Thermal Analysis of RFETS SS&C

by

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SUMMARY

Thermal analysis of plutonium bearing mixtures was conducted as part of a SRTC effort to determine the gas generation rate and composition for Rocky Flats Environmental Technology Site (RFETS) residue which is expected to be shipped to SRS. A model has been used to estimate the amount of gas that may be generated due to radiolysis. The weight changes that occur at 210°C is of particular interest since this temperature corresponds to the practice of holding at 210°C until there is no weight change with time (i.e., zero slope) employed at Rocky Flats. Thermogravimetric analysis revealed that PuO_2 can adsorb approximately 3% water, while mixtures of CaO , Ca(OH)_2 , and CaCO_3 are difficult to characterize due to decomposition, and MgO adsorbs little water, if any. The composition of certain sand, slag, and crucible material (SS&C) can be determined from the decomposition products, while the presence of metallic species (e.g., Ca and Pu) can only be estimated from weight gains. To confound the weight gain data, calcium hydroxide adsorbs carbon dioxide from the air at temperatures above approximately 200°C to form CaCO_3 . Thermal drift and buoyancy adversely affected the instrument precision at the small weight losses that were exhibited by SS&C material.

BACKGROUND

In support of the gas generation test program (GGTP) for the 9975 shipping container, thermogravimetric analysis (TGA) was conducted. The objective of this activity was to determine the moisture content as an input to the gas generation model. Hydrogen produced from the radiolysis of water could adversely affect the shipping safety. The model uses moisture content, radiation dose, and quantity of matrix components to predict gas generation rates.

There are many unknowns within the model, especially with respect to the gas species formed and the effect of the composition. For instance, the SS&C materials are packaged in air and may contain other adsorbed species. Therefore, reactions that form products other than H_2 and O_2 are expected. The scientific studies that could be conducted are immense, but the overall task only considered these elements:

- Effects of matrix component variation in residue mixtures
- Effects of decreasing moisture content in the shipping matrix
- Effect of package temperature during shipping.

There is a complex matrix of materials and mixtures that need to be transported between DOE sites to stabilize Pu for disposition. However, even with defined content limits for the number and type of matrix materials, the possible combinations are large in number. The initial activity

evaluated combinations of materials that produced larger than expected quantities of hydrogen to establish "worst case" conditions.

TEST PROCEDURE

Equipment Description

Thermogravimetric analysis (TGA) was conducted on a Thermal Analyzer (TA) instruments model 951 TGA. The instrument continuously measures the amount and rate of weight change of material either isothermally, as a function of time, or as a function of increasing temperature or some combination thereof in a controlled atmosphere. A photo of the instrument is shown in Figure 1. The TGA module is comprised of a furnace, a balance and an electronics housing. The furnace is resistance wound and has a maximum temperature capability of 1200°C with a heating rate range of 0.5 to 50°C/min. A platinum thermocouple inside the furnace wall is used to control the temperature.

The balance assembly consists of the balance housing, a control chamber, and a sample chamber. Mounted in the balance housing, a taut-band meter movement is attached to the balance beam. The ends of the beam extend into the sample and control chambers. The control chamber contains a shutter assembly and a counterweight arm mounted on the beam, and a beam stop pin, a lamp, and photodiodes mounted on a member extending from the balance housing. The sample chamber contains a quartz support rod which is attached to the beam, a sample pan attached to the rod, and a sample thermocouple. A quartz furnace tube encloses the sample chamber.

The TGA 951 operates on a null-balance principle. The beam is held in a horizontal position by an optically actuated servo loop. A light shutter is attached to the control end of the balance beam.

A constant intensity lamp is focused through an aperture slit in the shutter to strike two vertically mounted photodiodes. When the balance is in the null position, the focussed light strikes both diodes

Figure 1. Model 951 TGA and computer controller and data acquisition system.

equally. As the sample gains or loses weight, the beam becomes unbalanced and the intensity of the light hitting each photodiode is different. The voltage output from the more intensely illuminated photodiode increases and unbalances the photodiode amplifier, producing a current flow in the taut-band meter which acts as a restoring force on the balance beam, driving it back to its null position. The magnitude of the restoring force (current) is directly proportional to the sample weight loss or gain. After the signal is conditioned, the sample weight loss or gain is displayed in a real time plot as either percent weight change or absolute weight change.

Figure 2. Schematic of the TGA. Note the variable placement of the thermocouple in relation to the sample pan.

A personal computer is used for control, set-up, and data acquisition. The desired heating profile and pertinent features for the test and sample are entered prior to starting the test. The data acquisition interval is selected so that an appropriate amount of data are collected. The data for this work were collected every 3 seconds during the heating and cooling cycle.

The instrument in 773-A, D0115 was calibrated by an outside source while the instrument in 773-A, C163 was calibrated by comparison to the one in D0115. Based on a sample of four runs of $\text{Ca}(\text{OH})_2$ decomposition, the repeatability of the tests is estimated at 3.2% of the measurement (two sigma/average).

Figure 3. Typical heating and cooling profile (right hand scale) used to determine moisture contents (uncorrected data) and decomposition temperatures.

SAMPLE TESTING

Constituent powders and mixtures of powder samples were obtained from the tests that were described in the task technical plan (1). The samples had been mixed to desired compositions or subjected to a variety of moisture conditions. The TGA was used to determine the actual uptake/release of moisture and the decomposition temperatures of adsorbed species and components. Radioactive samples were tested in a radiological glove box in lab 773-A, C163 while "clean" samples were tested in lab 773-A, D0115. The sample size was between 15 and 20 mg for either instrument. The sample was heated from room temperature to 200°C (programmed temperature, actual was $\pm 15^\circ\text{C}$) at a rate of 10°C/min, the sample was held at 200°C for 30 minutes and subsequently heated to 900°C at 10°C/min and held for 5 minutes prior to being cooled at 20°C/min (programmed) to room temperature. The typical heating and cooling cycle for a sample of RFETS SS&C is shown in Figure 3.

A baseline sample was run to determine the extent of thermal buoyancy and instrument heating on the mass change. A nominal linear increase in mass was determined with increasing temperature. The observed weight change was approximately 0.120 mg on heating to 900°C, while a slope of 0.0002 mg/°C was calculated. The baseline data are shown in Figure 4a while the corrected runs are shown in Figure 4b and 4c. The correction to the data was somewhat

involved since the TA instrument DAQ does not output data in a format that can be read by standard DOS or windows programs. In order to make the correction, a hardcopy of the data was plotted and scanned. This scanned image was subsequently converted to x and y coordinates by a program entitled UN-SCAN-IT (2). This program generated x and y data compatible with Microsoft Excel®. The data were manipulated to determine the corrected weight losses and percent weight change. It is apparent that the correction is important for samples which exhibit small weight changes and less important for samples exhibiting large weight changes. Since the samples in this study exhibited small to moderate weight changes, it was necessary to make the corrections so that the data exhibited the correct weight change. The necessity of these data modifications revealed that the instrument was not sensitive enough to measure the small weight changes that occurred.

Table I. Slope and intercept for the baseline corrections of samples run on the TGA in the radiologic glovebox in "C" wing.

	Run 1	Run 2	Run 3	Run 4	Average	Standard dev.
Slope	0.000199	0.000189	0.000207	0.000224	0.000205	0.000015
Intercept	-0.0481	-0.0343	-0.1505	-0.0754	-0.0771	0.0518

RESULTS

In order to better understand the effects of chemical compounds likely to be found in SS&C mixtures on the evolution of gases during thermal decomposition, several raw materials were examined, (i.e., RF MgO, SRS MgO, reagent grade $\text{Ca}(\text{OH})_2$, and CaF, both individually and in mixtures). The raw and corrected data are attached in Appendix A. Summaries of the important features of the data are presented in Tables II through X.

There was little if any adsorbed moisture present on the MgO material and only a small amount of chemically bound moisture was detected. This chemically bound water was evolved at about 380°C. The total amount of water evolved was on the order of 0.09%, maximum.

$\text{Ca}(\text{OH})_2$ had a higher affinity for water both physically adsorbed and chemically combined. The amount of adsorbed moisture on $\text{Ca}(\text{OH})_2$ due to exposure to ambient condition varied from 0.5 to 1.5%. A characteristic scan of reagent grade $\text{Ca}(\text{OH})_2$ for heating and cooling is shown in Figure 5. This material exhibited a weight gain at about 200°C and two weight losses, one at 400 and the other at 650°C. The weight loss at approximately 400°C can be attributed to H_2O evolution while the second loss at approximately 650°C can be attributed to CO_2 evolution. As can be seen from the cooling cycle, the sample started to gain weight on cooling below about 425°C with a large increase at about 250°C. Tests to determine the compounds formed under cooling conditions were not conducted. There was no 200°C hold for these samples, although the 200°C hold (3) has been shown to encourage the formation of CaCO_3 . To determine the rate of formation of the reaction product, a TGA scan to 500°C was used. The sample gained weight immediately on cooling to below 250°C; there was no incubation period required for the weight

to increase, as shown in Figure 6. The sample was not subsequently heated to decompose the reaction product to determine its chemical nature.

Weight change data for the samples tested are presented in Tables II through VII. The weight loss at 210°C represents the loosely bound physically adsorbed moisture. In cases where no chemical decomposition occurred between RT and 500°C, a weight loss at 500°C and higher, if relevant, is presented. This high temperature weight change is used to determine the maximum amount of water that was adsorbed. The ratio of the amount of water removed

at 210° compared to that removed at 500°C is also presented. What is apparent from this data is that anywhere from 43 to 90% of the total adsorbed water on PuO₂ was desorbed at 210°C.

Since materials containing Mg(OH)₂, Ca(OH)₂, and CaCO₃, have decomposition temperatures below 500°C, no "total" moisture estimates can be made. In addition, the samples containing Ca(OH)₂ can gain weight in this temperature range due to a reaction with CO₂ in the air to form CaCO₃. For samples containing only Pu cake (as a mixture of 70% PuF₂, bal PuO₂), the 210°C thermal treatment accounted for 23 and 58% of the total weight loss through heating to 500°C. The weight change for samples comprised of Pu cake and NaF exhibited only 17% of the total weight loss after the 210°C – 30 minute hold (Table IV). The Pu cake can have a complex weight change since it is possible to hydrolyze the PuF₄ to form PuO₂ and HF. Samples containing both MgO and PuO₂ exhibited very little weight loss (from essentially 0 to about over 0.5%) at 210°C (Table V). Several of the samples continued to lose weight on heating to 500°C and beyond. These samples have total weight loss ratios that range from 5 to 84% depending on the constituents and treatment. This data suggests that a variable amount of moisture was removed by the thermal exposure at 210°C. The samples that contained both PuO₂ and Ca compounds, Table VI, could only be evaluated at 210°C due to the decomposition reactions, i.e., calcium hydroxide and calcium carbonate decomposition. The samples generally exhibited fractions of a percent weight loss on heating to 210°C and small weight gains during the hold at 210°C. This weight gain can be attributed to the reaction of Ca(OH)₂ with CO₂ to form CaCO₃. The RF SS&C samples, Table VII, exhibited weight losses that ranged from 0.2 to 2.5%, with a preponderance near 0.5%. The SS&C material contains Ca(OH)₂ and CaCO₃ in addition to some Ca metal and MgO. The calcium constituents make it difficult to determine the total fraction of water that is adsorbed on the surface of the powders since there are competing reactions to

desorb water from CaO , Ca(OH)_2 , and PuO_2 and to decompose Ca(OH)_2 , oxidize residual calcium metal, convert Ca(OH)_2 to CaCO_3 , and decompose CaCO_3 depending on the temperature.

With further analysis of the TGA plots, it may be possible to estimate the composition of the RF SS&C samples. The weight gains can be used to roughly estimate the amount of calcium metal present, if all the weight gain is attributed to oxidation

of Ca metal; this simplified approach ignores the well known reaction of Ca(OH)_2 and CO_2 to form CaCO_3 . In addition, the fractions of Ca(OH)_2 and CaCO_3 can be estimated from the weight losses at approximately 450 and 650°C. As a first step towards this estimate, a sample of calcium metal was oxidized at 210°C. The weight gain data are shown in Figure 7. These data may be used to bracket the oxidation rate. Only a rough estimate can be obtained since the rate will strongly depend on surface condition, moisture content of air, and particle size.

The tested samples of SS&C that exhibited weight gains on heating were examined to estimate the fraction of residual Ca metal present. All of the weight gain was attributed to Ca oxidation to CaO rather than the more likely Ca(OH)_2 , and further ignored the formation of CaCO_3 . This condition was assumed to provide the most hydrogen potential. The weight gain that is possibly attributable to Ca oxidation is presented in Table IX. These calculations may be used as an upper bound for the Ca metal content and indicate, in a very rough manner, that less than 1% Ca metal was present in this 17 mg sample of RF SS&C.

The more significant consideration for this work is the comparison of the Savannah River Site (SRS) data and the Rocky Flats (RF) data. This comparison for selected runs is presented in Table X. There are differences in the measured weight losses with the RF data indicating a higher loss than the SRS TGA data. These differences are difficult to rationalize. Some possible explanations include furnace temperature drift, real variations in the content, measurement technique, relative humidity during packaging, etc. The most likely causes are the variation in the sample composition and the measurement technique. RF use soil drying scale that heats a 2-3 g (2000-3000 mg) sample to 210°C and subsequently holds the sample until a constant slope

is attained. In all cases in this work, the sample (10-20 mg) was held for 30 minutes at 210°C. During this time, the following competing reactions may have been occurring: oxidation of Ca metal, conversion of CaO to Ca(OH)_2 , conversion of Ca(OH)_2 to CaCO_3 , and water desorption. In addition, the samples tested at both RF and SRS were not obtained in a manner consistent with standard practices for powder sampling. This sampling method may have a dramatic influence on the character of the samples tested.

CONCLUSIONS

1. There is not a one to one correspondence of the TGA data to the RF "Loss on ignition" "(LOI)" data. In general, the SRS values are smaller than those determined by RFETS.
2. Sampling may contribute significant error in weight loss measurement – especially for the relatively small samples used for TGA.
3. Plutonium oxide can adsorb about 3.7% water from humid air that may be available for hydrogen generation by radiolysis.
4. The RF SS&C samples that were examined may contain up to 1% Ca metal based on the weight gain on heating to 900°C.
5. Calcium hydroxide can rapidly form calcium carbonate at temperatures above 250°C.
6. A drying treatment of 210°C for 30 minutes removed from 40 to 88% of the available moisture on plutonium oxide depending on the total moisture adsorbed on the powder.

Table II Weight loss of PuO₂ at 210°C, 500°C, and the temperature of minimum weight.

Run	Sample ID	Temp (°C)	% loss	Temp (°C)	% loss	Temp (°C)	% loss "T"	$\frac{Wt_{210^{\circ}C}}{Wt_{500^{\circ}C}} * 100\%$	(T)
19	PuO ₂ As Received	210	0.746	500	1.262			59.1	
75	FB line PuO ₂	217	0.618	500	1.285	661	1.589	48.1	38.9
22	PuO ₂ Dessicator	211	0.162	500	0.306	526	0.373	82.04	43.25
32	PuO ₂ + 10% H ₂ O vac. P6	210	0.325	500	0.369			88.0	
38	PuO ₂ + 100%RH	232	0.554	500	0.944			58.63	
95	PuO ₂ + 100%RH 2 weeks	210	1.381	510	1.839			75.09	
97	PuO ₂ + 100%RH 2 weeks	224	2.525	501	3.441	545	3.373	74.85	
39	PuO ₂ + 76%RH	210	0.937	500	2.136	731	2.306	43.87	40.64
80	PuO ₂ + 76%RH	211	0.643	500	1.272			50.54	
94	PuO ₂ + 2.5% H ₂ O	225	1.836	500	2.410	520	2.462	76.18	74.56
83	PuO ₂ + 5% H ₂ O	210	3.254	501	4.032	610	4.244	80.70	76.67
84	PuO ₂ + Can + 5% H ₂ O	210	3.796	501	4.558			83.30	
88	PuO ₂ + Cu wire + 5% H ₂ O	210	2.786	500	3.634			76.67	
24	PuO ₂ + 10% H ₂ O	213	4.531	504	5.119			88.52	
25	PuO ₂ + 10% H ₂ O	210	2.068	500	2.423			85.35	

Table III. Weight loss of starting materials without PuO₂ at 210°C and the minimum weight for materials that do not exhibit decomposition transitions.

run	Sample ID	Temp (°C)	% loss	Temp (°C)	% loss	Temp (°C)	% loss "T"	$\frac{Wt_{210^{\circ}C}}{Wt_{500^{\circ}C}} * 100\%$	(T)
1	RF SAND	211	-0.042						
3	SRS MgO	211	0.032						
6	CaF	210	0.079						
7	MgO Reagent	210	-0.001						
9	CaO Reagent	210	0.818						
20	CaO-Ca(OH) ₂ Start	210	-0.186						
29	MgO + Ca(OH) ₂	210	0.279						
30	MgO Crucible	210	1.085	500	1.536			70.7	
44	Mg(OH) ₂	211	0.464			324	0.747		62.1
45	NaF Reagent Grade	210	0.129						
46	NaF Reagent Grade	211	0.351						

Table IV. Weight loss of samples containing Pu cake.

run	Sample ID	Temp (°C)	% loss	Temp (°C)	% loss	Temp (°C)	% loss "T"	$\frac{W_{t_{210}^{\circ}\text{C}}}{W_{t_{500}^{\circ}\text{C}}} * 100$ (T)
15	Pu Cake + MgO	210	0.628	500	1.728			36.3
17	Pu Cake +before test	210	3.089			487	14.955	20.7
26	Pu Cake + MgO	206	1.026	500	3.427			29.9
73	Pu Cake as received	210	-0.350					
62	Pu Cake + NaF No H2O	237	0.728	506	2.872	665	3.645	25.4 20.0
85	Pu Cake No added H2O	210	3.266	500	14.090			23.2
86	Pu Cake No added H2O	210	0.730	500	1.254			58.3
89	Pu Cake + NaF	223	0.594	500	1.977	790	3.581	30.1 16.6

Table V. Weight loss of samples containing PuO2 and MgO exposed to varying conditions.

run	Sample ID	Temp (°C)	% loss	Temp (°C)	% loss	Temp (°C)	% loss "T"	$\frac{W_{t_{210}^{\circ}\text{C}}}{W_{t_{500}^{\circ}\text{C}}} * 100$ (T)
31	PuO2 + MgO	212	0.401	502	0.475			84.3
40	PuO2 + MgO 100% RH	224	0.294	501	0.768			38.3
34	PuO2 + MgO + Ca(OH)2	210	-0.376					
35	PuO2 + MgO + 100% RH	220	0.098					
43	PuO2 + MgO + Ca(OH)2	214	0.264			313	0.500	52.7
47	PuO2 + MgO	211	0.266	500	0.759	767	1.242	35.0 21.4
49	PuO2 + Mg(OH)2	210	0.446			323	0.613	72.8
57	PuO2 + MgO + Ca(OH)2	222	0.516					
61	PuO2 + MgO + low moisture	210	0.537					
64	PuO2 + MgO + High H2O	237	0.215	500	1.032	665	1.184	20.8 18.1
65	PuO2 + MgO + Med. H2O	205	-0.003					
67	PuO2 + MgO + 100% RH	210	0.195	500	0.807	591	0.850	24.1 22.9
77	PuO2 + MgO	210	0.305					
78	PuO2 + MgO + Ca(OH)2	210	0.360			409	0.481	74.8
79	PuO2 + MgO + 100% RH	210	0.040	500	0.801	577	0.919	5.0 4.4
82	PuO2 + MgO + 100% RH	211	-0.098	501	0.731	643	0.978	-13.5 -10.1

Table VI Weight loss of PuO₂ samples containing calcium oxide, hydroxide, or fluoride.

run	Sample ID	Temp (°C)	% loss	Temp (°C)	% loss	Temp (°C)	% loss "T"	$\frac{Wt_{210^{\circ}C}}{Wt_{500^{\circ}C}} * 100$ (T)
76	PuO ₂ + CaO + Ca(OH) ₂ Start	206	-0.310					
21	PuO ₂ + CaO + Ca(OH) ₂	210	-0.146					
28	PuO ₂ + CaO + Ca(OH) ₂	210	-0.273					
36	PuO ₂ + Ca(OH) ₂ 100% RH	210	-0.003					
48	PuO ₂ + CaO Moist	198	-0.322					
50	PuO ₂ + CaF	210	0.025					

Table VII Weight loss of samples comprised of RF SS&C materials.

run	Sample ID	Temp (°C)	% loss	Temp (°C)	% loss	Temp (°C)	% loss "T"	$\frac{Wt_{210^{\circ}C}}{Wt_{500^{\circ}C}} * 100$ (T)
12	RF SS & C 1	210	0.428	499	1.233	763	2.392	34.7 17.9
13	RF ETS H6 As Recd	236	0.589					
14	RF ETS H6 As Recd	210	0.568					
74	RF H6 as Received	210	0.482			319	0.506	95.2
18	RFETS H6 after test	210	0.596					
27	H6 after test	210	0.220					
51	H6	229	0.405					
58	H2	211	0.446					
59	H7	203	0.534					
60	H8	210	0.586			689	1.402	41.8
90	13304 RF	213	0.837	506	11.178			7.5
91	13303 RF	172	1.477	500	3.125	671	7.368	47.3 20.0
96	P-3	224	2.481	500	3.397	542	3.427	73.0 72.4

Table VIII. Reaction temperatures for significant changes in weight.

Run	Identification	Temp	% reading	Temp	%	Temp	%	Temp	%
0.003	SRS MgO	226	99.95	339	99.86	339	99.86	408	99.52
0.009	CaO Reagent	391	98.86	400	79.61	538	79.45	658.9	75.3
0.044	Mg(OH) ₂	352	98.00	433	73.16				
0.029	MgO + Ca(OH) ₂	395	99.50	448	93.00	553	92.70	642	91.2
0.015	Pu Cake + MgO	274	99.07	363	98.46				
0.017	Pu Cake + before test	242	95.55	292	94.91	292	94.91	481	84.95
0.026	Pu Cake + MgO	241	98.72	427	96.70				
0.062	Pu Cake + NaF No H ₂ O	333	98.80	541	96.96				
0.073	Pu Cake as received	615	99.56	677	99.09				
0.085	Pu Cake No added H ₂ O	218	96.22	298	95.28	241	96.1	472	86.24
0.089	Pu Cake + NaF	199	99.90			365	99.09	558	97.38
0.028	PuO ₂ + CaO + Ca(OH) ₂	374	100.60	467	94.10	560	93.94	677	92.4
0.034	PuO ₂ + MgO + Ca(OH) ₂	383	100.40	429	97.40	541	97.20	652	96.2
0.036	PuO ₂ + Ca(OH) ₂ 100% RH	411	99.65	485	87.50	591	87.00	717	85.00
0.038	PuO ₂ + 100% RH	211	99.49	227	99.39				
0.039	PuO ₂ + 76% RH	210	99.01	223	98.88	223	98.8	444	97.98
0.040	PuO ₂ + 100% RH	318	99.60	372	99.3				
0.081	PuO ₂ + 76% RH	155	99.48	171	99.07	499	98.2	553	97.8
0.043	PuO ₂ + MgO + Ca(OH) ₂	381	99.55	421	98.33	550	98.7	650	97
0.048	PuO ₂ + CaO Moist	413	100.40	485	92.8	591	92.7	728	89.7
0.049	PuO ₂ + Mg(OH) ₂	346	98.70	428	81.8				
0.057	PuO ₂ + MgO + Ca(OH) ₂	390	99.68	426	98.88	554	96.9	650	95.22
0.013	RF.ETS	406	99.73	455	97.91	550	97.81	675	95.4
0.018	RFETS H6 after test	416	99.94	458	98.87	591	98.52	660	96.75
0.058	H2	403	99.44	458	94.49	556	94.65	633	92.96
0.059	H7	404	99.87	462	93.88	566	93.84	636	92.2
0.060	H8	500	99.00	587	98.76				
0.074	RF H6 as Received	424	99.29	489	96.74	588	96.54	730	94.07
0.061	PuO ₂ + MgO + low moisture	397	99.10	432	98.96				
0.064	PuO ₂ + MgO + High H ₂ O	334	99.70	397	99.11				
0.065	PuO ₂ + MgO + Medium H ₂ O	337	99.90	415	99.61				
0.067	PuO ₂ + MgO + 100% RH	327	99.68	374	99.4				
0.078	PuO ₂ + MgO + Ca(OH) ₂	423	99.37	483	96.72	602	96.48	714	95.4
0.079	PuO ₂ + MgO + 100% RH	361	99.69	390	99.4	211	99.91		
0.082	PuO ₂ + MgO + 100% RH	367	99.50	413	99.1				
0.090	13304 RF	332	98.20	501	88.78				
0.091	13303 RF	173	98.50	156	99.01				

Table. IX Weight gain attributed to oxidation of Ca Metal (uncorrected for buoyancy and thermal effects and ignores Ca(OH)_2 conversion to CaCO_3).

Run	sample	Size (mg)	Temperature over which weight changes occurred. TA convention is (-) is weight gain								Total mg	Total %
			210 mg	210 %	200-400 mg	200-400 %	450-550 mg	450-550 %	640 + mg	640 + %		
18	H6	17.966	-0.0116	-0.065	-0.1214	-0.676	-0.0355	-0.1976			-0.1685	-0.938
27	H6	17.321	-0.0143	-0.084	-0.0602	-0.347	-0.0467	-0.2693			-0.1211	-0.700
51	H6	18.056	0.0002	0.001	-0.0586	-0.325	-0.0324	-0.1795	-0.0096	-0.053	-0.1004	-0.556
74	H6	18.167	-0.0004	-0.002	-0.0230	-0.127	-0.0094	-0.0519			-0.0328	-0.181
58	H2	16.683	-0.0129	-0.077	-0.0257	-0.015	-0.0553	-0.3313			-0.0938	-0.424
59	H7	17.497	-0.0312	-0.179	-0.0912	-0.521	-0.0268	-0.1533			-0.1493	-0.853
5*	H7	15.948	-0.0531	-0.333	-0.0714	-0.448	-0.0034	-0.0211			-0.1279	-0.802
8	Reag CaO	15.826	-0.0710	-0.449	-0.0433	-0.274	0.0064	0.0407	0.0038	0.024	-0.1041	-0.658

Table X Comparison of RF weight loss data at 210 compared to SRS TGA weight loss data.

RFETS Sample	RFETS 210 (LOI)	SRS 210 (TGA)	Difference	SRS 500 (TGA)
IDC390 1880392 (H2)	0.16	0.45	+0.29	5.48
IDC398 1880438 (H6)	1.08	0.59 (Run 13) 0.48 (Run 74)	-0.9 -0.60	2.21 (Run 13) 3.28 (Run 74)
IDC395 1880280 (H7)	0.86	0.53	-0.33	6.16
IDC392 1880443 (H8)	1.53	0.59	-0.94	1.30*
IDC090 13304H	1.69	0.84	-0.85	11.2
IDC093 13303	3.74	1.48	-2.26	3.16

* Actual temperature approximately 590°C.

REFERENCES

1. Task Technical and Quality Assurance Plan - Gas Generation Test Support for Revision of 9975 SARP, WSRC-RP-99-00164, R. R. Livingston. January 1999, SRS.
2. Art Jurgensen, XRD determination of the species present in CaO. SRS, 1999.
3. SRT-MTS-99-3021, Thermal Analysis of Constituents Comprising SS&C and Low Pu Containing SS&C Samples, P. Korinko, July 1999, SRS, Aiken, SC.

Distribution List WRSC-TR-99-00236

Paul Blanton	773-54A
Greg Chandler	773-A
Steve Hensel	773-42A
Natraj Iyer	773-41A
Sue King	773-54A
Ronald Livingston	773-A
Jim Marra	773-43A
Alice Murray	773-A
Jeffrey Schaade	703-F
Scott West	773-A
Clinton Wolfe	773-A
Site Records (4)	773-52A
MTS Records (L. Hillary)	773-41A
Paul Korinko	773-A