A Statistical Review of the Oxygen Data from the Gas Generation Tests Supporting the Revision of the 9975 SARP

by

T. B. Edwards

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

DOE Contract No. **DE-AC09-96SR18500**

This paper was prepared in connection with work done under the above contract number with the U. S. Department of Energy. By acceptance of this paper, the publisher and/or recipient acknowledges the U. S. Government's right to retain a nonexclusive, royalty-free license in and to any copyright covering this paper, along with the right to reproduce and to authorize others to reproduce all or part of the copyrighted paper.

A STATISTICAL REVIEW OF THE OXYGEN DATA FROM THE GAS GENERATION TESTS SUPPORTING THE REVISION OF THE 9975 SARP (U)

T. B. Edwards

Westinghouse Savannah River Company
Savannah River Technology Center
Aiken, SC 29808

SAVANNAH RIVER SITE

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT NO. DE-AC09-96SR18500

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available for sale to the public, in paper, from: U.S. Department of Commerce, National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, phone: (800) 553-6847

fax: (703) 605-6900

email: orders@ntis.fedworld.gov

online ordering: <http://www.ntis.gov/ordering.htm>

Available electronically at <http://www.doe.gov/bridge>

Available for a processing fee to U.S. Department of Energy and its contractors, in paper, from: U.S. Department of Energy, Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831-0062, phone: (865) 576-8401

fax: (865) 576-5728

email: reports@adonis.osti.gov

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

A STATISTICAL REVIEW OF THE OXYGEN DATA FROM THE GAS GENERATION TESTS SUPPORTING THE REVISION OF THE 9975 SARP (U)

T. B. Edwards

July 15, 1999

Document Approvals

_____ T. B. Edwards, Author Statistical Consulting Section	_____ Date
_____ J. H. Weber, Technical Reviewer Statistical Consulting Section	_____ Date
_____ R. C. Tuckfield, Manager, Statistical Consulting Section	_____ Date
_____ C. P. Holding-Smith, Manager Engineering Modeling & Simulation Group Authorized Derivative Classifier	_____ Date

This page intentionally left blank.

INTRODUCTION

The Chemical and Hydrogen Technology Section (CHT) of the Savannah River Technology Center (SRTC) has conducted a series of gas generation tests in support of the revision of the safety analysis report for packaging (SARP) for the 9975 container, developed at the Savannah River Site (SRS). The Packaging and Transportation Group of SRTC is coordinating the revision to this SARP. A Task Technical and Quality Assurance Plan directing this work was issued by CHT in February 1999 [1].

Initially, the primary interest in this testing was hydrogen generation. From [1], these gas "generation tests can be tracked in real-time by measuring the pressure of a sealed container of the materials being studied. Because multiple gas phase reactions are produced in the radiation field of the sample, material pressure measurements do not necessarily define the quantity of H_2 generated. However, the change in total molecules of gas can be calculated using the ideal gas law from the pressure measurement, known container volume and sample temperature. A measurement of the actual headspace gases must be completed to calculate the H_2 generation rate for a particular sample."

As the results from these tests were reviewed, however, questions arose regarding the oxygen in the headspace gases. Specifically, do the data from some tests indicate that oxygen was generated for those tests? And do the data for other tests indicate that the oxygen was depleted for them? A statistical analysis of the oxygen data derived from these tests is provided in this report to help answer these questions.

This analysis was conducted using JMP® Version 3.2.2, a commercial software package of the SAS Institute, Inc., in Cary, NC [2].

DISCUSSION

In this section various plots of the oxygen data are reviewed; the tests are grouped into similar types of experiments, which are then studied; measurement and analytical uncertainties are propagated through the equations used to determine the change in oxygen; and finally, the test results are judged for significant O_2 changes based upon their corresponding uncertainties. As part of this final assessment, the results from those tests that are thought to be most representative of the operational window for the 9975 package are summarized.

The data from these tests are provided in Table A1 of the Appendix. A test identifier is provided in the first column of this table. This identifier is used throughout this report to designate specific tests. Table A2 in the Appendix provides additional information for these tests designated using these same test identifying numbers.

As described above for H₂, the determination of interest for O₂ is the change in total O₂ molecules from the beginning to the end of each test in the sample vessel.

This determination is made using the following equation

$$r = n_2 - n_1 = \frac{mP_2V}{RT_2} - \frac{aP_1V}{RT_1} \quad (1)$$

where n_1 is an estimate of the initial moles of O₂ in the headspace gases of the container and n_2 is an estimate of the final moles of O₂ in the headspace gases. Equation (1) also shows how these estimates are determined using the ideal gas law. In equation (1), the term "a" represents the initial O₂ as a fraction of the headspace gas volume, V (in cubic centimeters, cc), at an initial pressure of P₁ (in torr) and temperature of T₁ (in degrees Kelvin, °K). R is the universal gas constant, $62324.73 \frac{\text{torr} \cdot \text{cc}}{\text{K} \cdot \text{mole}}$. The "m" term in equation (1) represents the final

O₂ concentration as a fraction of the headspace gases of volume V at a pressure of P₂ (in torr) and temperature T₂ (°K).

The value of "a" for each experiment is the fraction of O₂ in air, for which the reference value is 0.20946. The value for "m" was determined by gas chromatography utilizing a sample of the headspace gases taken by syringe at the end of the test. These values along with results from equation (1) are provided in Table A1.

INITIAL LOOK AT THE OXYGEN DATA

Exhibit A1 in the Appendix provides a plot of the initial O₂ molar value versus the corresponding final value by test. Some of those tests with final O₂ values larger than their initial values are labeled with their test number in this plot. Exhibit A2 in the Appendix provides a plot of the change in O₂ (final minus initial) by test number. A reference line at zero is also shown on this plot. Two questions of interest in this last plot are: "Which of these test results indicate a significant increase in O₂ (if any)?" and "Which of these tests indicate a significant decrease in O₂ (if any)?"

In order to answer these questions, an idea of the repeatability of the outcomes from similar tests must be determined. The test descriptions in Table A2 of the Appendix were used to group the tests into the categories indicated in Table 1.

Table 1: Types of Tests

Type	Tests
PuO ₂ + MgO	1, 11, 23H, 25, 26
PuO ₂ + CaF	9, 14, 19
Pu Cake	17, 44, 50, 52
PuO ₂	12, 16H, 41, 51
PuO ₂ + CaO	2, 8, 15H
Other	3, 4, 5, 6, 7, 10, 13, 18, 20, 21, 22, 24, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41, 42, 43, 45, 46, 47, 48, 49, 53, 54, 55, 56, 57, 58, 59

An "H" following a test number indicates that that particular test was heated, possibly differentiating it from the other tests in its group.

Exhibit A3 in the Appendix provides a look at the change in O_2 by group type. Descriptive statistics for each of these groups are also provided in this exhibit. Note that the heated tests (those with the "H" suffix in Table 1) are labeled (in addition to those that were labeled earlier) in this exhibit and that they result in a change in O_2 that appears to be different from their unheated counterparts. The average and standard deviation of each group containing a heated test are computed including and excluding this test.

The repeatability of a test of the 9975 package may be represented by the standard deviation in the measurements of set of identical experiments. Each of the first 5 groups from Table 1 (excluding the heated tests) is to be considered as a set of nearly identical experiments. Their standard deviations range from 0.000071 to 0.000002 moles of O_2 with a pooled estimate of the standard deviation across all five groups of 0.00004 moles. When considered on a percentage basis as a relative (to the mean O_2 change) standard deviation, they range from about 21% to about 101% of the average O_2 change for the group with a pooled relative standard deviation of about 88% (using a one-way analysis of variance approach). In summary, a 2-sigma uncertainty determined from these results would lead to a limit of ± 0.00008 moles and $\pm 176\%$ of reading for absolute and relative errors, respectively.¹ Obviously, assessing the O_2 changes using the relative errors leads to insignificant differences between the initial and final oxygen for all of the tests. For those tests showing a potential O_2 increase, the absolute error 2-sigma limit leads to only 28, 48, and 49 being significant at an approximate 95% confidence level.

Part of the variation seen among the results for each of these groups is from measurement and analytical uncertainty and part is from differences in the conditions among each set of repeated tests (i.e., the conditions of the tests in each group may not have been as "identical" as desired). The next section looks at the variation due to the first of these two categories via the approach of error propagation. Before moving on to that topic, consider two additional looks at these data.

The first is a plot of the O_2 change versus headspace volume (in cc) across all of the tests, which is provided in Exhibit A4 in the Appendix. The volume of headspace gases in the container of each test is plotted along the x-axis. Along the y-axis the initial moles of O_2 are plotted as a solid light-gray line. The final moles of O_2 are also plotted on the y-axis, and their values are indicated by the symbol "x". While several of the tests showing a potential increase in O_2 have a headspace gas volume in the interval from 65 to 75 cc, other tests with volumes of only 30 cc also show a potential increase in O_2 . Thus, there is no obvious

¹ Absolute and relative errors are discussed later in this section.

relationship between the initial headspace gas volume and the generation or depletion of O₂.

One of the key steps in measuring the final O₂ value is injecting a sample from the headspace gas of the test container into a gas chromatograph. The recovery of this sample as reflected by its total sample volume percent determination is a critical measurement in this procedure. The target value for this determination is 100%. Exhibit A5 in the Appendix provides a plot of the O₂ changes versus the total sample volume percent determinations for these tests. There is no obvious relationship between the total sample volume percent determination and the generation or depletion of O₂.

PROPAGATION OF MEASUREMENT UNCERTAINTIES

As evident in equation (1), the determination of r , the difference in moles of O₂ from the beginning to the end of a test, is dependent on several measurements conducted over the course of the test. One question of interest as the results of these tests are evaluated is the potential impact of the uncertainties of these measurements on the derived values for r . Propagating the measurement uncertainties or errors afflicting the terms of equation (1) into the resultant r reveals this potential impact.

The errors in experimental measurements fall into the two broad categories of bias errors and random errors. Bias is related to accuracy, the closeness of agreement between the measured value and the true value.² Randomness of a measurement is related to the precision or repeatability of the measurement. Appropriately taken together, these two errors yield the uncertainty of the measurement. The error propagation for equation (1) conducted here follows the framework provided in reference [3]. This reference also provides a full discussion of the practice of and underlying theory for uncertainty analysis in an experimental setting. It is a linearized approach based on the use of a Taylor series expansion of equation (1).

Using the approach and terminology of [3], a 95% coverage estimate of the uncertainty in the result r , U_r , can be expressed as

$$U_r^2 = B_r^2 + (tS_r)^2 \quad (2)$$

where B_r represents the 95% confidence bound of the bias limit for r , S_r represents the estimate of the standard deviation of the random errors, and t is the two-tailed, 95% Student t value (i.e., the 97.5th percentile, with degrees of freedom equal to those associated with the estimate of S_r).

²

In the situation where bias errors are known, the corresponding measurements are frequently corrected for these biases. Some random errors may be induced into the measurement by this process, however. In addition, the durations of the biases often vary. Some bias or systematic errors may remain constant over only a few measurements while others may afflict an entire experimental study. For this study, the approach taken is to select bounding values (at a 95% confidence level) for the potential measurement biases.

The bias (B_r) and precision (S_r) errors for r are estimated by propagating the bias (B_i) and precision (S_i) errors of each measurement term i of equation (1) using a Taylor's series expansion as indicated in the following equations:

$$B_r^2 = \sum_{i=1}^J \theta_i^2 B_i^2 + 2 \sum_{i=1}^{J-1} \sum_{j=i+1}^J \theta_i \theta_j \rho_{ij} B_i B_j \quad (3)$$

and

$$(tS_r)^2 = \sum_{i=1}^J \theta_i^2 (tS_i)^2 + 2 \sum_{i=1}^{J-1} \sum_{j=i+1}^J \theta_i \theta_j \rho'_{ij} (tS_i)(tS_j) \quad (4)$$

where the summations are over the J ($=7$) terms of equation (1), which are listed in Table 2 and which contribute uncertainty to the resultant r , each $\theta_i = \frac{\partial r}{\partial i^{\text{th}} \text{ term}}$ represents the partial derivative of equation (1) with respect to the i^{th} term of that equation, each B_i represents the 95% confidence estimate of the bias for the i^{th} such term, S_i represents the standard deviation of the precision errors for this i^{th} term, the corresponding Student t value is chosen for 95% (two-tailed) confidence and reflecting the degrees of freedom associated with S_i , the ρ_{ij} expression represents the correlation between the biases for terms i and j , and the ρ'_{ij} expression represents the correlation between the random errors for terms i and j .

For the approach being used here, the degrees of freedom associated with each estimated S_i is assumed large enough to justify using $t=2$ in the analyses that follow.

Table 2 provides a list of the partial derivative of equation (1) with respect to each term of interest and provides initial estimates of the bias and precision errors for each of these terms.

Table 2: Information Associated with the Propagation of Errors

Term		Units	Partial Derivative	95% Bound for Bias, B_i (relative)	95% Bound for Bias, B_i (absolute)	1 Standard Deviation Precision, S_i (relative)	1 Standard Deviation Precision, S_i (absolute)
1	a		$\frac{-P_1 \cdot V}{R \cdot T_1}$	2%	0.004	2%	0.004
2	V	cc	$\frac{m \cdot P_2}{R \cdot T_2} - \frac{a \cdot P_1}{R \cdot T_1}$	3%	1	3%	1
3	m		$\frac{P_2 \cdot V}{R \cdot T_2}$	5%	0.01	5%	0.01
4	P_1	torr	$\frac{-a \cdot V}{R \cdot T_1}$	1%	7	1%	7
5	P_2	torr	$\frac{m \cdot V}{R \cdot T_2}$	1%	7	1%	7
6	T_1	°K	$\frac{a \cdot P_1 \cdot V}{R \cdot T_1^2}$	0.33%	1	0.33%	1
7	T_2	°K	$\frac{-m \cdot P_2 \cdot V}{R \cdot T_2^2}$	0.33%	1	0.33%	1

The estimates of the uncertainties presented in Table 2 will be the basis for deriving the uncertainties, via error propagation, for the r 's resulting from these 9975 package tests.³ As indicated in this table, relative and absolute errors will be considered in this analysis for each measurement.

There is a need to understand the difference between using relative and absolute errors to represent or model these uncertainties. Let the true value for a measurement be represented by M_T . If the size of the errors afflicting M_T is affected by the size of M_T itself, then the use of relative errors would be appropriate. If the size of the errors is unaffected by the size of M_T , then the use of absolute errors is appropriate.

For example, the value of the sample volume, V , varies from a low of 14.43 cc to a high of 123.67cc over these tests. The bias, when considered as a relative 3% error, would range from $14.43 \times 0.03 = 0.433$ cc to $123.67 \times 0.03 = 3.71$ cc. The bias, when considered as an absolute error, would be a constant 1 cc. The standard deviation of the random errors afflicting V , when considered as a relative error, would range from $14.43 \times 0.03 = 0.433$ cc to $123.67 \times 0.03 = 3.71$ cc. The standard deviation would be a constant (an absolute) 1 cc when these random errors are assumed to be unaffected by the size of V .

It is not unusual for the absolute errors to dominate when M_T is at the low end of the range of its possible values while relative errors dominate when M_T is at the high end of its range. In the discussion that follows, error propagations are conducted that consider various combinations of these errors as relative or absolute, since the true models for these uncertainties are not known.

³

These estimates of bias and precision were developed during discussions of these issues and are felt to be representative of these measurement uncertainties.

Correlations among the biases and any among the random errors afflicting the measurements for any one test would be limited to the beginning and ending temperatures and the beginning and ending pressures. It is expected that such correlations would be positive in sign. Note, however, that the partial derivatives for the two pressures and those for the two temperatures are of different signs. This implies that any such correlations among these biases and/or among these random errors would tend to reduce the uncertainty of the resultant, r .

SIGNIFICANT DIFFERENCES IN MOLES OF OXYGEN

In the analyses that follow, the focus is on two primary situations. The first assumes that there are no correlations among these measurements and that the uncertainties of Table 2 are representative. In this situation, the error propagation is to be developed (i.e., the choice between each relative and absolute error model is made) to provide an upper bound on the uncertainties for the r values (at a 95% confidence level). The second situation reflects highly correlated errors between the two temperature measurements, highly correlated errors between the two pressure measurements, and uncertainties for all measurements smaller than those appearing in Table 2. The intent is to use the second situation to conduct a sensitivity analysis on the assumptions used in the first situation.

Table 3 provides the results for the first situation, the upper bounding case. It identifies each of the 9975 package tests, provides values for the 7 measurement terms from equation (1), evaluates the corresponding value for r , and propagates the uncertainties of the measurements into the resulting r , selecting the maximum of the relative and absolute error models for each measurement. No correlations among the biases or random errors are considered in this table.

The next to the last column of this table provides the uncertainty of the measured difference, r , in O_2 , at a 95% confidence level. This is a $\pm$ uncertainty for the derived value of r . The last column indicates the results of assessing the difference between the calculated value of r and zero in light of r 's uncertainty. Three labels are used for the rows of this last column. If the value of r is sufficiently greater than zero, the label " O_2 generated" is used for the row. If the value of r is sufficiently less than zero, the label used is " O_2 depleted." Otherwise, the label "No difference" is used for the row.

Exhibit A6 in the Appendix provides a plot of the results of Table 3 by test. It shows the upper and lower error bounds for each test plotted around zero. If the calculated value of r falls within this interval, there is no significance difference between the initial and final moles of O_2 . A plotted value about the upper limit, indicates that the final moles of O_2 is significantly greater than the initial for that test, and a plotted value below the lower limit indicates that the final O_2 moles are significantly less than the initial moles. Thus, this plot provides a graphical representation of the significance of the O_2 difference relative to the 95% uncertainty limits.

Table 3: Results of Bounding Error Propagation by Test Number

Test	a	P1	V	T1	m	P2	T2	R	r	95%	r value
										Uncertainty for r	relative to zero at 95%
1	0.20946	752.2	34.11	296.6	0.188	743.2	295.7	62324.73	-0.000032	0.00003470	No difference
2	0.20946	741.9	63.86	294.7	0.179	711.8	297.2	62324.73	-0.000101	0.00006254	O ₂ Depleted
3	0.20946	740.9	30.50	299.3	0.211	741.3	295.9	62324.73	0.000005	0.00003222	No difference
4	0.20946	748.3	30.90	298.5	0.201	751.5	295.4	62324.73	-0.000007	0.00003182	No difference
5	0.20946	755.5	29.81	297.3	0.228	891.3	295.5	62324.73	0.000074	0.00004012	O ₂ Generated
6	0.20946	775.7	62.33	295.5	0.039	774.7	294.5	62324.73	-0.000447	0.00007169	O ₂ Depleted
7	0.20946	649.4	49.71	296.2	0.036	808.1	331.0	62324.73	-0.000296	0.00005145	O ₂ Depleted
8	0.20946	750.2	64.10	295.6	0.196	735.1	295.7	62324.73	-0.000046	0.00006467	No difference
9	0.20946	734.5	62.91	299.1	0.202	733.3	298.2	62324.73	-0.000018	0.00006303	No difference
10	0.20946	746.5	43.46	298.5	0.202	746.5	295.8	62324.73	-0.000010	0.00004463	No difference
11	0.20946	755.7	38.51	296.4	0.197	749.7	295.9	62324.73	-0.000022	0.00003948	No difference
12	0.20946	756.6	30.72	297.5	0.2	753.7	296.1	62324.73	-0.000012	0.00003161	No difference
13	0.20946	762.3	51.66	302.1	0.031	655.9	295.9	62324.73	-0.000381	0.00005323	O ₂ Depleted
14	0.20946	765.1	64.37	296.3	0.167	975.2	329.5	62324.73	-0.000048	0.00007500	No difference
15	0.20946	754.1	65.63	299.6	0.117	806.3	327.6	62324.73	-0.000252	0.00006695	O ₂ Depleted
16	0.20946	760.2	30.78	296.1	0.155	753.2	314.3	62324.73	-0.000082	0.00003058	O ₂ Depleted
17	0.20946	762.2	42.04	298.0	0.206	779.1	297.2	62324.73	0.000003	0.00004546	No difference
18	0.20946	760.0	30.34	298.0	0.155	1179.8	296.4	62324.73	0.000040	0.00004593	No difference
19	0.20946	751.7	52.40	298.4	0.182	744.5	297.4	62324.73	-0.000061	0.00005307	O ₂ Depleted
20	0.20946	753.4	53.82	297.6	0	1178.3	321.3	62324.73	-0.000458	0.00008059	O ₂ Depleted
21	0.20946	744.0	25.98	298.4	0.06	803.2	323.0	62324.73	-0.000155	0.00002898	O ₂ Depleted
22	0.20946	749.4	43.03	298.3	0.154	742.1	298.0	62324.73	-0.000099	0.00004357	O ₂ Depleted
23	0.20946	758.2	31.95	299.0	0.119	806.3	307.8	62324.73	-0.000112	0.00003417	O ₂ Depleted
24	0.20946	764.4	43.83	298.1	0.197	764.3	297.8	62324.73	-0.000022	0.00004545	No difference
25	0.20946	749.5	14.43	295.9	0.126	800.2	324.7	62324.73	-0.000051	0.00001633	O ₂ Depleted
26	0.20946	752.6	25.67	296.2	0.162	789.2	317.1	62324.73	-0.000053	0.00002617	O ₂ Depleted
28	0.20946	758.2	68.91	296.7	0.242	829.8	297.9	62324.73	0.000153	0.00009085	O ₂ Generated
29	0.20946	768.4	27.47	371.7	0.133	341.5	419.5	62324.73	-0.000143	0.00001726	O ₂ Depleted
30	0.20946	755.8	63.26	297.8	0.165	724.6	297.6	62324.73	-0.000132	0.00006300	O ₂ Depleted
31	0.20946	733.8	26.22	369.5	0.205	738.8	367.5	62324.73	-0.000002	0.00002169	No difference
32	0.20946	753.9	91.65	297.5	0.129	772.2	296.9	62324.73	-0.000287	0.00009684	O ₂ Depleted
33	0.20946	760.0	38.54	298.8	0.202	751.8	296.8	62324.73	-0.000013	0.00003982	No difference
34	0.20946	768.9	38.98	298.9	0.202	760.5	296.9	62324.73	-0.000013	0.00004072	No difference
35	0.20946	745.8	53.04	296.4	0.189	755.3	297.4	62324.73	-0.000040	0.00005432	No difference
36	0.20946	748.5	61.32	297.9	0.013	3843.2	324.5	62324.73	-0.000366	0.00026301	O ₂ Depleted
37	0.20946	741.6	26.49	297.4	0.2	813.1	316.1	62324.73	-0.000003	0.00002739	No difference
38	0.20946	744.9	44.90	297.4	0.141	802.2	297.9	62324.73	-0.000104	0.00004835	O ₂ Depleted
39	0.20946	752.4	46.04	297.4	0.208	758.3	297.8	62324.73	0.000000	0.00004889	No difference
40	0.20946	759.7	46.27	297.9	0.201	805.8	298.0	62324.73	0.000007	0.00005028	No difference
41	0.20946	750.0	19.25	298.2	0.199	742.32	296.4	62324.73	-0.000009	0.00001952	No difference
42	0.20946	751.6	72.90	301.3	0.197	739.1	296.9	62324.73	-0.000038	0.00007337	No difference
43	0.20946	742.4	70.24	303.0	0.209	730.8	296.9	62324.73	0.000001	0.00007241	No difference
44	0.20946	750.5	123.67	300.6	0.186	722.7	297.4	62324.73	-0.000141	0.00012212	O ₂ Depleted
45	0.20946	756.9	37.51	298.5	0.21	782.5	297.4	62324.73	0.000013	0.00004130	No difference
46	0.20946	763.9	38.12	298.9	0.211	788.3	296.9	62324.73	0.000015	0.00004253	No difference
47	0.20946	749.7	18.96	297.9	0.073	1197.3	370.0	62324.73	-0.000088	0.00002573	O ₂ Depleted
48	0.20946	752.4	71.59	297.7	0.227	1095.2	336.7	62324.73	0.000240	0.00010292	O ₂ Generated
49	0.20946	744.4	69.90	297.8	0.297	1092.7	333.9	62324.73	0.000503	0.00013241	O ₂ Generated
50	0.20946	748.8	122.76	298.2	0.189	736	298.4	62324.73	-0.000118	0.00012278	No difference
51	0.20946	755.7	18.74	298.1	0.199	753.4	298.1	62324.73	-0.000008	0.00001917	No difference
52	0.20946	762.5	45.15	298.8	0.2014	747.9	298.4	62324.73	-0.000022	0.00004616	No difference
53	0.20946	754.9	18.74	297.6	0.13	872.6	338.2	62324.73	-0.000059	0.00002052	O ₂ Depleted
54	0.20946	754.7	31.56	298.1	0.2	775.2	315.6	62324.73	-0.000020	0.00003153	No difference
55	0.20946	743.3	71.49	298.4	0.19	744.9	298.1	62324.73	-0.000054	0.00007213	No difference
56	0.20946	759.8	37.50	298.4	0.2	774.6	297.5	62324.73	-0.000008	0.00003931	No difference
57	0.20946	774.9	18.49	298.3	0.101	1048.9	300.6	62324.73	-0.000057	0.00002561	O ₂ Depleted
58	0.20946	750.0	31.27	298.8	0.00126	750.3	299.3	62324.73	-0.00000594	0.00003234	No difference
59	0.20946	742.4	59.55	298.2	0.162	1019	322.6	62324.73	-0.000009	0.00007296	No difference

Table 4 repeats the conclusions from Table 3 and adds the results from three more approaches to the error propagation for equation (1). The columns of Table 4 headed "Correlations With Uncertainties" reflects the results of an error propagation using the information of Table 2 and assuming perfect positive correlations between the biases for the two pressure measurements, between the biases for the two temperature measurements, between the random errors for the two pressure measurements, and between the random errors for the two temperature measurements. The next pair of columns shows the results from the

approach of assuming these same correlations but reducing the uncertainties of Table 2 by a factor of 2. The last pair shows the results from the error propagation assuming these same correlations and reducing the uncertainties of Table 2 by a factor of 5. For these correlated situations, the minimum of the relative and absolute error models is selected to represent each and every uncertainty term of equations (3) and (4) involved in the error propagation.

Table 4: Comparisons Among The Error Propagation Approaches

Test	r	Bounding Situation		Perfectly Correlated Situation		Correlated With Uncertainties Reduced by a Factor of 2		Correlated With Uncertainties Reduced by a Factor of 5	
		95%	r value	95%	r value	95%	r value	95%	r value
		Uncertainty for r	relative to zero at 95%	Uncertainty for r	relative to zero at 95%	Uncertainty for r	relative to zero at 95%	Uncertainty for r	relative to zero at 95%
1	-0.000032	0.00003470	No difference	0.00003139	O2 Depleted	0.00001569	O2 Depleted	0.00000628	O2 Depleted
2	-0.000101	0.00006254	O2 Depleted	0.00005424	O2 Depleted	0.00002712	O2 Depleted	0.00001085	O2 Depleted
3	0.000005	0.00003222	No difference	0.00002935	No difference	0.00001468	No difference	0.00000587	No difference
4	-0.000007	0.00003182	No difference	0.00003017	No difference	0.00001509	No difference	0.00000603	O2 Depleted
5	0.000074	0.00004012	O2 Generated	0.00003407	O2 Generated	0.00001704	O2 Generated	0.00000681	O2 Generated
6	-0.000447	0.00007169	O2 Depleted	0.00003197	O2 Depleted	0.00001598	O2 Depleted	0.00000639	O2 Depleted
7	-0.000296	0.00005145	O2 Depleted	0.00002306	O2 Depleted	0.00001153	O2 Depleted	0.00000461	O2 Depleted
8	-0.000046	0.00006467	No difference	0.00006046	No difference	0.00003023	O2 Depleted	0.00001209	O2 Depleted
9	-0.000018	0.00006303	No difference	0.00005957	No difference	0.00002978	No difference	0.00001191	O2 Depleted
10	-0.000010	0.00004463	No difference	0.00004214	No difference	0.00002107	No difference	0.00000843	O2 Depleted
11	-0.000022	0.00003948	No difference	0.00003708	No difference	0.00001854	O2 Depleted	0.00000742	O2 Depleted
12	-0.000012	0.00003161	No difference	0.00003006	No difference	0.00001503	No difference	0.00000601	O2 Depleted
13	-0.000381	0.00005323	O2 Depleted	0.00002698	O2 Depleted	0.00001349	O2 Depleted	0.00000540	O2 Depleted
14	-0.000048	0.00007500	No difference	0.00006116	No difference	0.00003058	O2 Depleted	0.00001223	O2 Depleted
15	-0.000252	0.00006695	O2 Depleted	0.00004227	O2 Depleted	0.00002114	O2 Depleted	0.00000845	O2 Depleted
16	-0.000082	0.00003058	O2 Depleted	0.00002399	O2 Depleted	0.00001200	O2 Depleted	0.00000480	O2 Depleted
17	0.000003	0.00004546	No difference	0.00004217	No difference	0.00002108	No difference	0.00000843	No difference
18	0.000040	0.00004593	No difference	0.00003499	O2 Generated	0.00001750	O2 Generated	0.00000700	O2 Generated
19	-0.000061	0.00005307	O2 Depleted	0.00004669	O2 Depleted	0.00002334	O2 Depleted	0.00000934	O2 Depleted
20	-0.000458	0.00008059	O2 Depleted	0.00002909	O2 Depleted	0.00001454	O2 Depleted	0.00000582	O2 Depleted
21	-0.000155	0.00002898	O2 Depleted	0.00001593	O2 Depleted	0.00000797	O2 Depleted	0.00000319	O2 Depleted
22	-0.000099	0.00004357	O2 Depleted	0.00003370	O2 Depleted	0.00001685	O2 Depleted	0.00000674	O2 Depleted
23	-0.000112	0.00003417	O2 Depleted	0.00002258	O2 Depleted	0.00001129	O2 Depleted	0.00000452	O2 Depleted
24	-0.000022	0.00004545	No difference	0.00004266	No difference	0.00002133	O2 Depleted	0.00000853	O2 Depleted
25	-0.000051	0.00001633	O2 Depleted	0.00001018	O2 Depleted	0.00000509	O2 Depleted	0.00000204	O2 Depleted
26	-0.000053	0.00002617	O2 Depleted	0.00002098	O2 Depleted	0.00001049	O2 Depleted	0.00000420	O2 Depleted
28	0.000153	0.00009085	O2 Generated	0.00007288	O2 Generated	0.00003644	O2 Generated	0.00001458	O2 Generated
29	-0.000143	0.00001726	O2 Depleted	0.00001366	O2 Depleted	0.00000683	O2 Depleted	0.00000273	O2 Depleted
30	-0.000132	0.00006300	O2 Depleted	0.00005112	O2 Depleted	0.00002556	O2 Depleted	0.00001022	O2 Depleted
31	-0.000002	0.00002169	No difference	0.00002024	No difference	0.00001012	No difference	0.00000405	No difference
32	-0.000287	0.00009684	O2 Depleted	0.00006468	O2 Depleted	0.00003234	O2 Depleted	0.00001294	O2 Depleted
33	-0.000013	0.00003982	No difference	0.00003755	No difference	0.00001878	No difference	0.00000751	O2 Depleted
34	-0.000013	0.00004072	No difference	0.00003838	No difference	0.00001919	No difference	0.00000768	O2 Depleted
35	-0.000040	0.00005432	No difference	0.00004931	No difference	0.00002466	O2 Depleted	0.00000986	O2 Depleted
36	-0.000366	0.00026301	O2 Depleted	0.00003163	O2 Depleted	0.00001582	O2 Depleted	0.00000633	O2 Depleted
37	-0.000003	0.00002739	No difference	0.00002605	No difference	0.00001302	No difference	0.00000521	No difference
38	-0.000104	0.00004835	O2 Depleted	0.00003480	O2 Depleted	0.00001740	O2 Depleted	0.00000696	O2 Depleted
39	0.000000	0.00004889	No difference	0.00004501	No difference	0.00002251	No difference	0.00000900	No difference
40	0.000007	0.00005028	No difference	0.00004764	No difference	0.00002382	No difference	0.00000953	No difference
41	-0.000009	0.00001952	No difference	0.00001849	No difference	0.00000924	No difference	0.00000370	O2 Depleted
42	-0.000038	0.00007337	No difference	0.00006895	No difference	0.00003447	O2 Depleted	0.00001379	O2 Depleted
43	0.000001	0.00007241	No difference	0.00006652	No difference	0.00003326	No difference	0.00001330	No difference
44	-0.000141	0.00012212	O2 Depleted	0.00010926	O2 Depleted	0.00005463	O2 Depleted	0.00002185	O2 Depleted
45	0.000013	0.00004130	No difference	0.00003771	No difference	0.00001885	No difference	0.00000754	O2 Generated
46	0.000015	0.00004253	No difference	0.00003865	No difference	0.00001933	No difference	0.00000773	O2 Generated
47	-0.000088	0.00002573	O2 Depleted	0.00001215	O2 Depleted	0.00000607	O2 Depleted	0.00000243	O2 Depleted
48	0.000240	0.00010292	O2 Generated	0.00008658	O2 Generated	0.00004329	O2 Generated	0.00001732	O2 Generated
49	0.000503	0.00013241	O2 Generated	0.00008592	O2 Generated	0.00004296	O2 Generated	0.00001718	O2 Generated
50	-0.000118	0.00012278	No difference	0.00011134	O2 Depleted	0.00005567	O2 Depleted	0.00002227	O2 Depleted
51	-0.000008	0.00001917	No difference	0.00001815	No difference	0.00000907	No difference	0.00000363	O2 Depleted
52	-0.000022	0.00004616	No difference	0.00004361	No difference	0.00002181	No difference	0.00000872	O2 Depleted
53	-0.000059	0.00002052	O2 Depleted	0.00001369	O2 Depleted	0.00000685	O2 Depleted	0.00000274	O2 Depleted
54	-0.000020	0.00003153	No difference	0.00002994	No difference	0.00001497	O2 Depleted	0.00000599	O2 Depleted
55	-0.000054	0.00007213	No difference	0.00006578	No difference	0.00003289	O2 Depleted	0.00001316	O2 Depleted
56	-0.000008	0.00003931	No difference	0.00003739	No difference	0.00001870	No difference	0.00000748	O2 Depleted
57	-0.000057	0.00002561	O2 Depleted	0.00001397	O2 Depleted	0.00000698	O2 Depleted	0.00000279	O2 Depleted
58	-0.00000594	0.00003138	No difference	0.00003015	No difference	0.00001508	No difference	0.00000603	No difference
59	-0.000009	0.00007296	No difference	0.00005799	No difference	0.00002900	No difference	0.00001160	No difference

Table 5 summarizes the results from the various approaches to propagating the errors of Table 2 into equation (1). This table identifies those tests (by number) that reveal an O₂ change significantly nonzero (at the 95% confidence level). The average of the uncertainties across all of the tests is also provided for each situation.

Table 5: Summary Information from Error Propagations

Description of Propagation Approach	Tests showing an increased O ₂ level	Tests showing a decreased O ₂ level	Average Uncertainty in O ₂ Change (moles)
Bounding Case Using Maximum of Relative versus Absolute Errors	5, 28, 48, 49	2, 6, 7, 13, 15, 16, 19-23, 25, 26, 29, 30, 32, 36, 38, 44, 47, 53, 57	0.00005541
Correlated Errors Using Minimum of Relative versus Absolute Errors	5, 18, 28, 48, 49	1, 2, 6, 7, 13, 15, 16, 19-23, 25, 26, 29, 30, 32, 36, 38, 44, 47, 50, 53, 57	0.00004151
Correlated Errors Using Minimum of Relative versus Absolute Errors with Initial Uncertainties Reduced by a Factor of 2	5, 18, 28, 48, 49	1, 2, 6-8, 11, 13-16, 19-26, 29, 30, 32, 35, 36, 38, 42, 44, 47, 50, 53-55, 57	0.00002075
Correlated Errors Using Minimum of Relative versus Absolute Errors with Initial Uncertainties Reduced by a Factor of 5	5, 18, 28, 45, 46, 48, 49	1, 2, 4, 6-16, 19-26, 29, 30, 32-36, 38, 41, 42, 44, 47, 50-57	0.00000830

In comparing the test conditions indicated in Table A2 to the anticipated operational window for the 9975 package, several of the tests are deemed unrepresentative of the operational conditions or are considered to have led to questionable outcomes. The tests deemed unrepresentative include 29, 35, 40, 46, and 52 (due to under injection of the sample in the gas chromatography measurement), 5 and 18 (due to excessive moisture), and 28, 35, 42, 43, 48, and 49 (due to the samples not having sand, slag, and crucible, SS&C, components).

Exhibit A7 in the Appendix provides a plot the O₂ changes for those remaining tests that are considered to be representative of the 9975-package operational conditions. Also, shown on this plot are the corresponding upper uncertainty limits for these oxygen differences. This plot shows that none of these tests have O₂ changes indicating significant (at the 95% confidence level) increases in oxygen.

The average O₂ change for these tests is -0.00009 moles, and the average uncertainties corresponding to the error propagation approaches of Table 5 range from a high of 0.000053 moles to a low of 0.0000094 moles. Expressed as relative uncertainties, these high and low values are 59% and 10%, respectively.

Exhibit A8 in the Appendix provides a plot of the initial versus final moles of oxygen for these tests. A paired t-test for these data (assuming that these test results are representative of --- i.e., a random sample from --- the intended operational window for the 9975) is also provided in this exhibit. This statistical test reveals that the null hypothesis of a zero mean difference between the initial

and final moles of oxygen (final – initial) for these experiments is strongly rejected (the probability of obtaining these results by chance alone is 1 in 100,000 if there is no difference in the quantity of O_2) in favor of the hypothesis that the mean oxygen difference is less than zero; i.e., in favor of the hypothesis that on average oxygen is depleted for these tests.

CONCLUSIONS

A review of the oxygen data from the gas generation tests supporting the 9975 SARP has been conducted including the development of an error propagation of the measurement uncertainties into the equation for the change in moles of O_2 . This analysis revealed tests with significant (at a 95% confidence level) differences between their beginning and ending moles of O_2 based upon the measurement and analytical uncertainties assumed in this report.

REFERENCES

- [1] Livingston, Ronald R., Gas Generation Test Support for Revision of 9975 SARP: Task Technical and Quality Assurance Plan," WSRC-RP-99-00164, February 18, 1999.
- [2] SAS Institute, **JMP® Statistics and Graphics Guide, Version 3 of JMP**, SAS Institute Inc., Cary, NC, 1995.
- [3] Coleman, Hugh W. and W. Glenn Steele, Jr., **Experimentation and Uncertainty Analysis for Engineers**, John Wiley & Sons, Inc., New York, 1989.

This page intentionally left blank.

APPENDIX:

TABLES AND EXHIBITS

This page intentionally left blank.

Table A1: Test Measurements by Test Number⁴

Test	Volume (cc)	Initial Pressure (torr)	Initial Temperature (°K)	Initial Total moles	Estimate Initial N ₂ moles	Estimate Initial O ₂ moles	Final Pressure (torr)	Final Temperature (°C)	Final Temperature (°K)	Final Total moles	%H ₂	%O ₂	%N ₂	Final H ₂ moles	Final O ₂ moles	Final N ₂ moles	Δ O ₂ moles (final-initial)
1	34.11	752.2	296.6	1.388E-03	1.084E-03	2.907E-04	743.2	22.7	295.7	1.376E-03	0.55	18.80	84.00	7.566E-06	2.586E-04	1.155E-03	-3.212E-05
2	63.86	741.9	294.7	2.579E-03	2.014E-03	5.403E-04	711.8	24.2	297.2	2.454E-03	0.00	17.90	84.50	0.000E+00	4.393E-04	2.074E-03	-1.010E-04
3	30.50	740.9	299.3	1.211E-03	9.459E-04	2.537E-04	741.3	22.9	295.9	1.226E-03	0.00	21.10	82.30	0.000E+00	2.587E-04	1.009E-03	4.942E-06
4	30.90	748.3	298.5	1.243E-03	9.705E-04	2.603E-04	751.5	22.4	295.4	1.261E-03	0.00	20.10	82.50	0.000E+00	2.535E-04	1.041E-03	-6.814E-06
5	29.81	755.5	297.3	1.215E-03	9.491E-04	2.546E-04	891.3	22.5	295.5	1.443E-03	10.80	22.80	61.70	1.558E-04	3.289E-04	8.901E-04	7.434E-05
6	62.33	775.7	295.5	2.625E-03	2.050E-03	5.499E-04	774.7	21.5	294.5	2.631E-03	4.20	3.90	81.60	1.105E-04	1.026E-04	2.147E-03	-4.473E-04
7	49.71	649.4	296.2	1.749E-03	1.365E-03	3.663E-04	808.1	105.5	331.0	1.947E-03	3.70	3.60	87.00	7.206E-05	7.011E-05	1.694E-03	-2.962E-04
8	64.10	750.2	295.6	2.610E-03	2.038E-03	5.467E-04	735.1	22.7	295.7	2.557E-03	0.05	19.60	86.00	1.278E-06	5.011E-04	2.199E-03	-4.560E-05
9	62.91	734.5	299.1	2.479E-03	1.936E-03	5.192E-04	733.3	25.2	298.2	2.482E-03	0.08	20.20	85.10	1.986E-06	5.014E-04	2.112E-03	-1.780E-05
10	43.46	746.5	298.5	1.744E-03	1.362E-03	3.653E-04	746.5	22.8	295.8	1.760E-03	0.62	20.20	83.80	1.091E-05	3.555E-04	1.475E-03	-9.794E-06
11	38.51	755.7	296.4	1.575E-03	1.230E-03	3.300E-04	749.7	22.9	295.9	1.566E-03	0.26	19.70	88.10	4.070E-06	3.084E-04	1.379E-03	-2.157E-05
12	30.72	756.6	297.5	1.254E-03	9.788E-04	2.626E-04	753.7	23.1	296.1	1.255E-03	0.54	20.00	84.80	6.775E-06	2.509E-04	1.064E-03	-1.164E-05
13	51.66	762.3	302.1	2.092E-03	1.633E-03	4.381E-04	655.9	22.9	295.9	1.837E-03	6.19	3.10	90.80	1.137E-04	5.696E-05	1.668E-03	-3.811E-04
14	64.37	765.1	296.3	2.667E-03	2.082E-03	5.586E-04	975.2	97.8	329.5	3.057E-03	0.89	16.70	80.00	2.721E-05	5.105E-04	2.446E-03	-4.809E-05
15	65.63	754.1	299.6	2.651E-03	2.070E-03	5.552E-04	806.3	111.7	327.6	2.592E-03	0.00	11.70	82.60	0.000E+00	3.032E-04	2.141E-03	-2.520E-04
16	30.78	760.2	296.1	1.268E-03	9.901E-04	2.656E-04	753.2	109.2	314.3	1.184E-03	0.22	15.50	78.20	2.604E-06	1.835E-04	9.256E-04	-8.211E-05
17	42.04	762.2	298.0	1.725E-03	1.347E-03	3.614E-04	779.1	24.2	297.2	1.768E-03	1.64	20.60	79.60	2.900E-05	3.643E-04	1.408E-03	2.889E-06
18	30.34	760.0	298.0	1.242E-03	9.694E-04	2.600E-04	1179.8	23.4	296.4	1.938E-03	24.15	15.50	53.10	4.680E-04	3.003E-04	1.029E-03	4.029E-05
19	52.40	751.7	298.4	2.118E-03	1.654E-03	4.436E-04	744.5	24.4	297.4	2.105E-03	0.00	18.20	83.80	0.000E+00	3.831E-04	1.764E-03	-6.057E-05
20	53.82	753.4	297.6	2.186E-03	1.707E-03	4.579E-04	1178.3	25.1	321.3	3.167E-03	80.00	0.00	23.60	2.534E-03	0.000E+00	7.474E-04	-4.579E-04
21	25.98	744.0	298.4	1.039E-03	8.115E-04	2.177E-04	803.2	94.7	323.0	1.037E-03	2.60	6.00	95.60	2.695E-05	6.220E-05	9.910E-04	-1.555E-04
22	43.03	749.4	298.3	1.734E-03	1.354E-03	3.633E-04	742.1	25	298.0	1.719E-03	0.13	15.40	86.30	2.235E-06	2.648E-04	1.484E-03	-9.853E-05
23	31.95	758.2	299.0	1.300E-03	1.015E-03	2.723E-04	806.3	96.9	307.8	1.343E-03	1.60	11.90	88.60	2.149E-05	1.598E-04	1.190E-03	-1.125E-04
24	43.83	764.4	298.1	1.803E-03	1.408E-03	3.777E-04	764.3	24.8	297.8	1.805E-03	5.00	19.70	76.60	9.024E-05	3.556E-04	1.383E-03	-2.216E-05
25	14.43	749.5	295.9	5.865E-04	4.579E-04	1.228E-04	800.2	92.6	324.7	5.705E-04	2.17	12.60	82.70	1.238E-05	7.189E-05	4.718E-04	-5.095E-05
26	25.67	752.6	296.2	1.047E-03	8.172E-04	2.192E-04	789.2	94.4	317.1	1.025E-03	1.19	16.20	79.40	1.220E-05	1.661E-04	8.140E-04	-5.312E-05
28	68.91	758.2	296.7	2.825E-03	2.206E-03	5.918E-04	829.8	24.9	297.9	3.080E-03	1.82	24.20	68.20	5.605E-05	7.453E-04	2.100E-03	1.535E-04
29	27.47	768.4	371.7	9.112E-04	7.115E-04	1.909E-04	341.5	99.3	419.5	3.588E-04	0.46	13.30	85.80	1.650E-06	4.772E-05	3.078E-04	-1.431E-04
30	63.26	755.8	297.8	2.576E-03	2.011E-03	5.396E-04	724.6	24.6	297.6	2.471E-03	1.27	16.50	75.80	3.139E-05	4.078E-04	1.873E-03	-1.318E-04
31	26.22	733.8	369.5	8.355E-04	6.524E-04	1.750E-04	738.8	94.5	367.5	8.457E-04	0.03	20.50	76.00	2.537E-07	1.734E-04	6.428E-04	-1.621E-06
32	91.65	753.9	297.5	3.726E-03	2.910E-03	7.805E-04	772.2	23.9	296.9	3.825E-03	8.13	12.90	78.50	3.109E-04	4.934E-04	3.002E-03	-2.872E-04
33	38.54	760.0	298.8	1.573E-03	1.228E-03	3.294E-04	751.8	23.8	296.8	1.566E-03	1.60	20.20	76.40	2.506E-05	3.164E-04	1.197E-03	-1.304E-05
34	38.98	768.9	298.9	1.609E-03	1.256E-03	3.370E-04	760.5	23.9	296.9	1.602E-03	0.20	20.20	76.90	3.204E-06	3.236E-04	1.232E-03	-1.339E-05
35	53.04	745.8	296.4	2.141E-03	1.672E-03	4.485E-04	755.3	24.4	297.4	2.161E-03	0.00	18.90	55.20	0.000E+00	4.085E-04	1.193E-03	-4.003E-05
36	61.32	748.5	297.9	2.472E-03	1.930E-03	5.178E-04	3843.2	106.3	324.5	1.165E-02	74.70	1.30	17.90	8.703E-03	1.515E-04	2.086E-03	-3.663E-04
37	26.49	741.6	297.4	1.060E-03	8.276E-04	2.220E-04	813.1	106.1	316.1	1.093E-03	0.40	20.00	64.20	4.373E-06	2.187E-04	7.019E-04	-3.334E-06
38	44.90	744.9	297.4	1.804E-03	1.409E-03	3.780E-04	802.2	24.9	297.9	1.940E-03	7.40	14.10	77.70	1.436E-04	2.735E-04	1.507E-03	-1.044E-04
39	46.04	752.4	297.4	1.869E-03	1.459E-03	3.915E-04	758.3	24.8	297.8	1.881E-03	6.50	20.80	72.10	1.223E-04	3.913E-04	1.356E-03	-2.066E-07
40	46.27	759.7	297.9	1.893E-03	1.478E-03	3.966E-04	805.8	25.0	298.0	2.007E-03	3.00	20.10	54.40	6.022E-05	4.035E-04	1.092E-03	6.940E-06
41	19.25	750.0	298.2	7.769E-04	6.066E-04	1.627E-04	742.32	23.4	296.4	7.736E-04	0.70	19.90	81.20	5.415E-06	1.539E-04	6.282E-04	-8.780E-06
42	72.90	751.6	301.3	2.918E-03	2.278E-03	6.112E-04	739.1	23.9	296.9	2.912E-03	0.10	19.70	80.00	2.912E-06	5.736E-04	2.330E-03	-3.754E-05
43	70.24	742.4	303.0	2.761E-03	2.156E-03	5.784E-04	730.8	23.9	296.9	2.774E-03	0.00	20.90	79.50	0.000E+00	5.798E-04	2.205E-03	1.384E-06
44	123.67	750.5	300.6	4.954E-03	3.868E-03	1.038E-03	722.7	24.4	297.4	4.822E-03	0.20	18.60	82.40	9.644E-06	8.969E-04	3.973E-03	-1.408E-04
45	37.51	756.9	298.5	1.526E-03	1.192E-03	3.196E-04	782.5	24.4	297.4	1.583E-03	2.30	21.00	76.80	3.642E-05	3.325E-04	1.216E-03	1.289E-05

⁴ Test 27 was unsuccessful due to equipment failure.

Table A1: Test Measurements by Test Number (continued)

Test	Volume (cc)	Initial Pressure (torr)	Initial Temperature (°K)	Initial Total moles	Estimate Initial N ₂ moles	Estimate Initial O ₂ moles	Final Pressure (torr)	Final Temperature (°C)	Final Temperature (°K)	Final Total moles	%H ₂	%O ₂	%N ₂	Final H ₂ moles	Final O ₂ moles	Final N ₂ moles	Δ O ₂ moles (final-initial)
46	38.12	763.9	298.9	1.563E-03	1.221E-03	3.274E-04	788.3	23.9	296.9	1.624E-03	1.20	21.10	62.10	1.949E-05	3.427E-04	1.009E-03	1.524E-05
47	18.96	749.7	297.9	7.654E-04	5.977E-04	1.603E-04	1197.3	97	370.0	9.842E-04	3.40	7.30	81.80	3.346E-05	7.185E-05	8.051E-04	-8.847E-05
48	71.59	752.4	297.7	2.903E-03	2.267E-03	6.081E-04	1095.2	63.7	336.7	3.736E-03	0.00	22.70	74.00	0.000E+00	8.480E-04	2.765E-03	2.399E-04
49	69.90	744.4	297.8	2.804E-03	2.189E-03	5.872E-04	1092.7	60.9	333.9	3.670E-03	1.50	29.70	66.40	5.506E-05	1.090E-03	2.437E-03	5.029E-04
50	122.76	748.8	298.2	4.946E-03	3.862E-03	1.036E-03	736	25.4	298.4	4.858E-03	0.40	18.90	79.20	1.943E-05	9.182E-04	3.848E-03	-1.178E-04
51	18.74	755.7	298.1	7.623E-04	5.952E-04	1.597E-04	753.4	25.1	298.1	7.599E-04	1.50	19.90	73.40	1.140E-05	1.512E-04	5.578E-04	-8.435E-06
52	45.15	762.5	298.8	1.848E-03	1.443E-03	3.872E-04	747.9	25.4	298.4	1.815E-03	0.55	20.14	78.36	9.985E-06	3.656E-04	1.423E-03	-2.154E-05
53	18.74	754.9	297.6	7.627E-04	5.956E-04	1.598E-04	872.6	65.2	338.2	7.758E-04	1.32	13.00	85.00	1.024E-05	1.009E-04	6.594E-04	-5.891E-05
54	31.56	754.7	298.1	1.282E-03	1.001E-03	2.686E-04	775.2	42.6	315.6	1.244E-03	0.00	20.00	74.00	0.000E+00	2.488E-04	9.205E-04	-1.977E-05
55	71.49	743.3	298.4	2.857E-03	2.231E-03	5.985E-04	744.9	25.1	298.1	2.866E-03	0.36	19.00	84.00	1.032E-05	5.446E-04	2.408E-03	-5.389E-05
56	37.50	759.8	298.4	1.532E-03	1.196E-03	3.209E-04	774.6	24.5	297.5	1.567E-03	0.25	20.00	84.00	3.917E-06	3.133E-04	1.316E-03	-7.580E-06
57	18.49	774.9	298.3	7.707E-04	6.018E-04	1.614E-04	1048.9	27.6	300.6	1.035E-03	1.80	10.10	81.50	1.863E-05	1.046E-04	8.437E-04	-5.687E-05
58	31.27	750.0	298.8	1.259E-03	9.83E-04	2.64E-04	750.3	26.3	299.3	1.258E-03	0.00	20.50	78.10	0.000E+00	2.58E-04	9.82E-04	-5.945E-06
59	59.55	742.4	298.2	2.379E-03	1.857E-03	4.983E-04	1019	49.6	322.6	3.018E-03	0.70	16.20	81.60	2.113E-05	4.890E-04	2.463E-03	-9.281E-06

Table A2: Description of 9975 Test by Test Number⁵

Run #	Description	Time (days)	Container Volume	Grams PuO ₂	Bulk Weight(g)	Measured Volume	Temp (°C)	Run #	Sample #	H ₂ (vol%)	O ₂ (vol%)	N ₂ (vol%)	CO ₂ (vol%)	N ₂ O (vol%)	CO (vol%)	sum (vol%)
2/20/99 Start -- Set 1																
1	PuO ₂ + RF MgO sand & crucible	8.83	25cc	9.59	37.83	34.11	ambient	1	3-123637	0.55	18.8	84	0	0	0	103.35
2	PuO ₂ + calcined CaO + Ca(OH) ₂	8.92	50cc	8.85	35.80	63.86	ambient	2	3-123636	0.00	17.9	84.5	0	0	0	102.40
3	PuO ₂ Stored over dessicant	9.65	5cc	7.65	7.65	30.50	ambient	3	3-123635	0.00	21.1	82.3	0.05	0	0	103.45
4	PuO ₂ stored in 76%RH humidior	9.71	5cc	8.54	8.54	30.90	ambient	4	3-123634	0.00	20.1	82.5	0.16	0	0	102.76
5	PuO ₂ ~ 10% H ₂ O added	6.66	5cc	7.57	7.57	29.81	ambient	5	3-123633	10.80	22.8	61.7	0.1	0	0	95.40
6	Standard Gas	3.73	50cc	0.00	0.00	62.33	ambient	6	3-123638	4.20	3.9	81.6	1.8	0.5	1.2	93.20
3/1/99 Start -- Set 2																
7	Standard Gas + Heat	6.31	50cc	0.00	0.00	49.71	90	7	3-123639	3.70	3.6	87	NR	NR	4.06	98.36
8	#2 stored at 100% RH	6.14	50cc	7.73	31.30	64.10	ambient	8	3-123640	0.05	19.6	86	0.1	0	0	105.75
9	PuO ₂ + 30g CaF ₂	6.83	50cc	9.83	39.85	62.91	ambient	9	3-123641	0.08	20.2	85.1	0.09	0.02	0	105.49
10	PuO ₂ +Ca(OH) ₂ & MgO Crucible	5.75	25cc	7.90	15.80	43.46	ambient	10	3-123642	0.62	20.2	83.8	0.06	0	0	104.68
11	#1 stored at 100%RH	6.12	25cc	9.32	36.76	38.51	ambient	11	3-123643	0.26	19.7	88.1	0.05	0.04	4.28	112.43
12	PuO ₂ stored at 100%RH	6.82	5cc	9.52	9.52	30.72	ambient	12	3-123644	0.54	20	84.8	0.12	0.12	0	105.58
3/8/99 Start -- Set 3																
13	44g of RF SS&C sample H6	7.88	50cc	15.50	43.84	51.66	ambient	13	3-123645	6.19	3.1	90.8	0.03	1.6	0	101.72
14	#9 heated	7.88	50cc	9.83	39.85	64.37	90	14	3-123646	0.89	16.7	80	5.2	0.25	0	103.04
15	#8 heated	8.16	50cc	7.73	31.30	65.63	90	15	3-123647	0.00	11.7	82.6	0	0	0	94.30
16	PuO ₂ stored at 100%RH + Heat	7.77	5cc	7.26	7.26	30.78	90	16	3-123648	0.22	15.5	78.2	0.48	0.04	0	94.44
17	Pu Cake (no added moisture)	7.61	25cc	32.01	35.66	42.04	ambient	17	3-123649	1.64	20.6	79.6	0.06	0.05	0	101.95
18	#5 mixed with 2g Ca metal	7.56	5cc	4.88	6.93	30.34	ambient	18	3-123650	24.15	15.5	53.1	0.03	0	0	92.78
3/17/99 Start-- Set 4																
19	CaF ₂ at RT repeat #14 and #9	6.8	50cc	9.83	39.85	52.40	ambient	19	3-123651	0.00	18.2	83.8	0.37	0.08	0	102.45
20	#13 heated	6.8	50cc	15.50	43.84	53.82	90	20	3-123652	80.00	0	23.6	0	0	0	103.60
21	#10 heated	6.81	10cc	7.50	15.00	25.98	90	21	3-123653	2.60	6	95.6	0.16	0.2	0	104.56
22	PuO ₂ +MgO 1:1 ratio	6.81	25cc	9.73	19.45	43.03	ambient	22	3-123654	0.13	15.4	86.3	0.09	0.11	0	102.03
23	#11 heated	6.78	10cc	4.66	20.00	31.95	90	23	3-123655	1.60	11.9	88.6	0.22	0.17	0	102.49
24	PuO ₂ + Mg(OH) ₂	6.79	25cc	7.26	17.26	43.83	ambient	24	3-123656	5.00	19.7	76.6	0.03	0.09	0	101.42
3/26/99 Start-- Set 5																
25	PuO ₂ + MgO with lo water	10.11	10cc	6.60	25.00	14.43	90	25	3-123658	2.17	12.6	82.7	<1	<1	<1	97.47
26	PuO ₂ + MgO with med water	9.74	10cc	6.24	23.43	25.67	90	26	3-123659	1.19	16.2	79.4	<1	<1	<1	96.79
4/6/99 Start-- Set 6																
30	RFETS H2	7.69	50cc	8.18	43.10	63.26	ambient	30	3-125514	1.27	16.5	75.8	<1	<1	<1	93.57
31	PuO ₂ +MgO w/ hi water+heat	8.83	10cc	#REF!	23.09	26.22	90C	31	3-125515	0.03	20.5	76	<1	<1	<1	96.53
32	RFETS H7	7.71	100cc	6.77	14.60	91.65	ambient	32	3-125516	8.13	12.9	78.5	<1	<1	<1	99.53
33	#18 repeated	7.71	10cc	4.65	6.60	38.54	ambient	33	3-125517	1.6	20.2	76.4	<1	<1	<1	98.20
34	RFETS H8	7.69	10cc	8.49	10.80	38.98	ambient	34	3-125518	0.2	20.2	76.9	<1	<1	<1	97.30
4/16/99 Start -- Set 7																
36	RFETS Sample H2	5.61	50cc	8.18	43.10	61.32	90	36	3-125520	74.70	1.3	17.9	0.02	<1	<1	93.92
37	#31 repeated	5.66	10cc	6.15	23.09	26.49	90	37	3-125521	0.40	20	64.2	0.03	0.02	<1	84.65
38	PuO ₂ + 5%H ₂ O	5.69	25cc	9.66	10.29	44.90	ambient	38	3-125522	7.40	14.1	77.7	0.08	0.04	<1	99.32
4/23/99 Start -- Set 8																
41	PuO ₂ +100%RH -2weeks	5.6	10cc	10.08	10.08	19.25	ambient	41	3-125525	0.7	19.9	81.2	NR	NR	<0.1	101.80
44	PuCake - no treatment like #17	5.77	100cc	27.65	30.80	123.67	ambient	44	3-125528	0.2	18.6	82.4	0.32	<0.1	<0.1	101.52
45	PuO ₂ + 2.5% water	5.77	10cc	12.17	12.50	37.51	ambient	45	3-125529	2.3	21	76.8	<0.1	<0.1	<0.1	100.10
46	PuO ₂ + 2.5% water	5.84	10cc	9.83	10.32	38.12	ambient	46	3-125530	1.2	21.1	62.1	<0.1	<0.1	<0.1	84.40

⁵ Samples 28, 29, 35, 40, 42, 43, 48 and 49 contained materials that do not conform to the content description of SS&C residues.

Table A2: Descriptions of Tests by Test Number (continued)

Run #	Description	Time (days)	Container Volume	Grams PuO ₂	Bulk Weight(g)	Measured Volume	Temp (°C)	Run #	Sample #	H ₂ (vol%)	O ₂ (vol%)	N ₂ (vol%)	CO ₂ (vol%)	N ₂ O (vol%)	CO (vol%)	sum (vol%)
4/29/99 Start -- Set 9																
47	PuO ₂ +2.5%H ₂ O+Heat	6.77	10cc	12.17	12.50	18.96	90	47	3-125531	3.4	7.3	81.8	<0.1	<0.1	<0.1	92.50
50	PuCake - replicate1	6.76	100cc	27.34	30.46	122.76	ambient	50	3-125534	0.4	18.9	79.2	<0.1	<0.1	<0.1	98.50
51	#41 repeated	6.76	10cc	10.08	10.08	37.11	ambient	51	3-125535	1.5	19.9	73.4	<0.1	<0.1	<0.1	94.80
52	PuCake - replicate #2	6.77	25cc	14.98	16.69	45.15	ambient	52	3-125536	0.55	20.14	78.36	<0.1	0.05	<0.1	99.10
5/18/99 Start -- Set 10																
53	#51 heated	7.95	10cc	10.08	10.08	18.74	90	53	3-127169	1.32	13	85	<0.1	<0.1	<0.1	99.32
54	Blank+Heat	7.95	10cc	0.00	0.00	31.56	90	54	3-127170	0	20	74	<0.1	<0.1	<0.1	94.00
55	PuO ₂ +13mg H ₂ O	7.95	50cc	12.57	12.70	71.49	ambient	55	3-127171	0.36	19	84	<0.1	<0.1	<0.1	103.36
56	PuO ₂ +15mg H ₂ O	7.95	10cc	10.42	10.57	37.50	ambient	56	3-127172	0.25	20	84	<0.1	<0.1	<0.1	104.25
5/26/99 Start -- Set 11																
57	PuO ₂ +15mg H ₂ O	7.75	10cc	10.42	10.57	18.49	90	57	3-127173	1.8	10.1	81.5	<0.1	<0.1	<0.1	93.40
58	Blank	8	10cc	0.00	0.00	31.27	ambient	58	3-127174	0	20.5	78.1	<0.1	<0.1	<0.1	98.60
59	PuO ₂ +13mg H ₂ O	7.78	50cc	12.57	12.70	59.55	90	59	3-127175	0.7	16.2	81.6	2.8	0.31	<0.1	101.61

Exhibit A1: A Plot of the Initial versus Final O₂ by Test Number

Exhibit A2: A Plot of the Change in O₂ (Delta O₂) by Test Number

delta O₂ (M)

(Symbols used to identify tests as defined by the categories of Table 1)

Exhibit A3: A Plot of the Change in O₂ (Delta O₂) by Type Group **Means and Std Deviations**

Level	Number	Mean	Std Dev	Excluding Heated Tests		
				Number	Mean	Std Dev
Other	39	-0.00006	0.000176			
Pu Cake	4	-0.00007	0.000071			
PuO2	4	-0.00003	0.000036	3	-0.00001	0.000002
PuO2+CaO	3	-0.00013	0.000107	2	-0.00007	0.000039
PuO2+CaF	3	-0.00004	0.000022			
PuO2+MgO	5	-0.00005	0.000035	4	-0.00004	0.000015

Exhibit A4: A Plot of the Change in O₂ (Delta O₂) by Headspace Volume (cc)

Symbols are shaded to designate the categories of Table 1 (i.e., they are shaded as in Exhibit A3).

Exhibit A5: A Plot of the Change in O₂ (Delta O₂) by Total Sample Volume Percent

Exhibit A6: A Plot of the Change in O₂ (Delta O₂) by Test Number with Bounding Uncertainties

Bold symbols are used to designate statistically significant (at the 95% confidence level) test results.

**Exhibit A7: A Plot of the Change in O₂ (Delta O₂) by Test Number
with Bounding Uncertainties for Representative Tests**

**Exhibit A8: A Plot of the Initial Versus Final Moles of Oxygen
for Representative Tests****initial O2 (M) By final O2 (M)**

≡ Paired t-Test

Paired t-Test

final O2 (M) - initial O2 (M)

Mean Difference	-0.00009	Prob > t	0.0000080
Std Error	0.000018	Prob > t	0.9999960
t-Ratio	-5.0429	Prob < t	0.0000040
DF	45		

Symbols are shaded to designate the categories of Table 1 (i.e., they are shaded as in Exhibit A3).

Distribution

N. M. Askew, 773-A
A. L. Blancett, 773-A
P. S. Blanton, 773-54A
N. E. Bibler, 773-A
M. A. Ebra, 773-42A
T. B. Edwards, 773-42A
F. R. Graham, 773-A
S. J. Hensel, 773-42A
S. M. King, 773-54A
R. R. Livingston, 773-A
J. E. Marra, 704-T
D. B. Moore-Shedrow, 773-A
A. M. Murray, 773-A
J. B. Shaade, 703-F
R. C. Tuckfield, 773-42A
J. H. Weber, 773-42A
J. C. Williams, 707-F
C. R. Wolfe, 773-A
D. C. Wood, 704-F
J. E. Young, 773-A