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**Gas Generation and Migration Studies
Involving Recently Generated
²³⁸Pu-Contaminated Waste
for the TRU Waste Sampling Program**

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MASTER

GAS GENERATION AND MIGRATION STUDIES
INVOLVING RECENTLY GENERATED
²³⁸Pu-CONTAMINATED WASTE
FOR THE
TRU WASTE SAMPLING PROGRAM

by

Al Zerwekh and John L. Warren

ABSTRACT

This study is part of the multicontractor TRU Waste Sampling Program. Radiolytically generated gases were vented through a filtering device to determine its effectiveness in maintaining hydrogen concentrations within acceptably safe levels.

In the second part of the study measurements were made to determine the ability of these gases, particularly hydrogen, to migrate through a sealed rigid polyethylene drum liner.

Void volumes in these drums were found to be generally in excess of 90%. The carbon composite filter was found to satisfactorily vent hydrogen up to moderately high levels of alpha activity in the waste substrate. The sealed 90-mil liner was found to inhibit, but not prevent, the migration of hydrogen and other radiolytically generated gases.

INTRODUCTION

Radiolytic gas generation in transuranic (TRU) waste has been studied previously under controlled laboratory conditions and under actual retrievable storage conditions in the Los Alamos waste disposal area.¹ It has been found to be a potential but controllable hazard in the storage and handling of such waste.

The current study is part of the TRU Waste Sampling Program, a multicontractor program to evaluate parameters of various stored and newly generated TRU waste for eventual disposal at the Waste Isolation Pilot Plant (WIPP). Results of an earlier portion of the work conducted at Los Alamos involving TRU waste drums retrieved from storage for varying periods ranging from 8 months to 9 years recently were reported.² In that work it was observed that none of the drums examined had gas pressure in excess of atmospheric; all had vented radiolytically generated gases through the porous gasket. However, several of the 8-month-old drums of ^{238}Pu waste still contained significant levels of hydrogen.

This report presents results of a second portion of the Los Alamos work conducted for the TRU Waste Sampling Program involving measurements of the generation and migration of radiolytic gases from drums of TRU wastes. In one part of this study, drums were vented through a filtering device to determine its effectiveness in maintaining hydrogen concentrations within acceptable levels. In a second part of the experiment, waste was contained in a sealed rigid polyethylene drum liner. Measurements were made to

determine the ability of the radiolytic gases generated to migrate through the liner and subsequently from the drum through a filtering device.

TASK OBJECTIVES AND DESCRIPTION

The work accomplished was described as "Task 10: ^{238}Pu Gas Generation and Leak-Rate Studies" in the Implementation Plan.³ This task was described as follows:

"Task 10: ^{238}Pu Gas-Generation and Leak-Rate Studies. Six drums of high-activity, newly generated ^{238}Pu waste will be evaluated (a) to determine gas-generation rates of worst gas-generating content codes, compared to experimentally obtained laboratory yields, and (b) to ensure the carbon composite filter maintains hydrogen levels within a drum below hazardous levels. Waste of a known type and activity level will be placed in a drum. The study will be set up and conducted as described in RFP Task 5.³

Two drums of high-activity, newly generated ^{238}Pu waste will be evaluated (a) to determine the leakage rate of hydrogen through the 90-mil rigid polyethylene drum liner, and (b) to provide information on whether or not the rigid liner should be punctured to provide an escape pathway for hydrogen. Information on the gas-generation rate will also be obtained, if possible. Waste of a known type (high gas-generating potential) and activity level will be used. Parameters described for RFP Task 5 will be used, with the following exceptions:

1. Waste will be placed in drums retrofitted with a carbon composite filter, a sampling port that extends inside the 90-mil rigid liner, and a separate port that extends inside the drum lid.
2. The 90-mil rigid liner insert plug will be used and sealed in the rigid liner lid.
3. Rigid liners and procedures for sealing the rigid liner will be comparable to those used by RFP and SRP.

4. Void volume of the rigid liner will be determined.
5. The following will be recorded for each sample when obtained: drum and rigid liner pressures, atmospheric pressures, and temperatures (room).

Gas samples will be collected and analyzed as described above. The experiment will be conducted in two three-month phases. During the first phase, the drum will be in the unvented state with the filter sealed. Gas samples will be taken from each sampling port weekly for three months to determine (a) gas concentrations in the rigid liner and the drum, (b) leakrates of hydrogen from the rigid liner, and (c) gas generation rates. During the second phase, the drum will be allowed to vent. Gas samples will be taken weekly for three months to determine the rate of reduction in gas concentration in the waste container."

EXPERIMENTAL PREPARATION AND PROCEDURE

Waste Description

Data on the eight experimental drums prepared are contained in Table I. All of the drums contained varying mixtures of combustible wastes, which were thought to be the most significant potential generators of radiolytic gas at Los Alamos. In each case, the waste originated from work in the Los Alamos Plutonium Facility. The ^{238}Pu contamination is "heat source" grade material, having the isotopic composition listed in Table II.

Waste Assay

Waste contained in the original 30-gallon waste drums provided for this study was assayed by segmented gamma scan techniques. The drums contained a number of smaller waste

Table I

Experimental Waste Drum Characteristics

Drum Number	Date Waste Generated	Listed Waste Description	238Pu Contamination		Date Drum Prepared
Grams	Curies				
(Venting through carbon composite filter)					
BFB-109	1/27/84	Plastic	3.0	42.0	3/26/84
BFB-112	7/28/83	Mixed Combustibles	1.2	16.8	12/8/83
BFB-116	10/27/83	Mixed Combustibles	2.28	31.9	12/8/83
BFB-118	10/27/83	Mixed Combustibles/ Noncombustibles	4.92	68.9	12/8/83
BFB-120	1/3/84	Rubber	1.6	22.4	3/26/84
BFB-122	1/12/84	Mixed Combustibles	11.9	166.6	3/26/84
(Venting through 90-mil liner)					
BFB-113	9/26/83	Rubber	2.2	30.8	12/8/83
BFB-114	9/23/83	Mixed Combustibles	15.6	218.4	12/8/83

Table IIIsotopic Composition of Heat-Source ^{238}Pu

Isotope	Weight %
^{238}Pu	80.0
^{239}Pu	16.3
^{240}Pu	3.0
^{241}Pu	0.6*
^{242}Pu	0.1

* Not an alpha emitter.

packages, generally bagout bags, and the segmented gamma scan was conducted on each package, with the drum content then being determined as the sum of the individual assays.

Experimental Drum Preparation

All of the waste transferred to the 55-gallon experimental drums was double-bagged; that is, in two 5-mil polyethylene bags, one inside the other, each individually sealed with vinyl tape. In December of 1983, the contents of three 30-gallon drums of recently generated ^{238}Pu -contaminated waste were transferred to sealed 55-gallon drums which had been fitted with a valve, a pressure gauge, and a carbon composite filter. Each of these drums was lined with a 90-mil high density polyethylene liner, but the liner was not sealed and the sealing plug in the liner lid was left out of them. In March 1984, three additional drums of newly generated ^{238}Pu -contaminated waste were prepared in the same manner; again, each was fitted with a valve, a pressure gauge, and a carbon composite filter. These six drums constituted the first part of the experiment. The setup of the experimental 55-gallon drums is shown in Figure 1.

Figure 1. Experimental drum setup for gas migration study through carbon composite filter.

Two additional waste drums were prepared for part two of this experiment. Each of these drums, as seen in Figure 2, involved containing the waste in a sealed 90-mil high-density polyethylene liner. Both this inner liner and the drum were fitted with valves, pressure gauges, and the carbon composite filters.

General Electric white silicon calking compound (GE-SE-1202) was applied to assure a complete seal of the drum and liner lids, and to seal the measuring/sampling devices to the inner 90-mil liners, as noted above. To seal the measuring/sampling devices to the drum lid, a 0.25 in. pipe coupling first was welded to the lid, and the device was screwed into it, sealed with Teflon tape.

Pressure gauges used in all drums were Ashcroft combination vacuum or pressure gauges. These are calibrated in units of 0.5 psig (25.9 mm Hg), thus allowing a measurement reading sensitivity of no better than approximately 0.1 psig (5.2 mm Hg).

Pressure Testing

Each drum was pressure tested twice during the experimental procedure. The first of these tests occurred when the drums were initially sealed following being loaded with waste material. For this first test, a manometer was attached to each drum, one at a time, and then the system pressurized to approximately 200 mm Hg with compressed air.

Figure 2. Experimental drum setup for gas migration study through sealed 90-mil liner.

The drum was then checked at 30 and 60 minutes' elapsed time. The drums were found to be satisfactorily sealed. After the completion of the gas-generating and filter-testing periods, the drums were again leak-checked, this time employing a Wallace and Tiernan differential pressure gauge with a certified accuracy of <1 mm Hg. Two of the drums, BFB-109 and BFB-122, allowed the pressurized gas to diffuse at a rate of 12.5 mm Hg/min or more. These drums were considered to be unsatisfactory for subsequent void volume determinations.

Gas Sampling

Drums for both experimental parts were sampled at approximately two to three week intervals, although some longer intervals were subsequently established for some drums. This frequency was determined, in part, by the availability of needed support personnel, and also when the collected data indicated that changes in gas composition were occurring at a very slow rate.

To obtain a gas sample in each instance a 1 liter gas sampling flask was attached directly to the measuring/sampling device on the drum. The flask was evacuated to <0.02 mm Hg prior to sampling. A very small error is associated with each sample due to the small volume of air in the adapter where the sampling flask was attached. Based upon the relative volume of this adapter, this error in each case is less than 0.5%, and thus is considered negligible.

Sample Analysis

Each day, before any samples were analyzed, a new instrument sensitivity factor was determined for each gas of interest. This factor was determined by measuring the electrical response of the instrument to a known amount of each gas. Daily determination of the sensitivity factors is standard procedure for Los Alamos mass spectrometric work and avoids reliance on a secondary standard. The gases included in the daily calibration and the approximate sensitivity factor for each were: hydrogen, 41 mV/ μ m; helium, 15 mV/ μ m; carbon monoxide, 2 mV/ μ m; nitrogen, 7 mV/ μ m; methane, 82 mV/ μ m; argon 95 mV/ μ m; and carbon dioxide, 74 mV/ μ m.

A certified gas sample was supplied to Los Alamos by EG&G, Idaho. After the daily sensitivity factors were determined, the sample was analyzed and results reported by telephone to Tom Clements at EG&G, Idaho; the results of the analysis were pronounced acceptable. Table III contains results of the analyses of the standard gas sample as determined throughout most of the experimental period.

Experimental Procedure

Part 1 - Drum Venting Through Carbon Composite Filter

The first three drums utilized in this part of the experiment initially were left vented through the carbon filters to the atmosphere from day 1 through day 124. The drums were then subjected to a special purge treatment

DATE	H ₂	He	CO	N ₂	CH ₄	O ₂	Ar	CO ₂
12-29-83	2.6	<0.1	1.6	65.9	<0.1	18.5	<0.1	11.4
1-20-84	Cancelled							
2-21-84	3.0	<0.1	1.4	65.3	<0.1	18.7	<0.1	11.7
3-27-84	3.0	<0.1	1.0	65.8	<0.1	18.6	<0.1	11.6
3-30-84	2.3	<0.1	0.4	66.7	<0.1	18.8	<0.1	11.8
4-10-84	3.6	<0.1	1.4	65.0	<0.1	18.6	<0.1	11.4
5-04-84	2.7	<0.1	1.0	65.6	<0.1	19.0	<0.1	11.7
5-30-84	1.9	<0.1	1.1	66.3	<0.1	19.0	<0.1	11.7
6-14-84	2.4	<0.1	0.9	65.9	<0.1	19.0	<0.1	11.8
7-10-84	2.5	<0.1	0.5	66.1	<0.1	19.1	<0.1	11.8
8-03-84	<u>2.5</u>	<u><0.1</u>	<u>2.9*</u>	<u>64.9</u>	<u><0.1</u>	<u>18.4</u>	<u><0.1</u>	<u>11.3</u>
	x 2.65		1.03	65.7		18.8		11.6
	o <u>+0.46</u>		<u>+0.40</u>	<u>+0.57</u>		<u>+0.25</u>		<u>+0.19</u>

*Outlier not included in calculation.

which consisted of partial evacuation to approximately 300 mm Hg below atmospheric pressure, returning to atmospheric, pressurizing the drum to approximately 300 mm Hg above atmospheric with compressed air, and then returning to atmospheric. This cycle was repeated three times. A gas sample then was extracted from the drum. The valve allowing the drum to vent through the carbon composite filter then was closed for the remainder of the experiment.

For the second set of three drums, the procedure generally was similar, though with some slight variations. These drums were initially left open to the atmosphere through their carbon composite filters for a period of 137 days. They were then given a special purge treatment consisting of partially evacuating them to -300 mm Hg below atmospheric pressure, returning to atmospheric pressure, pressurizing to 300 mm Hg above atmospheric, and returning to atmospheric pressure. This cycle was repeated four times. A sample then was obtained from each drum, and the valves were closed.

Part 2 - Gas Migration Through the 90-mil Liner

Both phases of both parts of these experiments were extended in time beyond the 3 month period specified. This was done primarily because of the apparent low rate of gas generation in several drums, and to assure that the results observed were representative of "steady-state" conditions during the period the drums were vented.

Void Volume Determination

Void volume measurements in the eight experimental drums were attempted following completion of all of the gas sampling measurements. No attempt could be made to determine the void volumes in two of the drums that at that time were found not to be adequately sealed. For the void volume measurement, the six sealed drums were pressurized to 200 mm Hg above atmospheric with compressed air, and then the gaseous contents expanded into a 3-gallon tank whose volume had been previously determined to be 11.813 liters. This volume had been accurately measured by completely filling, with water, the smaller tank and all of its associated tubing for connecting with the waste drum, then weighing the unit. Comparison of this weight with the empty weight of the unit gave the volume. This procedure was repeated several times, and each time produced exactly the same result.

The procedure followed to measure void volume was adapted from that of E.L. D'Amico (AL583.993), reproduced in Reference 4. The void volume measurement and consequent determination was repeated five times for each drum. We found, as D'Amico had earlier, that the void volumes are reasonably reproducible. We also verified what we had suspected, viz., that in waste consisting of paper, plastic, rubber and other loosely packed combustibles, the void volumes are generally very high, equal to or greater than 90% in 5 of the 6 drums measured. D'Amico's work also showed that a "55-gallon" drum as produced for Rocky Flats Plant, chosen at random, had a volume greater than 208.2 liters. His standards laboratory found the volume to be 220.3 ± 1

liter. Our void volume measurement of a randomly chosen 55-gallon drum, identical to those at Rocky Flats, found the volume to be 219.9 liters. Therefore, we have used a value of 220 liters for the volume of a "55-gallon" drum in computing the void volume percentages. The mean void volume and the standard deviation for each drum is reported in Table IV.

Table IV

Void Volumes

<u>DRUM</u>	<u>VOID VOLUME (LITERS)</u>	<u>ACTUAL CONTENTS</u>
BFB-112	198.1 $\pm$ 14	Plastic materials and leaded gloves
BFB-113	168.3 $\pm$ 12.7	Leaded gloves
BFB-114	201.1 $\pm$ 11.7	Rags and plastic
BFB-116	210.3 $\pm$ 7	Leaded gloves
BFB-118	201.3 $\pm$ 7.5	Rags, plastic, metal furnace, Al & Zr oxides
BFB-120	215.6 $\pm$ 5.2	Leaded gloves

As noted previously, drums BFB-113 and BFB-114 contained the waste in sealed 90-mil high density polyethylene liners. The void volumes for these two drums were from measurements made through the connection to the inside of this sealed liner. Thus, for these two drums, the result is considered to be the requested rigid liner void volume.

RESULTS AND CONCLUSIONS

Venting Capability Through Carbon Composite Filter

Experimental results showing the pressures and gas

compositions of the six drums in this part of the experiment are presented in Appendix 1.

For the entire period during which these drums were vented through the carbon composite filter there was no detectable pressure difference between the interior of the drum and external atmospheric. As noted previously, the minimum pressure differential that could have been measured, based upon the pressure gauges utilized, was approximately 5 mm Hg.

The carbon composite filter appears to adequately vent radiolytic hydrogen to maintain a safe (noncombustible/non-explosive) gas mixture in the drums in all but the highest activity situations. Realizing that 1 g of heat-source ^{238}Pu has approximately the same total alpha radioactivity (and energy input) as the maximum 200 g loading of weapons grade ^{239}Pu , we conclude that the carbon composite filter should be quite adequate for venting and maintaining a safe atmosphere in drums of combustible waste contaminated up to this maximum loading level.

In all instances, once the drum was sealed the hydrogen content was seen to build. In the two highest activity drums, BFB-118 and BFB-122, potentially combustible/explosive gas mixtures were obtained within less than one month after closing the drum vent. It also should be noted, however, that the decreasing oxygen concentrations in the several drums having higher hydrogen levels, in most cases, would render them noncombustible/nonexplosive within a several month period.

Recall that drums BFB-109 and BFB-122 were found, following the gas sampling, not to be sealed gas tight. We

postulate that the damage to the drum seal most likely occurred during the purge cycling procedure; consequently, we believe the data valid for these drums during the initial period of venting through the carbon filter. Drum BFB-109, following closure of the vent, never did appear to accumulate any quantity of hydrogen gas. On the other hand, drum BFB-122, containing the highest ^{238}Pu loading of these six, did show a buildup of hydrogen, although at a much slower rate than drum BFB-118 which contained half as much ^{238}Pu .

Venting Capability Through 90-mil Liner

Results showing the pressures and gas compositions in the two experimental drums in which the waste also was contained in a sealed 90-mil high density polyethylene liner are presented in Appendix 2.

Comparison of the data for the annular space versus volume inside the liner shows a consistent slight lag in the diffusion of radiolytic gases from the liner. Hydrogen values in the annular space, for example, typically are slightly lower than inside the liner. The oxygen concentration in the annular space, on the other hand, is generally higher than inside the liner. These results are not unexpected.

At the time the vent to the annular space was opened on the two drums, the pressure measured in the annular space immediately fell to atmospheric. By the time of the next sampling, 11 days later, the pressure within the sealed liner also had reduced to a reading of 0.0 on the gauge.

Drum BFB-114 contained the largest quantity of ^{238}Pu of all eight experimental drums, with 15.6g. As seen in Appendix 2, Table 2-IV and Figure 2-3, with the vent open there clearly remains an explosive gas mixture within this container.

Gas Generation Analysis

G (gas) is defined as molecules of gas produced by each 100 eV of energy deposited in the waste substrate. It will vary with the physical state of the radioactive contaminant, its distribution in the waste, and the waste matrix. It slowly decreases with time because the contact of the contaminant with the substrate becomes less effective, a phenomenon sometimes referred to as depletion. Given sufficient time, the contaminant falls out of effective contact with the waste, and gas generation essentially ceases. Earlier studies of 3- and 9-year old waste drums retrieved from storage are in agreement with this statement.²

G values were calculated for the six drums which pressure retesting showed to be still sealed at the end of the experimental period. These were the same drums for which void volumes were determined, because the void volume of the drum is a needed component of the G value calculation. Because some of the testing involved a study of whether the carbon filters were adequate to vent gases radiolytically generated in these drums, they were open to the atmosphere (through the filter) for part of the experimental period. Obviously it was not possible to determine G values for these times. However, each drum was sealed for a portion

of the experimental period; the G values were determined for these times. Half of these six drums built up positive pressures during the closed periods; two developed negative pressures (with respect to atmospheric), and one remained at atmospheric. For the drums which showed less than atmospheric pressure at the end of the sealed period, an effort was made to account for this by the generation/interaction of H_2 , CO, CO_2 , and O_2 . It was possible to account for most, but not all, of the pressure decrease. The balance is probably attributable to the formation of iron rust and water, and to temperature changes.

Parameters utilized in calculating G values are presented in Tables V and VI. The basic equation required for calculation of the G values is the following:

$$G = \frac{(\text{moles gas generated}) (6.02 \times 10^{23})}{\text{eV} / 100}$$

In this equation,

moles gas generated = moles gas(final) - moles gas(initial),
where

$$\text{moles gas} = \frac{\left(\frac{\text{mol}\%}{100} \right) \left(\frac{P}{760} \right) \left(\frac{273}{T} \right) \left(\frac{\text{Void Volume}}{22.4} \right)}{22.4},$$

and where

P = Absolute Pressure in mm of Hg

T = Temperature in $^{\circ}\text{K}$, i.e. $273 + ^{\circ}\text{C}$

Normal atmospheric pressure at Los Alamos was taken as 579 mm Hg in these calculations.

Total energy input to each drum, eV, was determined according to the equation

Table V
Gas Generation Data

DRUM NO.	ACTUAL WASTE MATRIX	^{238}Pu		GAS GENERATION TIME (DAYS)	eV (TOTAL)	VOID VOL. (LITERS)
		g	Ci			
BFB-112	Plastic, leaded gloves	1.2	16.8	318	9.55×10^{25}	198.1
BFB-113	Leaded gloves	2.2	30.8	75	4.13×10^{25}	168.3
BFB-114	Rags, plastic	15.6	218.4	75	2.93×10^{26}	201.1
BFB-116	Leaded gloves	2.28	31.9	318	1.82×10^{26}	210.3
BFB-118	Rags, plastic furnance, Al & Zr oxides	4.92	68.9	294	3.62×10^{26}	201.3
BFB-120	Leaded gloves	1.6	22.4	208	8.35×10^{25}	215.6

Table VI

Additional Gas Generation Data

DRUM NO.	ABSOLUTE PRESSURE (mm Hg)		TEMPERATURE (°C)		MOL% H ₂		MOL% GAS**	
	INIT	FINAL	INIT	FINAL	INIT	FINAL	INIT	FINAL
BFB-112	579	582.2	18.3	3.1	0.1	5.2	0.8	13.7
BFB-113	579*	579	-5.5*	4.4	1.2*	2.9	3.1*	6.6
BFB-114	579*	984.7	-5.5*	4.4	11.5*	26.3	24.7*	56.4
BFB-116	579	553.6	18.3	3.1	0.3	6.8	1.3	16.7
BFB-118	579	708.3	18.3	7.8	0.9	20.8	2.1	42.0
BFB-120	579	579	25.6	6.7	0.3	2.7	0.7	6.1

* Measured inside liner 12/29/83, with gauge pressure at 0.0 immediately after venting.

** Gas includes H₂, CO₂, CO, CH₄.

$$\text{eV} = (\text{Ci})(3.7 \times 10^{10} \text{dis/Ci-sec})(5.59 \text{ MeV/dis})(10^6 \text{eV/MeV})(\text{sec})$$

Using this calculational method and assumptions, values were calculated for G (H₂) and G (gas) for the six sealed experimental drums. These values are presented in Table VII.

Table VII
Calculated G Values

DRUM NO.	G (H ₂)	G (gas)
BFB-112	0.20	0.49
BFB-113	0.14	0.28
BFB-114	0.40	0.86
BFB-116	0.15	0.34
BFB-118	0.27	0.54
BFB-120	0.12	0.28

Values for G (gas) found in this study are in particularly good agreement with the earlier laboratory results of Kazanjian⁵ for the mixed combustibles and leaded gloves. The work of Kazanjian involved both ²³⁹Pu and ²³⁸Pu contamination. The current results also appear to be quite consistent with one another. Here, in particular, the similarity of results for the three drums containing leaded gloves is noted.

Drum Leak Rate Analysis

An analysis of the capability of the carbon composite filter and 90-mil liner to vent radiolytically generated

hydrogen is presented in Figure 3. Data points for the drums appear here as bars based upon the range of values measured for mol% hydrogen. Three outlying points, one of 5% for drum BFB-118, one of <0.1% for drum BFB-116, and one of 0.1% for drum BFB-122 have been omitted. A best fit "upper limit" line through these points, and through 0,0 which is assumed to be a valid point, crosses the 4% hydrogen concentration "explosive limit" at approximately 108 alpha curies content of a drum. However, reference to the earlier work of Zerwekh¹ shows a more realistic value to be 6% hydrogen as an explosive limit based upon actual gas compositions in waste drums. The line crosses the 6% hydrogen concentration at approximately 160 alpha curies.

Two of the experimental drums, BFB-113 and BFB-114, also were sampled inside the sealed 90-mil liner after the annular space between the drum and liner was vented through the carbon composite filter. The two data points for the hydrogen concentration in the sealed liner also are included on Figure 3. Again, the point 0,0 is considered valid. These two points show the holdup of gas migration by the liner. However, radiolytic gases are capable of migrating from these drums, although at a slightly slower rate. The best fit "upper limit" line through these points crosses the 4% hydrogen concentration line at approximately 76 alpha curies, and crosses the 6% hydrogen concentration limit at approximately 114 alpha curies.

The 200 g limit of weapons grade plutonium in a drum corresponds only to approximately 14.6 alpha curies. We conclude, therefore, that the carbon composite filter is quite adequate for venting hydrogen from drums of Los Alamos combustible wastes containing up to the allowed contamination limit of 200 g of weapons grade material.

ENERGY INPUT vs HYDROGEN GENERATION

Figure 3. Energy Input vs Hydrogen Generation

However, this analysis can be considered applicable only for wastes having $G (H_2)$ values comparable to or less than those measured in this experiment for the Los Alamos combustible wastes. A more wide-ranging, useful analysis, potentially applicable to all TRU Wastes, would be to show the relationship between a rate of hydrogen generation and the percent hydrogen in a vented drum. This is attempted in Figure 4. The rate of hydrogen generation in a waste container will be a function of both the waste matrix (G value) and quantity of alpha contamination present (αCi). Thus in Figure 4, we have shown the rate generation as the product of these two values as determined for each experimental drum. Unfortunately, because $G (H_2)$ could not be determined for the two drums found to leak at the end of the experiment, this plot contains two less data points than Figure 3. The same assumptions and ranges of values for equilibrium hydrogen concentrations in the vented drums are used as were in Figure 3.

As in Figure 3, the two lines drawn between data points, one for the vented drum spaces and one for the volume inside the sealed 90-mil liner (with the drum vented), are somewhat arbitrary. While there does appear to be some relationship between rate and percent hydrogen present, it is not clear from these limited data points whether this is a linear relation (as illustrated). Based upon the relationship shown, however, it would appear that a vented drum having a rate of generation $[\alpha Ci \times G (H_2)]$ of less than 42 will maintain the hydrogen concentration below 4%. This would correspond, for example for a $G (H_2)$ of 1.0, to an allowable alpha activity loading of 42 Ci. Conversely, for the maximum alpha curie loading in a drum

HYDROGEN CONCENTRATION IN DRUM vs RATE OF GENERATION

Figure 4. Hydrogen Concentration in Drum vs Rate of Generation

of weapons plutonium of approximately 14.6 Ci, the G (H₂) for the waste matrix could be almost 3, a value far in excess of the values determined in laboratory studies for most waste matrices.

Similar evaluations for the hydrogen concentration in a sealed 90-mil liner (with the drum vented) could be made which would yield lower allowable values. In order to clarify and validate this relationship between rate of hydrogen generation and hydrogen concentration in a vented drum, we suggest further studies be completed. In particular, additional data points corresponding to rates of hydrogen generation in the range between 20 and 80 [α Ci x G (H₂)] would appear to be of considerable benefit.

SUMMARY

In summary, we believe that this work has conclusively demonstrated the following points:

- o The carbon composite filter may be employed to satisfactorily maintain drums of TRU waste at atmospheric pressure.
- o Void volumes in drums of combustible TRU wastes are quite high, and generally are 90% or higher.
- o The carbon composite filter allows for sufficient venting of radiolytic hydrogen gas from combustible wastes in drums without the sealed 90-mil liner up to moderately high levels of alpha activity, considerably in excess of the levels associated with the maximum allowed 200 g of weapons grade plutonium.
- o The sealed 90-mil liner does inhibit the migration of hydrogen. However, it would appear that there still is an ability to vent radiolytically generated gases through it.

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APPENDIX 1

**GAS SAMPLING RESULTS FOR 6 DRUMS
VENTED THROUGH THE CARBON COMPOSITE
FILTER AND NOT HAVING A SEALED
INNER 90-MIL LINER**

Table 1-I

Drum BFB-109. Generated 01-27-84. Contains 3.0g (42 μ Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H ₂	O ₂	CO	CO ₂	CH ₄	N ₂	He	Ar
03-26-84*	0.0									
04-10-84	0.0	65	0.1	20.7	0.5	0.4	<0.1	77.4	<0.1	0.9
05-04-84	0.0	73	<0.1	20.7	0.4	0.4	<0.1	77.6	<0.1	0.9
05-25-84	0.0	91.5	<0.1	19.6	<0.1	0.4	<0.1	79.1	<0.1	0.9
06-14-84	0.0	85	0.2	20.4	0.3	0.4	<0.1	77.8	<0.1	0.9
07-06-84	0.0	92.5	0.1	20.5	0.1	0.5	<0.1	77.9	<0.1	0.9
07-27-84	0.0	84	0.1	20.3	0.4	0.5	<0.1	77.9	<0.1	0.9
08-10-84**	0.0	78	<0.1	21.2	0.3	0.1	<0.1	77.4	<0.1	0.9
08-24-84	Positive; <25.9	68	<0.1	21.1	0.3	0.2	<0.1	77.1	<0.1	1.2
03-06-85	0.0	44	0.2	21.3	0.4	0.3	<0.1	76.9	<0.1	0.9

* Drum prepared; vented to atmosphere through carbon filter.

** Drum purged of gaseous contents, sampled, valve closed to seal contents of drum from atmosphere

Drum BFB-109

Gen. 1/27/84, 3.0g Pu238

Figure 1-1.

Table 1-II

Drum BFB-112. Generated 07-28-83. Contains 1.2g (16 μ Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H ₂	O ₂	CO	CO ₂	CH ₄	N ₂	He	Ar
12-08-83*	0.0									
12-30-83	0.0	22	0.6	19.1	0.2	0.6	<0.1	78.5	<0.1	0.9
01-20-84	0.0	27	0.5	20.0	0.4	0.5	<0.1	77.6	<0.1	0.9
02-14-84	0.0	58	0.5	20.0	0.1	0.5	<0.1	78.0	<0.1	0.9
03-13-84	0.0	64	0.4	20.4	0.3	0.5	<0.1	77.5	<0.1	0.9
03-30-84	0.0	40	0.3	19.8	0.1	0.6	0.1	78.2	<0.1	0.9
04-10-84**	0.0	65	0.1	20.7	0.4	0.3	<0.1	77.6	<0.1	0.9
05-04-84	0.0	73	0.7	18.3	0.3	1.0	<0.1	78.8	<0.1	0.9
05-25-84	0.0	91.5	0.6	16.9	0.5	1.7	0.1	79.3	<0.1	0.9
06-14-84	0.0	85	1.6	15.2	0.4	2.3	0.1	79.5	<0.1	0.9
07-06-84	0.0	92.5	2.0	13.6	0.6	2.9	0.1	79.9	<0.1	0.9
07-27-84	0.0	84	2.6	11.8	1.8	3.3	0.1	79.5	<0.1	0.9
11-13-84	-50.8	53	4.2	7.4	1.7	5.4	0.1	80.2	<0.1	1.0
12-20-84	-50.8	35	4.6	6.6	1.3	5.9	0.1	80.4	<0.1	1.1
01-29-85	-38.1	46	5.2	5.7	1.6	6.4	0.1	80.0	<0.1	1.0
02-22-85	-50.8	37.5	5.2	5.3	2.1	6.3	0.1	80.0	<0.1	1.0

* Drum prepared; vented to atmosphere through carbon filter.

** Drum purged of gaseous contents, sampled, valve closed to seal contents of drum from atmosphere.

Drum BFB-112

Gen. 7/28/83, 1.2g Pu238

Figure 1-2.

Time - Weeks

Table 1-III

Drum BFB-116. Generated 10-27-83. Contains 2.28g (31.9 α Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H_2	O_2	CO	CO_2	CH_4	N_2	He	Ar
12-08-83*	0.0									
12-30-83	0.0	22	0.6	19.7	0.4	0.7	<0.1	77.6	<0.1	0.9
01-20-84	0.0	27	0.7	19.8	0.5	0.8	<0.1	77.3	<0.1	0.9
02-14-84	0.0	58	0.6	19.6	0.1	0.8		77.9		0.9
03-13-84	0.0	64	<0.1	20.5	0.3	0.9	<0.1	77.4	<0.1	0.9
03-30-84	0.0	40	0.6	19.6	0.2	1.0	<0.1	77.7	<0.1	0.9
04-10-84**	0.0	65	0.3	20.5	0.5	0.5	<0.1	77.3	<0.1	0.9
05-04-84	0.0	73	1.6	18.6	0.6	1.5	<0.1	76.8	<0.1	0.9
05-25-84	25.9	91.5	1.5	16.2	<0.1	2.4	<0.1	79.0	<0.1	0.9
06-14-84	25.5	85	2.4	15.5	0.8	3.1	0.1	77.2	<0.1	0.9
07-06-84	25.9	92.5	2.9	14.1	0.8	3.9	0.1	77.3	<0.1	0.9
07-27-84	0.0	84	3.7	12.6	1.9	4.4	0.1	76.4	<0.1	0.9
09-10-84	25.9	84	No gas sample taken							
10-12-84	0.0	43	No gas sample taken							
11-13-84	-25.4	53	No gas sample taken							
01-29-85	-25.4	46	6.7	7.0	1.8	7.7	0.1	75.8	<0.1	0.9
02-22-85	-25.4	37.5	6.8	6.5	2.0	7.8	0.1	75.9	<0.1	0.9

* Drum prepared; vented to atmosphere through carbon filter.

** Drum purged of gaseous contents, sampled, valve closed to seal contents of drum from atmosphere.

Drum BFB-116

Gen. 10/27/83, 2.28g Pu238

Figure 1-3.

Table 1-IV

Drum BFB-118. Generated 10-27-83. Contains 4.92g (68.9 α Cl) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H ₂	O ₂	CO	CO ₂	CH ₄	N ₂	He	Ar
12-08-83*	0.0									
12-30-83	0.0	22	2.3	18.9	1.2	1.6	0.1	75.1	<0.1	0.9
01-20-84	0.0	27	5.0	17.0	2.4	3.2	0.1	71.4	<0.1	0.8
02-14-84	0.0	58	2.8	19.0	1.0	2.0	0.1	74.0	<0.1	0.9
03-13-84	0.0	64	2.6	18.6	1.1	1.9	0.1	74.9	<0.1	0.8
03-30-84	0.0	40	2.3	19.0	0.7	2.0	0.1	75.0	<0.1	0.9
04-10-84**	0.0	65	0.9	20.6	0.5	0.7	<0.1	76.4	<0.1	0.9
05-04-84	0.0	73	5.1	17.4	2.0	3.1	0.1	71.5	<0.1	0.8
05-25-84	64.6	91.5	5.3	15.0	1.8	4.7	0.2	72.2	<0.1	0.8
06-14-84	77.6	85	8.1	13.9	3.6	6.1	0.3	67.2	<0.1	0.8
07-06-84	103.4	92.5	9.8	12.6	3.0	7.6	0.3	65.8	<0.1	0.9
07-27-84	103.4	84	12.1	11.1	3.7	8.8	0.4	63.2	<0.1	0.7
11-13-84	155.1	53	16.5	5.9	4.5	14.4	0.5	57.5	<0.1	0.7
12-20-84	155.1	35	14.7	7.8	2.8	12.7	0.4	60.8	0.1	0.7
01-29-85	129.3	46	20.8	3.0	3.3	17.3	0.6	54.4	0.1	0.6

* Drum prepared; vented to atmosphere through carbon filter.

** Drum purged of gaseous contents, sampled, valve closed to seal contents of drum from atmosphere.

Drum BFB-118 Gen. 10/27/83, 4.92g Pu238

Figure 1-4.

Time - Weeks

Table 1-V

Drum BFB-120. Generated 01-03-84. Contains 1.6g (22.4 α Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components						
			H ₂	O ₂	CO	CO ₂	CH ₄	N ₂	He
03-26-84*	0.0								
04-10-84	0.0	65	0.3	20.6	0.4	0.5	<0.1	77.3	<0.1
05-04-84	0.0	73	0.2	20.6	0.5	0.4	<0.1	77.4	<0.1
05-30-84	0.0	91.5	<0.1	19.7	<0.1	0.4	<0.1	79.0	<0.1
06-14-84	0.0	85	0.5	19.9	0.1	0.6	<0.1	78.0	<0.1
07-06-84	0.0	92.5	0.3	20.8	0.2	0.5	<0.1	77.3	<0.1
07-27-84	0.0	84	0.1	20.8	0.2	0.4	<0.1	77.6	<0.1
08-10-84**	0.0	78	0.3	20.7	<0.1	0.4	<0.1	77.6	<0.1
08-24-84	Positive; <25.9	68	0.6	20.2	0.2	0.5	<0.1	77.2	<0.1
03-06-85	0.0	44	2.7	16.2	0.9	2.5	<0.1	76.8	<0.1

* Drum prepared; vented to atmosphere through carbon filter.

** Drum purged of gaseous contents, sampled, valve closed to seal contents of drum from atmosphere.

Drum BFB-120

Gen. 1/3/84, 1.8g Pu238

Figure 1-5.

Table 1-VI

Drum BFB-122. Generated 01-12-84. Contains 11.9g (166.6 μ Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H ₂	O ₂	CO	CO ₂	CH ₄	N ₂	He	Ar
03-26-84*	0.0									
04-10-84	0.0	65	0.1	21.0	0.6	0.4	0.1	76.9	<0.1	0.
05-04-84	0.0	73	5.3	17.8	3.7	4.4	0.2	67.8	<0.1	0.
05-25-84	0.0	91.5	4.9	16.8	3.0	4.8	0.2	69.5	<0.1	0.
06-14-84	0.0	85	5.5	17.3	3.8	4.7	0.2	67.7	<0.1	0.
07-06-84	0.0	92.5	5.2	17.6	3.3	4.9	0.2	68.0	<0.1	0.
07-27-84	0.0	84	5.5	17.4	3.7	4.6	0.2	67.8	<0.1	0.
08-10-84**	0.0	78	2.1	20.3	0.6	0.9	<0.1	75.1	<0.1	0.
08-24-84	-25.4	68	4.2	18.2	2.0	2.2	0.1	72.1	<0.1	1.
11-13-84	-25.4	53	9.7	14.4	4.6	7.5	0.3	62.7	<0.1	0.
12-20-84	-25.4	35	10.9	13.9	4.1	8.5	0.4	61.5	<0.1	0.
01-29-85	-25.4	46	10.4	14.0	4.4	8.2	0.4	61.9	<0.1	0.
02-22-85	-25.4	37.5	8.7	14.5	4.9	7.1	0.3	63.7	<0.1	0.

* Drum prepared; vented to atmosphere through carbon filter.

** Drum purged of gaseous contents, sampled, valve closed to seal contents of drum from atmosphere.

Drum BFB-122 Gen. 1/12/84, 11.9g Pu238

Figure 1-6.

APPENDIX 2

GAS SAMPLING RESULTS FOR 2 DRUMS
VENTED THROUGH THE CARBON COMPOSITE
FILTER AND HAVING AN INNER SEALED
90-MIL LINER

Table 2-I

Drum BFB-113 (Annular Space). Generated 09-26-83. Contains 2.2g (30.8 μ Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H_2	O_2	CO	CO_2	CH_4	N_2	He	Ar
12-08-83*	0.0									
12-29-83**	181.0	22	1.0	18.0	0.4	0.8	0.1	77.9	<0.1	0.9
01-20-84	25.4	27	1.4	18.2	0.7	1.3	0.1	77.4	<0.1	0.9
02-14-84	25.8	58	2.0	17.0	0.6	1.8	0.1	77.7	<0.1	0.9
03-13-84	38.8	64	0.5	17.4	0.8	2.5	0.1	77.8	<0.1	0.9
03-30-84***	0.0	40	2.7	15.7	0.5	2.8	0.2	77.2	<0.1	0.9
04-10-84	0.0	65	2.0	17.9	0.4	1.9	0.1	76.8	<0.1	0.9
05-04-84	0.0	73	1.2	19.3	1.0	1.0	<0.1	76.6	<0.1	0.9
05-25-84	0.0	91.5	0.7	18.4	<0.1	1.0	<0.1	79.0	<0.1	0.9
06-14-84	0.0	85	0.7	19.7	0.7	0.9	<0.1	77.1	<0.1	0.9
07-06-84	0.0	92.5	0.6	20.0	0.2	0.8	<0.1	77.5	<0.1	0.9
07-27-84	0.0	84	1.2	18.5	0.6	1.4	<0.1	77.4	<0.1	0.9
11-13-84	0.0	53	<0.1	21.3	0.2	0.1	<0.1	77.5	<0.1	0.9

* Drum prepared; vents sealed.

** Vented after sampling, but resealed.

*** After sampling, valve opened to vent annular space to atmosphere through carbon composite filter.

Drum BFB-113 (Annular Space)

Gen. 9/26/83, 2.2g Pu238

Figure 2-1.

Table 2-II

Drum BFB-113 (Inside Liner). Generated 09-26-83. Contains 2.2g (30.8 α Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H ₂	O ₂	CO	CO ₂	CH ₄	N ₂	He	Ar
12-08-83*	0.0									
12-29-83**	181.0	22	1.2	18.4	0.7	1.2	<0.1	77.5	<0.1	0.9
01-20-84	25.4	27	1.7	17.8	0.8	1.7	<0.1	77.1	<0.1	0.9
02-14-84	7.8	58	2.2	16.5	0.7	2.1	0.1	77.5	<0.1	0.9
03-13-84	7.8	64	2.9	16.2	1.0	2.7	0.1	76.2	<0.1	0.9
03-30-84***	0.0	40	2.9	15.2	0.5	3.1	0.1	77.3	<0.1	0.9
04-10-84	0.0	65	3.2	15.4	1.3	3.0	0.1	76.1	<0.1	0.9
05-04-84	0.0	73	2.6	17.0	0.8	2.1	<0.1	76.6	<0.1	0.9
05-25-84	0.0	91.5	1.2	17.2	<0.1	1.8	<0.1	78.9	<0.1	0.9
06-14-84	0.0	85	1.4	18.5	0.2	1.7	<0.1	77.3	<0.1	0.9
07-06-84	0.0	92.5	1.2	18.3	<0.1	1.6	<0.1	78.0	<0.1	0.9
07-27-84	0.0	84	0.5	19.2	0.4	0.7	<0.1	78.2	<0.1	0.9
11-13-84	-38.1	53	1.0	19.4	0.2	1.0	<0.1	77.5	<0.1	0.9
12-20-84	-38.1	35	1.0	19.6	0.1	1.0	<0.1	77.4	<0.1	0.9
01-29-85	-38.1	46	1.1	19.3	0.3	1.0	<0.1	77.4	<0.1	0.9
03-06-85	-38.1	44	1.1	20.1	0.5	0.9	<0.1	76.5	<0.1	0.9

* Drum prepared; vents sealed.

** Vented after sampling, but resealed.

*** After sampling, valve opened to vent annular space to atmosphere through carbon composite filter.

Drum BFB-113 (Inside Liner)

Gen. 9/26/83, 2.2g Pu238

Figure 2-2.

Table 2-III

Drum BFB-114 (Annular Space). Generated 09-23-83. Contains 15.6g (218.4 α Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H_2	O_2	CO	CO_2	CH_4	N_2	He	Ar
12-08-83*	0.0									
12-29-83	38.8	22	10.7	15.7	4.3	5.6	0.3	62.6	<0.1	0.7
01-20-84	77.6	27	16.0	13.2	7.3	9.7	0.5	52.8	<0.1	0.6
02-14-84	129.3	58	22.1	10.5	8.5	13.2	0.6	44.6	<0.1	0.5
03-13-84	258.5	64	27.0	8.8	9.3	15.8	0.8	37.8	<0.1	0.5
03-30-84**	299.9	40	25.2	8.0	9.2	18.2	0.9	38.1	<0.1	0.4
04-10-84	0.0	65	20.1	11.6	7.0	11.4	0.6	48.7	<0.1	0.6
05-04-84	0.0	73	9.5	16.3	4.6	6.1	0.3	62.5	<0.1	0.7
06-14-84	0.0	85	8.1	16.7	3.9	5.6	0.3	64.7	<0.1	0.7
07-06-84	0.0	92.5	7.3	16.8	3.5	5.5	0.3	65.8	<0.1	0.8
07-27-84	0.0	84	7.1	17.3	3.9	4.4	0.3	66.2	<0.1	0.8
11-13-84	0.0	53	5.4	18.5	2.2	3.4	0.2	69.5	<0.1	0.9
03-06-85	0.0	44	6.0	18.6	2.5	3.3	0.2	68.6	<0.1	0.8

* Drum prepared; vents sealed.

** After sampling, valve opened to vent annular space to atmosphere through carbon composite filter.

Drum BFB-114 (Annular Space)

Gen. 9/23/83, 15.8g Pu238

Figure 2-3.

Table 2-IV

Drum EFB-114 (Inside Liner). Generated 09-26-83. Contains 15.6g (218.4 α Ci) ^{238}Pu .

Sample Date	Gauge Pressure (mm Hg)	Temp. ($^{\circ}\text{F}$)	Mol% of Gaseous Components							
			H ₂	O ₂	CO	CO ₂	CH ₄	N ₂	He	Ar
12-08-83*	0.0									
12-29-83	38.8	22	11.5	15.1	5.3	7.5	0.4	59.6	<0.1	0.7
01-20-84	103.4	27	17.4	12.4	8.4	11.7	0.5	49.1	<0.1	0.6
02-14-84	155.1	58	23.1	10.0	9.0	14.8	0.7	42.0	<0.1	0.5
03-13-84	284.4	64	27.0	8.3	10.3	16.8	0.8	36.4	<0.1	0.4
03-30-84**	325.7	40	26.3	7.5	10.0	19.2	0.9	35.7	<0.1	0.4
04-10-84	0.0	65	27.3	6.9	10.4	17.8	0.9	36.3	<0.1	0.4
05-04-84	0.0	73	14.8	13.0	7.6	11.7	0.6	51.7	<0.1	0.6
05-30-84	0.0	91.5	12.7	13.3	5.3	10.2	0.5	57.3	<0.1	0.7
06-14-84	0.0	85	11.3	14.0	5.8	9.7	0.5	58.0	<0.1	0.7
07-06-84	0.0	92.5	10.5	14.7	5.1	9.3	0.5	59.2	<0.1	0.7
07-27-84	0.0	84	10.4	14.3	6.1	8.9	0.5	59.1	<0.1	0.7
11-13-84	-25.4	53	9.0	15.6	4.4	7.1	0.4	62.7	<0.1	0.8
12-20-84	-38.1	35	9.6	15.5	4.0	7.1	0.4	62.6	<0.1	0.8
01-29-85	-50.8	46	9.7	15.7	3.7	6.8	0.4	63.0	<0.1	0.8
02-22-85	-50.8	37.5	8.8	15.9	4.4	6.0	0.3	63.8	<0.1	0.8

* Drum prepared; vents sealed.

** After sampling, valve opened to vent annular space to atmosphere through carbon composite filter

Drum BFB-114 (Inside Liner)

Gen. 8/23/63, 15.6g Pu238

Figure 2-4.

Time - Weeks