the acetate and acetate mixtures

Sample	Sample weight, mg	Heating rate, °C/min	Onset temperature, ¹ °C	Peak temperature for sodium nitrite phase transition, °C
Sodium acetate	2.1	10	445	-
Sodium acetate	4.3	10	435	-
Sodium acetate	4.3	5	426	-
Sodium acetate	4.0 ²	5	-	-
S-1-1	2.8	10	218	164.5
S-1-2	5.5	10	213	164.3
S-1-3	2.5	10	217	164.3
S-1-4	1.9	10	219	164.3
S-1-5	3.8	10	235	165.7
S-1-7	4.1	10	215	165.4
S-1-8	3.0	5	214	164.8
S-2-1	NA	10	213	³
S-2-2	NA	5	219	164.3
S-2-3	3.6	10	219	165.4
S-2-4	2.9	10	223	165.9
S-3-1	1.8	10	228	166.9
S-3-2	3.2	5	219	164.5
S-3-3	2.8	20	232	³
S-4-1	2.7	10	230	164.4

NA Not available; sample was not weighed.

¹ Based on initial deviation of the decomposition step from baseline.

² Nitrogen flow.

³ Was not detected in the thermogram.

with calculated weight loss being 35.4 pct. A second sample, heated at 20° C/min, lost 35.4 pct of its initial weight and the onset temperature was 340° C. The residue in both tests was mostly black, with small gray portions, showing evidence of a side reaction forming small amounts of carbon or char.

A thermogram for sample S-1, in a test conducted at 10° C/min, is shown in figure 18. A small loss of moisture, of 0.5 percent of initial weight is complete at about 47° C. Weight loss then proceeds in two stages. The first stage is initiated at 220° C and ends at about 294.5° C, with weight loss equal to 4.2 pct. The second stage, which follows immediately, is complete at 420° C, with total weight loss being 9.9 pct of original weight. Conversion of the sodium acetate in the mixture to sodium carbonate would result in a weight loss of 6.6 pct. Thus, it is seen that there is weight loss from the other compounds, as is expected. Thermograms obtained for the three other mixtures, S-2 to S-4, are shown in figures 19 to 22. Some of the features of note in these thermograms are the following: (1) the increase of moisture content with increase in sodium hydroxide content; (2) the increased complexity in the decomposition scheme with increased acetate and hydroxide content; and, (3) the presence of carbon in the residues from mixtures S-2 to S-4. These residues were gray or black, while the residues from mixture S-1 were white, on account of their small content of acetate. Weight losses from the various mixtures were highly reproducible. Calculations of weight losses, based on anhydrous contents of all the samples, also indicate that weight loss was not only due to conversion of acetate to carbonate. Conversion of the nitrate to nitrite via the reaction with the fuel also leads to some weight loss. Weight loss due to other reactions is also possible. The results for the tests in the TGA are summarized in table 3.

Table 3.- TGA thermal analysis results for the acetate and acetate mixtures

Sample	Sample weight, mg	Heating rate, °C/min	Onset temperature, ¹ °C	Weight loss, pct	
				Total loss	Loss based on dry weight
Sodium acetate	2.74	10	396	35.6	35.1
Sodium acetate	2.48	20	402	35.5	35.1
S-1-1	5.69	10	236	9.9	9.4
S-1-2	4.30	10	237	9.4	9.3
S-2-1	5.10	10	218	15.4	14.5
S-3-1	3.60	10	218	22.4	19.3
S-3-2	2.48	10	223	28.2	22.3
S-4-1	5.54	10	227	25.5	23.1
S-4-2	5.53	5	216	26.3	23.0

¹ Based on initial deviation of the decomposition step from baseline.

As shown in reaction 1, acetone (CH_3COCH_3), a flammable gas, is a product of sodium acetate decomposition (2). Sodium acetate can also react with sodium hydroxide as follows,

Methane (CH_4), another flammable gas, is therefore another possible product when the mixtures with acetate are heated. Small amounts of methane are produced in the laboratory by this method. The consecutive and/or simultaneous reactions of sodium acetate with sodium nitrate and with sodium nitrite (3),

and,

can be combined into one, overall reaction,

Based on reaction 5, there is enough sodium nitrate in mixtures S-1 to S-3 to completely react with the acetate. In addition, the sodium nitrite in these mixtures can react with the leftover, unreacted acetate in the event that the reaction with nitrate is not complete. The combination of nitrate and nitrite in mixture S-4 is not sufficient for a complete reaction, but in alkaline medium oxygen can easily convert the nitrite to nitrate (4), and thereby provide additional oxidant for the reaction with the acetate. To verify complete reaction of the acetate, the residue should be analyzed for its contents. Since the acetate can react with the nitrate, the nitrite and the hydroxide, as well as decompose by itself in the temperature range of interest, and form carbonate in each reaction, it would be difficult to determine which reaction takes precedence nor the reactions that predominate from a chemical analysis of the final products. Sodium carbonate can also form in the reaction of sodium hydroxide with carbon dioxide (CO_2), one of the products in reaction 3. For a more detailed picture of the various events, analysis of intermediate products, at different stages of the overall

reaction, is required. If the reactions producing methane and acetone predominate, hazardous conditions can occur if concentrations of these gases are within their flammable limits and temperatures are high enough for autoignition.

The thermal decomposition of acetate in fused potassium nitrate was studied by Shams El Din and El Hosary (5). They represented the results of the decomposition as follows:

In addition to carbonate and nitrite, gaseous nitromethane (CH_3NO_2) is a product. Nitromethane was not detected by other researchers.

Sodium Stearate and Stearate Mixtures

Sodium stearate was heated in the DSC in a flow of air at 5°, 10°, and 20° C/min, and the resultant thermograms are illustrated in figures 23 to 25. After undergoing a very small endothermic phase, a series of exothermic reactions occurs with onset temperatures for exothermic behavior for the three tests being 176°, 185°, and 193° C, respectively. The residues from the three tests were black. The thermograms for the mixtures containing the sodium stearate, S-5 to S-10, are shown in figures 26 to 33. They all have similar features, including the endotherm due to moisture loss from sodium hydroxide, and the small endotherm arising from the solid phase change of sodium nitrite. A third and quite substantial endothermic region in turn reverts into a large exotherm. Figure 27 presents a thermogram generated by a large sample from mixture S-5 in a test conducted at 20° C/min, which clearly delineates all

these stages. The third endotherm in figure 29, for a sample from mixture S-7, is smaller in size, but otherwise the thermogram contains all the detailed phases. The figure also displays the horizontal baseline which was used to identify the various events in the thermogram. Mixture S-9 did not contain sodium hydroxide, and indeed, the endotherm due to loss of moisture from the hydroxide is not present in the thermogram for the mixture in figure 32. The sharp exotherm in the figure, with onset near 220° C and peak at 233° C, is not present in the other thermograms for the samples with sodium stearate. However, a small exotherm, with onset at 217° C and peak at 228° C is present in figure 31, for a sample from mixture S-8. The thermogram for a sample from mixture S-10, seen in figure 33, also has some unique features; the sharp endotherm at 65° C represents the release of water from sodium hydroxide monohydrate, $\text{NaOH} \cdot \text{H}_2\text{O}$, while the physical and/or chemical events giving rise to the three endotherms in the temperature range of 225° to 260° C are not known. The same three endotherms were seen in duplicate tests. The onset temperatures for the start of exothermic behavior for the sodium stearate and the mixtures with stearate are recorded in table 4.

In the tests with samples S-7 to S-10, a very shallow exotherm was noted in the thermograms, and respective onset temperatures are also given in table 4. With the increased amounts of sodium stearate in these four mixtures, perhaps a reaction is observed that was not evident in the thermograms with mixtures S-5 and S-6 which contain smaller amounts of the stearate. At the same time, it should be noted that as the mixtures are heated and the components undergo various crystalline phase changes, melting and/or mixing and dissolution, their thermal properties change. Thermal

Table 4.- DSC thermal analysis results for stearate and stearate mixtures

Sample	Sample weight, mg	Heating rate, °C/min	Onset temperature, ¹ °C	Onset of very shallow exotherm, ³ °C	Peak temperature for sodium nitrite phase transition, °C
Sodium stearate	2.1	10	185	-	-
Sodium stearate	2.3	5	176	-	-
Sodium stearate	1.9	20	193	-	-
S-5-1	3.2	10	268	-	164.3
S-5-2	6.2	20	280	-	167.0
S-6-1	4.8	10	252	-	165.4
S-7-1	3.6	10	249	-	165.4
S-7-2	4.8	5	246	141	164.2
S-8-1	2.2	10	248	220	²
S-9-1	4.1	10	214	147	165.7
S-9-3	1.5	5	209	160	164.5
S-10-1	3.9	10	212	²	²
S-10-2	3.1	10	217	165	164.5

¹ Based on initial deviation of the decomposition step from baseline.

² Was not detected in the thermogram.

properties also change with changes in temperature, even if physical changes do not take place. Such changes, for example in heat capacity, are reflected in the baseline of a thermogram. Finally, the temperatures for the peaks of the solid phase transition of sodium nitrite are also presented in the table. For the eight tests, the average value for the peak is $165.1 \pm 0.9^\circ \text{C}$.

Sodium stearate and the mixtures with sodium stearate were tested in the TGA. The stearate is a very fine powder of low density, and a small amount filled the sample pan. Thermograms showing the various steps of weight loss are presented in figures 34 and 35 for the sodium stearate, and in figures 36 to 42 for the mixtures. In figure 34, for a test at a heating rate of 10°C/min , three main steps of weight loss are seen. The derivative curve shows additional peaks which indicate the various changes in mechanism and in the kinetics of the complete decomposition. For the test at 5°C/min , figure 35, weight loss was somewhat more uniform. Total weight loss in the two tests was 80.4 and 78.7 pct of original sample weight, respectively. Conversion to sodium carbonate would result in a weight loss of 82.7 pct. The residues from the tests were black, containing some carbon or char, thus explaining the small difference between the experimental and theoretical weight loss values.

The thermograms for the mixtures show weight loss of moisture from sodium hydroxide, other than the thermogram for sample S-9, in figure 42, which did not contain sodium hydroxide. Moisture loss was then followed, at higher temperature, by one major step of weight loss. A small amount of weight gain, prior to the main loss, is seen in some of the thermograms. Onset temperatures for decomposition for the mixtures are shown in table 5. It is clearly evident that the stearate itself starts to decompose at a lower temperature than the onset temperatures for the mixtures. Stearate decomposition starts at 190° to 210°C , depending on heating rate. Mixture S-9, containing only the stearate, nitrate and nitrite salts and no sodium hydroxide, starts to decompose at about 240°C , while the other

Table 5.- TGA thermal analysis results for stearate and stearate mixtures

Sample	Sample weight, mg	Heating rate, °C/min	Onset temperature, °C	Weight loss, pct	
				Total loss	Loss based on dry weight
Sodium stearate	2.23	10	211	80.4	80.4
Sodium stearate	1.67	5	191	78.7	78.7
S-5-1	6.43	10	264	6.3	3.4
S-6-1	7.65	10	277	8.7	5.6
S-6-2	3.57	20	307	10.3	6.4
S-7-1	6.79	10	304	11.1	6.9
S-8-1	4.14	10	302	16.1	11.8
S-8-2	5.49	10	298	13.2	11.3
S-8-3	5.12	5	295	14.4	12.2
S-9-1	5.02	10	241	11.8	11.4
S-10-1	4.55	10	302	10.1	3.2

¹ Based on initial deviation of the decomposition step from baseline.

mixtures, all containing hydroxide, begin to decompose at higher temperatures. These onset temperatures range from 264° to 304° C for the tests at 10° C/min. A similar trend was seen in the DSC results. Lowest onset temperatures for decomposition were observed for the stearate, and mixture S-9 had the lowest onset temperature among the mixtures. However, in general, onset temperatures for the stearate and for the mixtures were higher in the TGA than in the DSC by 20° to 90° C. Reactions can take place initially without a loss of large amounts of gas. The change in energy due to the reaction would then be noted first, at a lower temperature. Also, evolution of gas from melts is not

generally a fast event and depends on the viscosity of the melts. Perhaps the sodium hydroxide increases the viscosity of the melts with sodium stearate; this would partly explain the lowest onset temperature for mixture S-9 which does not contain sodium hydroxide.

It is of interest to compare results from tests with mixtures S-7, S-9 and S-10, all three containing the same amount of sodium stearate. Mixture S-9 which contains the largest amount of nitrate and nitrite, but no hydroxide, lost the most weight. Mixture S-7 with somewhat less nitrate and nitrite, and with some hydroxide, lost about half the amount of weight lost by mixture S-9 (6.9 vs. 11.4 pct). Mixture S-10, with the least amount of oxidants and the largest amount of hydroxide, lost the least weight. For the sodium stearate to decompose completely to carbonate, assuming no changes in the three other components, weight loss for the three mixtures would be 5.61 pct, based on the following reaction (6):

In addition to carbonate, stearone $[(C_{17}H_{35})_2CO]$ forms. Stearone has a low melting point, 88° C, and at higher temperatures it vaporizes. The reaction of nitrate and/or nitrite with the stearate will increase the weight loss even more, due to conversion of the nitrate to nitrite, and the conversion of the nitrite to the oxide and eventually to carbonate. Finally, anhydrous sodium salts of fatty acids, such as the stearic acid, can react with sodium hydroxide (7), forming sodium carbonate and a hydrocarbon, as follows:

Some of the hydrocarbons may be flammable.

In general, fatty acids are reported to be resistant to oxidation, but prolonged heating with oxidizers produces carbon dioxide and water (7). The results for the three mixtures indicate that when sufficient oxidant is present, weight loss is at a maximum. A similar conclusion can be drawn from comparison of weight loss values for mixtures S-5 and S-10. The same values, of 3.4 to 3.2 pct, were found, even though the amount of stearate in S-5 is only a third of that in S-10. These results indicate that mixture S-10 did not contain enough oxidant for reaction with the stearate. On the other hand, mixture S-8, with the largest amounts of stearate and hydroxide, but without comparably large amounts of oxidant(s), registered the greatest weight loss of the six mixtures. Thus, the conclusion is that the reactions are complex and involve all the components, and that the results depend on the ratios between the components and the rates of the various reactions. From the TGA thermograms it is evident that by 550° C, the final temperature in the tests, equilibrium has been attained. At much higher temperatures, the carbonates and unreacted oxidants will decompose.

Sodium Oxalate and Oxalate Mixtures

Sodium oxalate was tested in the main series of tests, as well as in this series. When heated in the DSC to 550° C, it did not undergo any significant reaction, as is seen in figure 43. A thermogram for a sample from mixture S-11 is shown in figure 44. In addition to the endotherm due to loss of moisture, there is the endotherm due to the combined melting of the two oxidants and possibly also the hydroxide. It is followed by a wide, shallow

exotherm, and then by the sharp exotherm due to the reaction of the oxalate with the oxidants. Figures 45 and 46 contain thermograms, obtained at 10° and 5° C/min, for samples from mixture S-12. Instead of one endotherm for eutectic melting, two endotherms are present. The shape of the first endotherm, due to melting, suggests a step-wise process. Both figures also contain a wide but shallow exotherm. It may arise from a reaction between the oxalate and the hydroxide. The thermogram for the test at 5° C/min, in figure 46, shows a more drastic change in the slope at about 304° C and three small exotherms superimposed on the shallow exotherm, in the temperature range between 330° and 370° C. The slow heating rate may help in the resolution of events. The residue from this last test was cooled in the sample pan, and then reheated at 20° C/min. The resultant thermogram, figure 47, shows only a variety of endotherms; the oxalate was completely consumed in the first test. Another sample from mixture S-12 was heated at 20° C/min. The thermogram is shown in figure 48, and is very similar to figure 45 for the test at 10° C/min. The onset temperatures for the first exothermic reaction in this mixture are about the same and vary between 261° and 268° C, while the onset temperatures for the final exothermic reaction vary from 391° to 437° C as the heating rate varies from 5° to 20° C/min.

Two thermograms for mixture S-13, for tests conducted at 10° and 5° C/min, are given in figures 49 and 50; they differ from the thermograms for mixtures S-11 and S-12. Starting at 233° and 238° C, respectively, for the tests at 10° and 5° C/min, a series of two or three major exotherms is seen, possibly due to a reaction between the oxalate and sodium hydroxide. A few small endotherms, superimposed on the first exotherm, indicate the fusion of the small amounts of the nitrate and the nitrite. Lastly, towards the end of

the thermograms, an array of small exotherms between -418° and 480° C represent the reaction between the unreacted sodium oxalate and the unreacted nitrate and/or nitrite. On the other hand, the last few peaks may be a result of a reaction of sodium oxide (formed in the decomposition of sodium nitrite) with either oxalate or carbon dioxide, with sodium carbonate being the product of either reaction. Similar small peaks were observed when a mixture of sodium oxalate and sodium nitrite was heated at 10° C/min, as shown in figure 51. Following the main reaction between the two compounds, a series of small exotherms appears. In the main reaction, the sodium nitrite converts to sodium oxide, while in the additional small steps the sodium oxide may combine with evolved carbon dioxide to form sodium carbonate, as well as react with leftover oxalate. Onset temperatures for exothermic behavior for the mixtures with oxalate are given in table 6.

The three mixtures with oxalate were also tested in the TGA. Figures 52 and 53 are for mixture S-11, heated at 10° and 20° C/min, and figures 54 and 55 are for mixtures S-12 and S-13, both obtained at a heating rate of 10° C/min. The thermograms for mixtures S-11 and S-12 show three stages of weight loss, the first due to loss of water and the next two due to chemical reactions. Thus, the shallow exotherms in the DSC thermograms represent a chemical reaction corresponding with the second phase of weight loss in the TGA thermograms, whereas, the last and sharp weight loss is due to the reaction of the unreacted portion of the oxalate with the oxidants or with their products. Finally, figure 55, for mixture S-13, shows a variety of reactions as seen in both the thermogram and the derivative curve. The final reaction of oxalate with the small amount of oxidant(s), starting at 483° C, is followed by another continuous weight loss which corresponds with the array

Table 6.- DSC thermal analysis results for oxalate mixtures

Sample	Sample weight, mg	Heating rate, °C/min	Onset temperature for the shallow, wide exotherm, ¹ °C	Onset temperature for exothermic reaction, ¹ °C	Onset temperature for final exothermic reaction, ¹ °C	Peak temperature for sodium nitrite phase transition, ² °C
S-11-1	3.5	10	271	-	409	165.9
S-12-1	3.5	10	262	-	409	166.5
S-12-2	2.5	5	261	-	391	163.9
S-12-4	2.3	20	268	-	437	-
S-13-1	3.1	10	132	233	429	165.9
S-13-3	2.4	5	115	238	418	165.3

¹Based on initial deviation of the decomposition step from baseline.

²Average peak temperature: 165.5°±1.0° C.

reaction of oxalate with the small amount of oxidant(s), starting at 483° C, is followed by another continuous weight loss which corresponds with the array of small peaks as seen in the DSC thermograms for samples of mixture S-13 (figures 49 and 50). Onset temperatures for exothermic behavior for the mixtures with oxalate are presented in table 7.

Table 7.- TGA thermal analysis results for oxalate mixtures

Sample	Sample weight, mg	Heating rate, °C/min	Onset temperature, ¹ °C	Onset temperature for final exothermic reaction, ¹ °C	Weight loss, pct	
					Total loss	Loss based on dry weight
S-11-1	3.28	10	322	418	10.1	5.2
S-11-2	2.74	10	322	424	7.8	4.5
S-11-3	4.83	20	284	438	5.6	3.2
S-12-1	4.81	10	255	423	12.2	7.6
S-12-2	4.56	10	258	413	13.4	9.6
S-13-1	5.71	10	104	483	13.6	8.5

¹ Based on initial deviation of the decomposition from baseline.

CONCLUSIONS

A series of tests were conducted for Westinghouse Hanford Company on a set of thirteen compositions. These tests evaluated the thermal characteristics of these compositions, which included combinations of three fuels with two oxidants and one base, at various weight ratios. The fuels were sodium salts of acetate, stearate and oxalate. The oxidants were sodium nitrate and sodium nitrite, and the base was sodium hydroxide.

In the thermal tests, using the differential scanning calorimeter and the thermogravimetric analyzer, the onset temperatures for initiation of exothermic behavior were determined for the mixtures. Onset temperatures for the acetate mixtures ranged from 213° to 235° C in the DSC tests, and from 216° to 237° C in the TGA tests. Considering the variation in the

concentration of the components, the variation in onset temperatures is small. Onset temperatures for the stearate mixtures varied from 214° to 268° C for the DSC tests in which the heating rate was 10° C/min; for the TGA tests, respective values were between 241° to 304° C. In the mixtures that contained larger amounts of stearate and hydroxide and smaller amounts of the oxidants (S-7 to S-10), a very shallow exotherm was noted, starting at as low a temperature as 141° C. A corresponding loss of weight was not noted in the TGA thermograms. It could be that the amount of gas released during this phase was minimal, or that this reaction was more of a mixing-type or dissolution reaction. Nonetheless, any exothermic event where heat is produced, even if in small amounts, should be noted. In these stearate mixtures, the concentration of sodium hydroxide varied by more than an order of magnitude, and the concentration of stearate also changed by a larger margin than in the mixtures with sodium acetate. The larger spread in onset values for the stearate mixtures can be understood on this basis.

Finally, for the three oxalate mixtures in which the concentration of all four components changed substantially, shallow exotherms were observed for all the mixtures (S-11 to S-13). Onset temperatures for mixtures S-11 and S-12 were at 260° to 270° C; for mixture S-13 they were at 115° to 130° C. A corresponding weight loss was noted in the TGA thermograms at equivalent or higher temperatures. In these initial exothermic reactions, only a portion of the oxalate was consumed; the rest underwent a fast reaction with the nitrate and/or nitrite that was also seen in the previous series of tests.

In addition to exothermic phenomena, the release of flammable gases during the decomposition reactions for some of these mixtures should be of great concern. The presence of sodium hydroxide affects the behavior of the

mixtures in at least two ways. First, some onset temperatures are quite low, due to reaction of the fuel(s) with the hydroxide or with eutectic melts containing hydroxide. Second, reactions of fuel(s) with sodium hydroxide result, in some cases, in the evolution of flammable gas (5, 7). A recent publication (8) concerning the formation of hydrogen in some of the waste tanks also notes the role of hydroxide. In mixtures containing, for example, EDTA (ethylenediamine tetraacetic acid), formaldehyde forms and is converted through a series of steps to hydrogen, according to the authors. Alkaline hydroxide is necessary for these reactions.

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APPENDIX

At the end of the test series, a check on the calibration of the DSC was conducted with both indium and zinc standards. Their melting points were found to be 157.6° and 419.6° C, respectively. Literature values are 156.4° and 419.5° C. Both experimental values are well within the accepted variation for this test series. Also, as can be seen for zinc, the fine tuning conducted during the initial calibration was maintained. In addition, the balance and slope of the DSC were also checked at the initiation and at the end of the test series, and both were fine.

At the end of the test series, a check on the calibration of the TGA was conducted with the 100-mg weight and with pure nickel. The Curie temperature for nickel was 353.2° C, well within the literature value of 354° C.

The solid phase transition of sodium nitrite was used as an internal standard in the DSC tests. Temperatures found were 165.0°±0.8° C, 165.1°±0.9° C, and 165.5°±1.0° C, for the three fuel mixtures. Variation was small, considering the fact that sodium nitrite was one of four ingredients.

Onset temperatures for exothermic decomposition or reaction in the DSC tests, and onset temperatures for initiation of weight loss due to decomposition or reaction in the TGA tests for all the mixtures varied by ±10° C.

Curve 1: DSC
File info: Cadmium Wed Sep 1 17:35:00 1993
Sample Weight: 5.200 mg
Cadmium

Glass Beads in Reference
DSC 21:8 8 TIMES: 0.0 min RATE: 10.0 °C/min
YH
PERKIN-ELMER
7 Series Thermal Analysis System
Wed Sep 1 17:39:09 1993

Figure 1. - DSC thermogram for cadmium

WMC-MR-0455, Supp. 1

Curve 1: TGA
File info: N1-Check-2 Wed Oct 13 10:41:06 1993
Sample Weight: 3.780 mg
Nickel Curie Point Check

1 Nickel Curie Point Check
Weight (mg)

WMC-MR-0455, Supp. 1

TIME: 0.0 min RATE: 10.0 C/min

YM
PERKIN-ELMER
7 Series Thermal Analysis System
Wed Oct 13 11:02:39 1993

Figure 2. - TGA thermogram for nickel

ve 1: DSC

le info: Ace21A-2 Fri Sep 3 11:36:23 1993

ple Weight: 4.320 mg

dium Acetate (2)

1 Sodium Acetate (2)

Heat Flow (mW)

Glass Beads in Reference

TIME: 57.8 s TIME: 0.0 min RATE: 10.0 C/min

Temperature (°C)

YM & PE

PERKIN-ELMER

7 Series Thermal Analysis System

Fri Sep 3 11:48:57 1993

Figure 3. - DSC thermogram for sodium acetate

Curve 1: DSC

File info: Ace21A-3 Fri Sep 3 13:39:25 1993

Sample Weight: 4.320 mg

Sodium Acetate (3)

1 Sodium Acetate (3)

Heat Flow (mW)

Glass Beads in Reference

TIME: 0.0 min RATE: 5.0 C/min

Temperature (°C)

YM & PE

PERKIN-ELMER

7 Series Thermal Analysis System

Fri Sep 3 13:46:36 1993

Figure 4. - DSC thermogram for sodium acetate

Curve 1: DSC
File info: Ace21A-4 Fri Sep 3 15:47:10 1993
Sample Weight: 4.010 mg
Sodium Acetate (4)

Glass Beads in Reference

WE 22.8 g TIME: 0.0 min RATE: 5.0 C/min

Temperature (°C)

YM & PE
PERKIN-ELMER
7 Series Thermal Analysis System
Fri Sep 3 16:16:12 1993

Figure 5. - DSC thermogram for sodium acetate

File 1: DSC
Info: S-1-2 Wed Sep 8 15:43:09 1993
Sample Weight: 5.550 mg
Element (2)

1 Supplement (2)
Heat Flow (mW)

Glass Beads Reference Used

TIME: 0.0 min RATE: 10.0 C/min

Temperature (°C)

YM

PERKIN-ELMER

7 Series Thermal Analysis System

Wed Sep 8 16:08:31 1993

Figure 6. - DSC thermogram for sodium acetate mixture S-1

MHC-MR-0455, Supp. 1

urve 1: DSC

ile info: S-1-7

Fri Oct 8 14:12:00 1993

ample Weight: 4.100 mg

upplement 1 (7)

1 Supplement 1 (7)

Heat Flow (mW)

Glass Beads Reference Used

Temperature (°C)

YM

PERKIN-ELMER

7 Series Thermal Analysis System

Fri Oct 8 14:36:32 1993

Figure 7. - DSC thermogram for sodium acetate mixture S-1

File 1: DSC
Info: S-1-8 Fri Oct 8 16:32:43 1993
Sample Weight: 3.000 mg
Element 1 (8)

1 Supplement 1 (8)
Heat Flow (mW)

Glass Beads Reference Used
TIME: 0.0 min RATE: 5.0 C/min
Temperature (°C)
YM
PERKIN-ELMER
7 Series Thermal Analysis System
Fri Oct 8 16:33:26 1993

Figure 8. - DSC thermogram for sodium acetate mixture S-1

WHC-MR-0455, Supp. 1

Curve 1: DSC

File info: S-1-7 Fri Oct 8 14:12:00 1993

Sample Weight: 4.100 mg

Supplement 1 (7)

Supplement 1 (7)

Glass Beads Reference Used
TEMP: 21.0 °C TIME: 0.0 min RATE: 10.0 °/min

Temperature (°C)

YM
PERKIN-ELMER
7 Series Thermal Analysis System
Fri Oct 8 14:36:32 1993

Figure 7A. - DSC thermogram for sodium acetate mixture S-1

ve 1: DSC
e info: S-1-B Fri Oct 8 16:32:43 1993
ple Weight: 3.000 mg
plement 1 (8)

Supplement 1 (8)

Glass Beads Reference Used
E 21.0 g TIME: 0.0 min RATE: 5.0 C/min

Temperature (°C)

YM
PERKIN-ELMER
7 Series Thermal Analysis System
Fri Oct 8 16:33:26 1993

Figure 8A. - DSC thermogram for sodium acetate mixture S-1

File info: S-1-5 Mon Sep 27 10:51:53 1993

Sample Weight: 3.800 mg

Supplement 1 (5)

1 Supplement 1 (5)

Heat Flow (mW)

Glass Beads Reference Used

Temperature (°C)

TIME: 55.0 s TIME: 0.0 min RATE: 10.0 C/min

YH

PERKIN-ELMER

7 Series Thermal Analysis System

Mon Sep 27 10:53:28 1993

Figure 9. -- DSC thermogram for sodium acetate mixture S-1

--- thermogram for residue from sodium acetate mixture S-1

Curve 1: DSC
File info: S-2-3 Tue Sep 21 15:51:02 1993
Sample Weight: 3.600 mg
Supplement 2 (3)

1 Supplement 2 (3)
Heat Flow (mW)

Glass Beads In Reference

Temperature (°C)

YH
PERKIN-ELMER
7 Series Thermal Analysis System
Tue Sep 21 15:51:39 1993

MHC-MR-0455, Supp. 1

Figure 11. - DSC thermogram for sodium acetate mixture S-2

Curve 1: DSC

File info: S-2-4 Fri Oct 8 18:27:56 1993

Sample Weight: 2.900 mg

Supplement 2 (4)

Supplement 2 (4)

WHC-MR-0455, Supp. 1

Figure 12. - DSC thermogram for sodium acetate mixture S-2

Curve 1: DSC

File info: S-3-2

Wed Sep 22 15:10:33 1993

Sample Weight: 3.200 mg

Supplement 3 (2)

1 Supplement 3 (2)

Heat Flow (mW)

Glass Beads In Reference

TIME: 20.0 min RATE: 5.0 C/min

YM

PERKIN-ELMER

7 Series Thermal Analysis System

Fri Oct 8 13:34:02 1993

WMC-MR-0455, Supp. 1

Figure 13. - DSC thermogram for sodium acetate mixture S-3

Curve 1: DSC

File info: S-3-1 Tue Sep 21 17:34:56 1993

Sample Weight: 1.800 mg

Supplement 3 (1)

1 Supplement 3 (1)

Heat Flow (mW)

Glass Beads In Reference

Temperature (°C)

YN

PERVOTM-ELMER

7 Series Thermal Analysis System

Tue Sep 21 17:35:19 1993

TEMP: 33.0 °C TIME: 0.0 min RATE: 10.0 °/min

Figure 14.- DSC thermogram for sodium acetate mixture S-3

Curve 1: DSC

File info: S-3-3

Wed Sep 22 15:54:42 1993

Sample Weight: 2.800 mg

0.0

1 Supplement 3 (3)

Heat Flow (mW)

Glass Beads In Reference

Temperature (°C)

DATE: 09.22.93 TIME: 0.0 min RATE: 20.0 C/min

YM

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Wed Sep 22 15:56:40 1993

Figure 15. - DSC thermogram for sodium acetate mixture S-3

Curve 1: DSC

File info: S-4-1 Wed Sep 22 17:20:15 1993

Sample Weight: 2.700 mg

Supplement 4 (1)

1 Supplement 4 (1)

Heat Flow (mW)

Glass Beads In Reference

Temperature (°C)

WEIGHT: 2.700 g TIME: 0.0 min RATE: 10.0 C/min

YM

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Wed Sep 22 17:24:22 1993

Figure 16.- DSC thermogram for sodium acetate mixture S-4

Curve 1: TGA

File info: Ace21A-1 Wed Oct 13 12:14:48 1993

Sample Weight: 2.739 mg

Na Acetate 21A (1)

1 Na Acetate 21A (1)

% Weight (Wt. %)

TEMP: 55.0 C TIME: 0.0 min RATE: 10.0 C/min

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Wed Oct 13 12:16:10 1993

WHC-MR-0455, Supp. 1

Figure 17. - TGA thermogram for sodium acetate

Curve 1: TGA

File info: S-1-1

Wed Oct 13 14:22:46 1993

Sample Weight: 5.689 mg

Supplement 1 (1)

1 Supplement 1 (1)

% Weight (Wt. %)

WHC-MR-0455, Supp. 1

Figure 18. - TGA thermogram for sodium acetate mixture S-1

Curve 1: TGA
 File Info: S-2-1 Wed Oct 13 16:48:46 1993
 Sample Weight: 5.103 mg
 Supplement 2 (1)

Figure 19. - TGA thermogram for sodium acetate mixture S-2

Curve 1: TGA

File info: S-3-1

Wed Oct 13 16:02:09 1993

Sample Weight: 3.602 mg

Supplement 3 (1)

1 Supplement 3 (1)

1st Derivative (%/min x 10⁻¹)

2 % Weight (Wt. %)

Figure 20. - TGA thermogram for sodium acetate mixture S-3

Curve 1: TGA

File info: S-4-1

Thu Oct 14 12:10:17 1993

Sample Weight: 5.542 mg

Supplement 4 (1)

Supplement 4 (1)

Figure 21. - TGA thermogram for sodium acetate mixture S-4

Curve 1: TGA

File info: S-4-2

Thu Oct 14 14:13:24 1993

Sample Weight: 5.528 mg

Supplement 4 (2)

1 Supplement 4 (2)

1st Derivative (%/min $\times 10^{-4}$)

Air

TEMP: 87.8 C TIME: 0.0 min RATE: 5.0 C/min

Temperature (°C)

YH

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Figure 22. - TGA thermogram for sodium acetate mixture S-4

Curve 1: DSC

File info: Stear22A-2 Fri Oct 8 10:50:07 1993

Sample Weight: 2.300 mg

Na Stearate 22A (2)

1 Na Stearate 22A (2)

Heat Flow (mW)

Air

Glass Beads in Reference

TEMP: 25.0 °C

TIME: 0.0 min

RATE: 5.0 °C/min

Temperature (°C)

YM

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Fri Oct 8 10:50:35 1993

WHC-MR-0455, Supp. 1

Figure 23. - DSC thermogram for sodium stearate

urve 1: DSC

ile info: Stear22A-1 Thu Sep 23 13:04:33 1993

ample Weight: 2.100 mg

a Stearate 22A (1)

1 Na Stearate 22A (1)

Heat Flow (mW)

Glass Beads in Reference
EXP: 21.8 g TIME: 0.0 min RATE: 10.0 C/min
DSC: 000.0 g

Temperature (°C)

YM
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Thu Sep 23 13:04:56 1993

Figure 24. - DSC thermogram for sodium stearate

Curve 1: DSC

File info: Stear22A-3 Fri Oct 8 11:50:54 1993

Sample Weight: 1.900 mg

Na Stearate 22A (3)

1 Na Stearate 22A (3)

Heat Flow (mW)

Glass Beads in Reference

TEMP: 21.0 °C TIME: 0.0 min RATE: 20.0 °C/min

Temperature (°C)

YM

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Fri Oct 8 11:54:41 1993

Figure 25. - DSC thermogram for sodium stearate

Curve 1: DSC

File info: S-5-1

Thu Sep 23 14:18:41 1993

Sample Weight: 3.200 mg

Supplement 5 (1)

1 Supplement 5 (1)

Heat Flow (mW)

Glass Beads In Reference

Temperature (°C)

NP1: 21.8 g TIME1: 0.0 min RATE1: 10.0 C/min

YM

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Thu Sep 23 14:36:36 1993

Figure 26. - DSC thermogram for sodium stearate mixture S-5

Curve 1: DSC

File info: S-5-2

Thu Sep 23 15:22:32 1993

Sample Weight: 6.200 mg

Supplement 5 (2)

1 Supplement 5 (2)

Heat Flow (mW)

Glass Beads In Reference

Temperature (°C)

TIME: 0.0 min RATE: 20.0 C/min

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Thu Sep 23 15:23:27 1993

Figure 27. - DSC thermogram for sodium stearate mixture S-5

urve 1: DSC

ile info: S-6-1

Thu Sep 23 16: 41: 24 1993

ample Weight: 4.800 mg

upplement 6 (1)

1 Supplement 6 (1)

Heat Flow (mW)

Glass Beads In Reference

Temperature (°C)

YM

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Thu Sep 23 16: 42: 42 1993

Figure 28. - DSC thermogram for sodium stearate mixture S-6

Curve 1: DSC

File info: S-7-1 Mon Sep 27 13:58:42 1993

Sample Weight: 3.600 mg

Supplement 7 (1)

Supplement 7 (1)

Glass Beads in Reference
TEMP: 21.0 °C TIME: 0.0 min RATE: 10.0 °C/min
TEMP: 464.0 °C

Temperature (°C)

YM
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Mon Sep 27 15:01:32 1993

WMC-MR-0455, Supp. 1

Figure 29. - DSC thermogram for sodium stearate mixture S-7

Curve 1: DSC

File info: S-7-2

Mon Sep 27 15: 48: 35 1993

Sample Weight: 4.800 mg

Supplement 7 (2)

1 Supplement 7 (2)

Heat Flow (mW)

Glass Beads in Reference

Temperature (°C)

TEMP: 31.0 °C TIME: 0.0 min RATE: 5.0 °C/min

YM

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Mon Sep 27 15: 57: 54 1993

Figure 30. - DSC thermogram for sodium stearate mixture S-7

WMC-MR-0455, Supp. 1

Curve 1: DSC

File info: S-8-1 Mon Sep 27 17:02:41 1993

Sample Weight: 2.200 mg

Supplement 8 (1)

Supplement 8 (1)

Figure 31. - DSC thermogram for sodium stearate mixture S-8

Curve 1: DSC

File info: S-9-1

Tue Sep 28 10:53:07 1993

Sample Weight: 4.100 mg

Supplement 9 (1)

1 Supplement 9 (1)

Heat Flow (mW)

Glass Beads in Reference

Temperature (°C)

YM

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Tue Sep 28 10:53:42 1993

Figure 32. - DSC thermogram for sodium stearate mixture S-9

Curve 1: DSC

File info: S-10-1

Tue Sep 28 14: 47: 59 1993

Sample Weight: 3.900 mg

Supplement 10 (1)

1 Supplement 10 (1)

Heat Flow (mW)

Glass Beads in Reference

TIME: 31.8 s TIME: 0.0 min RATE: 10.0 C/min

Temperature (°C)

YM

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Tue Sep 28 14: 48: 25 1993

Figure 33. - DSC thermogram for sodium stearate mixture S-10

Curve 1: TGA
File info: Stear22A-1 Thu Oct 14 16:26:36 1993
Sample Weight: 2.230 mg
Na Stearate 22A (1)

Figure 34. - TGA thermogram for sodium stearate

Na Stearate 22A (2)

% Weight (Wt. %)

2 1st Derivative (%/min)

Figure 35. – TGA thermogram for sodium stearate

Curve 1: TGA

File info: S-5-1

Fri Oct 15 13:07:21 1993

Sample Weight: 6.427 mg

Supplement 5 (1)

Curve 1: TGA

File info: S-6-1

Fri Oct 15 14:23:31 1993

Sample Weight: 7.648 mg

Supplement 6 (1)

1 Supplement 6 (1)

1st Derivative (%/min $\times 10^{-2}$)

2 % Weight (Wt. %)

WHC-MR-0455, Supp. 1
(% Wt) TGA10M

Air
TEMP1: 20.0 C
TEMP2: 550.0 C
TIME1: 0.0 min
RATE1: 10.0 C/min

Temperature (°C)

YM
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Fri Oct 15 15:05:06 1993

Figure 37. - TGA thermogram for sodium stearate mixture S-6

Curve 1: TGA

File info: S-6-2

Fri Oct 15 16:44:31 1993

Sample Weight: 3.573 mg

Supplement 6 (2)

Figure 38. - TGA thermogram for sodium stearate mixture S-6

Curve 1: TGA

File info: S-7-1

Fri Oct 15 15:52:44 1993

Sample Weight: 6.787 mg

Supplement 7 (1)

1 Supplement 7 (1)

% Weight (Wt. %)

Air
TEMP: 25.0 C
TEMP: 550.0 C
TIME: 0.0 min RATE: 10.0 C/min

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WHC-MR-0455, Supp. 1

Figure 39. - TGA thermogram for sodium stearate mixture S-7

Curve 1: TGA

File info: S-8-2

Mon Oct 18 11:17:51 1993

Sample Weight: 5.493 mg

Supplement 8 (2)

1 Supplement 8 (2)

% Weight (Wt. %)

Figure 40. - TGA thermogram for sodium stearate mixture S-8

Curve 1: TGA

File info: S-9-1

Mon Oct 18 12:49:11 1993

Sample Weight: 5.018 mg

Supplement 9 (1)

1 Supplement 9 (1)

% Weight (Wt. %)

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Figure 41. - TGA thermogram for sodium stearate mixture S-9

Curve 1: TGA

File info: S-10-1 Mon Oct 18 14:06:20 1993

Sample Weight: 4.549 mg

Supplement 10 (1)

1 Supplement 10 (1)

% Weight (Wt. %)

Air
TEMP: 88.0 °C
TIME: 0.0 min RATE: 10.0 °C/min

Temperature (°C)

YH
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Mon Oct 18 14:09:58 1993

WHC-MR-0455, Supp. 1

Figure 42. - TGA thermogram for sodium stearate mixture S-10

Curve 1: DSC

File info: 0x4A-6

Tue Nov 23 09:29:16 1993

Sample Weight: 3.000 mg

Na Ox 4A (6)

1 Na Ox 4A (6)

Heat Flow (mW)

Glass Beads Reference Used

Temperature (°C)

TEMP1: 25.0 C TIME1: 0.0 min RATE1: 10.0 C/min

TEMP2: 550.0 C

YM

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Figure 43. - DSC thermogram for sodium oxalate

Curve 1: DSC

File info: S-11-1 Tue Sep 28 17:08:27 1993

Sample Weight: 3.500 mg

Supplement 11 (1)

1 Supplement 11 (1)

Heat Flow (mW)

Glass Beads in Reference

Wt: 31.0 g TIME: 0.0 min RATE: 10.0 °C/min

Temperature (°C)

YH

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Tue Sep 28 17:08:48 1993

Figure 44. -.DSC thermogram for sodium oxalate mixture S-11

Curve 1: DSC

File info: S-12-1 Tue Sep 28 18:23:12 1993

Sample Weight: 3.500 mg

Supplement 12 (1)

1 Supplement 12 (1)

Heat Flow (mW)

Glass Beads in Reference

Temperature (°C)

YM

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Figure 45. - DSC thermogram for sodium oxalate mixture S-12

WHC-MR-0455, Supp. 1

Curve 1: DSC

File info: S-12-2 Wed Sep 29 14:18:13 1993

Sample Weight: 2.500 mg

Supplement 12 (1)

1 Supplement 12 (1)

Heat Flow (mW)

Glass Beads in Reference

Temperature (°C)

TEMP: 88.0 °C TIME: 0.0 min RATE: 5.0 °C/min

YM

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Wed Sep 29 14:18:35 1993

WHC-MR-0455; Supp. 1

Figure 46. = DSC thermogram for sodium oxalate mixture S-12

Curve 1: DSC

File Info: S-12-3

Wed Sep 29 14: 49: 01 1993

Sample Weight: 2.100 mg

Supplement 12 (3)

1 Supplement 12 (3)

Heat Flow (mW)

Glass Beads in Reference

Temperature (°C)

TEMP: 31.8 °C TIME: 0.0 min RATE: 20.0 °C/min

YM

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Wed Sep 29 14: 51: 09 1993

Figure 47. - DSC thermogram for residue from sodium oxalate mixture S-12

Curve 1: DSC

File info: S-12-4 Wed Sep 29 15:29:27 1993

Sample Weight: 2.300 mg

Supplement 12 (4)

1 Supplement 12 (4)

Heat Flow (mW)

Glass Beads in Reference

Temperature (°C)

YM

TIME: 31.0 g TIME: 0.0 min RATE: 20.0 C/min

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Wed Sep 29 15:30:16 1993

Figure 48. - DSC thermogram for sodium oxalate mixture S-12

Curve 1: DSC

File info: S-13-1 Wed Sep 29 16: 58: 00 1993

Sample Weight: 3.100 mg

Supplement 13 (1)

1 Supplement 13 (1)

Heat Flow (mW)

Glass Beads in Reference

Temperature (°C)

TEMP: 21.0 °C TIME: 0.0 min RATE: 10.0 °C/min

YM

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Wed Sep 29 16: 58: 30 1993

Figure 49. - DSC thermogram for sodium oxalate mixture S-13

Curve 1: DSC
File info: S-13-3 Thu Oct 7 17:46:40 1993
Sample Weight: 2.400 mg
Supplement 13 (3)

1 Supplement 13 (3)
Heat Flow (mW)

Glass Beads in Reference Temperature (°C) YM
TIME: 21.0 g TIME: 0.0 min RATE: 5.0 C/min PERKIN-ELMER
TEMP: 550.0 g 7 Series Thermal Analysis System
Thu Oct 7 17:47:05 1993

Figure 50. - DSC thermogram for sodium oxalate mixture S-13

WHC-MR-0455, Supp. 1

ve 1: DSC

le info: Ox19A-2 Tue Aug 31 15: 09: 33 1993

ample Weight: 3.100 mg

Ox/Na Nitrite 19A (2)

1 Na Ox/Na Nitrite 19A (2)

Heat Flow (mW)

Glass Beads Reference Used

P1: 31.0 C TIME1: 0.0 min RATE1: 10.0 C/min
P2: 500.0 C

Temperature (°C)

YM
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Tue Aug 31 15: 09: 55 1993

Figure 51. - DSC thermogram for a mixture of sodium oxalate with sodium nitrite

Curve 1: TGA

File info: S-11-2 Tue Oct 19 14:25:09 1993

Sample Weight: 2.744 mg

Supplement 11 (2)

1 Supplement 11 (2)

1st Derivative (%/min $\times 10^{-1}$)

Figure 52. - TGA thermogram for sodium oxalate mixture S-11

Curve 1: TGA

File info: S-11-3 Tue Oct 19 15:17:34 1993

Sample Weight: 4.830 mg

Supplement 11 (3)

1 Supplement 11 (3)

% Weight (Wt. %)

Figure 53. - TGA thermogram for sodium oxalate mixture S-11

ve 1: TGA
e Info: S-12-2 Tue Oct 19 16:36:44 1993
ple Weight: 4.555 mg
plement 12 (2)

1 Supplement 12 (2)
% Weight (Wt. %)

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Tue Oct 19 16:37:31 1993

Figure 54. TGA thermogram for sodium oxalate mixture S-12

Curve 1: TGA

File Info: S-13-1 Mon Oct 18 16:10:48 1993

Sample Weight: 5.714 mg

Supplement 13 (1)

1 Supplement 13 (1)

1st Derivative (%/min $\times 10^{-1}$)

2 % Weight (Wt. %)

WHC-MR-0455, Supp. 1

Air

DATE: 10/18/93 TIME: 00:00:00 RATE: 10.0 C/min

Temperature (°C)

YH PERKIN-ELMER

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Figure 55. - TGA thermogram for sodium oxalate mixture S-13

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