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Gas Analyses from Headspace of Plutonium-Bearing Materials Containers

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Abstract

The Savannah River National Laboratory (SRNL) 3013 destructive examination program performs surveillances on 3013 containers originating from multiple sites across the DOE complex. The bases for the packaging, storage, and surveillance activities are derived from the Department of Energy's 3013 Standard (DOE-STD-3013-2004). During destructive examination, headspace gas samples are obtained from the 3013 inner container and the annulus between the outer and inner containers. To characterize gas species, the samples are analyzed by gas chromatography (GC), direct-inlet mass spectrometry (DIMS), and Fourier-transform infrared spectroscopy (FTIR). The GC results, as well as other parameters, are utilized as input into the gas evaluation software tool (GEST) program for computation of pre-puncture gas compositions and pressures.

Over 30 containers from the Hanford Site and the Rocky Flats Environmental Technology Site (RFETS) have been examined in the first three years of the surveillance program. Several containers were shown to have appreciable hydrogen content (some greater than 30 mol %), yet little or no oxygen was detected in any of the containers, including those exhibiting high hydrogen concentrations. Characteristics including moisture content, surface area, and material composition, along with the headspace gas composition, are utilized in an attempt to explain the chemical behavior of the packaged materials.

Introduction

THE 3013 DESTRUCTIVE EXAMINATION PROGRAM - HEADSPACE GAS ANALYSIS

Plutonium-bearing materials packaged at the Savannah River Site (SRS) and other sites across the DOE complex, including Rocky Flats Environmental Technology Site (RFETS), Hanford, Los Alamos National Laboratory (LANL), and Lawrence Livermore National Laboratory (LLNL), are presently being stored at SRS in 3013 containers. The Department of Energy (DOE) 3013 Standard (DOE-STD-3013-2004) requires periodic surveillance of 3013 containers to verify container integrity during storage over their design life of 50 years.[1] As part of the surveillance requirements, a portion of the stored inventory is destructively examined under the 3013 surveillance program. Container selection is based upon random statistical sampling and engineering judgment provided by the Materials Identification and Surveillance (MIS) working group, a counsel of inter-laboratory experts in the field of packaging, corrosion, and radiolysis. More than 30 containers from Hanford and RFETS have been destructively examined at SRS during the first three years of the program.

Sampling and analysis of headspace gas from within 3013 containers is performed as part of the destructive examination of selected samples within the surveillance program. The potential presence of high container pressure in combination with the presence of hydrogen and oxygen within the container system is a primary concern because of the potential for container over-pressurization. A secondary concern is combustion upon opening the container.[2] During surveillance, headspace gas is extracted from the sealed 3013 container by using a specially designed can puncture device (CPD). Analysis of the gas is subsequently performed by gas chromatography (GC), Fourier-transform infrared spectroscopy (FTIR), and direct-inlet mass spectrometry (DIMS). Gas chromatography results and other input parameters are entered into the Gas Evaluation Software Tool (GEST), a program to calculate pre-puncture gas composition and pressure conditions of the inner container.[3] This suite of data provides valuable information regarding the behavior of 3013 containers during storage and is summarized in the paper.

GAS GENERATION IN HANFORD AND RFETS CONTAINERS PACKAGED TO THE 3013 STANDARD

DOE-STD-3013-2004 requires containers of plutonium-bearing oxide to be welded and leak tight with a moisture content of less than 0.5 wt %. Based on the Standard requirements, it is possible that up to 25 g of water could be present, distributed throughout the container based on a maximum allowed material content of 5 kg. Although this is a small amount of water compared to the total amount of packaged material, radiolytic decomposition of any adsorbed moisture and waters of hydration, could have a significant effect on the hydrogen and oxygen generation rates and the pressure of hydrogen and oxygen ultimately developed in the sealed container.[4-7]

The decomposition of water by radiolysis may produce hydrogen and oxygen, and ideally should produce them in stoichiometric quantities;[1] however, stoichiometric hydrogen and

oxygen production was rarely observed in sealed container studies when the moisture content of the packaged material was less than 0.5 wt %.[8] Rather, oxygen, if seen at all, was observed in sub-stoichiometric amounts. This observation may be explained by the formation of a superstoichiometric plutonium oxide or corrosion reactions that produce metal oxides or hydroxides and release hydrogen.[1] The decomposition of water can occur by solid-state chemical reaction on the plutonium oxide surface to produce hydrogen, yet retain oxygen as PuO_{2+x}.[1,9] However, oxygen and hydrogen can also be removed from the headspace through recombination to form water. Recombination can be catalyzed by PuO_2,[10] other impurity oxides,[1,11] or even the stainless steel container itself.[11] Radiolytic recombination from alpha and gamma radiation has also been reported.[12]

Studies were performed by the Materials Identification and Surveillance (MIS) program on representative materials previously stabilized at Hanford and RFETS and packaged in small containers designed to mimic the behavior of packaged 3013 containers. Because of the material origin and the fact that no additional water was intentionally added by exposure to high humidity environments before packaging, these experiments are considered to be most closely related to material currently stored under the 3013 Standard.[8] The material water content in these small-scale MIS packages was estimated to be less than 0.2 wt %. The experiments demonstrated that over a few years, oxygen present in test containers sealed in air was consumed, and little hydrogen was produced. These trends have also been observed for aged Hanford containers that were sealed for up to 18 years.[13,14]

In a separate set of experiments, thirty-nine MIS small-scale containers of plutonium-bearing oxide material representative of the inventory of materials at RFETS, Hanford, and LANL were targeted at 0.5 wt % moisture to create a bounding study for the effects of moisture. Nearly all of the MIS containers that produced hydrogen (as high as ~73 mol %) did not produce much oxygen, or, if initially produced, the oxygen was later consumed, and therefore the gas content seldom reached the flammability region.[8] Two containers from this study were observed to reach flammable mixtures of hydrogen and oxygen. Sample 011589A had a hydrogen content of 46.0 mol % at a maximum of 14.0 mol % oxygen and 1000089 had a hydrogen content of 12.0 mol % at a maximum of 7.5 mol % oxygen. Sample 011589A reached approximately a four times greater maximum pressure increase and maximum partial pressure of oxygen and hydrogen than sample 1000089.[8]

Following this study, a database survey was performed for the items in the 3013 storage inventory that had the highest probability of being similar to the composition of sample 011589A. The evaluation, based on variables such as binning classification, prompt gamma analyses, moisture content, and process knowledge, was performed on the Integrated Safety Program (ISP) database and identified 15 items originating from Hanford, RFETS, and SRS that are of specific interest.[15] Item R602498 has been analyzed and the results are communicated in this article.

SRNL has examined the headspace gas recovered from more than 30 Hanford and RFETS containers that had been sealed for between 4 and 6 years. Physical properties,

such as moisture content, as measured by thermogravimetric analysis – mass spectrometry (TGA-MS), surface area as measured by the Brunauer-Emmett-Teller (BET) method, and chemical composition are compared for hydrogen generating materials and non-hydrogen generating materials in an effort to deduce how hydrogen and oxygen are generated during storage.

Experimental

CAN PUNCTURE DEVICE

The ability to drill into a 3013 container, measure pressure, and collect a pure gas sample at the Savannah River Site (SRS) was achieved through the development and implementation of the can puncture device (CPD). The CPD consists of three main parts: the pressure vessel, the drill press, and the piping system. The pressure vessel component of the CPD has a design pressure of 700 psig and is capable of holding the gases released up to the 699 psig design pressure of a 3013 outer container.[1] A quarter inch titanium drill bit is used for penetrating each wall of the 3013 container. Differential pressures are used as the motive force to move gases out of the 3013 container and into the gas sample cylinder. A picture of the CPD is shown in Figure 1.

The CPD is completely enclosed within a radiological glovebox that contains an air atmosphere. The air must be removed from the CPD prior to drilling to prevent contamination of the gas samples. The entire CPD system is evacuated to a pressure of less than 1.0 psia and then backfilled with N_2 to a pressure of 14.5 psia; this evacuation and backfill process is repeated two additional times. After this operation, the oxygen content in the system should be less than 0.008 mol %. After a fourth evacuation, nitrogen is then added to a pressure between 2.25 psia and 6.25 psia and a blank sample is collected in a 150-cc Swagelok stainless steel gas sample cylinder (Figure 2). The blank sample provides a positive means for demonstrating air has been removed from the CPD and that the system diagnostics are performing as expected prior to puncturing the 3013 container.[16,17]

Next, a sample of the gas between the outer and inner container is collected. A new Swagelok gas sample cylinder is connected to the piping system and the evacuation and back-fill process described above is repeated. A final pressure of 0.5 psia or less is established in the CPD, and a 10-minute leak check is also performed to ensure that in-leakage can be quantitatively addressed when analyzing the sample results. Once an acceptable leak rate of 0.004 psia/min has been established, the wall of the outer 3013 container is punctured. Gases expand from the 3013 annulus into the evacuated CPD pressure vessel and piping manifold, and the gas is subsequently drawn into a Swagelok gas sample cylinder. The evacuation, backfilling, and drilling procedure is repeated for penetration of the inner container and collection the associated gas sample. The inner container (IC), annulus (OI), and blank (BL) samples are sent to SRNL for analysis.[16,17]

GAS EVALUATION SOFTWARE TOOL

The Gas Evaluation Software Tool (GEST - Version 2.0) was developed to calculate the pre-puncture gas conditions within the 3013 inner container. GEST calculates nitrogen, oxygen, hydrogen, helium, methane, and carbon dioxide percentages and inner container pressure. GEST input includes gas composition results from GC analysis and pressure data acquired from the CPD. The GEST accounts for pre-puncture glovebox in-leakage and post-puncture mixing of gases within the CPD. The actual transport or mixing of gases in the CPD following puncture of the inner 3013 container is extremely complex. The transport process is complicated by the tortuous path that the gases must follow to get to the sample cylinder (i.e., passing through the drill bit assembly, metal sintered filter, and quarter inch piping). Immediately after the container is punctured, the pressure will rapidly equilibrate and individual gas species will be quickly mixed with the residual gases left in the CPD. The gas composition becomes homogeneous through diffusion. A statistically designed test was developed to evaluate the transport of gases in the CPD. The test used various combinations of inner can pressures, CPD pressures and inner can free gas volumes. Results from the testing indicated that the "uniform mixing model," in which the gaseous composition of all volumes is identical after puncture of the inner container, best models the behavior of the gases inside the CPD system.[3]

HEADSPACE GAS ANALYSIS

The primary method of characterizing headspace gas from within 3013 containers is by GC. GC measurements are performed using a Varian CP-4900 micro-gas chromatograph that contains two columns, a 10-meter PoraPLOT Q column heated to 45 ºC using helium as the carrier gas and a 25-meter Molsieve 5Å column heated to 60 ºC using argon as the carrier gas. Gas samples are analyzed quantitatively to determine helium, hydrogen, oxygen, nitrogen, methane, carbon dioxide, carbon monoxide, and nitrous oxide concentrations. The GC is installed in a radiological glovebox and controlled by an external computer. It is connected to a gas manifold utilized for evacuation, purging, and sample introduction. The GC is configured to measure gas sample pressures between 2 and 34.7 psia.

A Swagelok gas sample cylinder containing gas sampled by the CPD is connected to the gas manifold to be initially analyzed by GC. The manifold is flushed with argon and evacuated below 1×10^{-5} torr. Initially, analysis of argon passed through the manifold is performed to verify a blank background. After evacuation of the manifold, the sample gas is expanded into the system; if necessary, argon gas is added immediately before sampling to provide a constant inlet pressure of approximately 20 psia. A plugged flow configuration precludes the dilution of the gas sampled by the GC. The GC samples a 1-µL aliquot of the gas with a one second injection time. Sample gases are identified by comparing retention times to previously analyzed NIST-traceable gas standards. Peak areas are integrated to calculate mole percentages of each gas. Qualitative measurements from direct-inlet mass spectrometry (DIMS) and Fourier-transform infrared spectroscopy (FTIR) analyses are used to compliment the GC analysis. These measurements are performed using a MIDAC series 1 model I1301 FTIR with a 10 m beam path and a Pfeifer Omnistar 301 Quadrupole DIMS each connected to the gas manifold.

Results and Discussion

A selection of thirty-one 3013 material containers has been destructively examined at SRS after being stored for approximately 4 to 6 years, as of this writing. When attempting to understand gas behavior in a sealed container, the original gas composition and pressure at packaging is important; unfortunately, it was not possible to characterize the initial headspace gas compositions and pressures. There is, however, some knowledge of the packaging conditions.

The 3013 containers discussed herein were prepared at two different facilities. The average glovebox pressure at Hanford was near 742 torr, whereas the RFETS glovebox pressures averaged approximately 620 torr.[13] Variation in packaging conditions is clearly evident in the helium to nitrogen ratios observed between facilities. Containers packaged at both the Hanford RMC line and RFETS were purged with helium in an air glovebox atmosphere before sealing, in an attempt to substitute inert gas (He) for air, and to reduce the oxygen content of the headspace gas to below 5 %. The GC analyses of the inner can gas samples showed that most Hanford containers contained a $He:N_2$ ratio of approximately 1:1 upon opening, whereas most RFETS containers had a $He:N_2$ ratio of about 5:1. Other gases measured by GC included hydrogen, oxygen, methane, carbon dioxide, carbon monoxide, and nitrous oxide. A summary of GC results for the blank, annulus, and inner container, and final GEST reported gas compositions and pressures for the inner container are presented in Tables 1-3.

The headspace gas composition during storage of plutonium oxide is important because the radiolytic production of gas can result in can pressurization and/or flammability. Pressures measured by the CPD and calculated by GEST for RFETS containers ranged from 11.7 to 13.7 psia and Hanford containers ranged from 11.1 to 20.6 psia. From an over-pressurization standpoint, helium production from alpha decay is expected to be small (less than 1 psia in five years).[1] Postulating RFETS and Hanford RMC containers were sealed having helium equal to that measured at opening less helium produced, the remainder of container contents at packaging would be air at packaging pressure. An analysis of pre-puncture container pressures calculated by the GEST software showed that GEST-calculated pressures for containers with hydrogen were less than predicted based on these assumptions (Table 4), even after accounting for oxygen depletion during storage. A mechanism for nitrogen gas removal by reaction of nitrogen and hydrogen gas to form ammonia and subsequently ammonium chloride solid is hypothesized as a possible explanation for the lower than predicted pressures. Crystalline ammonium chloride has been detected in the corrosion product of H004111 on the inside walls of its convenience container in the headspace region.[18]

The GC results for the 3013 containers packaged at Hanford measured a hydrogen content as high as 36.3 mol %. Seven containers that produced hydrogen over 0.1 % are compiled in Table 4. Oxygen contents for each of these containers were below 0.1 mol %, precluding any in-container flammability concerns. The material contents of the seven containers were very similar with respect to moisture content, surface area, and chloride content. Moisture analysis was performed by TGA-MS on samples of material

taken from each container immediately after opening.[19] Moisture levels ranged from 0.19 to 0.33 wt %. Based on measured hydrogen concentration and moisture content at opening, up to approximately 0.02 wt %, or up to ~7% of the calculated original moisture content was decomposed to hydrogen during storage (Table 4). BET surface area measurements from a representative sampling of each container ranged from 0.99 to 1.30 m^2/g.[19] Approximately 5.7-7.4 wt % chloride, originating from pyrochemical operations, was measured by ion chromatography (IC) of solutions from representative material leached with nanopure deionized water for 3 hours at 90 °C. Because of the presence of hygroscopic alkaline earth chlorides remaining after calcination in these materials, such as magnesium and calcium chlorides that form hydrates, most of the moisture is likely associated with the chloride salt.

Twenty four remaining containers that were examined did not produce hydrogen greater than 0.01 mol % (Tables 1-3). Of these containers, only four, H002088, H003157, R610735, and R602498, contained moisture at levels greater than or equal to 0.1 wt % after opening. Therefore, only these four containers had moisture contents in the range anticipated for hydrogen producing containers. (The moisture contents for each of these materials, as measured by TGA-MS, were 0.18, 0.10, 0.19, and 0.13 wt %, respectively). Chloride contents measured from leached solutions by IC were at or below detection limits for H002088 and H003157. R610735 and R602498 measured 20 wt % and 0.7 wt % chloride, respectively. BET surface area measurements for these materials were 2.02, 1.98, 0.58, and 0.93 m^2/g, respectively.[19] R610735 was most similar to the hydrogen-generating containers in total chloride content and reported moisture. However, the material from R610735 was observed to have increased hygroscopic properties, and possibly sorbed moisture between opening of the 3013 container and TGA-MS analysis of the initial moisture sample, giving an overestimation of moisture. H003157 measured 1.7 mol % oxygen, the highest oxygen concentration observed.

One container, R602498, was identified through a study of the ISP database as a possible candidate for hydrogen and oxygen generation similar to 011589A (from the MIS investigation). Oxygen was measured at <0.1 mol % and hydrogen was not detected. Analysis of material from R602498, by ICP-ES, gave 0.7 % Ca and 0.5 % Mg, while Na and K were below detection limits.[20] While R602498 was determined to be similar to 011589A based on Query 1,[15] the moisture content of 0.13 wt % was well below 0.3 wt % suggested to permit oxygen generation from MIS 011589A experiments.[21]

Conclusions

Gas composition and container pressure results obtained from the destructive surveillance of 3013 containers packaged at Hanford and RFETS provide important data on the gas generation characteristics of plutonium-bearing material from actual 3013 storage inventory. Containers having greater than trace hydrogen had less than 0.1 mol % oxygen, averting flammability concerns for similar containers in storage. Their material moisture levels were at least 0.19 wt % upon opening. All observed pressures are well below the 699 psig design pressure for a 3013 outer container. The ISP database was utilized to identify 3013 containers in storage with a potential to have a composition

similar to test material in MIS item 011589A which generated both hydrogen and oxygen when loaded with 0.5 wt % moisture. R602498, identified by the MIS program as possibly being similar to that of 011589A, did not contain a flammable gas mixture in the headspace. R602498, with a moisture content of 0.13 wt %, had no detectable hydrogen and an oxygen content <0.1 mol %, supporting the conclusion of MIS 011589A experiments, that greater than 0.3 wt % moisture is needed for oxygen to be generated for 011589A-like materials.[21]

Monitoring gas composition and pressure of containers with plutonium-bearing oxide materials during storage provides a key piece of information to evaluate potential container behavior. Headspace gas results from the surveillance program to date support the ability of 3013 containers to store these materials safely over their expected life of 50 years. However, more data from the continued destructive examination of 3013 containers as well as further experimental work are needed to meet the established statistical evaluation basis[22] and to satisfactorily explain differences between observed and predicted pressures for hydrogen-generating materials. Additional work is also needed to understand why observed hydrogen pressures are much lower than might be predicted based on moisture content and experimentally determined rates of hydrogen generation. Finally, multiple small-scale studies in which simultaneous hydrogen and oxygen generation were observed for materials with moisture contents allowed by the 3013 standard point to the importance of continued surveillance of the 3013 inventory to ensure these materials can continue to be safely stored.

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List of Tables and Figures.

Table 1. FY07 Gas Analysis Results from Gas Chromatography and GEST Report

Sample Information	N_2 (%)	O_2 (%)	H_2 (%)	CO_2 (%)	CH_4 (%)	He (%)	CO (%)	He:N_2	GEST Pressure (psia)
FY07-01 (R600885)									
BL-0009	98.30	2.31	Trace	Trace	ND	ND	ND		
OI-0009	28.20	0.53	0.12	0.19	ND	70.20	ND		
IC-0009	10.30	0.11	Trace	Trace	ND	89.40	ND		
GEST (IC)	**9.2**	**0.0**	**0.0**	**0.0**	**0.0**	**90.8**	**NC**	**9.9**	**12.8**
FY07-02 (R601722)									
BL-0016	93.70	2.62	0.03	Trace	ND	ND	ND		
OI-0016	37.80	4.20	0.10	0.14	ND	54.70	ND		
IC-0015	15.90	0.05	Trace	ND	ND	82.20	ND		
GEST (IC)	**12.7**	**<0.1**	**0.0**	**0.0**	**0.0**	**87.2**	**NC**	**6.9**	**12.5**
FY07-03 (R601957)									
BL-0014	93.50	0.12	0.08	Trace	Trace	Trace	ND		
OI-0014	17.90	ND	0.12	0.22	ND	77.90	ND		
IC-0014	11.90	ND	Trace	Trace	ND	86.20	ND		
GEST (IC)	**10.4**	**0.0**	**0.0**	**0.0**	**0.0**	**89.6**	**NC**	**8.6**	**12.8**
FY07-04 (R600719)									
BL-0005	80.00	0.02	Trace	Trace	ND	Trace	ND		
OI-0005	NM	NM	NM	NM	NM	NM	NM		
IC-0005	18.30	ND	Trace	Trace	ND	80.00	ND		
GEST (IC)	**16.2**	**0.0**	**0.0**	**0.0**	**0.0**	**83.8**	**NC**	**5.2**	**12.9**
FY07-05 (R610735)									
BL-0002	96.80	0.04	Trace	Trace	ND	Trace	ND		
OI-0002	23.50	ND	0.10	0.10	Trace	72.80	Trace		
IC-0001	19.20	ND	Trace	0.14	ND	75.80	ND		
GEST (IC)	**17.5**	**0.0**	**0.0**	**0.2**	**0.0**	**82.3**	**NC**	**4.7**	**12.4**
FY07-06 (R610697)									
BL-0017	97.00	ND	0.05	Trace	Trace	0.05	ND		
OI-0017	24.70	0.70	0.05	0.10	Trace	73.40	Trace		
IC-0017	18.60	ND	0.01	Trace	Trace	80.80	ND		
GEST (IC)	**18.0**	**0.0**	**0.0**	**0.0**	**0.0**	**82.0**	**NC**	**4.6**	**12.5**
FY07-07 (R601285)									
BL-0013	67.80	0.03	0.03	Trace	ND	0.03	ND		
OI-0013	21.80	ND	0.13	0.11	Trace	76.90	0.02		
IC-0013	24.40	ND	Trace	Trace	0.02	75.40	ND		
GEST (IC)	**22.5**	**0.0**	**0.0**	**0.0**	**<0.1**	**77.4**	**NC**	**3.4**	**13.4**

RFETS site origin contains the letter R or H<1000, Hanford site origin contains H>1000, BL = blank, OI = annulus, IC = inner container, ND = not detected, NC = not calculated, NM = not measured, trace = <0.01 mol %

Table 2a. FY08 Gas Analysis Results from Gas Chromatography and GEST Report

Sample Information	N_2 (%)	O_2 (%)	H_2 (%)	CO_2 (%)	CH_4 (%)	He (%)	CO (%)	He:N_2	GEST Pressure (psia)
FY08-01 (R602731)									
BL-0001	100.10	ND	ND	Trace	ND	ND	ND		
OI-0007	31.20	0.90	0.02	0.12	Trace	67.20	ND		
IC-0006	18.70	0.07	Trace	Trace	Trace	81.30	ND		
GEST (IC)	**16.1**	**<0.1**	**Trace**	**Trace**	**Trace**	**83.9**	**NC**	**5.2**	**12.3**
FY08-02 (R601318)									
BL-0004	100.20	ND	ND	Trace	ND	ND	ND		
OI-0008	24.20	0.46	0.08	0.06	Trace	74.70	ND		
IC-0007	11.20	ND	Trace	Trace	ND	87.50	ND		
GEST (IC)	**8.5**	**0.0**	**Trace**	**Trace**	**0.0**	**91.5**	**NC**	**10.8**	**12.6**
FY08-03 (H000898)									
BL-0011	101.90	ND	Trace	Trace	ND	ND	ND		
OI-0010	31.80	ND	0.15	0.09	Trace	69.30	ND		
IC-0008	23.00	0.05	Trace	0.25	ND	76.30	ND		
GEST (IC)	**19.3**	**<0.1**	**Trace**	**0.3**	**0.0**	**80.3**	**NC**	**4.2**	**12.3**
FY08-04 (R610327)									
BL-0012	100.30	ND	0.02	Trace	ND	ND	ND		
OI-0011	23.50	0.17	0.04	0.09	Trace	74.00	ND		
IC-0018	19.70	ND	Trace	0.41	ND	78.20	ND		
GEST (IC)	**16.7**	**0.0**	**Trace**	**0.4**	**0.0**	**82.8**	**NC**	**5.0**	**12.4**
FY08-05 (R610298)									
BL-0007	97.90	1.02	ND	Trace	ND	ND	ND		
OI-0001	25.10	ND	0.18	0.11	Trace	72.70	ND		
IC-0019	17.80	ND	Trace	Trace	ND	79.40	ND		
GEST (IC)	**16.0**	**0.0**	**Trace**	**Trace**	**0.0**	**84.0**	**NC**	**5.3**	**13.0**
FY08-06 (R610324)									
BL-0018	102.30	ND	ND	Trace	ND	ND	ND		
OI-0018	29.10	0.03	0.11	0.07	Trace	72.00	ND		
IC-0020	21.00	ND	Trace	ND	ND	78.70	ND		
GEST (IC)	**17.7**	**0.0**	**Trace**	**0.0**	**0.0**	**82.3**	**NC**	**4.6**	**13.1**
FY08-07 (H001992)									
BL-0019	99.40	ND	ND	Trace	ND	ND	ND		
OI-0019	17.80	0.04	0.25	0.02	Trace	79.50	ND		
IC-0021	43.30	ND	Trace	ND	ND	54.40	ND		
GEST (IC)	**42.2**	**0.0**	**Trace**	**0.0**	**0.0**	**57.8**	**NC**	**1.4**	**13.3**
FY08-08 (H003157)									
BL-0021	101.40	0.01	Trace	Trace	ND	Trace	ND		
OI-0021	15.70	Trace	0.38	Trace	Trace	83.60	ND		
IC-0023	45.60	1.57	Trace	Trace	ND	52.10	ND		
GEST (IC)	**44.0**	**1.7**	**Trace**	**Trace**	**0.0**	**54.3**	**NC**	**1.2**	**12.4**

RFETS site origin contains the letter R or H<1000, Hanford site origin contains H>1000, BL = blank, OI = annulus, IC = inner container, ND = not detected, NC = not calculated, NM = not measured, trace = <0.01 mol %

Table 2b. FY08 Gas Analysis Results from Gas Chromatography and GEST Report

Sample Information	N_2 (%)	O_2 (%)	H_2 (%)	CO_2 (%)	CH_4 (%)	He (%)	CO (%)	He:N_2	GEST Pressure (psia)
FY08-09 (R610584)									
BL-0020	94.10	6.34	Trace	<0.1	ND	ND	Trace		
OI-0020	26.20	ND	0.13	0.20	Trace	73.00	0.04		
IC-0022	18.00	ND	Trace	0.64	ND	80.90	ND		
GEST (IC)	**14.8**	**0.0**	**Trace**	**0.7**	**0.0**	**84.6**	**NC**	**5.7**	**13.7**
FY08-10 (R610578)									
BL-0022	97.10	3.40	Trace	0.01	ND	Trace	ND		
OI-0022	22.90	Trace	0.11	0.06	Trace	76.40	ND		
IC-0025	21.00	ND	Trace	0.02	ND	77.80	ND		
GEST (IC)	**17.8**	**0.0**	**Trace**	**<0.1**	**0.0**	**82.1**	**NC**	**4.6**	**12.5**
FY08-11 (H001916)									
BL-0023	101.40	0.37	Trace	Trace	ND	ND	ND		
OI-0023	19.00	Trace	0.20	0.03	Trace	80.60	ND		
IC-0024	48.90	ND	Trace	Trace	ND	52.40	ND		
GEST (IC)	**45.6**	**0.0**	**Trace**	**Trace**	**0.0**	**54.5**	**NC**	**1.2**	**13.3**
FY08-12 (H002088)									
BL-0024	101.80	ND	Trace	Trace	ND	ND	ND		
OI-0024	16.40	ND	0.62	Trace	Trace	83.80	ND		
IC-0026	44.40	ND	Trace	Trace	ND	56.80	ND		
GEST (IC)	**41.6**	**0.0**	**Trace**	**Trace**	**0.0**	**58.4**	**NC**	**1.4**	**12.4**
FY08-13 (H003409)									
BL-0025	100.30	ND	Trace	ND	ND	ND	ND		
OI-0025	17.50	ND	0.28	Trace	Trace	81.10	ND		
IC-0027	38.10	ND	18.10	Trace	Trace	45.60	ND		
GEST (IC)	**34.2**	**0.0**	**18.7**	**Trace**	**Trace**	**47.1**	**NC**	**1.4**	**13.4**
FY08-14 (H002573)									
BL-0026	103.10	ND	Trace	Trace	ND	ND	ND		
OI-0026	18.40	Trace	0.22	0.01	Trace	80.90	ND		
IC-0028	35.10	ND	28.80	0.31	Trace	41.20	ND		
GEST (IC)	**33.9**	**0.0**	**27.9**	**0.3**	**Trace**	**37.9**	**NC**	**1.1**	**17.6**
FY08-15 (H002534)									
BL-0027	101.50	ND	Trace	Trace	ND	ND	ND		
OI-0027	NM	NM	NM	NM	NM	NM	NM		
IC-0029	29.30	ND	30.10	2.62	0.39	40.40	0.75		
GEST (IC)	**24.0**	**0.0**	**30.8**	**2.7**	**0.4**	**41.4**	**0.8**	**1.7**	**13.7**
FY08-16 (R610679)									
BL-0028	100.30	ND	ND	Trace	ND	ND	ND		
OI-0028	14.10	ND	0.27	0.17	Trace	83.80	ND		
IC-0030	30.50	ND	Trace	2.76	ND	65.60	0.07		
GEST (IC)	**26.7**	**0.0**	**Trace**	**3.0**	**0.0**	**70.3**	**NC**	**2.6**	**11.7**
FY08-17 (H002750)									
BL-0029	101.00	ND	ND	Trace	ND	ND	ND		
OI-0029	19.50	Trace	0.21	0.01	Trace	79.70	ND		
IC-0031	40.50	0.34	Trace	Trace	ND	58.50	ND		
GEST (IC)	**36.8**	**0.1**	**Trace**	**Trace**	**0.0**	**63.1**	**NC**	**1.7**	**13.1**

RFETS site origin contains the letter R or H<1000, Hanford site origin contains H>1000, BL = blank, OI = annulus, IC = inner container, ND = not detected, NC = not calculated, NM = not measured, trace = <0.01 mol %

Table 3. FY09 Gas Analysis Results from Gas Chromatography and GEST Report

Sample Information	N_2 (%)	O_2 (%)	H_2 (%)	CO_2 (%)	CH_4 (%)	He (%)	CO (%)	He:N_2	GEST Pressure (psia)
FY09-01 (H004099)									
BL-0030	100.00	1.11	ND	ND	ND	Trace	Trace		
OI-0030	19.94	<0.1	0.59	<0.1	ND	78.28	ND		
IC-0032	55.10	0.19	Trace	0.34	ND	41.11	Trace		
GEST (IC)	**53.0**	**0.2**	**Trace**	**0.4**	**0.0**	**45.0**	**NC**	**0.8**	**11.7**
FY09-02 (H004111)									
BL-0031	102.00	ND	ND	<0.1	ND	ND	ND		
OI-0031	26.54	Trace	0.28	<0.1	ND	72.64	ND		
IC-0033	47.82	<0.1	20.43	Trace	ND	32.30	ND		
GEST (IC)	**44.4**	**<0.1**	**21.5**	**Trace**	**0.0**	**34.1**	**NC**	**0.8**	**14.4**
FY09-03 (H002554)									
BL-0032	100.00	ND	ND	<0.1	ND	Trace	ND		
OI-0032	33.26	Trace	19.31	Trace	Trace	49.44	ND		
IC-0034	NM	NM	NM	NM	NM	NM	NM		
GEST (IC)	**32.6**	**Trace**	**22.1**	**Trace**	**Trace**	**45.3**	**NC**	**1.4**	**12.4**
FY09-04 (H001941)									
BL-0033	101.55	ND	ND	Trace	ND	ND	ND		
OI-0033	4.66	<0.1	0.31	Trace	Trace	95.97	ND		
IC-0035	49.72	ND	Trace	Trace	ND	49.16	ND		
GEST (IC)	**49.8**	**0.0**	**Trace**	**Trace**	**0.0**	**50.2**	**NC**	**1.0**	**13.2**
FY09-05 (R602498)									
BL-0034	102.60	Trace	ND	Trace	ND	ND	ND		
OI-0034	18.34	0.24	<0.1	0.15	Trace	83.04	ND		
IC-0036	13.65	<0.1	ND	Trace	ND	88.78	ND		
GEST (IC)	**12.5**	**<0.1**	**0.0**	**0.0**	**0.0**	**87.5**	**NC**	**7.0**	**12.3**
FY09-06 (H002509)									
BL-0035	103.10	0.31	ND	Trace	ND	Trace	ND		
OI-0035	3.61	0.19	0.29	<0.1	Trace	98.70	ND		
IC-0037	31.18	<0.1	34.00	0.41	<0.1	31.95	ND		
GEST (IC)	**29.2**	**<0.1**	**36.3**	**0.4**	**0.0**	**34.1**	**NC**	**1.2**	**20.6**
FY09-07 (H002565)									
BL-0036	103.36	<0.1	ND	Trace	ND	ND	ND		
OI-0036	3.69	0.11	0.23	<0.1	Trace	98.29	ND		
IC-0038	41.37	<0.1	4.45	<0.1	Trace	56.27	ND		
GEST (IC)	**39.8**	**<0.1**	**4.4**	**<0.1**	**Trace**	**55.8**	**NC**	**1.4**	**11.1**

RFETS site origin contains the letter R or H<1000, Hanford site origin contains H>1000, BL = blank, OI = annulus, IC = inner container, ND = not detected, NC = not calculated, NM = not measured, trace = <0.01 mol %

Table 4. Results for Hydrogen Containers

Sample Information	N_2 (%)	O_2 (%)	H_2 (%)	CO_2 (%)	CH_4 (%)	He (%)	CO (%)	He:N_2	GEST Pressure (psia)	Predicted Pressure (psia)	Measured Moisture (wt %)	Calculated Moisture at Packaging (wt %)	Packaging Moisture Consumed (%)	Leached Chloride (wt %)
FY08-13 (H003409)														
GEST (IC)	34.2	ND	18.7	Trace	Trace	47.1	NC	1.4	13.4	15.4	0.29	0.30	2.2	6.4
FY08-14 (H002573)														
GEST (IC)	33.9	ND	27.9	0.3	Trace	37.9	NC	1.1	17.6	18.1	0.33	0.34	3.9	6.3
FY08-15 (H002534)														
GEST (IC)	24.0	ND	30.8	2.7	0.4	41.4	0.8	1.7	13.7	17.7	0.19	0.20	5.7	6.3
FY09-02 (H004111)														
GEST (IC)	44.4	<0.1	21.5	Trace	0.0	34.1	NC	0.8	14.4	15.9	0.26	0.27	3.2	5.8
FY09-03 (H002554)														
GEST (IC)	32.6	Trace	22.1	Trace	Trace	45.3	NC	1.4	12.4	15.7	0.22	0.23	3.2	6.7
FY09-06 (H002509)														
GEST (IC)	29.2	<0.1	36.3	0.4	ND	34.1	NC	1.2	20.6	20.8	0.26	0.28	7.3	5.7
FY09-07 (H002565)														
GEST (IC)	39.8	<0.1	4.4	<0.1	Trace	55.8	NC	1.4	11.1	13.6	0.25	0.25	0.5	7.4

RFETS site origin contains the letter R or H<1000, Hanford site origin contains H>1000, BL = blank, OI = annulus, IC = inner container, ND = not detected, NC = not calculated, NM = not measured, trace = <0.01 mol %

Figure 1. Can Puncture Device
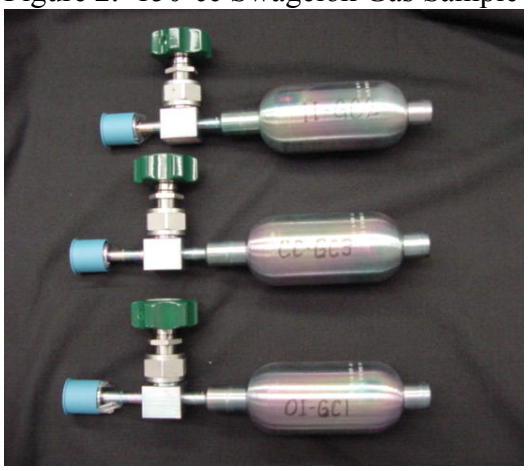
Figure 2. 150-cc Swagelok Gas Sample Cylinders