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TRU WASTE SAMPLING PROGRAM:
VOLUME II-GAS GENERATION STUDIES

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- o Gas Generation Results of Venting for Transuranic Waste Drums,
RFP-3739, April 1985.

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- o Gas Generation and Migration Studies Involving Recently Generated ^{238}Pu -Contaminated Waste for the TRU Waste Sampling Program,
July 1985.

The peer reviews of this document by the following individuals are appreciated:

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ABSTRACT

Volume II of the TRU Waste Sampling Program report contains the data generated from evaluating the adequacy of venting/filtering devices for maintaining safe hydrogen levels in plutonium contaminated waste drums. Additional studies reported in this volume include gas generation rates, selected waste form monitoring, and evaluation of hydrogen migration from sealed 90-mil rigid polyethylene drum liners containing ^{238}Pu -contaminated wastes. All wastes used in the studies were newly-generated, and the waste drums were setup under controlled, experimental conditions.

Studies using ^{239}Pu -contaminated wastes were conducted at the Rocky Flats Plant. Studies using ^{238}Pu -contaminated wastes were conducted at the Los Alamos National Laboratory.

SUMMARY

Volume II of the TRU Waste Sampling Program final report contains the data generated from: (1) evaluating the effectiveness of the carbon composite filter, Hanford vent clip, and semi-permeable styrene-butadiene gasket for maintaining internal drum pressure at or near atmospheric pressure, maintaining hydrogen concentrations within safe limits, and calculating gas generation rates for 16 newly-generated ^{239}Pu waste drums; (2) evaluating the effectiveness of the carbon composite filter on seven 12-year-old waste drums that were vented, purged, and transported from the INEL to the RFP; (3) evaluation of the carbon composite filter and the calculation of gas generation rates for six newly-generated high-activity ^{238}Pu contaminated waste drums; and (4) gas migration from sealed 90-mil rigid polyethylene drum liners.

It should be noted that it was not the intent of the TRU Waste Sampling Program to make policy decisions concerning the management and certification of stored transuranic wastes. However, this report does contain information useful for making such site specific policy decisions.

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Pu Studies

For all 16 drums tested, the carbon composite filter, Hanford vent clip, and the semi-permeable styrene-butadiene gasket performed well in maintaining internal drum pressures at or near atmospheric pressure, and maintaining hydrogen concentrations within safe limits for the three-month test period. The styrene-butadiene gasket did not maintain the internal drum pressure as close to atmospheric pressure as did the carbon composite filter or the Hanford vent clip. However, the slight pressure differential measured (up to 6 torr) for these drums appears to be negligible. The maximum hydrogen concentration observed in any of the vented test drums was 1.9%.

After completion of the venting study, the 16 ^{239}Pu waste drums were sealed and, under controlled experimental conditions, monitored for a three-month period to determine radiolytic gas generation values for these drums. The calculated gas generation values were then compared with values previously obtained in laboratory experiments. The waste forms evaluated included sludges, wet and dry combustibles, plastics, and leaded rubber.

The calculated values, with few exceptions, are in reasonable agreement with previously obtained gas generation values. Care should be exercised when comparing the values for experimentally controlled waste drums with data from actual waste drums. Actual waste drums are generally not controlled in the same manner done for these experiments. Many factors, such as bacterial action, corrosion, and radionuclide distribution, influence gas generation values. Much higher-than-expected G(total gas) values were obtained for drums containing solidified organic wastes. Resampling these drums for radiochemical assay still resulted in G(total gas) values of 15.1-22.5. Other reactions, such as corrosion of the mild steel drum, may be responsible.

Seven 12-year-old retrieved waste drums were monitored weekly for two months to ensure the carbon composite filter maintained internal drum pressures at or near atmospheric pressure and maintained hydrogen concentrations within safe limits. The drums were vented, purged and retrofitted with a carbon composite filter after retrieval. The selected drums contained known gas-generating waste forms having the highest measured concentrations of hydrogen. Originally, the drums were to be shipped from the INEL to the RFP in the vented state. However, due to delays in obtaining the required approvals to ship vented drums in ATMX railcars, the drums were resealed prior to shipment. Upon receipt at the RFP, the drums were resampled for gas composition and then allowed to vent through the carbon composite filter for the test period. Even after being resealed for approximately 5 weeks, the maximum hydrogen level observed during transportation was 1.05%. For the test period, the carbon composite filter

performed well in maintaining internal drum pressures at or near atmospheric pressure. Hydrogen concentrations did not exceed 1.1%. The purge cycle for the INEL prototype drum venting system performed well in removing high hydrogen concentrations from 12-year-old sealed waste containers.

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Pu Studies

Six ^{238}Pu waste drums were evaluated under controlled experimental conditions to determine the effectiveness of the carbon composite filter and to determine gas generation values. Two additional drums were used to also evaluate gas migration from drums containing fully sealed 90-mil polyethylene rigid liners.

For ^{238}Pu waste drums, the carbon composite filter appears adequate to maintain internal drum pressures at or near atmospheric pressure and maintain hydrogen levels within safe limits in all but the highest activity drums. For vented drums using the carbon composite filter with sealed 90-mil rigid liners, up to 30 alpha curies could be present in the drum without hydrogen levels exceeding 4% provided the $G(\text{H}_2)$ value does not exceed 1.0. For an unsealed liner and a $G(\text{H}_2)$ value of 1.0, this limit would be increased to approximately 42 alpha curies. Since these limits were derived from a small number of data points, additional tests are recommended.

The $G(\text{total gas})$ values obtained for six experimentally controlled ^{238}Pu waste drums were in good agreement with previously obtained laboratory values. In particular, the results for drums containing leaded rubber were very consistent between drums.

The gas migration studies indicate a sealed 90-mil rigid liner inhibits hydrogen diffusion out of the liner. Over time, the gases will migrate through the liner, but at a slower rate.

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TRU WASTE SAMPLING PROGRAM:
VOLUME II-GAS GENERATION STUDIES

1. INTRODUCTION

1.1 Background

The TRU Waste Sampling Program was established to evaluate various types of wastes contaminated with transuranic (TRU) isotopes. The sampling program was conducted at the Idaho National Engineering Laboratory (INEL) and the Los Alamos National Laboratory (LANL). The Rocky Flats Plant (RFP) provided support for work associated with evaluation of ^{239}Pu waste stored at the INEL. LANL conducted all work associated with evaluating ^{238}Pu -contaminated wastes. EG&G Idaho was responsible for the overall program management, quality assurance, and final reporting of the national effort under the direction of the Joint Integration Office (JIO). Tasks conducted by INEL, RFP, and LANL were delineated in the Implementation Plan for the TRU Waste Sampling Program, EG&G-WM-6324, July 1983. Appropriate task descriptions are repeated to provide the reader with background information.

1.2 Objectives

The primary objectives of the overall TRU Waste Sampling Program were:

1. To provide information on gas generation in newly generated and stored TRU wastes
2. To evaluate the adequacy of venting/filtering devices (carbon composite filter, Hanford vent clip, and the styrene butadiene gasket) for maintaining safe hydrogen levels in sealed waste drums
3. To determine the adequacy of the INEL real-time radiography system as a nondestructive certification technique

4. To examine wastes for compliance with the Waste Isolation Pilot Plant-Waste Acceptance Criteria (WIPP-WAC).¹

1.3 Purpose

The TRU Waste Sampling Program report consists of two volumes. Volume I, Waste Characterization, consists of data obtained from sampling, opening and examining various aged transuranic wastes stored at INEL and LANL (objectives 1, 3, and 4 above).

The purpose of Volume II is to present the results obtained from gas generation rate studies, evaluation of the effectiveness of filtering/venting devices for maintaining safe hydrogen levels in waste drums, monitoring selected 12-year-old retrieved INEL waste drums to ensure they remain in a safe condition after being vented, purged, and retrofitted with a carbon composite filter, and results from studying hydrogen migration from sealed 90-mil rigid polyethylene drum liners (objective 2 above). In some cases, this data may be applicable to other Department of Energy (DOE) sites. In other cases, the data may be site specific.

This data is subdivided into two sections concerning ²³⁹Pu-contaminated waste studies (INEL/RFP data) and a section concerning ²³⁸Pu-contaminated waste (LANL) studies.

1.4 Quality Assurance and Quality Control

Quality assurance of the data generated by the TRU Waste Sampling Program was provided by management and internal and external review. External peer reviews were conducted by the JIO, RFP, LANL, Rockwell Hanford, Savannah River Plant, and the Sandia National Laboratory.

To ensure that gas sampling and analytical methods were consistent among the participating laboratories, the following special quality control measures were used in addition to established site laboratory procedures and standard practices:

1. Gas Bottles--Bottles or cylinders used to collect gas samples were initially cleaned by evacuating them to less than 2.0×10^{-2} torr (mm/Hg) at 100°C for 2 hours.
2. Measuring Instruments--Instruments (gauges, transducers) used for measuring pressures (subatmosphere and above-atmosphere) were calibrated, as specified by each participating laboratory's quality control program, and were accurate to a minimum of 0.2% of full scale. Pressure measurements will be reported in torr (mm Hg).
3. Drum Sealing--Drums required to be completely sealed were considered sealed if pressurized to 155 torr above atmospheric pressure for 3 hours with a pressure loss of no more than 5 torr.
4. Gas Sampling Pressure--The gas sampling manifold and gas sampling bottle were evacuated to less than 2.0×10^{-2} torr before a gas sample was drawn from the drum. After this pressure has been obtained, the valve to the pump was closed and the pressure observed to ensure that the sampling manifold was properly sealed to the drum lid. The seal was adequate if a pressure rise of less than 1.0×10^{-2} torr was observed for 1 minute.
5. Gas Analysis--Mass spectrometry or gas chromatography was used for analyzing gas samples. In either case, the lower limit of detection was 0.1 vol% for each gas. Sensitivity or response factors were established for each type of gas analyzed. At the beginning of each day, before any gas samples were analyzed, instruments were checked by using one of the gases as an analytical standard for comparison with the established sensitivity or response factor. Results of this test were required to be within $\pm 2\%$ of the established sensitivity or response factor.

6. Gas Standards--A certified gas standard was supplied to each participating laboratory for use when analyzing gas samples. The gas mixture and composition used was the same for each laboratory to allow comparison of analytical results.

After sensitivity or response factors were established for the analytical instrument, a certified gas sample was analyzed before any gas samples were analyzed. The results of this analysis were reported, by phone, to EG&G Idaho before any further analyses were conducted. A sample of the certified gas was then analyzed for approximately every eight field gas samples analyzed.

7. Other Analyses--Other types of analyses, such as those conducted for particulates and unknown materials were conducted on an as needed basis. For an unknown material, 90% of the total was required to be identified.
8. Documentation--Each participating laboratory was required to keep appropriate documentation such as: (a) analytical instrument output (spectra or chromatogram) used for establishing sensitivity or response factors; (b) environmental conditions when drums were sampled; (c) all analytical results, including daily instrument checks, variance from established factors, tolerances, certified gas standard results, and (d) appropriate internal analytical and quality control standards and procedures.

2. ^{239}Pu GAS GENERATION STUDIES

This section contains the task descriptions and data obtained from evaluating the effectiveness of venting/filtering devices (carbon composite filter, Hanford vent clip, and the semi-permeable styrene butadiene gasket) to maintain hydrogen concentrations at safe levels, maintain drum pressures at or near atmospheric pressure, and conduct gas-generation rate studies on experimentally controlled drums containing ^{239}Pu contaminated wastes. These studies were conducted at the Rocky Flats Plant. The data from these studies are contained in Rocky Flats report RFP-3739, Gas Generation Results and Venting for Transuranic Waste Drums, April 1985.

2.1 Gas Generation Study Overview

A total of 16 newly-generated ^{239}Pu waste drums were evaluated in two phases over a 6-month period. In Phase I, the drums were vented for three months using one of three venting/filtering devices to determine if venting was adequate to maintain hydrogen concentrations at safe levels and maintain drum pressure at or near atmospheric pressure. In Phase II, the drums were sealed for a second three-month period for determination of gas generation rates. These rates were then compared to rates previously determined in controlled laboratory experiments.¹⁻⁵

Twelve newly-generated waste drums were fitted the carbon composite filter (Task 5 of the TRU Waste Sampling Program implementation plan⁶). The carbon composite filter, which was developed at the Rocky Flats Plant, is a nuclear-grade filter. Two newly-generated waste drums were fitted with the Hanford vent clip and two drums were fitted with the semi-permeable styrene-butadiene gasket (Task 7 of the TRU Waste Sampling Program implementation plan⁶). All venting/filtering devices meet Type A packaging requirements for radioactive wastes.⁷

2.2 Task Descriptions

2.2.1 Carbon Composite Filter Study (Task 5)

Twelve drums of newly-generated ^{239}Pu wastes, representing six content codes (2 drums/code), were evaluated. The following content codes, which represent known gas generating waste forms, were selected:

(a) Content Code 1-combined sludge, (b) Content Code 3-organic setups, (c) Content Code 330-dry combustibles, (d) Content Code 336-wet combustibles, (e) Content Code 337-plastics, and (f) Content Code 339-leaded rubber (gloves). The following requirements were used:

1. Only newly generated waste was studied
2. High-activity waste (contaminated with americium), if available, was used to increase the probability of significant gas generation
3. Waste was assayed carefully as individual packages or by radiochemical analysis to determine total alpha activity
4. The level of packaging was recorded
5. Drums were full (to reduce the void volume)
6. Waste was contained in drums retrofitted with a carbon composite filter and a separate sampling port that extended inside the drum lid
7. A 100% seal of the drum lid was to be ensured
8. Drums were conditioned at least 24 hours for temperature stabilization before they were tested for void volume
9. Sludges were analyzed for water and nitrate content.

Experiments conducted at RFP have shown there is not a 100% seal on the 90-mil rigid polyethylene drum liner lid. Also, it was assumed that the vent/purge technique developed at INEL would provide a pathway for release of gases inside the liner. The 90-mil rigid liner insert plug was not to be used on the rigid liner lid. This allowed generated gases to flow from the liner through the carbon composite filter.

Gas samples were collected from the sampling port and analyzed periodically. Drum pressures were measured. The samples were analyzed to determine the volume percent of the following gases: hydrogen, oxygen, nitrogen, oxides of nitrogen, carbon dioxide, carbon monoxide and hydrocarbons.

As previously stated, the experiment was conducted in two, three-month phases. During the first phase, the drums were allowed to vent, and gas samples were taken weekly for three months. During the second phase, the filter was removed and the filter insert port was sealed to stop venting. Gas samples were originally to be taken every two weeks for a three month period for determination of the gas generation rate. To improve the quality of the results, gas samples were taken every week for the three month period.

2.2.2 Vent Clip/Gasket Study (Task 7)

Two drums fitted with the Hanford vent clip and two drums fitted with the semi-permeable styrene-butadiene gasket (currently used by Rocky Flats) were evaluated. Two drums of Content Code 330 (dry combustibles) and two drums of Content Code 1 (combined sludge) were used. One drum for each code was fitted with the Hanford vent clip and the other fitted with the styrene butadiene gasket.

The experiment was set up and conducted as described in Section 2.2.1.

2.3 Filtering/Venting Study Experimental Procedure

2.3.1 Drum Selection

The highest activity waste drums, of the required content codes, were selected by drum counter nondestructive assay personnel in Building 771 during a two week period. Content Code 1 and 3 sludge drums were obtained from Building 774, where these drums are generated. Two drums containing unwashed leaded-rubber gloves were obtained from drum storage. One of the drums contained plutonium above the economic discard limit, but was used in the study to prevent further delays in the project. Since the waste used for this study represents full drum quantities of actual TRU waste, the waste cannot be as carefully controlled for matrix composition, radionuclide assay, radioactive particle size and deposition, etc., as was done in the previous laboratory studies where carefully doped, discrete test matrices were used. The exact amount of time that the waste has been contaminated can not be determined since contaminated waste is continually generated and placed in drums. The drums may collect waste for several months before they are sealed. Drums selected for this study were significantly higher in activity than an average waste drum at Rocky Flats Plant and were therefore expected to produce proportionately more gas.

2.3.2 Drum Assay

All drums, except sludge drums (Content Codes 1 and 3), were assayed by segmented-gamma drum scan. The segmented-gamma drum counter uses a 20-minute count with high purity germanium detectors for 413, 662, and 185 keV gamma energy to assign plutonium, americium, and uranium gram values, respectively. Eight segments, approximately four inches high, were counted separately and matrix attenuation corrections were measured for each segment so that the drum need not be completely filled for consistent assay. The sludge and organic setup drums (Content Codes 1 and 3) were assayed by radiochemical analysis of a sample taken during the retrofitting of the drum with the sampling lid. The sludge weight was determined by

subtracting the packaging weights (as determined in stored waste examinations of the TRU Waste Sampling Program) from the gross drum weight to arrive at the net sludge weight.

2.3.3 Drum Setup

After selection and assay, the drums were retrofitted with sampling lids in the Building 776 Size Reduction airlock. The original lids were removed along with the rigid polyethylene drum liner (Type II) lid. At this point, a visual determination of the number of polyethylene or PVC drum bags used in packaging was made and the capacity utilized was determined. The sludge type drums, Codes 1 and 3, were sampled with a coring-type sampler and a representative sample taken for radiochemical assay, water and nitrate determinations. The rigid liner lid was not reinstalled to assure an escape path for the gases.

The 12 drums to be fitted with the carbon composite filter were refitted with modified drum lids. The modified drum lids consisted of a welded carbon-steel half-coupling, used for insertion of the carbon composite filter (for Phase I of the study) or a pipe plug (used to seal the drum for Phase II of the study), and a sampling port connection. The sampling port consisted of an O-ring sealed stainless steel tube connector, particulate filter, and a sampling valve. The drum lids were sealed to the drum using Permatex Form-A-Gasket (trade name).

The two drums using the semi-permeable gasket and the two drums using the Hanford vent clip were fitted with drum lids having the sampling ports only. Drums with the Hanford vent clip were sealed using nonpermeable rubber gaskets.

2.3.4 Void Volume Determination

The drum void volume was determined by measuring the drum pressure and then expanding the gas inside the drum into a calibrated expansion volume and measuring the new pressure. Void volumes were determined on the

12 drums fitted with the carbon composite filter, in a sealed state. The 4 drums fitted with the Hanford vent clip and styrene-butadiene gaskets could not be measured until after the completion of Phase I of the study.

The free volume inside the drum was calculated using the equation:

$$V_v = \frac{P_2 V_c}{P_1 - P_2}$$

where

V_v = void volume of the drum (L)

V_c = calibrated expansion volume (L)

P_1 = pressure inside drum before expansion (mm Hg)

P_2 = pressure inside drum after expansion (mm Hg).

The error associated with the void volume was determined by using the certified accuracy of the pressure gage, the calibrated expansion volume (13.67 liters), the certified volume of an empty, sealed 55-gal drum (220.3 liters) and using the average atmospheric pressure at the RFP of 615 mm Hg for P_1 . The lower and upper certified error limits were determined by RFP to be 10.9 and 13.6%, respectively.

An experimental error analysis was performed by determining the void volume six times on the certified empty drum. The mean and standard deviation of these tests was 233 and 1.3 L, respectively. These results fall within the certified error limits and indicate the actual error limits are much less than the certified error limits. Based on the good reproducibility (precision) of these tests, a correction factor of 0.944 was applied to the calculated drum void volume to obtain a corrected and

more accurate void volume value. Assuming that the correction factor is accurate over a wide range of void volumes and that temperature differences are negligible, the experimental error is $\pm 1.7\%$ (90% confidence).

2.3.5 Drum Sealing

All drums were pressure tested twice during the course of the study to ensure the drums were properly sealed. The drums were considered sealed if they held a pressure of 155 torr (3 psi) above atmospheric pressure for 3 hours with a pressure loss of no more than 5 torr. The 12 drums fitted with carbon composite filters were tested before the start of Phase I and after the conclusion of Phase II. Drums fitted with the Hanford vent clip or the styrene-butadiene gasket were tested before and after Phase II.

All drums remained sealed to the above criterion during the study, with the exception of two drums. Drums D31254 (Figure 5) and D31528 (Figure 2) developed leaks of 41 torr and 28 torr respectively in the six-month study. Even though these drums leaked when pressurized to 155 torr, the leak rate would be significantly less with the small (less than 15 torr) pressure differential encountered. In fact, the leaks may have developed at the drum gasket or pipe couplings as a direct result of applying 155 torr pressure differential. The leaks were not considered in determining the gas generation rates.

2.3.6 Gas Sample Analysis

All Phase I and Phase II drums were sampled weekly for both pressure and gas composition for 13 weeks. The gas sampling manifold and gas sampling bottle were evacuated to less than 0.02 torr before a gas sample was drawn. After this pressure was obtained, the valve to the vacuum pump was closed to ensure that there were no leaks in the sampling manifold as demonstrated by a pressure rise of less than 0.01 torr in one minute.

It should be noted that after conclusion of Phase I, some of the drums did not have gas concentrations representative of normal atmospheric air. Prior to initiating Phase II (gas generation rate study), all drums were "flushed" to return the drum to atmospheric air conditions. The flushing cycle consisted of evacuating each drum from atmospheric pressure to 300 torr, then pressurizing to 900 torr with bottled air. This was repeated three times to return the drums to normal atmospheric air conditions. This procedure of evacuating and pressurizing three times before the drum was returned to atmospheric pressure should have removed approximately 96.3% of the original atmosphere and replaced it with ambient air. The drums were then immediately resampled to obtain a time zero baseline.

Gas sample composition analysis was done on a DuPont Model CEC 21-104 mass spectrometer. Several rigid parameters were set up to assure better accuracy of the analysis. All samples were analyzed on the same instrument and by the same operator. All samples for a given week were analyzed on the same shift to minimize the effect to instrument drift and the need for recalibration. Calibration consisted of analyzing pure gases for H_2 , Ar, CO_2 , He, CO, CH_4 , N_2O , NO (to cover NO_x), and air (to cover N_2 and O_2). All other calibration gases and organics were determined at other times as necessary.

A certified gas standard, supplied by EG&G Idaho, was analyzed weekly by RFP each time the drums were sampled. The gas standard (Bottle 2) consisted of the following types of gases and concentrations: $H_2 = 2.57\%$; $O_2 = 18.7\%$; $N_2 = 66.7\%$; and $CO_2 = 12.0\%$. The results of the gas standard analysis for Phase I are presented in Table 1. The analysis indicate the relative error, when compared with the certified gas standard is: $H_2 = -1.95\%$; $O_2 = +3.21\%$; $N_2 = -1.50\%$; and $CO_2 = +3.33\%$.

The gas standard analyses conducted for Phase II are shown in Table 2. The relative error for Phase II, when compared with the certified gas standard is: $H_2 = -0.78\%$; $O_2 = +0.53\%$; $N_2 = -1.20\%$; and $CO_2 = +6.67\%$.

TABLE 1. RFP GAS STANDARD ANALYSIS-PHASE I

<u>Week No.</u>	<u>N₂</u>	<u>O₂</u>	<u>H₂</u>	<u>CO₂</u>
1	65.7	19.3	2.65	12.3
2	65.4	19.6	2.54	12.4
3	66.0	19.1	2.43	12.5
4	66.4	18.8	2.54	12.3
5	65.3	19.3	2.49	12.9
6	65.1	19.7	2.52	12.7
7	65.1	19.9	2.43	12.6
8	65.7	19.4	2.54	12.3
9	65.8	19.3	2.61	12.3
10	66.5	18.8	2.53	12.2
11	66.0	19.1	2.50	12.4
12	65.9	19.2	2.51	12.4
13	65.9	19.1	2.41	12.5
$\bar{x}^a =$	65.7	19.3	2.52	12.4
$\sigma^b =$	0.44	0.32	0.07	0.19
$\sigma/\bar{x}^c =$	0.68%	1.67%	2.71%	1.56%

a. Mean or Average.

b. Standard deviation.

c. Coefficient of variation.

TABLE 2. RFP GAS STANDARD ANALYSIS-PHASE II

Week No.	<u>N₂</u>	<u>O₂</u>	<u>H₂</u>	<u>CO₂</u>
0	66.4	18.4	2.56	12.7
1	65.4	19.2	2.64	12.8
2	65.2	19.6	2.54	12.7
3	65.5	18.9	2.53	13.1
4	66.6	18.2	2.52	12.6
5	65.5	18.9	2.58	13.1
6	66.0	18.7	2.55	12.7
7	65.9	18.5	2.61	13.0
8	65.8	18.6	2.58	13.0
9	66.1	18.8	2.57	12.6
10	66.6	19.0	2.50	11.9
11	65.7	19.1	2.56	12.6
12	66.1	18.8	2.51	12.6
13	65.2	18.5	2.50	13.8
$\bar{x}^a = 65.9$		18.8	2.55	12.8
$\sigma^b = 0.47$		0.36	0.04	0.42
$\sigma/\bar{x}^c = 0.72\%$		1.92%	1.61%	3.26%

a. Mean or Average.

b. Standard deviation.

c. Coefficient of variation.

TABLE 3. PHASE I DRUM IDENTIFICATION

<u>Figure</u>	<u>Drum Number</u>	<u>Content Code</u>	<u>Code Description</u>	<u>Filter Type</u>
1	D29258	1	Combined Sludge	Carbon Composite Filter
2	D31528	1	Combined Sludge	Carbon Composite Filter
3	D32180	1	Combined Sludge	Hanford Vent Clip
4	D32186	1	Combined Sludge	Styrene Butadiene Gasket
5	D31254	3	Organic Setup	Carbon Composite Filter
6	D31403	3	Organic Setup	Carbon Composite Filter
7	D24545	330	Dry Combustibles	Carbon Composite Filter
8	D31042	330	Dry Combustibles	Carbon Composite Filter
9	D26048	330	Dry Combustibles	Hanford Vent Clip
10	D31703	330	Dry Combustibles	Styrene-Butadiene Gasket
11	D25634	336	Wet Combustible	Carbon Composite Filter
12	D31216	336	Wet Combustible	Carbon Composite Filter
13	D25691	337	Plastic & Rubber	Carbon Composite Filter
14	D30638	337	Plastic & Rubber	Carbon Composite Filter
15	D29758	339	Leaded Rubber	Carbon Composite Filter
16	D30175	339	Leaded Rubber	Carbon Composite Filter
17	D32538	Empty	Control Drum	Sealed

2.4 Phase I Results

The results for Phase I, the vented portion of the study, are shown graphically in Figures 1 through 17 of Appendix A. Table 3 lists a figure number, drum identification number, Content Code, and Content Code description for each drum used in the study. For each drum, the internal drum pressure and atmospheric pressure, measured at the time of sampling, are graphically presented as the "a" figure for that drum. The composition of the sampled atmosphere within each drum is presented in Figures "b", "c", and "d" for each drum.

2.4.1 Drum Pressures

As indicated in Figures 1a-17a, Appendix A, excellent correlation exists between the drum pressures and atmospheric pressures, with one notable exception. The exception, Drum D31703 (Figure A-10a), which was fitted with a styrene-butadiene gasket, had a slight pressure differential of up to 6 torr (0.12 psi) during the study.

The control drum (D32538, Figure A-17), was an empty drum which was sealed after volume calibration, and contained a subambient atmospheric pressure. This pressure was expected to remain constant despite variations in atmospheric pressure. As can be seen from Figure 17a (Appendix A), this was the case. The slight pressure variations seen in the graph are within the calibrated gauge tolerance of ± 2 torr.

The carbon composite filter and the Hanford vent clip equilibrated the drum pressure with atmospheric pressure very well. All drums fitted with the styrene-butadiene semi-permeable gasket did not maintain drum pressures as close to atmospheric pressure as the carbon composite filter or the Hanford vent clip. However, for the test period of 3 months, this difference appears to be negligible.

2.4.2 Hydrogen Levels

All filtering/venting devices performed well in maintaining hydrogen levels within safe limits. The maximum hydrogen level observed was approximately 1.9% in Drum D30175 (Figure 16c, Appendix A). This drum contained leaded rubber and was fitted with a carbon composite filter. The drum atmosphere, at week 1, was 1.9% hydrogen. This level decreased to approximately 1.4% hydrogen at the end of the test. Drum D32180 (Figure 3c, Appendix A) contained combined sludge and was fitted with the Hanford vent clip. The drum atmosphere, at week 1, contained 0.8% hydrogen. This level increased slightly, with some fluctuation, to approximately 1.2% hydrogen by the end of the test.

2.5 Phase II Results

This section contains the data obtained from monitoring the 16 drums from Phase I for internal pressure, gas composition, and radiolytic gas yields for a 3-month period. The results of the sampling are graphically presented in Appendix B (Figures 1-17). The internal drum pressure is shown in the "a" figure for each drum. The composition of the sampled drum atmosphere is shown in the "b", "c", "d", and "e" (if necessary) figures for each drum. It should be noted that the gas compositions are graphed as partial pressures (volume percent x total drum pressure) and are also presented in tabular form as volume percent. Partial pressure graphs are commonly used to present this data. For example, as the drum pressure increases due to gas generation, the partial pressures of the inert gases such as nitrogen and argon should maintain relatively constant, while the volume percent of nitrogen and argon decrease. The partial pressures are subject to pressure measurement errors associated with the mass spectra analysis and the calibrated gauge tolerance of ± 2 torr.

The carbon composite filters were removed from the 12 Phase I drums and pipe plugs were installed into the coupling on the lid to seal the drum. The Hanford vent clips and styrene-butadiene gaskets were removed

from the remaining four drums. Nonpermeable gaskets, coated with Permatex Form-A-Gasket (trade name), were installed to seal the drums. The drums were pressure tested, as described in Section 2.3.5. All drums were flushed (see Section 2.3.6) to return the drums to atmospheric air conditions prior to initiating Phase II. The drums were sampled immediately after the flushing process to obtain time zero baseline data for drum internal pressure and gas composition.

All drums had various amounts of isopropanol [a solvent constituent in the Permatex Form-A-Gasket (trade name) used to seal the drums]. Drum D31703 (Figure 10 Appendix B) contained an organic compound that was not identified. It was calculated and included with the C_1 - C_4 hydrocarbons.

2.5.1 Gas Yield Evaluation

The radiolytic gas yields from the six waste categories were calculated from the sampling results and assay values. Table 4 shows the pertinent data used to determine the gas yields. The partial pressures shown were obtained from the slopes of the graphs in Appendix B, Figures 1-16. The moles of gas generated were calculated using the ideal gas law, sampling data and the drum void volume. The gas yields are given in Table 5 as G-values. G(gas) is defined as the number of gas molecules formed for each 100 eV of absorbed energy. The G-values were calculated using:

$$G = \frac{\text{Moles gas} \times (6.02 \times 10^{23} \text{ molecules/mole}) \times 100}{[(g \text{ Pu} \times 0.00211 \text{ watt/g}) + (g \text{ Am} \times 0.115 \text{ watt/g})] \times (6.24 \times 10^{18} \text{ eV/sec watt}) \times (7.86 \times 10^6 \text{ sec/13 week})}$$

In general the gas yields show satisfactory consistency within each waste category and are reasonable when compared to previously obtained laboratory values. A few of the gas yields (G-values) appear to be greater

TABLE 4. GAS GENERATION DATA

Appendix A or B Figure Number	Drum No.	Content Code	Pu(g)	Am(g)	Alpha Curies	ev ^a x10 ²⁴	Void Vol. (l)	Partial Pressure (Torr) ^b			Moles Generated		Weight %	
								H ₂	CO ₂	O ₂	H ₂	CO ₂	H ₂ O	NO ₃
1	D-29258	1st stage sludge-1	17.2	1.12	5.14	8.09	93	8.0	0.5	-16	0.0403	0.0025	48.9	8.4
2	D-31528	1st stage sludge-1	9.64	0.90	3.82	6.08	103	5.0	0	-20	0.0279	0	36.1	7.8
3	D-32180	1st stage sludge-1	68.9	3.03	15.6	24.2	102	14	0.2	+53	0.0773	0.0011	51.3	8.2
4	D-32186	1st stage sludge-1	19.5	1.65	7.14	11.3	110	4.9	0.2	-18	0.0292	0.0012	55.9	7.6
5	D-31254	Grease-3	3.19	0.033	0.35	0.517	60	40	0	90-0(1 wk)	0.130	0		
6	D-31403	Grease-3	1.45	0.026	0.20	0.299	86	24	0	90-0(1 wk)	0.112	0		
7	D-24545	Dry combustibles-330	16.4	0	1.21	1.69	204	5.3	4.2	-24	0.0585	0.0464		
8	D-31042	Dry combustibles-330	28.9	0	2.14	2.99	215	6.0	5.7	-47	0.0698	0.0663		
9	D-26048	Dry combustibles-330	28.3	0	2.09	2.93	182	3.9	2.3	-21	0.0384	0.0227		
10	D-31703	Dry combustibles-330	32.4	0	2.40	3.35	175	2.3	30	-80	0.0218	0.284		
11	D-25634	Wet combustibles-336	15.5	0	1.15	1.60	203	1.8	0.4	-19	0.0198	0.0044		
12	D-31216	Wet combustibles-336	26.1	0	1.93	2.70	213	2.0	1.1	-24	0.0231	0.0127		
13	D-25692	Plastics & rubber-337	28.8	0	2.13	2.98	182	5.4	11	-73	0.0532	0.108		
14	D-30688	Plastics & rubber-337	34.4	0	2.55	3.56	186	3.8	4.5	-27	0.0383	0.0453		
15	D-29758	Leaded gloves-339	2	--	0.15	0.207	202	0.1	2.0	-17	0.0011	0.0219		
16	D-30175	Leaded gloves-339	163.5	0.3	13.1	18.7	201	27	14	-75	0.294	0.152		

a. Energy released in 13 weeks.

b. Partial pressure change in 13 weeks (temperature 23°C).

TABLE 5. PHASE II GAS YIELDS

Fig. No.	Drum No.	Content Code	Code Description	G(H) 2	G(CO) 2	G(HC)	G (total)
1	D29258	1	Combined sludge	0.30	0.01	--	0.31
2	D31528	1	Combined sludge	0.28	0.01	--	0.29
3	D32180	1	Combined sludge	0.19	0.01	--	0.20
4	D32186	1	Combined sludge	0.16	0.01	0.02	0.19
5	D31254	3	Grease	15.1	0	--	15.1
6	D31403	3	Grease	22.5	0	--	22.5
7	D24545	330	Dry combustible	2.1	1.6	--	3.7
8	D31042	330	Dry combustible	1.4	1.3	0.9	3.6
9	D26048	330	Dry combustible	0.79	0.47	--	1.26
10	D31703	330	Dry combustible	0.39	5.1	--	5.49
11	D25634	336	Wet combustible	0.74	0.17	--	0.91
12	D31216	336	Wet combustible	0.52	0.28	0.25	1.05
13	D25691	337	Plastic & Rubber	1.1	2.2	--	3.3
14	D30688	337	Plastic & Rubber	0.65	0.77	--	1.42
15	D29758	339	Leaded Rubber	0.32	6.4	--	6.72
16	D30175	339	Leaded Rubber	0.95	0.49	0.07	1.51

than expected and these are mentioned in the below discussions for each waste category. Gas yields less than laboratory values can be explained by assuming that the actinides were not uniformly distributed on the organic matrix.

Oxygen was depleted at a relatively rapid rate as shown in Table 5. Some of these rates are too high to be attributed to radiolysis, and must be the result of another mechanism such as corrosion, bacterial action (to form CO_2), thermal decomposition or other chemical reactions. The G-values associated with oxygen depletion have not been included in Table 5 since the oxygen depletion may be caused by mechanisms other than radiolysis. The G(total) shown is the sum of $\text{G}(\text{H}_2)$, $\text{G}(\text{CO}_2)$ and $\text{G}(\text{hydrocarbons})$, where applicable. No increase in NO_x was noted in any of the drums during the study.

Some of the radiolytic yields obtained in previous studies are shown in Table 6.⁵ Table 6 is a partial listing of previously obtained laboratory values. The scope of this report did not include resolution of discrepancies which may exist. Table 6 shows that almost all the yields for organics and hydrogenous materials are within an order of magnitude. A two-fold difference in yields obtained in different laboratories is not extraordinary because of differences in materials, radiation sources (in alpha and gamma), and experimental techniques.

2.5.2 Waste Form Gas Yield Comparison

The gas generation yields for each waste form category sampled are discussed below.

2.5.2.1 Sludge. The H_2 yields for the four sludge drums appear reasonable for RFP generated sludge,¹ although they correspond to yields that would be obtained from sludge that contained 75% water. The water and nitrate contents of these sludges have been included in Table 4 because of their influence of the radiolytic yields. The presence of nitrate

TABLE 6. LABORATORY OBTAINED G (TOTAL GAS) VALUES

Matrix		G(Total Gas)
LANL:	cellulosics	1.3-2.9
	PVC	8-11*
	asphalt	0.2-1.0
	cellulosics, dry	1.6
	cellulosics, soaked	1.5
	composite	1.4
	polyethylene	1.9
	pump oil	1.3-1.8
SRL:	cellulosics	1.9
	pump oil	2.0
	octane	4.5
	concrete TRU ash	0.0003-0.6
RFP:	cellulosics (dry)	0.63
	cellulosics (wet)	0.31
	polyethylene	0.73
	PVC	0.43-0.96
	plexiglas	1.9
	rubbers (glovebox gloves)	0.37
	oil	3.1
First stage process sludge		1.0

Source: M. A. Molecke, Gas Generation for Transuranic Waste Degradation: Data Summary & Interpretation, SAND 79-1245, Sandia National Laboratory, Albuquerque, New Mexico, December 1979.

*At ambient temperature, the G(total gas) is approximately 1.0 (Reference 9).

significantly reduces the hydrogen yield. The primary radiolysis product from nitrate sludge is oxygen but only one drum D32180 (Figure B-3) showed a net increase in oxygen content. The $G(O_2)$ value of 0.7 is in agreement with previous values. Oxygen depletion in the other three drums must be due to another reaction (corrosion, etc.).

2.5.2.2 Grease. The H_2 yields for the two grease drums are much higher than the expected G-values of two to three.¹ The reason for the discrepancy may be either an incorrect plutonium or americium assay or another type of reaction, such as bacterial or corrosive action, as indicated by the complete depletion of oxygen in one week.

Radionuclide assay for each of the drums was completed twice. Each assay was determined by radiochemical analysis of a sample of grease. The original assay was determined from an approximate 2-inch core taken from the top of the drum. In an attempt to obtain a more representative sample, the drum was sectioned in a contamination control vault and a composite sample was obtained from three locations in a grease matrix. The apparent $G(H_2)$ for D31254 (Figure B-5) was reduced from 36 to 15.1, while D31403 (Figure B-6) increased from 13 to 22.5. The assay results indicate that while the grease may not be homogeneous, the hydrogen rates appear representative for these drums.

Another type of reaction may be responsible for the high G-values from these drums. The rate of oxygen depletion is too rapid to be attributed to radiolysis. Corrosion of the mild steel drum can produce hydrogen gas in an anoxic and wet atmosphere.⁵ The oxygen depletion after one week produced an anoxic atmosphere. These grease drums were analyzed by infrared and thermogravimetric analysis and were found to contain paraffin oils, silicates, sulfates and approximately 20% water. Therefore, the drum atmosphere is expected to be water saturated. A total hydrogen gas generation rate from corrosion of more than twice the rate from radiolysis is possible under certain conditions.⁵

2.5.2.3 Dry Combustibles. The gas yields for the four dry combustible drums are in the range of values previously obtained,¹⁻⁵ with one exception. The only anomalous value is the high CO_2 yield ($G=5.1$)

for D31703 (Figure B-10). A possible explanation for the high CO_2 yield is bacterial action. The total gas rate from bacterial action is highly variable but can be 2 to 3 orders of magnitude higher than the rate from radiolysis. Carbon dioxide is the primary component expected.⁵

2.5.2.4 Wet Combustibles. Very reasonable values have been obtained for both drums in this category.¹ The CO_2 yields are generally lower than the H_2 yields for cellulose and plastics as is demonstrated in these drums.

2.5.2.5 Plastics and Rubber. The gas yields obtained for this category are also well within the range of laboratory values. Complete characterization of the waste matrix, location and distribution of radionuclides on the matrix was beyond the scope of this study. The CO_2 value for D25691 (Figure B-13) appears to be somewhat higher than those previously obtained.

2.5.2.6 Leaded Gloves. The G-values for leaded rubber are in agreement with previous laboratory tests.¹ The $G(\text{CO}_2)$ for D29758 (Figure B-15) is much higher than can be attributed to radiolysis. This higher CO_2 value may be due to bacterial action as discussed in the section on dry combustibles.

3. MONITORING SELECTED CELL 5 WASTE

This section contains the results of monitoring seven 12-year-old waste drums retrieved from the INEL Transuranic Storage Area, Pad 1, Cell 5 (Task 8 of the TRU Waste Sampling Program implementation plan⁶). The purpose of this task was to evaluate retrieved drums that had been vented, purged and retrofitted with carbon composite filters to ensure that hydrogen concentrations remained at safe levels.

3.1 Experimental Procedure

The seven drums were selected for this task after analytical results were obtained from gas sampling the drums at the INEL. The drums were selected from known gas generating waste forms containing the highest concentrations of hydrogen. After venting and purging operations were completed, the drums were retrofitted with carbon composite filters. Originally, the drums were to be shipped to the RFP in the vented state. However, due to delays in obtaining the required approvals to ship vented waste containers in ATMX railcars, the drums were resealed by removing the carbon composite filter and installing a pipe plug. The drums were sealed for approximately 5 weeks.

Upon receipt of the sealed drums at RFP, each drum was resampled for internal pressure and gas composition. The drums were then retrofitted with new drum lids containing a carbon composite filter and a sampling port. The drums were then monitored weekly for a two month period.

Gas sampling was conducted as described in Section 2.3.6. Results for gas standard analysis are shown in Table 7. The analysis indicates the relative error, when compared to the certified gas standard is: $H_2 = -0.39\%$; $O_2 = -0.53\%$; $N_2 = -0.45\%$; and $CO_2 = +4.17\%$.

TABLE 7. RFP GAS STANDARD ANALYSIS (TASK 8)

<u>Date</u>	<u>Gas Composition (Volume %)</u>			
	<u>H₂</u>	<u>N₂</u>	<u>O₂</u>	<u>CO₂</u>
2-06-85	2.49	66.6	18.7	12.2
2-13-85	2.65	66.6	18.2	12.6
2-20-85	2.55	66.1	18.7	12.7
3-01-85	2.53	66.3	18.4	12.7
3-20-85	2.56	66.4	18.6	12.5
3-26-85	2.57	66.1	18.8	12.5
3-29-85	<u>2.57</u>	<u>66.8</u>	<u>18.5</u>	<u>12.1</u>
$\bar{x}^a$ = 2.56		66.4	18.6	12.5
σ^b = 0.05		0.27	0.21	0.24
$\sigma/\bar{x}^c$ = 1.90%		0.40%	1.12%	1.89%

a. Mean or average.

b. Standard deviation.

c. Coefficient of variation.

3.2 Results

The results for the initial sampling of the drums at INEL, after retrieval, are presented in Table 8. All drums had hydrogen concentrations greater than 4 volume %. The maximum hydrogen concentration was 25.9 volume %. Upon receipt at RFP, the drums were sampled. Results for the initial resampling at RFP, after the drums had been sealed for 5 weeks, are also included in Table 8. These results indicate that very little hydrogen was generated during the 5 weeks the drums were sealed for transportation to the RFP. These results also demonstrate the effectiveness of the venting and purging operation conducted by the INEL prototype drum venting system.

3.2.1 Drum Internal Pressure

Results of drum internal pressure monitoring for the 2-month period are presented in Figures 1 and 2, Appendix C. As indicated by Figures C-1 and C-2, excellent correlation exists between the drum internal pressure and the atmospheric pressure. The carbon composite filter performed well in maintaining drum pressure at atmospheric pressure. The results for control Drum D32538, which was an empty sealed drum (see Section 2.4.1), are included. The slight variations in pressure, which remained constant despite atmospheric pressure variations, are within the calibrated gauge tolerance.

3.2.2 Hydrogen Levels

Overall, the carbon composite filter performed well, maintaining hydrogen concentrations at less than 1.1 volume % for all drums during the test period. Six of the drums showed flat or decreasing hydrogen curves during the eight week study. A single drum, 743-11838 (see Appendix C, Figure 5c), showed an increase of hydrogen to a peak 1.1 volume percent in week 7, before falling off in week 8.

The increase in hydrogen in drum 743-11838 could be due to a corrosion mechanism causing the cathodic reduction of water to liberate hydrogen gas. This mechanism proceeds in an anoxic and wet atmosphere. The oxygen in this drum was rapidly depleted and fell below one vol % during week 2. The hydrogen increase started during week 2 also. Grease drums generally contain approximately 20 wt % water. Complete characterization of this waste drum was outside the scope of this study, however the drum atmosphere was expected to be water saturated. The drum apparently breathed during the course of the study since the oxygen level varied between 0.2 vol % and 0.8 vol %. The reason that the oxygen level remained low may be that atmospheric oxygen was still contributing to drum corrosion or because atmospheric oxygen could not readily diffuse inward through the filter while hydrogen and other gases were diffusing outward. The higher nitrogen levels (above normal atmospheric concentrations) can be attributed to depletion of oxygen in the drums.

TABLE B. SELECT CELL 5 WASTE FOR TASK 8 (12 YEAR OLD DRUMS)

Container ID No.	Content Code	Sample ^a Location	Code Description	H ₂	O ₂	N ₂	Ar	CO ₂	CO	NO _x	H.C. ^b	Trichl ^c	Other ^d H.C.	(mm Hg)	Gauge (mm Hg)	Corrected Drum Void Volume (e)	Storage Time (Days)	Total ^a Activity (Ci)
741-12858	1	INEL RFP	1st Stage Sludge	11.7 0.31	35.7 23.2	51.3 74.6	0.61 0.89	0.14 0.20	-- --	0.46 --	-- 0.35	0.04 --	0.01 ^e 0.48 ^e	591 617	+ 30 0.0	104 --	4,213 32 ^j	12.1 --
742-21396	2	INEL RFP	2nd Stage Sludge	8.63 0.10	0.13 14.5	89.9 83.4	1.00 1.03	0.07 --	-- --	-- --	0.15 0.52	0.08 --	0.03 ^e 0.53 ^e	490 611	-127 - 8.0	80 --	4,306 32 ^j	0.40 --
743-11838	3	INEL RFP	Organic Setup	13.7 0.04	1.60 8.89	83.2 82.1	0.96 1.01	0.05 0.03	-- --	-- --	-- 0.80	-- --	0.07 ^e 0.39 ^f 4.44 ^e 2.67 ^f	628 617	- 12.0 0.0	-- ^g -- ^g	4,239 32 ^j	0.12 --
744-2376	4	INEL RFP	Special Setup	25.9 0.17	0.09 9.89	72.1 88.4	0.86 1.05	0.09 0.05	-- --	0.55 --	0.44 0.22	-- --	-- 0.19 ^e	629 613	- 1.0 0.0	-- ^g -- ^g	4,305 35 ^j	0.02 --
90-00042	330	INEL RFP	Dry Combustible	13.6 1.05	0.10 13.8	76.4 79.4	0.94 0.97	8.45 4.38	-- --	-- --	0.22 0.40	-- --	0.32 ^h 0.07 ^f	621 675	- 15.0 + 69.0	137 --	4,215 36 ^j	1.18 --
90-00039	336	INEL RFP	Wet Combustible	7.33 0.79	0.29 3.57	90.4 92.8	1.0 1.14	0.74 0.80	-- --	-- --	0.17 0.34	-- --	-- 0.09 ^f 0.47 ⁱ	515 607	-122.0 + 1.0	165 --	4,227 36 ^j	1.76 --
22-00224	339	INEL RFP	Leaded Rubber	0.74 0.05	16.0 20.3	80.7 77.7	0.96 0.93	1.60 1.01	-- --	-- --	-- 0.02	-- --	-- --	-- 604	-- - 2.0	182 --	4,268 36 ^j	1.26 --

a. Drums samples at INEL after retrieval from storage; sealed and shipped to RFP where they were again sampled.

b. Hydrocarbons (saturated)--methane through propane.

c. Abbreviation for 1,1,1-trichloroethane.

d. Other hydrocarbons--as specified.

e. Trichloroethylene.

f. Freon.

g. Drum not sealed sufficiently to obtain a void volume.

h. Contains CF₃⁺ fragment.

i. Carbon tetrachloride.

j. Time between date sealed at INEL prior to shipment and date sampled at RFP.

4. ^{238}Pu GAS GENERATION STUDIES

This section contains the task descriptions and data generated from evaluating gas generation and migration studies conducted on newly-generated ^{238}Pu -contaminated wastes. These studies were conducted on experimentally controlled drums by the Los Alamos National Laboratory. This data was provided in LANL draft report, "Gas Generation and Migration Studies Involving Recently Generated ^{238}Pu -Contaminated Waste for the TRU Waste Sampling Program," A. Zerwekh and J. L. Warren, July 1985.

4.1 ^{238}Pu Gas Generation Study Overview

A total of eight high-activity newly-generated ^{238}Pu waste drums were evaluated in two phases over a 6-month period (Task 10 of the TRU Waste Sampling Program implementation plan⁶). Six drums were evaluated to determine the effectiveness of the carbon composite filter in maintaining hydrogen concentrations at safe levels and determining gas generation rates. Two drums were used to evaluate hydrogen migration from sealed 90-mil rigid polyethylene drum liners and determine gas generation rates.

4.1.1 Task Description

The following paragraphs define task requirements as stated in Task 10 of the TRU Waste Sampling Program implementation plan. Six drums of high-activity, newly generated ^{238}Pu waste were evaluated to: (a) 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 was placed in a drum. The study was set up and conducted as described in Section 2.2.1 (RFP Task 5).

Two drums of high-activity, newly generated ^{238}Pu waste were 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 was also obtained, if possible. Waste of known type (high gas generating potential) and activity level was used. Parameters described for RFP Task 5 were used, with the following exceptions.

1. Waste was placed in drums retrofitted with a carbon composite filter, a sampling port that extended inside the 90-mil rigid liner, and a separate port that extended inside the drum lid.
2. The 90-mil rigid liner insert plug was used and sealed on the rigid liner lid.
3. Rigid liners and procedures for sealing the rigid liner were comparable to those used by the RFP and the Savannah River Plant.
4. Void volume of the rigid liner was determined.
5. The following was recorded for each sample when obtained: drum and rigid liner pressures, atmospheric pressures, and temperatures (room).

Gas samples were collected and analyzed as described in Section 1.4. The experiment was to be conducted in two three-month phases. During the first phase, the drum was in the unvented state with the filter sealed. Gas samples were to be taken from each sampling port weekly for three months to determine (a) gas concentrations in the rigid liner and the drum, (b) leak rates of hydrogen from the rigid liner, and (c) gas generation rates. During the second phase, the drum was allowed to vent. Gas samples were to be taken weekly for three months to determine the rate of reduction in gas concentration in the waste container.

4.2 Experimental Procedure

4.2.1 Waste Selection

All waste drums selected for the gas generation and migration studies contained varying mixtures of combustible wastes. Combustible wastes are considered the most significant radiolytic gas generator at LANL. All wastes used in the study were generated from heat-source plutonium operations. Heat source plutonium has the following isotopic weight composition: ^{238}Pu -80.0%; ^{239}Pu -16.3%; ^{240}Pu -3.0%; ^{241}Pu -0.6% (not an alpha-emitter); and ^{242}Pu -0.1%. Data for the individual drums used in the studies are presented in Table 9.

4.2.2 Waste Assay

All wastes contained in 30-gallon drums were assayed by segmented gamma scan techniques. Each waste package (bagout bags) within the drum was individually scanned and the assay results for the individual packages were then totaled to give a drum total.

4.2.3 Experimental Drum Preparation

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. All waste transferred to the 55-gallon experimental drums was contained in two individually sealed (with vinyl tape) 5-mil polyethylene bags. All drums were lined with a 90-mil high density polyethylene liner. However, as required for the carbon composite filter study, the rigid liner lid was not sealed. The sealing plug in the rigid liner lid was also left out to provide a pathway for gases to escape. In March 1984, three additional drums of newly generated ^{238}Pu -contaminated waste were transferred to 55-gallon drums fitted with a valve, a pressure gauge, and a carbon composite filter. The above six drums constituted the drums used in the carbon composite filter study/gas generation rate experiment.

TABLE 9. LANL EXPERIMENTAL WASTE DRUM DATA

Drum Number	Waste Description	Generation Date	Drum Preparation Date	²³⁸ Pu	
				Grams	Activity (Ci)
BFB-109 ^a	Plastic	01/27/84	03/26/84	3.0	42.0
BFB-112 ^a	Mixed Combustibles	07/28/83	12/08/83	1.2	16.8
BFB-116 ^a	Mixed Combustibles	10/27/83	12/08/83	2.28	31.9
BFB-118 ^a	Mixed Combustibles/ Noncombustibles	10/27/83	12/08/83	4.92	68.9
BFB-120 ^a	Rubber	01/03/84	03/26/84	1.6	22.4
BFB-122 ^a	Mixed Combustibles	01/12/84	03/26/84	11.9	166.6
BFB-113 ^b	Rubber	09/26/83	12/08/83	2.2	30.8
BFB-114 ^b	Mixed Combustibles	09/23/83	12/08/83	15.6	218.4

a. Carbon Composite Filter and Gas Generation Study.

b. Migration and Gas Generation Study.

The two drums prepared for the migration/gas generation study were lined with 90-mil rigid polyethylene drum liners. Both the rigid liner and the drum lid were fitted with sampling valves, pressure gauges, and carbon composite filters. This allowed monitoring the gas inside the sealed 90-mil liner and the annular space between the 90-mil liner and the drum.

General Electric white silicon caulking compound (GE-SE-1202) was used to seal the drum lid and rigid liner lids, and also to seal the measuring/sampling devices to the inner 90-mil rigid liner lid. To seal the measuring/sampling device to the drum lid, a 0.25 in. pipe coupling was weld-sealed to the lid. The measuring/sampling device was fitted and sealed to the coupling.

4.2.4 Pressure Testing

All drums were pressure tested twice during the course of the study to ensure the drums were sealed. The first pressure test was conducted after the drums had been loaded with waste materials and initially sealed. The drums were pressurized to approximately 200 torr (3.9 psi) above atmospheric pressure. The drums were monitored for leakage after 30 and 60 minutes. The drums were found to be satisfactorily sealed. The drums were rechecked after completion of the carbon composite filter study and gas generation rate study. The drums, except for two, had remained sealed, within the certified accuracy of ± 1 torr for the pressure gauge, for the study period. Two drums, BFB-109 and BFB-122, were later found to have developed leaks equal to, or in excess, of 12.5 torr/minute. These drums were considered unsatisfactory for void volume determination.

4.2.5 Void Volume Determination

Void volume measurements were made after all gas sampling had been completed. Void volume could not be measured on Drums BFB-109 and BFB-122 due to leaks that developed in the drum seals. The procedure to measure void volume was adapted from the procedure used at RFP (see Section 2.3.4). The drums were pressurized to 200 torr above atmospheric

pressure with compressed air, and then expanding the gas inside the drum into a calibrated expansion volume of 11.818 liters. The volume of a randomly selected 55-gallon drum was found to essentially be identical (219.9L) to the RFP value (220.3L). As found by RFP, the void volume measurements were reproducible. The void volumes were measured five times for each drum. The void volumes for the six sealed drums are presented in Table 10.

4.2.6 Gas Sampling Analysis

All drums were sampled for both pressure and gas composition. A 1 liter gas sampling bottle was evacuated to less than 0.02 torr before a sample was taken. The bottle was attached directly to the drum measuring/sampling line. Based on the small volume of the sampling line, the error associated with taking the sample was less than 0.5%, was considered negligible. Drums were sampled at approximately two to three week intervals, although longer intervals were established for some drums. This frequency, which deviated from the implementation plan, was determined, in part, by the availability of needed support personnel, other assignments, and the availability and time for actual analysis. As noted, some longer intervals between samplings were established for some drums when the gas analysis indicated changes in gas composition were occurring at very slow rates.

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.

A certified gas standard (Bottle No. 1), supplied by EG&G Idaho, was also analyzed. The results of the analysis are presented in Table 11. The

TABLE 10. LANL ²³⁸PU WASTE DRUM VOID VOLUMES

<u>Drum Number</u>	<u>Drum Void Volume (L)</u>	<u>Rigid Liner Void Volume (L)</u>
BFB-112	198.1 $\pm$ 14	--
BFB-113	--	168.3 $\pm$ 12.7
BFB-114	--	201.1 $\pm$ 11.7
BFB-115	210.3 $\pm$ 7	--
BFB-118	201.3 $\pm$ 7.5	--
BFB-120	215.6 $\pm$ 5.2	--

TABLE 11. LANL GAS STANDARD ANALYSIS

Analysis	Date	Mol % of Constituents			
		H ₂	N ₂	O ₂	CO ₂
1	08-01-83	2.5	66.3	18.0	10.0
2	12-29-83	2.6	65.9	18.5	11.4
3	02-21-84	3.0	65.3	18.7	11.7
4	03-27-84	3.0	65.8	18.6	11.6
5	03-30-84	2.3	66.7	18.8	11.6
6	04-10-84	3.6	65.0	18.6	11.4
7	05-04-84	2.7	65.6	19.0	11.7
8	05-30-84	1.9	66.3	19.0	11.7
9	06-14-84	2.4	66.3	19.0	11.8
10	07-10-84	2.5	66.1	19.1	11.8
11	08-03-84	2.5	64.9	18.4	11.3
$\bar{x}^a =$		2.64	65.8	18.7	11.5
$\sigma^b =$		0.44	0.58	0.33	0.51
$\sigma/\bar{x}^c =$		16.8%	0.88%	1.76%	4.46%

a. Mean or average.

b. Standard deviation.

c. Coefficient of variation.

certified standard contained 2.56% H₂, 12.0% CO₂, 18.7% O₂, and 66.7% N₂. The relative error for the LANL analysis is: H₂ = +3.12%, CO₂ = -4.17%, O₂ = +0.0%, and N₂ = -1.35%.

4.2.7 Test Procedure-Carbon Composite Filter Venting Study

The first three ²³⁸Pu drums, utilized during the first phase of the experiment, initially were left vented through the carbon filters to the atmosphere from day 1 through day 124. At that time the drums were subjected to a special purge treatment consisting of partially evacuating them to approximately 250 to 300 mm Hg (absolute), 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 was then closed for the gas-generating rate study (second phase of the experiment).

For the second set of three ²³⁸Pu drums, the procedure generally was similar, through 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, returning to atmospheric pressure, pressurizing to 300 mm Hg, and returning to atmospheric pressure. This cycle was repeated four times. A sample then was obtained from each drum, and the valves were then closed for the gas generation rate study (second phase of the experiment).

4.2.8 Test Procedure-Gas Migration Study

In the two drums associated with this experiment, both the 90-mil liner and annular space between the drum and liner initially were sealed to the atmosphere (gas generation rate study) for the first 113 days of the experiment. At that time, the annular space was then opened through the carbon composite filter. The 90-mil liner within the drum remained sealed for the hydrogen migration study.

4.3 Results

4.3.1 Carbon Composite Filter Venting Capability

Analytical results for the pressures and gas compositions of the six drums for the experiment are presented in Appendix D, Tables D-1 to D-6 and corresponding Figures D-1 to D-6. It should be noted that the tests were conducted beyond the required two 3-month test periods.

During the first phase of the experiment, these drums were vented through the carbon composite filter. The 90-mil rigid liner vent plug was not used so a pathway was provided for gases to escape. There was no detectable pressure difference between the interior of the drum and external atmospheric. The minimum pressure differential that could have been measured, based upon the pressure gauges utilized, was approximately ± 5 torr.

The carbon composite filter appears to adequately vent radiolytically produced hydrogen to maintain concentrations within safe limits (noncombustible/nonexplosive) except in very high-activity drums. Drum BFB-122, which was not fully sealed (see Section 4.2.5), contained 166.6 alpha-curies.

The maximum hazardous gas mixture measured was 5.5% H_2 and 17.4 % O_2 . It should be noted that Drum BFB-118 (68.9 alpha Ci) had one H_2 measurement of 5.0% on January 20, 1984. The gas samples taken approximately 3 weeks before and after this date indicated H_2 values of 2.3% and 2.8%, respectively. This data point of 5.0% H_2 appears to be an anomaly in the data. It was interesting to note that during the gas generation rate (Section 4.3.2) study, the two highest activity ^{238}Pu waste drums tested, BFB-118 (68.9 alpha Ci) and BFB-122 (166.6 alpha Ci), obtained potentially combustible or explosive gas mixtures within one month after the filter was closed. In fact, hydrogen levels began to build for

all experimental drums once the carbon composite filter was closed. Decreasing oxygen concentrations in several of the drums would render them noncombustible/nonexplosive within several months.

One gram of heat-source ^{238}Pu has approximately the same total alpha activity (and energy input) as the 200 gram maximum drum loading allowed for weapons-grade ^{239}Pu . For ^{239}Pu waste drums, the carbon composite filter should be adequate for maintaining internal drum pressures near or at atmosphere pressures, and maintaining hydrogen concentrations within safe limits. For ^{238}Pu waste drums, the activity level appears to be a limiting factor. This will be further discussed in Section 4.3.3.

4.3.2 Gas Migration Study

4.3.2.1 Gas Migration Evaluation. Two of the experimental drums, BFB-113 and BFB-114, were used to conduct gas migration studies on a sealed 90-mil rigid polyethylene drum liner. For both phases of the experiment, the 90-mil rigid liner was sealed. During the first phase, the drum was also sealed to evaluate gas generation (Section 4.3.3). During the second phase, the drum annular space was vented through the carbon composite filter. The analytical results for these two drums are presented in Tables D-7 to D-10.

For Drums BFB-113 and BFB-114, the data points for the hydrogen concentration inside a sealed 90-mil rigid liner, indicate that a sealed liner does inhibit the migration of gas from the liner to the drum annular space and out the carbon composite filter. Over time, the gases will migrate through the liner, but at a slower rate.

Drum BFB-113 contained approximately 30 alpha curies and Drum BFB-114 contained approximately 218 alpha curies. Therefore, most of the evaluation of the data has been made from Drum BFB-114. When the drum was vented, the pressure inside the liner and the annular space decreased to zero by the time the next sample was taken. This indicates that the leak rate through the sealed liner is larger than the total gas generation

rate. The hydrogen and carbon dioxide concentrations also decreased indicating a leak rate faster than the generation rate. One month after the annular space was vented, the concentrations of all gases inside the liner and the annular space had reached an equilibrium. The hydrogen and carbon dioxide concentrations inside the liner, at this time, are higher than in the annular space. This is expected because the source of these gases is located inside the liner. The equilibrium concentration inside the liner depends on the generation rate of the gases (amount of alpha curies), the leak rate from the sealed liner and the concentration of these gases in the annular space.

The actual leak rate of hydrogen through the sealed liner will change as the pressure differential varies between the liner and the annular space. Since the pressure inside the liner and the annular space both dropped to atmospheric by the time the first vented sample was taken, it was not possible to calculate the leak rate of hydrogen as a function of pressure.

Since the hydrogen leak rate must be greater than the hydrogen generation rate, an estimation of the generation rate was made by INEL personnel based on the ideal gas law and an argon balance method. The argon balance method assumes that the quantity of argon present initially remains constant. The moles of hydrogen can then be estimated from the following:

$$\text{Moles of hydrogen} = \text{moles of argon} \times (\text{H}_2\%/\text{Ar}\%) .$$

The calculated generation rate was 5.9 grams of hydrogen per year for the ideal gas law and 9.3 grams of hydrogen per year based on an argon balance method. This is equivalent to a hydrogen generation rate of 9.4 to 14.7 cubic feet per year or 0.5 to 0.8 cubic centimeters per min. Therefore the leak rate through the inside liner must be greater than 0.5 to 0.8 cubic centimeters per minute.

4.3.3 Gas Generation Rate Study

G-values were calculated for six of the eight experimental drums. Two drums were found to be inadequately sealed during drum pressure re-testing at the end of the experiment. The two drums could not be used for the gas generation rate study since the void volume (which is determined on sealed drums) is a component of the G-value calculation. Since part of the testing involved a study of whether the carbon filters were adequate to vent gases radiolytically generated in these drums, the drums were initially 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 purged and sealed for a portion of the experimental period. The G-values were determined for these times. Half of the six drums built up positive pressures during the unvented 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 rust (iron oxide) and water, and to temperature changes.

4.3.3.1 Gas Generation Calculations. G(gas) is defined as molecules of gas produced by each 100 eV of energy deposited in the waste substrate. G will vary with the physical state of the radioactive contaminant, its distribution in the waste, and the waste matrix. The G-value can slowly decrease with time because the contact of the radioactive contaminant with the substrate becomes less effective due to matrix depletion. Given sufficient time, the radioactive contaminant may no longer be in effective contact with the waste, and gas generation essentially ceases. Earlier studies of 3- and 9-year-old drums of high-activity ^{238}Pu waste, retrieved from storage are in agreement with this statement.⁸

The parameters used to calculate the G-values for each waste drum are listed in Table 12. The equation used to calculate the G-values for LANL ^{238}Pu drums was:

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

In this equation:

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

where:

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

and where:

P = Absolute Pressure in mm of Hg

T = Temperature in °K, ie. 273 + °C.

Void Volume = liters

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:

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

TABLE 12. LANL GAS GENERATION DATA

Drum No.	Waste Matrix	g	^{238}Pu Ci	Time of Gas Generation (Days)	Total eV	Void Volume (Liters)	Absolute Pressure (mm Hg)		Temperature °C		Mol% H ₂		MOL% Gas ^b	
							Initial	Final	Initial	Final	Initial	Final	Initial	Final
BFB-112	Plastic, leaded gloves	1.2	16.8	318	9.55×10^{25}	198.1	579	582.2	18.3	3.1	0.1	5.2	0.8	13.7
BFB-113	Leaded gloves	2.2	30.8	75	4.13×10^{25}	168.3	579 ^a	579	-5.5 ^a	4.4	1.2 ^a	2.9	3.1 ^a	6.6
BFB-114	Rags, plastic	15.6	218.4	75	2.93×10^{26}	201.1	579 ^a	984.7	-5.5 ^a	4.4	11.5 ^a	26.3	24.7 ^a	56.4
BFB-116	Leaded gloves	2.28	31.9	318	1.82×10^{26}	210.3	579	553.6	18.3	3.1	0.3	6.8	1.3	16.7
BFB-118	Rags, plastic, furnace, Al & Zr oxides	4.92	68.9	294	3.62×10^{26}	201.3	579	708.3	18.3	7.8	0.9	20.8	2.1	42.0
BFB-120	Leaded gloves	1.6	22.4	208	8.35×10^{25}	215.6	579	579	25.6	6.7	0.3	2.7	0.7	6.1

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

b. Gas includes H₂, CO₂, CO, CH₄.

Using the above method and assumptions, G-values were calculated for hydrogen and total gas for the six drums. These results are presented in Table 13.

The G(total gas) values found in six experimental drums for this study are in particularly good agreement with the earlier laboratory results reported by Kazanjian¹ for the mixed combustibles and leaded gloves (0.31-0.73 and 0.37, respectively). The work of Kazanjian involved both ²³⁹Pu and ²³⁸Pu contamination. The current results are also consistent with each other. Here, in particular, the similarity of results of the three drums containing leaded gloves is noted.

4.3.4 Summary Analysis

The results of the carbon composite filter evaluation (Section 4.3.1) and the gas migration study (Section 4.3.2) were used in an attempt to determine drum loading limits to maintain safe hydrogen concentrations at safe levels. The rate of radiolytic gas generation is primarily dependent on the quantity of alpha-emitting radionuclides and the waste matrix (G-value).

Figure 1 is a graph that relates the alpha curie loading and the G(hydrogen) value of the test drums (gas generation rate) with the measured equilibrium hydrogen concentration in drums vented with a carbon composite filter. The results for BFB-109 and BFB-122 could not be used since these drums developed leaks during the experiments and gas generation rates could not be calculated. The following assumptions were used:

1. The best fit curve drawn through the data points are assumed to be valid for point 0,0 since this represents the point in time the drums were sealed. The drums at 0,0 were assumed to contain a normal air atmosphere.

TABLE 13. LANL ^{238}Pu WASTE DRUM G-VALUES

<u>Drum Number</u>	<u>Waste Description</u>	<u>G(H₂)</u>	<u>G(Total Gas)</u>
BFB-112	Plastics and leaded gloves	0.20	0.49
BFB-113	Leaded gloves	0.14	0.28
BFB-114	Plastics, rags	0.40	0.86
BFB-116	Leaded Gloves	0.15	0.34
BFB-118	Rags, plastic, metals, Al & Zr oxides	0.27	0.54
BFB-120	Leaded Gloves	0.12	0.28

Figure 1. Hydrogen Concentration in Drum vs. Rate of Generation.

2. The relationship between the rate of hydrogen generation and equilibrium concentration of hydrogen in the drum is linear.

The data presented in Figure 1 reflects two conditions: (a) an unvented (sealed) 90-mil rigid liner in a vented drum and (b) a vented 90-mil rigid liner in a vented drum. Based on Figure 1, if a drum contains a waste form with a $G(H_2)$ value = 1.0, the drum could contain approximately 42 alpha Ci to maintain H_2 concentrations below 4% if condition (b) above is met. For condition (a) above, the limit would be reduced to approximately 30 alpha Ci. It would appear that for ^{239}Pu wastes, at a maximum loading of 200 g (14.8 alpha Ci), waste forms with a $G(H_2)$ of nearly 3.0 for a vented rigid liner or 2.0 for an unvented rigid liner would maintain H_2 concentrations at, or below, 4%.

It should be noted that the above evaluations are based on very limited data. This is especially significant for high alpha Ci loadings. It is suggested that further work be done to increase the data points on this figure.

5. REFERENCES

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VOLUME II

Appendix A

Results of Phase I, vented, tests of
Tasks 5 and 7 for the TRU Waste Sampling Program

Appendix A

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DRUM INTERNAL PRESSURE

D-29258

DRUM
PRESSURE

ATM.
PRESSURE

FIG A-1a D29258 PRESSURE
VENTED-FILTER
CODE: 001-SLUDGE

DRUM ATMOSPHERE D-29258

FIG A-1b D29258 ATMOSPHERE
VENTED-FILTER
CODE: 001-SLUDGE

DRUM ATMOSPHERE

D-29258

FIG A-1c D29258 ATMOSPHERE
VENTED-FILTER
CODE: 001-SLUDGE

DRUM INTERNAL PRESSURE

D-31528

FIG A-2a D31528 PRESSURE
VENTED-FILTER
CODE: 001-SLUDGE

DRUM ATMOSPHERE

D-31528

FIG A-2b D31528 ATMOSPHERE
VENTED-FILTER
CODE: 001-SLUDGE

DRUM ATMOSPHERE D-31528

FIG A-2c D31528 ATMOSPHERE
VENTED-FILTER
CODE: 001-SLUDGE

DRUM INTERNAL PRESSURE

D-32180

DRUM
PRESSURE

ATM.
PRESSURE

DRUM ATMOSPHERE

D-32180

FIG A-3b D32180 ATMOSPHERE

VENTED-CLIP

CODE: 001-SLUDGE

DRUM ATMOSPHERE D-32180

FIG A-3c D32180 ATMOSPHERE
VENTED-CLIP
CODE: 001-SLUDGE

DRUM INTERNAL PRESSURE

D-32186

DRUM
PRESSURE

ATM.
PRESSURE

DRUM ATMOSPHERE

D-32186

Nitrogen

1

Oxygen

2

FIG A-4b D32186 ATMOSPHERE

VENTED-GASKET

CODE: 001-SLUDGE

DRUM ATMOSPHERE D-32186

FIG A-4c D32186 ATMOSPHERE
VENTED-GASKET
CODE: 001-SLUDGE

DRUM INTERNAL PRESSURE

D-31254

DRUM
PRESSURE

ATM.
PRESSURE

FIG A-5a D31254 PRESSURE

VENTED-FILTER

CODE: 003-GREASE

DRUM ATMOSPHERE

D-31254

Nitrogen

FIG A-5b D31254 ATMOSPHERE
VENTED-FILTER
CODE: 003-GREASE

DRUM ATMOSPHERE

D-31254

Argon (1)
Carbon Dioxide (2)
Hydrogen (3)
Oxygen (4)
Other Hydrocarbons (5)

FIG A-5c D31254 ATMOSPHERE
VENTED-FILTER
CODE: 003-GREASE

DRUM ATMOSPHERE

D-31254

FIG A-5d D31254 ATMOSPHERE
VENTED-FILTER
CODE: 003-GREASE

DRUM INTERNAL PRESSURE

D-31403

DRUM
PRESSURE

ATM.
PRESSURE

FIG A-6a D31403 PRESSURE

VENTED-FILTER

CODE: 003-GREASE

DRUM ATMOSPHERE

D-31403

Nitrogen

FIG A-6b D31403 ATMOSPHERE
VENTED-FILTER
CODE: 003-SLUDGE

DRUM ATMOSPHERE

D-31403

Argon ①
Carbon Dioxide ②
Hydrogen ③
Other Hydrocarbons ④
Helium ⑤

FIG A-6c D31403 ATMOSPHERE
VENTED-FILTER
CODE: 003-GREASE

DRUM ATMOSPHERE D-31403

FIG A-6d D31403 ATMOSPHERE
VENTED-FILTER
CODE: 003-GREASE

DRUM INTERNAL PRESSURE

D-24545

DRUM
PRESSURE

ATM.
PRESSURE

FIG A-7a D24545 PRESSURE

VENTED-FILTER

CODE: 330-DRY COMB

DRUM ATMOSPHERE D-24545

FIG A-7b D24545 ATMOSPHERE
VENTED-FILTER
CODE: 330-DRY COMB

DRUM ATMOSPHERE D-24545

Argon ①
Carbon Dioxide ②
Hydrogen ③
Other Hydrocarbons ④
Isopropanol ⑤

FIG A-7c D24545 ATMOSPHERE
VENTED-FILTER
CODE: 330-DRY COMB

DRUM INTERNAL PRESSURE

D-31042

FIG A-8a D31042 PRESSURE
VENTED-FILTER
CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-31042

FIG A-8b D31042 ATMOSPHERE
VENTED-FILTER
CODE: 330-DRY COMB

DRUM ATMOSPHERE D-31042

FIG A-8c D31042 ATMOSPHERE
VENTED-FILTER
CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-31042

FIG A-8d D31042 ATMOSPHERE
VENTED-FILTER
CODE: 330-DRY COMB

DRUM INTERNAL PRESSURE

D-26048

DRUM ATMOSPHERE

D-26048

FIG A-9b D26048 ATMOSPHERE
VENTED-CLIP
CODE: 330-DRY COMB

DRUM ATMOSPHERE D-26048

DRUM ATMOSPHERE

D-26048

Freon

FIG A-9d D26048 ATMOSPHERE

VENTED-CLIP

CODE: 330-DRY COMB

DRUM INTERNAL PRESSURE

D-31703

DRUM
PRESSURE

ATM.
PRESSURE

FIG A-10a D31703 PRESSURE
VENTED-GASKET
CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-31703

Nitrogen

FIG A-10b D31703 ATMOSPHERE
VENTED-GASKET
CODE: 330-DRY COMB

DRUM ATMOSPHERE D-31703

FIG A-10c D31703 ATMOSPHERE
VENTED-GASKET
CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-31703

FIG A-10d D31703 ATMOSPHERE
VENTED-GASKET
CODE: 330-DRY COMB

DRUM INTERNAL PRESSURE

D-25634

FIG A-11a D25634 PRESSURE
VENTED-FILTER
CODE: 336-WET COMB

DRUM ATMOSPHERE

D-25634

FIG A-11b D25634 ATMOSPHERE
VENTED-FILTER
CODE: 336-WET COMB

DRUM ATMOSPHERE

D-25634

FIG A-11c D25634 ATMOSPHERE

VENTED-FILTER

CODE: 336-WET COMB

DRUM INTERNAL PRESSURE

D-31216

DRUM
PRESSURE

ATM.
PRESSURE

FIG A-12a D31216 PRESSURE

VENTED-FILTER

CODE: 336-WET COMB

DRUM ATMOSPHERE D-31216

FIG A-12b D31216 ATMOSPHERE
VENTED-FILTER
CODE: 336-WET COMB

DRUM ATMOSPHERE D-31216

Argon ①
 Carbon Dioxide ②
 Hydrogen ③
 Other Hydrocarbons ④
 Isopropanol ⑤

FIG A-12c D31216 ATMOSPHERE
 VENTED-FILTER
 CODE: 336-WET COMB

DRUM INTERNAL PRESSURE

D-25691

DRUM
PRESSURE

ATM.
PRESSURE

FIG A-13a D25691 PRESSURE
VENTED-FILTER
CODE: 337-PLASTIC

DRUM ATMOSPHERE

D-25691

FIG A-13b D25691 ATMOSPHERE

VENTED-FILTER

CODE: 337-PLASTIC

DRUM ATMOSPHERE D-25691

FIG A-13c D25691 ATMOSPHERE
VENTED-FILTER
CODE: 337-PLASTIC

DRUM ATMOSPHERE

D-25691

Helium

FIG A-13d D25691 ATMOSPHERE
VENTED-FILTER
CODE: 337-PLASTIC

DRUM INTERNAL PRESSURE

D-30688

DRUM
PRESSURE

ATM.
PRESSURE

DRUM ATMOSPHERE

D-30688

FIG A-14b D30688 ATMOSPHERE
VENTED-FILTER
CODE: 337-PLASTIC

DRUM ATMOSPHERE D-30688

FIG A-14C D30688 ATMOSPHERE
VENTED-FILTER
CODE: 337-PLASTIC

DRUM INTERNAL PRESSURE

D-29758

DRUM
PRESSURE

ATM.
PRESSURE

DRUM ATMOSPHERE

D29758

FIG A-15b D29758 ATMOSPHERE
VENTED-FILTER
CODE: 339-Pb GLOVES

DRUM ATMOSPHERE

D-29758

Argon (1)
Carbon Dioxide (2)
Hydrogen (3)
Other Hydrocarbons (4)
Isopropanol (5)

FIG A-15c D29758 ATMOSPHERE

VENTED-FILTER

CODE: 339-Pb GLOVES

DRUM ATMOSPHERE

D-29758

Helium

FIG A-15d D29758 ATMOSPHERE
VENTED-FILTER
CODE: 339-Pb GLOVES

DRUM INTERNAL PRESSURE

D-30175

DRUM
PRESSURE

ATM.
PRESSURE

FIG A-16a D30175 PRESSURE

VENTED-FILTER

CODE: 339-Pb GLOVES

A-55

DRUM ATMOSPHERE

D-30175

FIG A-16b D30175 ATMOSPHERE
VENTED-FILTER
CODE: 339-Pb GLOVES

DRUM ATMOSPHERE D-30175

FIG A-16c D30175 ATMOSPHERE
VENTED-FILTER
CODE: 339-Pb GLOVES

DRUM INTERNAL PRESSURE

D-32538

FIG A-17a D32538 PRESSURE
SEALED CONTROL DRUM
EMPTY

DRUM ATMOSPHERE

D-32538

FIG A-17b D32538 ATMOSPHERE
SEALED CONTROL DRUM
EMPTY

DRUM ATMOSPHERE

D-32538

VOLUME II

Appendix B

Results of Phase II, gas generation,
tests of Tasks 5 and 7 for the
TRU Waste Sampling Program

Appendix B

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DRUM PRESSURE

D-29258

Drum
Pressure ①

Atm.
Pressure ②

FIG B-1a D29258 PRESSURE
SEALED
CODE: 001-SLUDGE

DRUM ATMOSPHERE

D-29258

Nitrogen

FIG B-1b D29258 ATMOSPHERE
SEALED
CODE: 001-SLUDGE

B-4

DRUM ATMOSPHERE

D-29258

Oxygen

FIG B-1c D29258 ATMOSPHERE

SEALED

CODE: 001-SLUDGE

DRUM ATMOSPHERE D-29258

FIG B-1d D29258 ATMOSPHERE
SEALED
CODE: 001-SLUDGE

TRU GAS GENERATION

Phase 2 Results

Drum ID 29258

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	76.20	22.88	0.82	0.05	0.05
1	76.44	22.51	0.83	0.03	0.13
2	76.13	22.73	0.77	0.05	0.25
3	76.82	21.90	0.78	0.05	0.38
4	76.14	22.37	0.79	0.04	0.48
5	76.79	21.67	0.81	0.02	0.56
6	77.33	21.03	0.80	0.04	0.66
7	76.39	21.95	0.77	0.05	0.72
8	76.01	22.19	0.81	0.05	0.83
9	76.46	21.71	0.79	0.05	0.87
10	77.08	20.83	0.83	0.08	1.09
11	76.85	21.00	0.79	0.08	1.21
12	76.83	21.18	0.76	0.07	1.09
13	77.84	19.95	0.81	0.10	1.23

<u>Week</u>	<u>Hc</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	---	---	---	---	---	---
1	0.02	---	---	---	---	0.04
2	0.04	---	---	---	---	0.03
3	0.03	---	---	---	---	0.04
4	0.13	---	---	---	---	0.05
5	0.14	---	---	---	---	0.01
6	0.13	---	---	---	---	0.01
7	0.11	---	---	---	---	0.01
8	0.10	---	---	---	---	0.01
9	0.11	---	---	---	---	0.01
10	0.08	---	---	---	---	0.01
11	0.06	---	---	---	---	0.01
12	0.06	---	---	---	---	0.01
13	0.07	---	---	---	---	---

TABLE B-1 D29258 VOL %
SEALED
CODE: 001-SLUDGE

DRUM PRESSURE

D-31528

Drum
Pressure ①

Atm.
Pressure ②

FIG B-2a D31528 PRESSURE
SEALED
CODE: 001-SLUDGE

DRUM ATMOSPHERE D-31528

Nitrogen

FIG B-2b D31528 ATMOSPHERE
SEALED
CODE: 001-STUDGE

DRUM ATMOSPHERE D-31528

Oxygen

FIG B-2c D31528 ATMOSPHERE
SEALED
CODE: 001-SLUDGE

DRUM ATMOSPHERE

D-31528

Partial Pressure (Torr)

- Argon ①
- Carbon Dioxide ②
- Hydrogen ③
- Other Hydrocarbons ④
- Isopropanol ⑤

FIG B-2d D31528 ATMOSPHERE

SEALED

CODE: 001-SLUDGE

TRU GAS GENERATION

Phase 2 Results

Drum ID 31528

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	75.80	23.29	0.80	0.07	0.04
1	75.64	23.24	0.79	0.05	0.22
2	76.77	22.03	0.76	0.06	0.29
3	77.03	21.75	0.77	0.03	0.33
4	76.70	21.69	0.77	0.09	0.62
5	77.68	21.47	0.79	0.05	0.43
6	77.02	21.62	0.76	0.07	0.46
7	76.58	21.98	0.77	0.06	0.49
8	76.62	21.90	0.79	0.06	0.57
9	77.27	21.15	0.79	0.06	0.65
10	77.68	20.54	0.79	0.07	0.83
11	77.34	20.94	0.81	0.09	0.78
12	77.06	21.26	0.78	0.06	0.78
13	78.45	19.86	0.80	0.09	0.80

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	---	---	---	---	---	---
1	0.03	---	---	---	---	0.03
2	0.09	---	---	---	---	---
3	0.09	---	---	---	---	---
4	0.12	---	---	---	---	0.01
5	0.07	---	---	---	---	0.01
6	0.07	---	---	---	---	---
7	0.10	---	---	---	---	0.02
8	0.05	---	---	---	---	0.01
9	0.08	---	---	---	---	---
10	0.09	---	---	---	---	---
11	0.04	---	---	---	---	---
12	0.05	---	---	---	---	---
13	---	---	---	---	---	---

TABLE B-2 D31528 VOL %
SEALED
CODE: 001-SLUDGE

DRUM PRESSURE

D-32180

FIG B-3a D32180 PRESSURE

SEALED

CODE: 001-SLUDGE

DRUM ATMOSPHERE D-32180

Nitrogen

FIG B-3b D32180 ATMOSPHERE
SEALED
CODE: 001-SLUDGE

B-14

DRUM ATMOSPHERE

D-32180

Oxygen

FIG B-3c D32180 ATMOSPHERE
SEALED
CODE: 001-SLUDGE

DRUM ATMOSPHERE D-32180

FIG B-3d D32180 ATMOSPHERE
SEALED
CODE: 001-SLUDGE

B-16

TRU GAS GENERATION

Phase 2 Results

Drum ID 32180

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	76.35	22.53	0.90	0.08	0.02
1	74.61	24.00	0.85	0.06	0.18
2	74.63	23.61	0.80	0.10	0.40
3	74.41	23.73	0.81	0.09	0.54
4	72.62	25.42	0.79	0.09	0.65
5	73.32	24.65	0.80	0.08	0.77
6	72.38	25.74	0.76	0.08	0.94
7	71.87	26.05	0.76	0.10	1.02
8	71.26	26.45	0.78	0.11	1.20
9	70.78	26.94	0.76	0.10	1.28
10	70.90	26.60	0.72	0.11	1.49
11	70.01	27.37	0.75	0.11	1.66
12	68.52	28.19	0.72	0.11	2.37
13	69.36	27.57	0.75	0.16	2.06

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	0.06	---	---	---	---	0.06
1	0.30	---	---	---	---	---
2	0.03	---	---	---	---	0.43
3	0.05	---	---	---	---	0.37
4	0.06	---	---	---	---	0.32
5	0.05	---	---	---	---	0.33
6	0.05	---	---	---	---	0.05
7	0.04	---	---	---	---	0.16
8	0.04	---	---	---	---	0.16
9	0.04	---	---	---	---	0.10
10	0.06	---	---	---	---	0.07
11	0.04	---	---	---	---	0.06
12	0.02	---	---	---	---	0.07
13	0.02	---	---	---	---	0.06

TABLE B-3 D32180 VOL %
SEALED
CODE: 001-SLUDGE
B16

DRUM PRESSURE

D-32186

FIG B-4a D32186 PRESSURE
SEALED
CODE: 001-SLUDGE

DRUM ATMOSPHERE

D-32186

Nitrogen

FIG B-4b D32186 ATMOSPHERE

SEALED

CODE: 001-STUDGE

B-19

DRUM ATMOSPHERE

D-32186

FIG B-4C D32186 ATMOSPHERE
SEALED
CODE: 001-SLUDGE

DRUM ATMOSPHERE

D-32186

FIG B-4d D32186 ATMOSPHERE
SEALED
CODE: 001-SLUDGE

DRUM PRESSURE

D-31254

FIG B-5a D31254 PRESSURE
SEALED
CODE: 003-GREASE

TRU GAS GENERATION

Phase 2 Results

Drum ID 32186

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	76.21	22.56	0.87	0.08	0.02
1	76.94	21.46	0.82	0.08	0.18
2	75.39	23.15	0.76	---	0.22
3	76.31	22.14	0.81	0.06	0.30
4	76.74	21.71	0.80	0.08	0.36
5	76.97	21.46	0.82	0.07	0.40
6	76.93	21.52	0.78	0.12	0.43
7	77.02	21.44	0.78	0.09	0.48
8	76.98	21.40	0.78	0.09	0.57
9	77.18	21.18	0.79	0.09	0.58
10	78.52	19.70	0.80	0.11	0.72
11	78.42	19.75	0.81	0.11	0.82
12	78.53	19.71	0.78	0.10	0.75
13	79.05	19.08	0.81	0.14	0.86

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	0.03	---	---	---	---	0.23
1	0.04	---	---	---	---	0.48
2	---	---	---	---	---	0.48
3	0.05	---	---	---	---	0.33
4	0.07	---	---	---	---	0.24
5	0.06	---	---	---	---	0.22
6	0.08	---	---	---	---	0.14
7	0.07	---	---	---	---	0.12
8	0.06	---	---	---	---	0.12
9	0.09	---	---	---	---	0.09
10	0.09	---	---	---	---	0.06
11	0.03	---	---	---	---	0.06
12	0.04	---	---	---	---	0.09
13	---	---	---	---	---	0.06

TABLE B-4 D32186 VOL 3
SEALED
CODE: 001-SLUDGE

DRUM ATMOSPHERE

D-31254

Nitrogen

FIG B-5b D31254 ATMOSPHERE
SEALED
CODE: 003-GREASE

B-24

DRUM ATMOSPHERE

D-31254

Oxygen

FIG B-5c D31254 ATMOSPHERE

SEALED

CODE: 003-GREASE

B-25

DRUM ATMOSPHERE

D-31254

DRUM ATMOSPHERE

D-31254

FIG B-5e D31254 ATMOSPHERE

SEALED

CODE: 003-GREASE

TRU GAS GENERATION

Phase 2 Results

Drum ID 31254

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	81.65	14.97	0.93	0.05	0.43
1	94.11	0.07	1.06	0.19	1.66
2	94.34	0.02	1.02	0.03	2.14
3	92.73	0.04	1.01	0.01	0.54
4	91.50	0.04	0.99	---	3.18
5	92.61	0.07	1.05	0.01	3.64
6	91.94	0.01	0.97	---	4.01
7	91.08	0.02	0.97	---	4.41
8	90.76	0.02	1.00	---	4.92
9	90.82	0.02	0.99	---	5.48
10	88.94	0.03	0.96	---	5.89
11	88.92	0.03	0.96	0.02	6.44
12	89.04	0.02	0.94	---	6.90
13	87.79	0.03	0.95	0.05	7.31

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂CL₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	0.19	0.35	0.23	1.09	0.11	---
1	0.36	0.47	0.35	1.55	0.15	0.03
2	0.51	0.39	0.27	1.14	0.14	---
3	0.54	0.56	0.41	1.91	0.12	0.01
4	0.60	0.68	0.49	2.37	0.17	0.02
5	0.39	0.43	0.31	1.39	0.13	0.02
6	0.50	0.46	0.34	1.61	0.14	0.01
7	0.53	0.55	0.41	1.89	0.13	0.01
8	0.46	0.56	0.40	1.79	0.14	---
9	0.36	0.43	0.30	1.41	0.17	0.02
10	0.35	0.51	1.08	2.01	0.22	0.01
11	0.31	0.55	0.37	2.12	0.26	0.01
12	0.24	0.49	0.31	1.78	0.30	0.01
13	0.36	0.60	0.40	2.18	0.31	0.04

TABLE B-5 D31254 VOL %

SEALED

CODE: 003-GREASE

DRUM PRESSURE

D-31403

Drum
Pressure ①

Atm.
Pressure ②

FIG B-6a D31403 PRESSURE
SEALED
CODE: 003-GREASE

DRUM ATMOSPHERE D-31403

FIG B-6b D31403 ATMOSPHERE
SEALED
CODE: 003-GREASE

DRUM ATMOSPHERE
D-31403

Oxygen

FIG B-6c D31403 ATMOSPHERE
SEALED
CODE: 003-GREASE

DRUM ATMOSPHERE D-31403

FIG B-6e D31403 ATMOSPHERE
SEALED
CODE: 003-GREASE

Tric ①
CC14 ②
Hydrogen ③
CH2C12 ④
Freon TF ⑤

DRUM ATMOSPHERE

D-31403

Argon ①
Carbon Dioxide ②
Other Hydrocarbons ③
Isopropanol ④

FIG B-6d D31403 ATMOSPHERE

SEALED

CODE: 003-GREASE

TRU GAS GENERATION

Phase 2 Results

Drum ID 31403

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	78.52	18.43	0.77	0.07	0.22
1	90.89	4.50	0.92	0.28	0.59
2	94.64	0.03	0.90	0.04	0.99
3	94.97	0.02	0.92	0.03	1.30
4	93.85	0.02	0.90	0.03	1.46
5	93.52	0.02	0.92	0.02	1.88
6	93.95	0.03	0.89	0.02	2.22
7	93.95	0.01	0.87	0.02	2.55
8	91.16	0.05	0.88	---	2.74
9	92.80	0.03	0.88	0.02	3.10
10	92.84	0.03	0.87	0.03	3.44
11	91.40	0.01	0.88	0.03	3.71
12	91.11	0.03	0.84	0.01	4.08
13	90.36	0.02	0.87	0.03	4.30

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	0.22	0.35	0.24	1.12	0.04	0.02
1	0.33	0.47	0.33	1.58	0.11	---
2	0.57	0.55	0.39	1.71	0.09	0.04
3	0.57	0.41	0.30	1.31	0.13	0.04
4	0.56	0.59	0.44	1.95	0.16	0.04
5	0.51	0.58	0.41	1.95	0.16	0.03
6	0.42	0.46	0.33	1.55	0.11	0.02
7	0.45	0.40	0.30	1.31	0.12	0.02
8	0.61	0.66	1.54	2.15	0.20	0.01
9	0.42	0.50	0.38	1.71	0.13	0.03
10	0.42	0.37	0.27	1.51	0.20	0.02
11	0.37	0.60	0.43	2.28	0.26	0.03
12	0.44	0.60	0.40	2.22	0.23	0.04
13	0.41	0.69	0.46	2.51	0.32	0.03

TABLE B-6 D31403 VOL %
SEALED
CODE: 003-GREASE

DRUM PRESSURE

D-24545

Drum Pressure ①

Atm. Pressure ②

FIG B-7a D24545 PRESSURE

SEALED

CODE: 330-DRY COMB

DRUM ATMOSPHERE D-24545

Nitrogen

FIG B-7b D24545 ATMOSPHERE
SEALED
CODE: 330-DRY COMB

DRUM ATMOSPHERE D-24545

Oxygen

FIG B-7c D24545 ATMOSPHERE

SEALED

CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-24545

FTB B-7d D24545 ATMOSPHERE
SEALED
CODE: 330-DRY COMB

B-38

TRU GAS GENERATION

Phase 2 Results

Drum ID 24545

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	76.50	22.54	.96	.05	0
1	76.13	22.52	.91	.13	.15
2	76.19	22.23	.86	.23	.23
3	77.35	20.97	.86	.25	.30
4	76.35	21.82	.86	.30	.33
5	77.03	21.00	.88	.33	.44
6	76.87	21.28	.82	.35	.39
7	77.08	20.89	.83	.41	.51
8	76.56	21.26	.87	.44	.56
9	77.00	21.11	.84	.44	.34
10	78.04	19.81	.86	.45	.68
11	79.52	18.01	.89	.56	.78
12	77.39	20.21	.85	.59	.74
13	78.65	18.74	.88	.69	.86

<u>Week</u>	<u>Hc</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	---	---	---	---	----	----
1	0.07	---	---	---	----	0.09
2	0.08	---	---	---	----	0.18
3	0.12	---	---	---	----	0.14
4	0.12	---	---	---	----	0.22
5	0.10	---	---	---	----	0.22
6	0.08	---	---	---	----	0.21
7	0.09	---	---	---	----	0.19
8	0.08	---	---	---	----	0.23
9	0.09	---	---	---	----	0.18
10	0.10	---	---	---	----	0.06
11	0.05	---	---	---	----	0.19
12	0.04	---	---	---	----	0.18
13	0.01	---	---	---	----	0.17

TABLE E-7 D24545 VOL %
SEALED
CODE: 330-DRY COMB

DRUM PRESSURE

D-31042

FIG B-8a D31042 PRESSURE
SEALED
CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-31042

Nitrogen

FIG B-8b D31042 ATMOSPHERE

SEALED

CODE: 330-DRY COMB

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DRUM ATMOSPHERE D-31042

Oxygen

FIG B-8c D31042 ATMOSPHERE
SEALED
CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-31042

Argon ①
Carbon Dioxide ②
Hydrogen ③
Other Hydrocarbons ④
Isopropanol ⑤

FIG B-8d D31042 ATMOSPHERE

SEALED

CODE: 330-DRY COMB

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DRUM ATMOSPHERE D-31042

TRU GAS GENERATION

Phase 2 Results

Drum ID 31042

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	75.57	23.01	0.91	0.08	0.02
1	73.95	21.34	0.78	0.19	0.06
2	73.96	21.43	0.71	0.28	0.21
3	74.24	20.21	0.72	0.32	0.23
4	75.90	20.68	0.79	0.34	0.31
5	73.95	19.20	0.71	0.44	0.40
6	74.99	19.03	0.69	0.52	0.48
7	74.60	18.63	0.68	0.56	0.56
8	74.25	18.12	0.69	0.59	0.59
9	75.40	17.66	0.69	0.67	0.64
10	76.31	16.96	0.69	0.78	0.81
11	76.33	16.69	0.70	0.86	0.90
12	76.55	16.08	0.66	0.95	0.91
13	76.26	15.24	0.69	1.12	0.99

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	0.05	0.16	0.14	---	---	0.06
1	0.22	1.10	1.83	0.02	---	0.51
2	0.16	1.21	1.98	0.01	0.01	0.04
3	0.58	1.19	1.92	0.01	0.02	0.56
4	0.29	0.56	0.94	---	---	0.19
5	0.81	1.38	2.26	0.02	---	0.83
6	0.39	1.22	1.94	0.02	---	0.72
7	0.71	1.34	2.13	0.03	---	0.76
8	0.89	1.55	2.40	0.05	---	0.87
9	0.72	1.32	2.10	0.03	---	0.77
10	0.67	1.40	1.79	---	---	0.59
11	0.46	1.42	1.96	---	---	0.68
12	0.42	1.53	2.09	---	---	0.81
13	0.46	1.82	2.44	0.03	---	0.95

TABLE B-8 D31042 VOL 3
SEALED
CODE: 330-DRY COMB

DRUM PRESSURE

D-26048

FIG B-9a D26048 PRESSURE
SEALED
CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-26048

Nitrogen

FIG B-9b D26048 ATMOSPHERE

SEALED

CODE: 330-DRY COMB

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DRUM ATMOSPHERE

D-26048

Oxygen

Partial Pressure (Torr)

FIG B-9C D26048 ATMOSPHERE
SEALED
CODE: 330-DRY COMB

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DRUM ATMOSPHERE

D-26048

FIG B-9d D26048 ATMOSPHERE

SEALED

CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-26048

FIG B-9e D26048 ATMOSPHERE
SEALED
CODE: 330-DRY COMB

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TRU GAS GENERATION

Phase 2 Results

Drum ID 26048

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	73.58	20.60	.93	.30	.05
1	72.52	20.83	.83	.34	.13
2	71.99	20.80	.77	.35	.21
3	72.47	20.31	.88	.28	.24
4	74.16	20.83	.83	.28	.21
5	73.95	18.96	.83	.42	.35
6	74.34	19.29	.78	.43	.40
7	74.17	19.11	.77	.46	.43
8	73.94	19.49	.80	.48	.45
9	74.37	18.92	.80	.53	.52
10	74.14	17.72	.80	.56	.46
11	72.98	17.83	.78	.60	.49
12	73.54	16.88	.76	.61	.55
13	72.96	16.99	.79	.71	.69

<u>Week</u>	<u>Hc</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	.19	---	.03	---	.82	3.50
1	.33	---	.09	---	2.20	2.73
2	.33	---	.11	---	3.03	2.41
3	.33	---	.14	.09	3.56	1.70
4	.17	---	.09	.06	2.58	.77
5	.31	---	.13	---	4.09	.96
6	.23	---	.15	---	3.67	.71
7	.20	---	.15	.03	4.04	.64
8	.21	---	.15	---	3.97	.51
9	.24	---	.15	---	3.88	.59
10	.22	---	.13	---	5.60	.37
11	.18	---	.15	---	6.52	.47
12	.18	---	.14	---	6.86	.48
13	.09	---	.15	---	6.99	.63

TABLE B-9 D26048 VOL %
SEALED
CODE: 330-DRY COMB

DRUM PRESSURE D-31703

Drum
Pressure ①
Atm.
Pressure ②

FIG B-10a D31703 PRESSURE
SEALED
CODE: 330-DRY COMB

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DRUM ATMOSPHERE

D-31703

Nitrogen

FIG B-10b D31703 ATMOSPHERE

SEALED

CODE: 330-DRY COMB

B-53

DRUM ATMOSPHERE

D-31703

Oxygen

FIG B-10C D31703 ATMOSPHERE
SEALED
CODE: 330-DRY COMB

DRUM ATMOSPHERE

D-31703

Carbon
Dioxide

FIG B-10d D31703 ATMOSPHERE
SEALED
CODE: 330-DRY COMB

DRUM ATMOSPHERE D-31703

TRU GAS GENERATION

Phase 2 Results

Drum ID 31703

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	76.40	21.53	0.90	0.50	0.05
1	76.52	18.80	0.86	2.27	0.22
2	76.88	17.21	0.78	2.89	0.29
3	77.60	16.14	0.80	3.35	0.33
4	77.93	15.15	0.80	3.87	0.40
5	78.89	13.35	0.82	4.37	0.39
6	79.35	13.16	0.79	4.56	0.42
7	79.15	12.52	0.80	5.22	0.52
8	79.43	11.71	0.83	5.51	0.54
9	80.25	10.72	0.81	5.79	0.51
10	82.09	9.57	0.87	5.58	0.47
11	81.58	9.15	0.85	6.45	0.59
12	81.54	9.64	0.83	6.19	0.38
13	81.25	9.25	0.84	6.87	0.37

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>Unident. Organic</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	0.17	---	---	---	---	0.45
1	---	---	---	0.56	---	0.77
2	---	---	---	1.00	---	0.95
3	---	---	---	1.07	---	0.71
4	---	---	---	1.18	---	0.67
5	---	---	---	1.41	---	0.77
6	---	---	---	1.22	---	0.50
7	---	---	---	1.33	---	0.46
8	---	---	---	1.50	---	0.48
9	---	---	---	1.47	---	0.45
10	---	---	---	1.13	---	0.29
11	---	---	---	1.05	---	0.33
12	---	---	---	1.10	---	0.32
13	---	---	---	1.15	---	0.28

TABLE B-10 D31703 VOL %
SEALED
CODE: 330-DRY COMB

DRUM PRESSURE

D-25634

Drum
Pressure ①

Atm.
Pressure ②

FIG B-11a D25634 PRESSURE
SEALED
CODE: 336-WET COMB

DRUM ATMOSPHERE

D-25634

Nitrogen

FIG B-11b D25634 ATMOSPHERE
SEALED
CODE: 336-WET COMB

DRUM ATMOSPHERE D-25634

Oxygen

Partial Pressure (Torr)

FIG B-11c D25634 ATMOSPHERE
SEALED
CODE: 336-WET COMB

DRUM ATMOSPHERE

D-25634

Argon (1)
Carbon Dioxide (2)
Hydrogen (3)
Other Hydrocarbons (4)
Isopropanol (5)

FIG B-11d D25634 ATMOSPHERE
SEALED
CODE: 336-WET COMB

TRU GAS GENERATION

Phase 2 Results

Drum ID 25634

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	---	---	---	---	---
1	76.58	22.35	0.94	0.10	0.02
2	76.49	22.39	0.86	0.12	0.10
3	76.65	22.12	0.87	0.10	0.08
4	79.63	17.58	0.91	1.28	0.39
5	77.46	21.26	0.88	0.13	0.11
6	77.18	21.58	0.85	0.12	0.12
7	77.47	21.24	0.86	0.14	0.14
8	77.02	21.43	0.88	0.13	0.39
9	77.11	21.49	0.87	0.15	0.22
10	78.41	19.96	0.90	0.16	0.23
11	78.51	20.17	0.88	0.17	0.18
12	78.71	19.86	0.85	0.17	0.28
13	79.57	18.91	0.88	0.18	0.26

<u>Week</u>	<u>Hc</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	---	---	---	---	---	---
1	0.01	---	---	---	---	---
2	0.03	---	---	---	---	0.01
3	0.16	---	---	---	---	0.02
4	0.16	---	---	---	---	0.05
5	0.14	---	---	---	---	0.02
6	0.14	---	---	---	---	0.01
7	0.13	---	---	---	---	0.02
8	0.14	---	---	---	---	0.01
9	0.15	---	---	---	---	0.01
10	0.14	---	---	---	---	---
11	0.08	---	---	---	---	0.01
12	0.12	---	---	---	---	0.01
13	0.16	---	---	---	---	0.04

TABLE B-11 D25634 VOL %
SEALED
CODE: 336-WET COMB

DRUM PRESSURE

D-31216

Drum
Pressure ①

Atm.
Pressure ②

FIG B-12a D31216 PRESSURE
SEALED
CODE: 336-WET COMB

DRUM ATMOSPHERE D-31216

Nitrogen

FIG B-12b D31216 ATMOSPHERE
SEALED
CODE: 336-WET COMB

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DRUM ATMOSPHERE

D-31216

Oxygen

FIG B-12c D31216 ATMOSPHERE

SEALED

CODE: 336-WET COMB

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DRUM ATMOSPHERE D-31216

FIG B-12d D31216 ATMOSPHERE
SEALED
CODE: 336-WET COMB

Argon ①
Carbon Dioxide ②
Hydrogen ③
Other Hydrocarbons ④
Isopropanol ⑤

TRU GAS GENERATION

Phase 2 Results

Drum ID 31216

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	75.94	22.95	0.93	0.08	--
1	76.48	22.33	0.98	0.12	--
2	76.02	22.46	0.89	0.19	0.08
3	77.34	21.05	0.95	0.16	0.12
4	76.79	21.69	0.92	0.21	0.09
5	77.06	21.22	0.94	0.22	0.15
6	78.23	20.07	0.93	0.22	0.24
7	77.15	21.10	0.93	0.22	0.26
8	77.19	21.00	0.93	0.24	0.28
9	77.52	20.70	0.93	0.24	0.29
10	78.70	19.43	0.94	0.25	0.39
11	78.04	20.34	0.94	0.22	0.22
12	78.88	19.39	0.92	0.31	0.13
13	79.11	19.03	0.95	0.28	0.35

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	0.04	---	---	---	---	0.06
1	0.06	---	---	---	---	0.03
2	0.06	---	---	---	---	0.30
3	0.16	---	0.73	---	---	0.22
4	0.17	---	---	---	---	0.13
5	0.16	---	---	---	---	0.25
6	0.13	---	---	---	---	0.18
7	0.20	---	---	---	---	0.14
8	0.17	---	---	---	---	0.19
9	0.15	---	---	---	---	0.17
10	0.17	---	---	---	---	0.12
11	0.11	---	---	---	---	0.13
12	0.14	---	---	---	---	0.23
13	0.12	---	---	---	---	0.16

TABLE B-12 D31216 VOL 3
SEALED
CODE: 336-WET COMB

DRUM PRESSURE

D-25691

Drum
Pressure ①

Atm.
Pressure ②

FIG B-13a D25691 PRESSURE
SEALED
CODE: 337-ELASTIC

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DRUM ATMOSPHERE

D-25691

Nitrogen

FIG B-13b D25691 ATMOSPHERE
SEALED
CODE: 337-PLASTIC

DRUM ATMOSPHERE

D-25691

Oxygen

FIG B-13c D25691 ATMOSPHERE

SEALED

CODE: 337-PLASTIC

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DRUM ATMOSPHERE

D-25691

FIG B-13d D25691 ATMOSPHERE
SEALED
CODE: 337-PLASTIC

TRU GAS GENERATION

Phase 2 Results

Drum ID 25691

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	76.04	22.84	0.93	0.14	0.05
1	77.47	20.45	0.96	0.77	0.24
2	78.15	19.52	0.88	1.05	0.27
3	78.96	18.39	0.91	1.19	0.35
4	77.03	21.65	0.88	0.13	0.11
5	80.41	16.57	0.94	1.42	0.46
6	80.54	16.54	0.91	1.38	0.45
7	81.14	15.76	0.91	1.49	0.52
8	81.77	15.01	0.95	1.55	0.58
9	82.71	13.91	0.93	1.58	0.68
10	83.74	12.79	0.96	1.51	0.88
11	83.74	12.63	0.95	1.65	0.91
12	84.36	12.33	0.92	1.60	0.66
13	84.70	11.34	0.98	2.01	0.85

<u>Week</u>	<u>Hc</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	---	---	---	---	---	---
1	0.06	---	---	---	---	0.05
2	0.08	---	---	---	---	0.05
3	0.14	---	---	---	---	0.06
4	0.18	---	---	---	---	0.02
5	0.13	---	---	---	---	0.07
6	0.11	---	---	---	---	0.07
7	0.11	---	---	---	---	0.07
8	0.07	---	---	---	---	0.07
9	0.15	---	---	---	---	0.04
10	0.12	---	---	---	---	---
11	0.09	---	---	---	---	0.03
12	0.09	---	---	---	---	0.04
13	0.08	---	---	---	---	0.04

TABLE B-13 D25691 VOL %
SEALED
CODE: 337-PLASTIC

DRUM PRESSURE

D-30688

Drum
Pressure ①

Atm.
Pressure ②

FIB 5-14a D30688 PRESSURE
SEALED
CODE: 337-PLASTIC

DRUM ATMOSPHERE

D-30688

FIG B-14b D30688 ATMOSPHERE
SEALED
CODE: 337-PLASTIC

DRUM ATMOSPHERE

D-30688

Oxygen

FIG B-14c D30688 ATMOSPHERE
SEALED
CODE: 337-PLASTIC

DRUM ATMOSPHERE

D-30688

FIG B-14d D30688 ATMOSPHERE
SEALED
CODE: 337-PLASTIC

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TRU GAS GENERATION
Phase 2 Results

Drum ID 30688

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	75.70	23.27	0.93	0.08	0.02
1	77.20	21.46	0.97	0.22	0.11
2	75.30	23.00	0.89	0.32	0.23
3	76.51	21.81	0.90	0.36	0.26
4	76.78	21.30	0.92	0.45	0.33
5	77.18	20.88	0.93	0.48	0.34
6	78.63	19.70	0.89	0.53	0.12
7	76.90	21.10	0.91	0.56	0.41
8	77.79	20.02	0.93	0.61	0.47
9	77.12	20.67	0.91	0.64	0.50
10	78.32	19.48	0.93	0.68	0.43
11	77.97	19.69	0.93	0.73	0.52
12	78.27	19.34	0.89	0.78	0.58
13	78.68	18.73	0.94	0.89	0.64

<u>Week</u>	<u>HC</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	---	---	---	---	---	---
1	0.04	---	---	---	---	---
2	0.14	---	---	---	---	---
3	0.13	---	---	---	---	0.02
4	0.18	---	---	---	---	0.03
5	0.15	---	---	---	---	0.04
6	0.11	---	---	---	---	0.04
7	0.08	---	---	---	---	0.02
8	0.15	---	---	---	---	0.04
9	0.13	---	---	---	---	0.03
10	0.13	---	---	---	---	0.03
11	0.13	---	---	---	---	0.03
12	0.11	---	---	---	---	0.03
13	0.10	---	---	---	---	0.02

TABLE B-14 D30688 VOL %
SEALED
CODE: 337-PLASTIC

DRUM PRESSURE

D-29758

Drum
Pressure ①
Atm.
Pressure ②

FIG B-15a D29758 PRESSURE
SEALED
CODE: 339-Pb RUBBER

DRUM ATMOSPHERE

D-29758

Nitrogen

FIG B-15b D29758 ATMOSPHERE

SEALED

CODE: 339-Pb RUBBER

DRUM ATMOSPHERE

D-29758

Oxygen

FIG B-15c D29758 ATMOSPHERE
SEALED
CODE: 339-Pb RUBBER

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DRUM ATMOSPHERE

D-29758

- Argon ①
- Carbon Dioxide ②
- Hydrogen ③
- Other Hydrocarbons ④
- Isopropanol ⑤

FIG B-15d D29758 ATMOSPHERE
SEALED
CODE: 339 PD RUBBER

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TRU GAS GENERATION

Phase 2 Results

Drum ID 29758

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	76.14	22.94	0.81	0.10	0.01
1	76.56	22.36	0.83	0.12	---
2	75.44	23.36	0.76	0.18	0.02
3	76.73	22.07	0.76	0.22	0.06
4	76.42	22.13	0.77	0.24	0.03
5	77.42	21.12	0.77	0.27	0.04
6	77.09	21.48	0.76	0.28	0.02
7	76.91	21.88	0.77	0.29	---
8	76.84	21.72	0.82	0.25	0.01
9	77.19	21.31	0.77	0.32	0.01
10	77.99	20.63	0.78	0.29	0.04
11	77.82	20.76	0.79	0.36	---
12	78.09	20.48	0.77	0.40	0.04
13	78.63	19.88	0.78	0.45	0.02

<u>Week</u>	<u>Hc</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	---	---	---	---	---	---
1	0.05	---	---	---	---	0.08
2	0.09	---	---	---	---	0.15
3	0.12	---	---	---	---	0.04
4	0.16	---	---	---	---	0.25
5	0.15	---	---	---	---	0.23
6	0.14	---	---	---	---	0.23
7	0.10	---	---	---	---	0.05
8	0.12	---	---	---	---	0.24
9	0.18	---	---	---	---	0.22
10	0.12	---	---	---	---	0.15
11	0.08	---	---	---	---	0.19
12	0.03	---	---	---	---	0.19
13	0.05	---	---	---	---	0.19

TABLE B-15 D29758 VOL %

SEALED

CODE: 339-Pb RUBBER

DRUM PRESSURE

D-30175

Drum
Pressure ①

Atm.
Pressure ②

FIG B-16a D30175 PRESSURE

SEALED

CODE: 339-Pb RUBBER

B-83

DRUM ATMOSPHERE

D-30175

Nitrogen

FIG B-16b D30175 ATMOSPHERE
SEALED
CODE: 339-Pb RUBBER

DRUM ATMOSPHERE

D-30175

Oxygen

FIG B-16c D30175 ATMOSPHERE

SEALED

CODE: 339-Pb RUBBER

DRUM ATMOSPHERE D-30175

TRU GAS GENERATION

Phase 2 Results

Drum ID 30175

Gas Analysis (Vol %)

<u>Week</u>	<u>N₂</u>	<u>O₂</u>	<u>Ar</u>	<u>CO₂</u>	<u>H₂</u>
0	75.74	23.12	0.94	0.12	0.08
1	76.62	21.45	1.00	0.32	0.45
2	75.99	21.50	0.90	0.56	0.80
3	76.97	19.93	0.93	0.73	1.17
4	78.60	18.03	0.95	0.79	1.27
5	77.73	17.89	0.97	1.11	1.87
6	77.99	17.15	0.92	1.29	2.26
7	78.32	16.44	0.93	1.43	2.56
8	78.28	16.03	0.95	1.58	2.80
9	79.08	14.47	0.97	1.78	3.26
10	79.93	13.29	0.98	1.77	3.62
11	79.44	13.03	0.95	2.08	4.12
12	80.12	11.93	0.94	2.38	4.26
13	80.21	13.07	1.02	2.26	3.04

<u>Week</u>	<u>Hc</u>	<u>Tric</u>	<u>CH₂Cl₂</u>	<u>CCl₄</u>	<u>Freon</u>	<u>C₃H₇OH</u>
0	---	---	---	---	---	---
1	0.14	---	---	---	---	0.02
2	0.20	---	---	---	---	0.05
3	0.23	---	---	---	---	0.04
4	0.32	---	---	---	---	0.04
5	0.38	---	---	---	---	0.05
6	0.34	---	---	---	---	0.05
7	0.28	---	---	---	---	0.04
8	0.33	---	---	---	---	0.03
9	0.39	---	---	---	---	0.05
10	0.38	---	---	---	---	0.03
11	0.35	---	---	---	---	0.03
12	0.34	---	---	---	---	0.04
13	0.37	---	---	---	---	0.03

TABLE B-16 D30175 VOL %
SEALED
CODE: 339-Pb RUBBER

DRUM PRESSURE

D-32538

Drum
Pressure ①

Atm.
Pressure ②

FIG B-17a D32538 PRESSURE
SEALED CONTROL DRUM
EMPTY

DRUM ATMOSPHERE

D-32538

Nitrogen

FIG B-17b D32538 ATMOSPHERE
SEALED CONTROL DRUM
EMPTY

DRUM ATMOSPHERE

D-32538

B-90

VOLUME II

Appendix C

Results of Task 8 for the
TRU Waste Sampling Program

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743-11838	C-11
744-2376	C-15
90-00042	C-17
90-00039	C-20
22-00224	C-23
D-32538	C-26

DRUM INTERNAL PRESSURE FIRST FOUR DRUMS

Figure 1

DRUM INTERNAL PRESSURE

SECOND FOUR DRUMS

Figure 2

DRUM INTERNAL ATMOSPHERE 741-12858

Figure 3a

DRUM INTERNAL ATMOSPHERE 741-12858

Figure 3b

DRUM INTERNAL ATMOSPHERE

741-12858

Figure 3c

C-7

DRUM INTERNAL ATMOSPHERE

742-21396

Figure 4a

DRUM INTERNAL ATMOSPHERE

742-21396

Figure 4b

DRUM INTERNAL ATMOSPHERE

742-21396

Figure 4c

Carbon
Dioxide
Hydrogen
Isopropanol
CCl4

DRUM INTERNAL ATMOSPHERE

743-11838

Nitrogen

Figure 5a

DRUM INTERNAL ATMOSPHERE

743-11838

Oxygen

Figure 5b

DRUM INTERNAL ATMOSPHERE

743-11838

Figure 5c

DRUM INTERNAL ATMOSPHERE

743-11838

Figure 5d

C-14

DRUM INTERNAL ATMOSPHERE

744-2376

Nitrogen

Oxygen

Figure 6a

DRUM INTERNAL ATMOSPHERE 744-2376

Figure 6b

DRUM INTERNAL ATMOSPHERE

90-00042

Figure 7a

DRUM INTERNAL ATMOSPHERE

90-00042

Figure 7b

DRUM INTERNAL ATMOSPHERE

90-00042

Figure 7c

C-19

DRUM INTERNAL ATMOSPHERE
90-00039

Nitrogen

Figure 8a

DRUM INTERNAL ATMOSPHERE

90-00039

Figure 8b

DRUM INTERNAL ATMOSPHERE

90-00039

Figure 8c

DRUM INTERNAL ATMOSPHERE

22-00224

Figure 9a

DRUM INTERNAL ATMOSPHERE

22-00224

Nitrogen

Oxygen

Figure 9a

DRUM INTERNAL ATMOSPHERE

22-00224

Figure 9b

DRUM INTERNAL ATMOSPHERE

D-32538

Nitrogen

Oxygen

Figure 10a

DRUM INTERNAL ATMOSPHERE

D-32538

Figure 10b

VOLUME II

Appendix D

Results of Task 10 for the
TRU Waste Sampling Program

Appendix D

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TABLE D-1

DRUM BFB-109. GENERATED 01-27-84. CONTAINS 3.0g (42 α Cl) ^{238}Pu .

Sampled	Gauge Pressure (mm Hg)	Temperature (°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 D-1

TABLE D-2

DRUM BFB-112. GENERATED 07-28-83. CONTAINS 1.2g (16 α Cl) ^{238}Pu .

Sampled	Gauge Pressure (mm Hg)	Temperature (°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

D-6

Figure D-2

TABLE D-3

DRUM BFB-116. GENERATED 10-27-83. CONTAINS 2.28g (31.9 α Ci) ^{238}Pu .

Sampled	Gauge Pressure (mm Hg)	Temperature (°F)	H ₂	O ₂	Mol% of Gaseous Components					
					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 D-3

TABLE D-4

DRUM BFB-118. GENERATED 10-27-83. CONTAINS 4.92g (68.9 α Cl) ^{238}Pu .

Sampled	Gauge Pressure (mm Hg)	Temperature (°F)	H ₂	O ₂	Mol% of Gaseous Components						He	Ar
					CO	CO ₂	CH ₄	N ₂				
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 D-4

TABLE D-5

DRUM BFB-120. GENERATED 01-03-84. CONTAINS 1.6g (22.4 αCi) ²³⁸Pu.

Sampled	Gauge Pressure (mm Hg)	Temperature (°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	0.3	20.6	0.4	0.5	<0.1	77.3	<0.1	0.9
05-04-84	0.0	73	0.2	20.6	0.5	0.4	<0.1	77.4	<0.1	0.9
05-30-84	0.0	91.5	<0.1	19.7	<0.1	0.4	<0.1	79.0	<0.1	0.9
06-14-84	0.0	85	0.5	19.9	0.1	0.6	<0.1	78.0	<0.1	0.9
07-06-84	0.0	92.5	0.3	20.8	0.2	0.5	<0.1	77.3	<0.1	0.9
07-27-84	0.0	84	0.1	20.8	0.2	0.4	<0.1	77.6	<0.1	0.9
08-10-84**	0.0	78	0.3	20.7	<0.1	0.4	<0.1	77.6	<0.1	0.9
08-24-84	Positive; <25.9	68	0.6	20.2	0.2	0.5	<0.1	77.2	<0.1	1.2
03-06-85	0.0	44	2.7	16.2	0.9	2.5	<0.1	76.8	<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-120

Gen. 1/3/84, 1.6g Pu238

Figure D-5

TABLE D-6

DRUM BFB-122. GENERATED 01-12-84. CONTAINS 11.9g (166.6 α Cl) ^{238}Pu .

Sampled	Gauge Pressure (mm Hg)	Temperature (°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	0.9
05-04-84	0.0	73	5.3	17.8	3.7	4.4	0.2	67.8	<0.1	0.8
05-25-84	0.0	91.5	4.9	16.8	3.0	4.8	0.2	69.5	<0.1	0.8
06-14-84	0.0	85	5.5	17.3	3.8	4.7	0.2	67.7	<0.1	0.8
07-06-84	0.0	92.5	5.2	17.6	3.3	4.9	0.2	68.0	<0.1	0.8
07-27-84	0.0	84	5.5	17.4	3.7	4.6	0.2	67.8	<0.1	0.8
08-10-84**	0.0	78	2.1	20.3	0.6	0.9	<0.1	75.1	<0.1	0.9
08-24-84	-25.4	68	4.2	18.2	2.0	2.2	0.1	72.1	<0.1	1.1
11-13-84	-25.4	53	9.7	14.4	4.6	7.5	0.3	62.7	<0.1	0.8
12-20-84	-25.4	35	10.9	13.9	4.1	8.5	0.4	61.5	<0.1	0.7
01-29-85	-25.4	46	10.4	14.0	4.4	8.2	0.4	61.9	<0.1	0.7
02-22-85	-25.4	37.5	8.7	14.5	4.9	7.1	0.3	63.7	<0.1	0.8

* Drum prepared; vented to atmosphere through carbon filter.

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

D-13

Drum BFB-122

Gen. 1/12/84, 11.9g Pu238

D-14

Figure D-6

TABLE D-7

DRUM BFB-113(Annular Space). GENERATED 09-26-83. CONTAINS 2.2g (30.8 gCi) ²³⁸Pu.

Sampled	Gauge Pressure (mm Hg)	Temperature (°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	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

D-16

Figure D-7

TABLE D-8

DRUM BFB-113(Inside Liner). GENERATED 09-26-83. CONTAINS 2.2g (30.8 μ Ci) ^{238}Pu .

Sampled	Gauge Pressure (mm Hg)	Temperature ($^{\circ}\text{F}$)	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	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

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Figure D-8

TABLE D-9

DRUM BFB-114(Annular Space). GENERATED 09-25-83. CONTAINS 15.6g (218.4 α C1) 238Pa.

Sampled	Gauge Pressure (mm Hg)	Temperature (°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.	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, 15.6g Pu238

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Figure D-9

TABLE D-10

DRUM BFB-114(Inside Liner). GENERATED 09-26-83. CONTAINS 15.6g (218.4 μ Ci) ^{238}Pu .

Sampled	Gauge Pressure (mm Hg)	Temperature (°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. 9/23/83, 15.6g Pu238

Figure D-10