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Test Results for Implementation of Hydrogen Getter in the DDF-1 Shipping Package

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Executive Summary

Implementation of a hydrogen getter for transportation of radioactive materials in the DDF-1 shipping package will support on-site transfers of radioactive materials (e.g., mixed oxide scraps) without extensive characterization or moisture analysis. The hydrogen getter developed for this application is an assemblage of commercial components that have been tested by the Savannah River Technology Center (SRTC) to demonstrate the required performance characteristics. The essential characteristics and test requirements were established for the hydrogen getter as part of a Task Technical and Quality Assurance Plan[1] issued prior to initiating getter assembly testing.

The test results for the DDF-1 hydrogen getter demonstrate:

- The getter assembly, consisting of a commercial polymer getter (140 grams), 4Å molecular sieve (100 grams) and porous nylon packaging is capable of maintaining the hydrogen pressure in the DDF-1 Primary Containment Vessel (PCV) at less than one volume percent (vol.%) in either an air or nitrogen environment.

- The getter assembly is capable of removing hydrogen at greater than two times the previously specified rate of 14 standard cubic centimeters per hour (std. cm^3 hr^{-1}) and has sufficient capacity for removing 1.5 times the amount of hydrogen associated with 30 days of continuous service at the specified rate.

Consequently, performance of the DDF-1 getter assembly provides a substantial safety margin by maintaining the hydrogen gas concentration below the lower flammable limit even under conditions that exceed the previously established content limits.

The maximum theoretical hydrogen generation rate for an aqueous solution containing one watt of radioactive material is 14 std. cm^3 hr^{-1}. This rate served as the basis for getter design. The maximum observed hydrogen generation rate[2] for weapons grade plutonium dioxide containing between 5 and 10% water is 1.4 std. cm^3 hr^{-1}W^{-1}. This rate is ten times lower than the getter design basis and higher than what is expected for materials to be shipped in the DDF-1.[3] Based on the demonstrated performance of the DDF-1 getter assembly, the nominal one-watt radioactive material content could be safely expanded to higher wattage by using more realistic estimates for the content's hydrogen generation rate.

The conclusions reached as a result of this effort and dialog with site experts in the related fields are summarized as follows:

- The current DDF-1 getter design is capable of removing hydrogen at greater than 30 std. cm^3 hr^{-1} for up to a total of 15,120 std. cm^3 (i.e., 90% of theoretical capacity). This performance should guide definition of limits for content wattage (based on the estimated hydrogen generation rate) and shipping time established during preparation of the DDF-1 On-Site Safety Assessment (OSA).

- The DDF-1 getter is not significantly impacted by the type or number of produce cans transported in the DDF-1 PCV. The PCV will easily accommodate the current getter

assembly along with one seven-inch and one five-inch tall produce can. With removal of the aluminum honeycomb impact limiter found at the bottom of the PCV, two seven-inch tall produce cans and the current getter assembly may be accommodated within the PCV. The getter assembly, as packaged for these tests, is approximately four inches wide, six inches tall, and 1.25 inches thick. Changing the position in the PCV or the shape of this getter assembly is not expected to affect getter performance.

- Establishing a hydrogen generation rate for the DDF-1 contents consistent with current expectations of the radioactive materials moisture content (i.e., no free liquid and less than 5% moisture) will support increasing the DDF-1 wattage limit. Based on a hydrogen generation rate of 5.6 std. cm^3 hr^{-1} W^{-1} (i.e., four times the maximum anticipated gas generation rate), the current getter assembly could support a 3.5-watt payload for a 30-day shipping window.

- Once the hydrogen generation rate for the DDF-1 content has been established (e.g., 5.6 std. cm^3 hr^{-1} W^{-1}), the payload must be limited to prevent hydrogen generation rates from exceeding 30 std. cm^3 hr^{-1}, and the shipping window duration must be restricted to prevent exceeding 15,120 std. cm^3 of hydrogen. For example, with two watts of radioactive material, the estimated bounding gas generation rate is 11.2 std. cm^3 hr^{-1}, and the shipping time could be extended to 56 days. Likewise, if a five-watt payload is to be shipped (~28 std. cm^3 hr^{-1}) the shipping duration is decreased accordingly to about 20 days (i.e., 15,120 std. cm^3/ (28 std. cm^3 hr^{-1} × 24 hr day^{-1}) = 22.5 days).

- The polymer getter selected for use in the DDF-1 has been demonstrated over a broad temperature range (-10 to 70°C) and is resistant to all expected poison vapors. Stability of the polymer getter material at 70°C was established as part of testing for getter use in the TRUPACT-II shipping package. This temperature range supports increasing the DDF-1 content wattage up to a point where the getter would reach 70°C. With revision of the estimated hydrogen generation rate, temperature is likely to become the limiting factor for implementation of the current hydrogen getter.

- No special handling of the getter assembly is required. To maintain the product shelf life, the getter assembly should be stored in a cool, dry location and opened just prior to installation in the DDF-1 PCV.

Introduction

Hydrogen gas may be produced by radioactive materials as a result of ordinary chemical reactions like corrosion of metals or radiation induced decomposition of hydrogenous materials (e.g. radiolysis of water or plastic). Previous testing at SRTC has demonstrated the difficulty of estimating hydrogen generation rates for impure plutonium materials based on moisture content.[2] Consequently the estimated hydrogen generation rates for contents in the DDF-1 are based on the maximum g-value for plutonium found in aqueous solutions.[4] This leads to a predicted hydrogen generation rate that is about 10 times the highest observed value for ~5% moisture adsorbed on plutonium dioxide.[2]

The getter assembly is able to function in either oxygen-containing or oxygen-deficient atmospheres. When oxygen is present, the getter assembly functions primarily as a recombination catalyst, and the 4Å molecular sieve adsorbs any water produced. During storage of radioactive materials, depletion of the oxygen gas from a container headspace is frequently observed. When oxygen is depleted from the DDF-1 headspace gas, the getter removes hydrogen primarily by hydrogenation of a butadiene polymer. Sufficient polymer is present to provide the getter assembly's rated capacity, and any hydrogen removed by recombination does not consume getter capacity.

The Task Technical and QA Plan developed for these tests provides a description of the necessary tests and related QA controls. These tests demonstrate that the getter assembly exceeds the minimum requirements for on-site transportation of radioactive materials. The getter performance requirements presented in the Task Technical and QA Plan are as follows:

- Maintain the hydrogen pressure of the DDF-1 PCV at less than 2 psia (~100 Torr) hydrogen in the presence of air or nitrogen. This pressure limit keeps the hydrogen concentration in the PCV at less than 12 volume percent (vol.%), assuming the PCV is packaged initially at one atmosphere pressure.

- Provide sufficient hydrogen removal rate and capacity for up to 14 std. cm^3 hr^{-1} over a period of 30 days. This hydrogen generation rate is calculated based on one watt of radioactive material producing hydrogen with an effective g-value of 1.6. This calculation is based on a conservative assumption that 100% of the alpha decay energy is absorbed by water.

The 12 vol.% hydrogen limit assures that the minimum cell size dimension for a hydrogen deflagration to detonation transition is sufficiently large to prevent a detonation in the DDF-1 package. The lower flammable limit (LFL) for hydrogen in air is considered to be 4 vol.%. To eliminate the potential for a hydrogen deflagration in the DDF-1 PCV, a hydrogen getter would need to maintain the hydrogen concentration below the LFL, or below about 0.6 psia (~30 Torr) at atmospheric pressure.

Five series of tests are described in the Task Technical and QA Plan for getter assembly evaluation. These include tests to validate hydrogen removal rate and capacity requirements, tests to show the effect of air or nitrogen headspace gas on hydrogen removal, tests to show getter assembly sensitivity to PCV configuration, and tests to demonstrate uniformity of the getter assemblies packaged as part of this test. Additional tests were added to improve temperature correction of pressure measurements used to estimate hydrogen concentration in the PCV during execution of these tests. The additional tests included an extended (> 20 hours) hydrogen flow test with the getter assembly loaded to 80% of theoretical capacity and two tests run to establish baseline PCV pressure changes as a function of temperature. These baseline tests were conducted because temperature corrections for PCV pressure based on the ideal gas law were unsatisfactory. Data from these tests were subsequently used to improve PCV pressure measurements.

In addition to a presentation and discussion of test results for the getter assembly, this report gives details of the test apparatus set-up and calibration, along with information on getter assembly fabrication.

Background

The development of a hydrogen getter for use in on-site transportation of radioactive materials has been funded by the Nuclear Materials Stabilization and Storage division as part of the DDF-1 On-Site Safety Assessment (OSA). Testing of the DDF-1 getter assembly was requested as part of Task Technical Request by the SRTC Radioactive Materials Packaging Technology Group.[5] Testing done to support development of the getter assembly was documented in an earlier manuscript.[6] These previous tests, along with more recent data, show that the getter functions effectively over a broad temperature range (-10°C to 70°C) with minimal effect on hydrogen removal rate, and that the getter material has good tolerance to poison vapors.

The hydrogen getter assembly designed for this application contains a commercially available polymer hydrogen getter manufactured by Vacuum Energy, Inc (VEI). This getter is placed outside the radioactive materials container (i.e. produce cans), so additional analysis is required to show that hydrogen from these containers will reach the getter. See Attachment 1, Figures 1-1 to 1-3, for pictures and drawings related to the DDF-1 design and configuration. This hydrogen getter has been evaluated previously as part of an extensive effort to examine the effects of temperature, pressure, poisons, radiation, and other factors on hydrogen removal rate and capacity.[6] The impact of vessel geometry and getter arrangement on hydrogen absorption rate were not addressed as part of the earlier study. Therefore, the purpose of this effort has been to demonstrate performance of the DDF-1 getter assembly inside an actual DDF-1 PCV to validate the getter assembly design.

Leaking hydrogen into the PCV (either a nitrogen or air atmosphere) simulates hydrogen generation within the PCV. Based on limited control of the hydrogen flow rate, this addition of hydrogen was typically at 2 to 4 times the maximum expected hydrogen generation rate of 14 std. $cm^3\ hr^{-1}$. In the absence of oxygen, the polymer hydrogen getter functions by an irreversible hydrogenation mechanism using a palladium catalyst. When oxygen is present, the polymer hydrogen getter can also function as a recombination catalyst. Therefore, the "getter assembly" contains both polymer hydrogen getter (~ 140 g) and zeolite (~ 100 g 4Å molecular sieve), to adsorb any water produced from recombination. The getter and molecular sieve are packaged separately but in close proximity to each other in a porous nylon fabric (CEREX®) that will contain the solid materials without restricting gas flow to the getter. The molecular sieve and getter are packaged separately to simplify potential recovery of the getter catalyst at a later date. Each test uses one prepackaged getter assembly to support evaluation of PCV geometry and gas composition on getter performance. The Material Safety Data Sheets (MSDS) for the polymer hydrogen getter and the molecular sieve are provided in Attachment 10.

Experimental

Hydrogen Getter Assembly Construction

The DDF-1 hydrogen getter assembly is constructed using two small CEREX® bags, which are heat sealed on three sides and have lay-flat dimensions of approximately 2.5 inches wide and 8 inches tall, and a third large CEREX® bag approximately 4 inches wide and 9 inches tall. One of the small CEREX® bags is filled with 140 g of VEI getter, and the second small bag is filled with 100 g of molecular sieve. The molecular sieve should be protected from extended exposure to moist air since it is supplied in a form guaranteed to contain less than two weight percent (wt%) moisture and will not be regenerated prior to installation in the DDF-1 PCV. Simple weight measurements are sufficient to demonstrate adequate moisture controls. Attachment 2 contains a list of suppliers for the getter assembly components and notes on the products selected. The open end of each small bag is heat-sealed to prevent spilling the contents of the bag. The length of these small bags when sealed is not viewed as critical, but should be optimized to support packaging of PCV content. These two bags are placed inside the larger CEREX® bag, which is also heat-sealed to hold the two components together. This three-bag arrangement provides an easy means for recycling the VEI getter and minimizing the potential for nuisance dust from the getter powder.

Attachment 1, Figures 1-4 and 1-5 show micrographs of VEI getter and CEREX® fabric used in assembling the DDF-1 getter assembly. Figures 1-6 to 1-10 show photographs of the molecular sieve, VEI getter, and completed getter assembly. The CEREX® fabric used is a two-ounce per square yard spunbonded nylon. A more detailed description of this product is reproduced in Attachment 3. The completed getter assemblies are stored in a foil-lined plastic bag known to have low transmission of moisture or organic vapors. This bag meets military specifications MIL-PRF-81705D, Type I. When packaged in this manner and stored in a cool, dry location, the getter assembly is anticipated to have a one-year shelf life. The heat sealer used at SRTC to prepare the test assemblies is a Vertrod (model 14HT-1/2 impulse heat sealer), which has adjustable settings for temperature and dwell time. The settings used for sealing these bags are shown in Table I.

Table I - Heat Sealer Settings for Preparation of DDF-1 Getter Assemblies

Bag Type	Heat Setting	Dwell Setting
CEREX®	7	7
Storage	3.5	7

Following heat sealing, the getter assemblies are labeled to include date of preparation and lot numbers of the getter and molecular sieve used. Each lot of getter material should be tested prior to use in preparation of getter assemblies to confirm baseline data like capacity and hydrogen removal rate in vacuum. Additional work is needed to develop future procurement specifications and qualification procedures. A separate document will be issued to provide details of how rate and capacity measurements should be made for getter qualification and to support the procurement specification. Variations that decrease either the rate or capacity of this should be examined carefully to assure getter performance is not negatively impacted. Increases in rate or capacity are beneficial and are not a concern for use in a DDF-1 getter assembly.

Description of the Test Apparatus

The experimental apparatus is constructed of an actual DDF-1 PCV (~ 5.1-L internal volume) modified with stainless steel tubing and fittings to allow for gas introduction, evacuation, and sampling. See Attachment 1, Figures 1-11 to 1-20 for photographs and drawings related to the test apparatus. All connections to the PCV are made at the vessel bottom to minimize changes to the vessel geometry. Prior to use for hydrogen getter testing the test apparatus was leak tested to assure any leaks were sufficiently small to have no impact on measurement accuracy. The pressure sensors are MKS Baratron® Series 690 sensors (0 – 5000 or 0 – 10,000 Torr). The apparatus is equipped with a Series 203 Variable Leak Valve (Granville-Phillips) to allow addition of hydrogen from a 1-L standard volume to the PCV at the desired rate to simulate hydrogen generation from within the nuclear material container. This standard volume is used to supply hydrogen to the variable leak. Three separate thermocouple sensors are positioned inside the PCV to monitor the temperature of the gas phase, getter, and molecular sieve. An additional thermocouple is positioned outside the PCV to monitor the temperature of the air in the laboratory.

The gases used as part of this testing include hydrogen, nitrogen and air. The hydrogen is supplied from a size b cylinder located within the C-064 lab module where these tests were completed. This hydrogen is supplied at a minimum purity of 99.95%. The nitrogen supply used in these tests is from the 773-A house gas system and is supplied from a cryogenic tank of liquid nitrogen. The air used in these tests is room air that has been filtered to remove any particles but is otherwise unprocessed.

Test Apparatus Calibration

The Task Technical and QA Plan (included as Attachment 8) prepared for evaluation of the DDF-1 getter assembly required use of Measurement and Test Equipment (M&TE) to confirm proper performance of the system components that are controlled as Measurement System and Equipment (MS&E). Completion of this effort relied on use of pressure, volume, temperature and mass standards. In Attachment 1 Figures 1-21 to 1-24 are pictures of the various M&TE used to assure acceptable performance of the test apparatus. Attachment 5 contains scanned images of the calibration certificates for each of these pieces of M&TE used in performance of the getter testing. A work instruction was prepared for calibration and control of the MS&E used as part of this task and is found in Attachment 6.

Temperature Calibration

The calibration checks for the four type J thermocouples used in this test were accomplished by comparing the response of the thermocouples with an M&TE calibrated temperature device. All five probes were inserted into the thermowell of a HART thermocouple calibrator. The calibrator was set at 5-degree increments from 15°C up to 30°C, and then in 10°C increments up to 70°C. The M&TE temperature probe has two ranges and the range setting was changed at 50°C.

Temperature readings from the test apparatus' data acquisition system were recorded and compared to readings from the M&TE temperature probe. The details of this procedure are included in Attachment 6. Temperature data from this calibration test were logged to file, then the file checked to confirm accurate recording of temperature data. In Attachment 7, Figures 1 and 3 show the temperature data with time. Figures 7-2 and 7-4 show the response of the four type J thermocouple readings versus standard data. A least squares linear fit to this data has an average slope of 1.000 ±0.006 indicating very little bias in the temperature reading. These calibration checks of the DDF-1 temperature measurement components were completed both before and after evaluation of the getter assemblies.

Pressure Calibration

Pressure calibration checks of the four Baratron pressure transducers used as part of this test were done by placing a M&TE calibrated Paroscientific pressure transducer on the apparatus manifold. The system was evacuated, then nitrogen gas metered into the manifold to raise pressure in various increments. This was done over the required range for the Baratron up to about 2000 Torr. The Paroscientific pressure readout and pressure readings from the Baratron readouts were recorded and compared against data logged by the data acquisition system. No discrepancies were noted as part of this test. Attachment 7, Figures 5 and 7 show the pressure change data plotted with time during the course of this test. Figures 6 and 8 show the standard pressure values versus the four Baratron readouts. A least squares linear fit to this data has an average slope of 1.000 ± 0.006 indicating very little bias in the pressure reading. Small amounts of nitrogen were also metered into the manifold during this calibration procedure to check Baratron resolution. Each Baratron pressure transducer was able to reliably detect changes as small as 0.1 Torr in the measured pressure. These calibration checks of the DDF-1 pressure measurement components were completed both before and after evaluation of the getter assemblies.

Volume Calibration

The various volumes of the test apparatus were determined using a one-gallon standard volume. Volumes of the test apparatus relative to the standard volume were determined at ambient temperature (~ 22°C) by one of two processes. In all cases, pressures were read from the Paroscientific M&TE controlled pressure transducer. Temperature remained essentially constant during the calibration process.

In the first process, nitrogen was introduced into the one-gallon standard volume V_1 at an initial pressure P_1, then expanded into the previously evacuated tubing V_2 and unknown volume V_3 to obtain a new pressure P_2. The apparatus was evacuated, then nitrogen was introduced into V_3 at a second initial pressure P_3 and expanded into V_2 and V_1 to obtain P_4. This process minimizes the propagated uncertainty in the unknown volume calculation because the volume of the connecting tubing V_2 can be algebraically canceled. V_3 is given by the following equation:

$$V_3 = P_1 \times P_4 \times V_1/(P_3 \times P_4).$$

The volumes of the one-liter vessel and the empty PCV were determined in this manner. Similarly, the manifold and hydrogen reservoir volumes were determined in this manner using the newly calibrated 1-liter volume as V_1.

The remaining volumes of the apparatus were determined by expanding nitrogen at a known pressure P_1 from a known volume V_1 into the unknown volume V_2 to obtain the final pressure P_2. V_2 was then calculated from Boyle's Law:

$$V_2 = P_1 \times V_1/P_2.$$

The results of these volume calibrations are provided in Table II. In most cases each volume calibration was performed a minimum of two times.

Table II - Test Apparatus Volume Calibration

Test Apparatus Component	Volume (cubic centimeters)
1 Gallon Standard Volume	**3778.9**
Manifold	**168.4**
A	**3.3**
B	**49.7**
Calibrated Volume	**1003.8**
Hydrogen Reservoir	**1048.2**
C	**11.1**
D	**28.1**
PCV Empty	**5148.6**
PCV 1 Can (7-inch)	**3603.5**
PCV w/ 2 Cans (7-inch and 5-inch) and stainless steel weight	**2386.2**

The uncertainties in the determined values are typically less than one percent. Only the hydrogen reservoir, "C" and the various PCV configurations are used in rate or concentration calculations. Table II shows the volume of each segment of the apparatus. Letters "A" to "D" refer to sections of tubing labeled on the test apparatus schematic found in Attachment 1, Figure 1-20.

Mass Calibration

Sample masses were measured using a Mettler model PM6100 balance, which is readable to 0.01 g. Standard weights were used to confirm balance performance before and after each use. These checks showed that mass measurements were consistently within ± 0.1 g of the nominal mass over the range required for measurements supporting this test.

Leak Check

All apparatus components have been leak checked by evacuating the components and measuring pressure rise with time (rate of rise). Leak rate Q (std. $cm^3\ s^{-1}$) is calculated using the following equation.[7]

$$Q = \frac{(P_{final} - P_{initial}) \times V \times 1.32}{(t_{final} - t_{initial})}$$

In this equation, P is pressure measured in Torr and corrected to standard temperature, V is the test volume in liters, and t is time in seconds. The factor 1.32 is used to convert from Torr•liter (at standard temperature) to std. cm^3. The leak rate limit for this apparatus was set at $1x10^{-5}$, which is equivalent to 1% of the minimum hydrogen addition rate. See Attachment 4 Figure 4-50 for data related to apparatus leak testing. By testing at close to one atmosphere delta P (i.e. test component evacuated), the measured leak rate is anticipated to be a factor of 10 greater than any leak observed with no delta P across a potential leak when there is little pressure differential between the apparatus and atmospheric pressure. All components used in construction of this apparatus are anticipated to have leak rates less than $1x10^{-7}\ cm^3\ sec^{-1}$. The leak rate data for key segments of the test apparatus are provided in Table III below:

Table III - Leak Rate for Components of DDF-1 Getter Test Apparatus

Component	Time Window	Pressure Change	Leak Rate ($cm^3 s^{-1}$)
PCV	**70 hrs**	**1.09 Torr**	**$1.8x10^{-5}$**
PCV	**40 hrs**	**0.00 Torr**	**$< 1.0x10^{-6}$**
Std. Leak	**70 hrs**	**0.04 Torr**	**$2.2x10^{-7}$**
Std. Leak	**40 hrs**	**0.04 Torr**	**$3.2x10^{-7}$**
Manifold	**70 hrs**	**0.37 Torr**	**$2.0x10^{-7}$**
Manifold	**40 hrs**	**0.03 Torr**	**$2.5x10^{-8}$**

The PCV pressure rise over the initial 20 hours of leak testing was 1.5 Torr, which is higher than anticipated for this system. This initial pressure rise may be attributed to having a produce can in the PCV during this test or other outgassing associated with the PCV. Typical leaks show a continuous rate of pressure rise that does not level out with time as seen in Figure 4-50. The leak rates measured for the last 40 hours of this leak test support the expectation of leak rates less than $1x10^{-7}\ cm^3\ sec^{-1}$.

Hydrogen Addition to the DDF-1 PCV

Evaluation of the hydrogen getter assembly requires leaking hydrogen into a nitrogen-only or air atmosphere within the PCV to demonstrate that the rate of hydrogen removal is sufficient to prevent the build-up of hydrogen in the PCV. The hydrogen addition rate into the PCV is calculated from the drop in pressure in the standard volume (hydrogen supply container). Attachment 4, Figures 4-2 and 4-3 show data related to calculating the hydrogen flow into the DDF-1 PCV. The pressure drop measurement shown in Figure 4-2 for the standard volume is associated with adding hydrogen at a leak rate close to 60 std. $cm^3\ hr^{-1}$ to the PCV. Figure 4-3 shows the calculated std. cm^3 of hydrogen added to the PCV based on the measured pressure drop. The slope of a linear least squares fit to this hydrogen addition rate is used to estimate the hydrogen flow rate in std. cm^3 hr^{-1}. This measurement of hydrogen addition rate is completed for each test conducted as part of the DDF-1 getter assembly evaluation; however, only the hydrogen addition rate is presented for each subsequent test. Figure 4-4 shows an example of the calculated percent hydrogen that would be found in the PCV if no hydrogen getter were present. This calculation is based on the hydrogen addition rate and the PCV measured volume.

Prior to beginning hydrogen flow, the hydrogen pressure in the standard leak section of the test apparatus is brought to a pressure slightly higher than the PCV pressure. Then the selected valve (V5 or V6) connecting the test apparatus variable leak section to the PCV is opened and the leak valve setting increased to initiate flow. The slight difference in pressure between the leak section and the PCV prevents air or nitrogen from the PCV from back filling the leak section. Once flow is initiated, the pressure drop of the standard volume is monitored to allow adjustment of the variable leak. These steps of the hydrogen addition are illustrated in Attachment 4, Figure 4-51.

While hydrogen is flowing into the PCV, the measured pressure increase of the PCV is used to estimate the hydrogen concentration. These hydrogen concentration estimates do not need to be corrected for the hydrogen volume of the supply line as originally described in the Task Technical and QA Plan. This improvement allows the hydrogen to enter the PCV almost immediately and removes the test variability associated with the time required to purge nitrogen from the variable leak section of the apparatus. Because this section of the test apparatus is started at nearly equal pressure with the PCV, and nitrogen is not displaced from most of the tube's length, hydrogen concentration in the PCV is easily estimated from the pressure rise associated with initiating hydrogen flow. Previous tests demonstrated that the volume of gas in the standard leak section of the test apparatus (component "C" and hydrogen reservoir in Figure 1-18) is not readily accessible to the hydrogen getter (i.e. diffusion past V5 or V6 is very slow).

For figures presented in Attachment 4 with the vertical axis labeled as "Hydrogen Concentration (vol.%)," the hydrogen concentrations in the PCV are calculated based on pressure rise of the PCV, assuming any increase in temperature corrected pressure results from hydrogen addition. With very small pressure increases observed in the PCV for these tests, the calculated hydrogen concentration is significantly below the established 12 vol.% limit and does not warrant gas sampling to confirm measurement accuracy. Typically the pressure increase during a hydrogen flow test is less than 3 Torr, which represents about 0.4 vol.% hydrogen in the PCV. The LFL for hydrogen in air is 4 vol.%, or about 30 Torr of hydrogen in the PCV. Consequently, the pressure rise observed during hydrogen flow testing is small compared with the LFL and much smaller than the 2 psia limit (~100 Torr) set for the DDF-1.

For tests that address recombination in the DDF-1 PCV, there are no graphs in Attachment 4 that address "Hydrogen Concentration (vol. %)." Because the pressure is dropping during the recombination tests from removal of oxygen from air, the hydrogen concentration reached during these tests is evaluated in discussion of each recombination test.

At a leakage rate of 14 std. cm^3 hr^{-1}, more than 30 days would be required to consume 80% of the theoretical getter capacity. To accelerate loading of the getter, testing alternates between two modes. The first mode consists of leaking hydrogen into the PCV at a rate of approximately 30 std. cm^3 hr^{-1} for several hours to demonstrate the getter exceeds the 14 std. cm^3 hr^{-1} performance requirement. In the second test mode, the hydrogen flow rate was increased up to about 9000 std. cm^3 hr^{-1} to accelerate loading of the getter to some percentage (e.g., 50%, 80%, and 90%) of its total capacity. This is possible because the hydrogen removal rate dramatically increases with increased hydrogen concentration and getter temperature. In Figures 4-5 and 4-6 with the getter temperature at 70 to 80°C and a hydrogen concentration of about 8 vol.%., the hydrogen flow rate can be maintained at more than 5000 std. cm^3 hr^{-1} . These two test modes are used to demonstrate that the getter can prevent hydrogen build-up within the PCV even when loaded to 90% of its theoretical capacity.

Results and Discussion

Temperature Corrected Pressure

Efforts to correct the pressure measurements of the PCV based on measured temperatures did not provide the expected degree of stability anticipated for the test apparatus. Tests 7 and 8 were completed to measure the effect of room temperature variation on PCV pressure without hydrogen flow. Attachment 4, Figure 4-49 shows the variations in pressure associated with changes in laboratory temperature over the course of many hours. Note that the uncorrected pressure measurements (P4) shown on this graph change about 5 Torr with a 3-degree temperature change. If a simple ratio of measured temperature to a nominal temperature in Kelvins is used to correct pressure (i.e. P/T=P'/T' or P'=PxT'/T when volume is constant) this improves the pressure stability to ± 1 Torr. However this variation in pressure is still greater than desired for evaluation of the DDF-1 hydrogen getter assembly. Figure 4-49 shows the temperature-corrected pressures (P4TC) using this ideal gas law temperature correction. Note that this approach tends to over correct the measured temperature change, so pressure measurements corrected using this method indicate a decrease in pressure as temperature increases.

To improve the pressure measurements used during hydrogen flow tests a pressure versus temperature calibration has been developed for the DDF-1 test apparatus. The data for this calibration was collected by adding 600 Torr of nitrogen to the PCV, then recording the changes in pressure and PCV gas temperature over time. The PCV gas temperature changes nearly as fast as the room temperature as seen in Figure 4-47. By plotting the pressure versus temperature in the PCV over the course of the test, a calibration curve for temperature correction of PCV pressure measurements has been developed. The slope of a least square linear fit of this data provides a correction factor to help minimize the observed variation in PCV pressure due to variation in gas temperature. The data for this correction factor is presented Figure 4-48 for the PCV containing only a produce can. Because data was collected for many hours, the correction factor uses the average slope from times of rapidly increasing or decreasing temperature and slowly increasing or decreasing temperature.

The molecular sieve used in the DDF-1 getter assembly tends to adsorb gases from the PCV. See Figure 4-44 for an example of nitrogen adsorption by the 100 g of 4Å molecular sieve identical to that used in the getter assembly. This weakly adsorbed gas will desorb with slight increases in temperature and re-adsorb with decreases in temperature, so the observed pressure changes are greater than predicted by the ideal gas laws for a given change in temperature. Figure 4-46 shows the change in pressure with temperature for the PCV containing 100 g of 4Å molecular sieve. With molecular sieve present in the PCV in a nitrogen atmosphere, the observed pressure fluctuation with changes in room temperature is pronounced than observed for the PCV with no molecular sieve in Figure 4-47. As for the empty PCV, a correction factor has been established using a least squares linear fit to a pressure versus temperature calibration curve. This data is plotted for the PCV containing molecular sieve in Figure 4-45.

The experimentally determined average correction for an empty PCV is 1.75 Torr $°C^{-1}$ and increases to 2.72 Torr $°C^{-1}$ for a PCV containing molecular sieve. The improvement gained by applying these temperature correction factors (P4corr) to the measured PCV pressure is demonstrated in Figure 4-49. Although this effort does not correct all the long term variations (e.g. 40 hours) observed for the pressure measurements in Figure 4-49, most of the short term variations in pressure where temperature changes are small have been fixed. This correction has been helpful in demonstrating that PCV hydrogen concentrations are very stable and not increasing gradually during the course of the hydrogen flow tests.

Getter Assembly Evaluation

A description of each test conducted as part of this effort is provided in the following pages along with a brief explanation of the test results. These tests are listed in sequential order of completion. Attachment 4 contains one figure for each test showing the recorded PCV pressure and temperature versus time; one figure for the amount of hydrogen added and the amount of hydrogen in the PCV over time; and the calculated vol.% hydrogen in the PCV with time*. Additional figures are provided as needed to demonstrate the results of a particular test. In all, eight tests were conducted. The final three tests were not anticipated during preparation of the Task Technical and QA Plan. These tests were added to help better characterize the test apparatus performance and demonstrate the effects of change in room temperature on pressure measurements. These additional tests were completed using the same QA measures described in the Task Technical and QA Plan. The first five sets of test were conducted to evaluate getter assembly performance in the DDF-1 PCV. These tests were outlined in the Task and QA Plan developed for this effort and presented in Table IV below.

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* The vol.% hydrogen graph is not presented for recombination experiments. The hydrogen concentration results for these tests will be presented as part of the discussion for recombination tests.

Table IV –Tests Required to Validate Hydrogen Getter Assembly Performance

Series	Description of Test	Equipment Configuration
1	H_2 flow rate ~30cm^{3}hr^{-1} with getter at ~0, 50 and 90% of theoretical capacity. These tests will be conducted with N_2 gas in the PCV.	Test with dummy produce can(s) in PCV. Demonstrate effect of H_2 leak location (i.e. bottom or middle of PCV)
2	Use H_2 saturated getter assembly from test 1. Remove dummy produce cans. Measure recombination rate.	Molecular sieve is present to adsorb water produced during recombination reaction.
3	Repeat Test 2. Replace getter assembly with one containing molecular sieve preloaded with water (10g) to demonstrate molecular sieve's capability to adsorb excess moisture.	Remove dummy product can(s) to maximize available O_2 for recombination. Recombination rate must remain at acceptable level.
4	Using a new getter assembly, repeat test 1 with getter assembly "crushed" to restrict available surface. Test needs to be conducted at only a single H_2 loading.	Same as Test 1 with heavy weight on top of "getter assembly." H_2 flow only required at bottom of PCV
5	Test 3 additional getter assemblies in the PCV for initial H_2 absorption rate to show that performance is similar.	Run these tests with a single PCV configuration with dummy product can(s) in place.

The figures discussed in this section are primarily from Attachment 4 and will be identified using the notation Figure X-Y, where X is the attachment number and Y is the figure number.

Hydrogen Flow Tests

Effects of Hydrogen Loading

Test 1A

Purpose: Evaluate impact of hydrogen loading on getter assembly performance.
Getter Assembly: 0% hydrogen loading
PCV Content: 7-inch produce can
PCV Headspace: Nitrogen
PCV Volume: 3.6 L
Hydrogen flow rate: ~60 std. cm^3 hr^{-1}
Figures: 4-1 to 4-4

The purpose of tests 1A to 1D was to demonstrate that the hydrogen getter assembly will maintain hydrogen pressure in the DDF-1 PCV at less than 2 psia (~100 Torr) with increased getter loading. These tests were conducted using about 600 Torr of nitrogen in the PCV to mimic the PCV content after consumption of the 20.9% oxygen from recombination to form water.

Test 1A utilized a freshly prepared hydrogen getter assembly, which had not previously been exposed to hydrogen. After loading the PCV with the getter assembly and one 7-inch produce can, the PCV was briefly evacuated then back filled with nitrogen. Hydrogen flow was initiated using previously established settings for the standard leak. The resulting flow rate was higher than anticipated, but presented no evident challenge for the getter assembly to maintain hydrogen concentration at less than 0.3%, even with flow at about four times the required rate. The point of hydrogen addition inside the PCV was changed from the bottom of the PCV to the middle of the PCV midway through this test by closing valve V5 and opening valve V6 (see Attachment 1, Figure 1-18). This change in flow position had no effect on the residual hydrogen concentration observed in the PCV. Figures 4-1 to 4-4 show the pressure, temperature, and calculated hydrogen concentration during this test.

Test 1B

Purpose: Evaluate impact of hydrogen loading on getter assembly performance.
Getter Assembly: 46% hydrogen loading
PCV Content: 7-inch produce can
PCV Headspace: Nitrogen
PCV Volume: 3.6 L
Hydrogen flow rate: > 30 std. cm^3 hr^{-1}
Figures 4-5 to 4-9

The getter assembly from test 1A was loaded to 46% capacity (see Figures 4-5 and 4-6) by flowing hydrogen at an accelerated rate into the PCV. Using this procedure, the getter can be tested at increasing levels of getter loading to confirm that the getter assembly maintains hydrogen at sufficiently low pressure as getter capacity is consumed. During loading, it should be noted that hydrogen addition rates were increased to greater than 5000 std. cm^3 hr^{-1} with hydrogen concentration maintained at about 10 vol.% in the nitrogen environment. The getter temperature increased to about 80°C as a result of loading to 50% capacity and was allowed to cool overnight to ambient temperature. Data for the hydrogen flow test at 50% capacity are presented in Figures 4-7 to 4-9. Hydrogen flow was maintained at greater than 30 std. cm^3 hr^{-1} during the course of this test, and the hydrogen concentration in the PCV did not exceed 0.4 vol.%. The test ran for about seven hours due to the observed slight increase in pressure observed during the test. This pressure rise was attributed to increasing PCV temperature during the test and limitations on our ability to correct for temperature changes. As a result of this slight pressure rise, a long flow test was conducted to assure that no gradual hydrogen pressure increases are overlooked during a 1 to 2 hour flow test. The description and results of this long flow test are found in Test 6.

Test 1C

Purpose: Evaluate impact of hydrogen loading on getter assembly performance.
Getter Assembly: 75 % hydrogen loading
PCV Content: 7-inch produce can
PCV Headspace: Nitrogen
PCV Volume: 3.6 L
Hydrogen flow rate: >30 std. cm^3 hr^{-1}
Figures: 4-10 to 4-12

The capacity of the VEI getter is about 120 std. cm^3 g^{-1}; however, the hydrogen removal rate slows as the getter is depleted. Preliminary tests indicated that the getter would have sufficient rate at 90% capacity. Because significant time is required to load a getter to this capacity, the testing at 80% was selected as an intermediate point to assure timely completion of the full scale tests. The getter assembly for test 1C was loaded to 75% capacity by flowing hydrogen at an accelerated rate to the PCV. As noted for test 1B, the getter temperature increased to about 70°C during loading and removed hydrogen very rapidly. The getter was allowed to cool to ambient temperature prior to testing. Data for the hydrogen flow test at 80% capacity are presented in Figures 4-10 to 4-12. Hydrogen flow is maintained at greater than 30 std. cm^3 hr^{-1} during the course of this test, which is about 2 times the required rate. The test ran for about 1.2 hours and the hydrogen concentration never exceeded 0.2 vol.% in the PCV.

Test 1D

Purpose: Evaluate impact of hydrogen loading on getter assembly performance.
Getter Assembly: 85% hydrogen loading
PCV Content: 7-inch produce can
PCV Headspace: Nitrogen
PCV Volume: 3.6 L
Hydrogen flow rate: >30 std. cm^3 hr^{-1}
Figures: 4-14 and 4-15

The getter assembly for test 1D was loaded to 85% capacity by flowing hydrogen at an accelerated rate to the PCV. The getter temperature increased to about 60°C during loading and was allowed to cool to ambient temperature prior to testing. During the hydrogen flow test, hydrogen flow was maintained at approximately 28 std. cm^3 hr^{-1}. The data for this test are presented in Figures 4-14 and 4-15. This test ran for about four hours and the hydrogen concentration never exceeded 0.3 vol.% in the PCV. During this test the PCV temperature increased from 21.5 to 23.5° as a result of changes in room temperature

Recombination Measurements for Hydrogen in Air

Test 2

Purpose: Demonstrate removal of hydrogen from air with hydrogen-saturated getter assembly.
Getter Assembly: 100% hydrogen loading
PCV Content: only Getter Assembly
PCV Headspace: Air
PCV Volume: 5.1 L
Hydrogen flow rate: ~ 30 std. cm^3 hr^{-1}
Figures: 4-16 to 4-19

The purpose of test 2 was to demonstrate that the hydrogen getter assembly will maintain hydrogen pressure in the DDF-1 PCV at less than 2 psia when being used as a recombination catalyst. These tests were conducted using about 760 Torr of air in the PCV to mimic the PCV upon initial loading of the PCV in air. The hydrogen getter was saturated with hydrogen to ensure that recombination alone was the mechanism for hydrogen removal.

Test 2A utilized the hydrogen getter assembly following test 1D, which was subsequently loaded to 100% of the theoretical capacity. The PCV was briefly evacuated then opened to the atmosphere to fill the PCV with air. The hydrogen flow was initiated using previously established settings for about 30 std. cm^3 hr^{-1} from the standard leak. These rates were confirmed by timing the pressure drop observed for the standard volume during one-minute intervals. Hydrogen flow was from the bottom of the PCV during this sequence of tests. Figures 4-16 and 4-17 illustrate the results of hydrogen flow test 2A. Because the getter was saturated with hydrogen with a residual pressure that was about 120 Torr, the palladium catalyst would be saturated with hydrogen similar to the application of metal hydrides for hydrogen storage. Once air is added to the PCV, the hydrogen will be removed from the palladium and combined with oxygen to form water. Consequently the net oxygen consumption shown in Figure 4-17 does not represent the expected stoichiometry for the amount of hydrogen added for the flow test. Note that this recombination required about 14 hours to consume all the available hydrogen.

Following Test 2A, air was added to the PCV to return to atmospheric pressure and the flow test repeated. The results of the repeated test are shown in Figures 4-18 and 4-19. After an initial pressure rise of about 0.3 Torr, the pressure began to drop due to the recombination of added hydrogen with oxygen from the air. The difference between initial and final pressure for this test corresponds to the amount of oxygen removed from the PCV. Figure 4-19 shows that ~ 44 std cm^3 of hydrogen were added to the PCV and ~ 22 std. cm^3 of oxygen were removed, as expected for the recombination reaction. The hydrogen concentration of the PCV when flow was stopped was 0.2 vol.%. This hydrogen concentration is estimated based on the pressure drop and volume decrease following termination of hydrogen flow. From Figure 4-18, a pressure drop of ~2 Torr occurs after hydrogen flow is stopped. Since this pressure drop is the result of recombination, stoichiometry requires that two-thirds of the pressure drop is a result of hydrogen and one third from oxygen. Consequently 1.3 Torr of the 762 Torr in the PCV was hydrogen or ~0.2 vol.%.

Effects of Moisture on Getter Performance

Test 3A

Purpose: Evaluate the impact of moisture-loaded molecular sieve on getter performance in air.
Getter Assembly: 0% Capacity and moisture loaded molecular sieve (~10g water)
PCV Content: only Getter Assembly
PCV Headspace: Air
PCV Volume: 5.1 L
Hydrogen flow rate: > 30 std. cm^3 hr^{-1}
Figures: 4-20 and 4-21

The purpose of this test was to demonstrate that the transition from recombination in the presence of air to hydrogenation of the polymer getter is not negatively impacted by the presence of water. To evaluate this condition, a getter assembly was made using molecular sieve with about 10 g of water absorbed. This amount of water is equivalent to about 45 days of operation at 14 std. cm^3 hr^{-1} with stoichiometric quantities of hydrogen and oxygen being generated. If only oxygen is used from the PCV, this would result in only about 1.8 g of water being produced. The hydration of this molecular sieve was accomplished by storing the getter in a one-gallon "paint" can along with 10 g of water in an evaporating dish. About 36 hours were required to absorb this quantity of water at room temperature. Adsorption of the water on the molecular sieve was confirmed using the change in mass before and after storage in the one-gallon "paint" can. The completed getter assembly was sealed in a foil bag until ready for use.

Figures 4-20 and 4-21 show the results for Test 3A. As in Test 2, the PCV was filled with room air before initiating hydrogen flow. The hydrogen flow was maintained at ~ 30 std. cm^3 hr^{-1} during the flow test. Initially the PCV pressure rose by about 3 Torr (0.4 vol.%) before the pressure began to drop due to the recombination reaction of added hydrogen with oxygen in the air. A total of about 83 std. cm^3 of hydrogen were added to the PCV, and the observed pressure drop in the PCV corresponded to the removal of about 39 std. cm^3 of oxygen. This test shows that even when the molecular sieve is loaded with about 10 wt.% water, the getter assembly is still capable of removing hydrogen from air by recombination at a rate greater than 30 std. cm^3 hr^{-1}.

Test 3B

Purpose: Evaluate the impact of moisture-loaded molecular sieve on getter performance in nitrogen.
Getter Assembly: 0% Capacity and moisture loaded Molecular sieve
PCV Content: only Getter Assembly
PCV Headspace: Nitrogen
PCV Volume: 5.1 L
Hydrogen flow rate: > 30 std. cm^3 hr^{-1}
Figures: 4-23 and 4-24

The purpose of this test was to show that moisture-loaded molecular sieve does not negatively impact the ability of the getter to remove hydrogen from a nitrogen atmosphere. This test was run for about 8 hours, and the results of this test are shown in Figures 4-23 and 4-24. As in other tests where the PCV has been opened to insert a new getter assembly containing molecular sieve, there is an indication of recombination upon initial flow of hydrogen. The PCV contains about 600 Torr of nitrogen for this test to represent the gas composition and pressure expected after recombination of oxygen from air in the PCV. During this test hydrogen flow was interrupted after about 2 hours and the pressure in the PCV allowed to equilibrate. The pressure between 2.4 and 4.7 hours is representative of recombination of about 22 std. cm^3 of oxygen and 44 std. cm^3 of hydrogen. When hydrogen flow was restarted at 4.7 hours, there was no evidence of recombination and PCV pressure quickly stabilized about 1 Torr higher than the starting pressure. When hydrogen flow was stopped after about 1.5 hours, the PCV pressure quickly returned to baseline. This observation suggests that the pressure drop following the termination of hydrogen flow is a good indication of residual hydrogen in the PCV, and could be helpful for understanding getter performance.

Results of this test show that hydrogen pressure did not increase more than 1 Torr (0.2 %) during hydrogen flow, which is very similar to Tests 1A - 1C. Consequently, we conclude that adsorption of water on the getter assembly molecular sieve does not reduce effectiveness of the hydrogenation reaction.

Evaluation of PCV Configuration on Getter Performance

Test 4

Purpose: Evaluate the effect of PCV configuration on getter assembly performance.
Getter Assembly: 0% Capacity with Getter Assembly Crushed
PCV Content: 7-inch produce can, 5-inch produce can, and 2-kg mass
PCV Headspace: Nitrogen
PCV Volume: 2.4 L
Hydrogen flow rate: > 30 std. $cm^3\ hr^{-1}$
Figures: 4-25 to 4-29

The purpose of this test was to demonstrate that changes in the PCV geometry do not prevent the getter assembly from functioning properly. The PCV was filled to capacity with the items used in this test. The photo in Attachment 1 Figure 1-25 best describes the arrangement of materials. A 7-inch produce can was in the PCV bottom, the getter pack was placed on top of this can (see Figure 1-26), then a 5-inch tall can was placed on top of the getter assembly. A 2.5-kg, 4-inch diameter stainless steel mass was placed on top of the 5-inch can, then the PCV lid was installed. When fully closed, the lid pressed on the PCV contents to further crush the getter assembly. These conditions were expected to restrict hydrogen flow to the getter assembly and represent worst case conditions for a DDF-1 turned upside-down or otherwise tightly packed.

Prior to running this test the PCV was evacuated, then back filled with nitrogen. Hydrogen was added at the rate of 30 std. cm^3 hr^{-1} for about 0.9 hr with the expectation that this would remove the trace of oxygen frequently observed when installing a new getter assembly in the PCV. The hydrogen flow was stopped after 0.9 hr and the pressure allowed to equilibrate. As seen in Figures 4-25 and 4-26, a pressure drop of ~ 1.5 Torr (corresponding to the removal of ~ 5 std. cm^3 of oxygen) was observed over the course of the test. We continued to monitor the pressure over the next 10 hours and a gradual pressure increase of ~ 1.5 Torr was observed. This slight pressure increase during a period of no hydrogen flow was most likely due to some off-gassing from the getter assembly, which was not thoroughly degassed prior to beginning the test.

After the initial addition of hydrogen to remove traces of oxygen, the hydrogen flow test with the crushed getter assembly was repeated without evacuating or opening the PCV to the atmosphere. Figures 4-27 to 429 give the results of the second flow test. Hydrogen was added to the PCV at the rate of 39 std. cm^3 hr^{-1} for a period of 7.2 hr. During this time the pressure in the PCV increased by less than 2 Torr, corresponding to a hydrogen concentration of about 0.3%. After the hydrogen flow was stopped the PCV pressure returned quickly to baseline confirming the maximum hydrogen concentration of about 0.3% during the test. Also, no further recombination or off-gassing was observed in the repeated test.

Evaluation of Getter Assembly Production

Test 5A

Purpose: Demonstrate consistent performance from multiple getter assemblies
Getter Assembly: 0% Capacity
PCV Content: 7-inch produce can
PCV Headspace: Nitrogen
PCV Volume: 3.6 L
Hydrogen flow rate: > 30 cm^3 hr^{-1}
Figures: 4-30 to 4-34

The purpose of Tests 5A through 5C was to determine variation between multiple getter assemblies manufactured at SRTC. Test 5A was run with a new getter assembly. After placing the getter assembly in the PCV and closing the lid, the PCV was evacuated for about 5 minutes, then back filled with nitrogen before initiating hydrogen flow. Figures 4-30 and 4-31 show the results of this flow test. Hydrogen was added to the PCV at the rate of 43 std. cm^3 hr^{-1} for about 1.3 hr. Similar to Test 4, an overall pressure drop in the PCV of about 2 Torr was observed when hydrogen was initially added. This was attributed to recombination of some of the added hydrogen with a small amount of oxygen not removed during evacuation. The hydrogen flow test was repeated (Figures 4-z to 4-w) with hydrogen being added at the rate of 32 std. cm^3 hr^{-1} for about 0.7 hr and no recombination was observed. Based on the observed pressure changes in the PCV, the hydrogen concentration did not exceed 0.2% during this test.

Test 5B

Purpose: Demonstrate consistent performance from multiple getter assemblies
Getter Assembly: 0% Capacity
PCV Content: 7-inch produce can
PCV Headspace: Nitrogen
PCV Volume: 3.6 L
Hydrogen flow rate: 39 std. cm^3 hr^{-1}
Figures: 4-35 to 4-37

A second new getter pack was used in this test, and the test was conducted in a manner similar to that described for Test 5A. The results are illustrated in Figures 4-35 to 4-37. Hydrogen was added to the PCV at the rate of 39 std. cm^3 hr^{-1} for about 1.9 hr. The results were very similar to those of Test 5B. Approximately 74 std. cm^3 of hydrogen were added to the PCV during this test. Initially the PCV pressure rose about 1 Torr (< 0.2%), then dropped back to the original pressure and remained steady during the remainder of the flow test. After the hydrogen flow was stopped, the PCV pressure dropped quickly to about 1 Torr below the initial PCV pressure. This pressure drop corresponds to the removal of about 5 std. cm^3 of oxygen by recombination and is also a good indication that the hydrogen concentration did not exceed 0.2% during this test.

Test 5C

Purpose: Demonstrate consistent performance from multiple getter assemblies
Getter Assembly: 0% Capacity
PCV Content: 7-inch produce can
PCV Headspace: Nitrogen
PCV Volume: 3.6 L
Hydrogen flow rate: 36 std. cm^3 hr^{-1}
Figures: 4-38 to 4-40

This test used the getter component (molecular sieve pack was removed) of the getter assembly from Tests 3A (recombination) and 3B (hydrogenation). This was deemed an acceptable choice because only about 0.5% of the getter capacity was consumed during Test 3. The results of this test may be found in Figures 4-38 to 4-40. After the getter pack was placed in the PCV, the vessel was evacuated for about 15 minutes before initiating hydrogen flow. During this test hydrogen flow to the PCV was maintained at 36 std. cm^3 hr^{-1} for about 1.2 hr. The PCV pressure increased by about 1.5 Torr (< 0.3%) over the first 0.5 hr before reaching a plateau. Once the hydrogen flow was stopped, the PCV pressure quickly returned to baseline with little or no evidence of recombination. This observation suggests that much of the oxygen remaining in the PCV after brief evacuation may be associated with the molecular sieve. The results of Tests 5A-C demonstrate very reproducible performance for multiple getter assemblies manufactured at SRTC. Because manufacture of the getter assembly is simple, finding a vendor capable of supplying getter assemblies for future on-site shipping should be fairly easy.

Extended Hydrogen Flow Test

Test 6

Purpose: Extended flow test to demonstrate minimum hydrogen build-up in PCV
Getter Assembly: 80% Capacity
PCV Content: Empty
PCV Headspace: Nitrogen
PCV Volume: 5.1 L
Hydrogen flow rate: > 28 std. cm^3 hr^{-1}
Figures: 4-41 to 4-43

Figures 4-41 to 4-43 show the results of this test, which was conducted to demonstrate that any small pressure increases observed during hydrogen flow testing were not related to slow build-up of hydrogen in the PCV. To accomplish this task, the PCV was evacuated and back filled with nitrogen. Hydrogen flow was initiated and maintained at greater than 24 std. cm^3 hr^{-1} for the first 2 hr, then increased and maintained at greater than 28 std. cm^3 hr^{-1} for about 20 hours. A total of 648 std. cm^3 of hydrogen were added to the PCV during this test while the maximum pressure increase in the PCV was only about 2 Torr (0.3vol.%). Therefore, even with the getter loaded to approximately 80% capacity and a lengthy flow test there was no evidence of PCV pressurization.

Test to Improve Temperature Corrected Pressure

Test 7

Purpose: Evaluate the PCV pressure change with temperature for 100 g of Molecular Sieve
Getter Assembly: No hydrogen getter – only molecular sieve
PCV Content: Empty
PCV Headspace: Nitrogen
PCV Volume: 5.1 L
Hydrogen flow rate: None
Figures: 4-44 to 4-46

Test 7 was conducted to evaluate the effect of temperature on pressure corrections with only molecular sieve present in the PCV. This test was discussed previously under the section on Temperature Corrected Pressure (p. 12) and will not be discussed further here. The test results are shown in Figures 4-44 to 4-46.

Test 8

Purpose: Evaluate the pressure change with temperature for empty PCV
Getter Assembly: None – no getter or molecular sieve
PCV Content: 7-inch produce can
PCV Headspace: Nitrogen
PCV Volume: 3.6 L
Hydrogen flow rate: None
Figures: 4-47 to 4-49

This test was conducted to show the effect of temperature on pressure corrections with only nitrogen at 600 Torr in the PCV. Test 8 was previously discussed in the section on Temperature Corrected Pressure (p. 12) and will not be discussed further here. The results are given in Figures 4-47 to 4-49.

Leak Tests

Test 9

Purpose: Leak test of DDF-1 Test Apparatus
Getter Assembly: No getter or molecular sieve
PCV Content: 7-inch produce can
PCV Headspace: Vacuum
PCV Volume: 3.6 L
Hydrogen flow rate: None
Figure: 4-50

This test was conducted to verify the components of the test apparatus used in the previous tests were effectively "leak-tight" for the purposes of these tests. Test 9 was discussed previously in the section on Leak Check (p. 9) and will not be discussed further here. The results of the leak test are shown in Figure 4-50.

Conclusions

This evaluation of the DDF-1 getter assembly, under conditions that simulate use in actual on-site transportation, demonstrates performance that exceeds the required 14 std. cm^3 hr^{-1} rate even at 90% of the theoretical capacity. Based on these results, we recommend extending the getter qualification to 30 std. cm^3 hr^{-1} and 15,120 std. cm^3 of hydrogen capacity. This capacity neglects the additional hydrogen that will be removed by recombination of hydrogen and oxygen to form water. Consequently, the rate limit for hydrogen generation may be determined separately and limits set appropriately on package content. These revised values reflect a 50% increase in useable hydrogen capacity and a 2X increase in acceptable gas generation rates.

For example, if the hydrogen generation rate for plutonium dioxide is set at 5.6 cm^3 hr^{-1} W^{-1} (four times the measured rate for PuO_2 with 5% moisture), the getter is sufficient for about 3.5 watts of PuO_2 up to 30 days. The getter material used in the DDF-1 getter assembly is being evaluated currently for stability at 70°C over a 60-day period. Unpublished data from this evaluation suggest the getter may be successfully incorporated into the DDF-1 under conditions that limit the PCV temperature to 70°C. Additional effort is required to establish a minimum recommended operating temperature, however, temperatures as low as –10°C should be acceptable even at 30 std. cm^3 hr^{-1} while maintaining hydrogen concentration under the LFL.

The number of cans present in the PCV and location of the getter assembly will not significantly impact the hydrogen removal rate. We have noted that removal of the aluminum honeycomb impact absorber may allow sufficient room for the getter assembly along with two seven inch tall produce cans in the PCV.

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- Gary Molen led a team of site experts including Steve Hensel, Jeff Schaade, Kurt Houghtaling, Neal Askew and Ron Livingston. This team developed the requirements for using a getter for on-site transportation.

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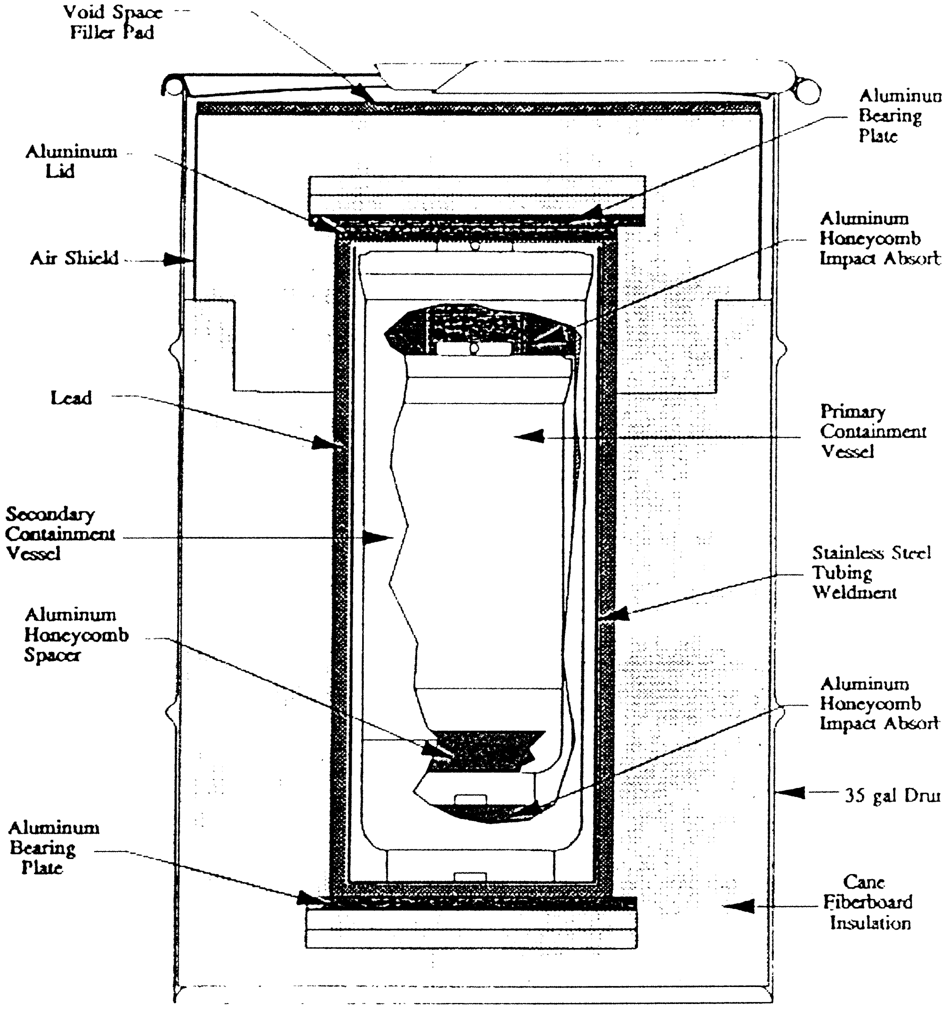

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Figure 1-1

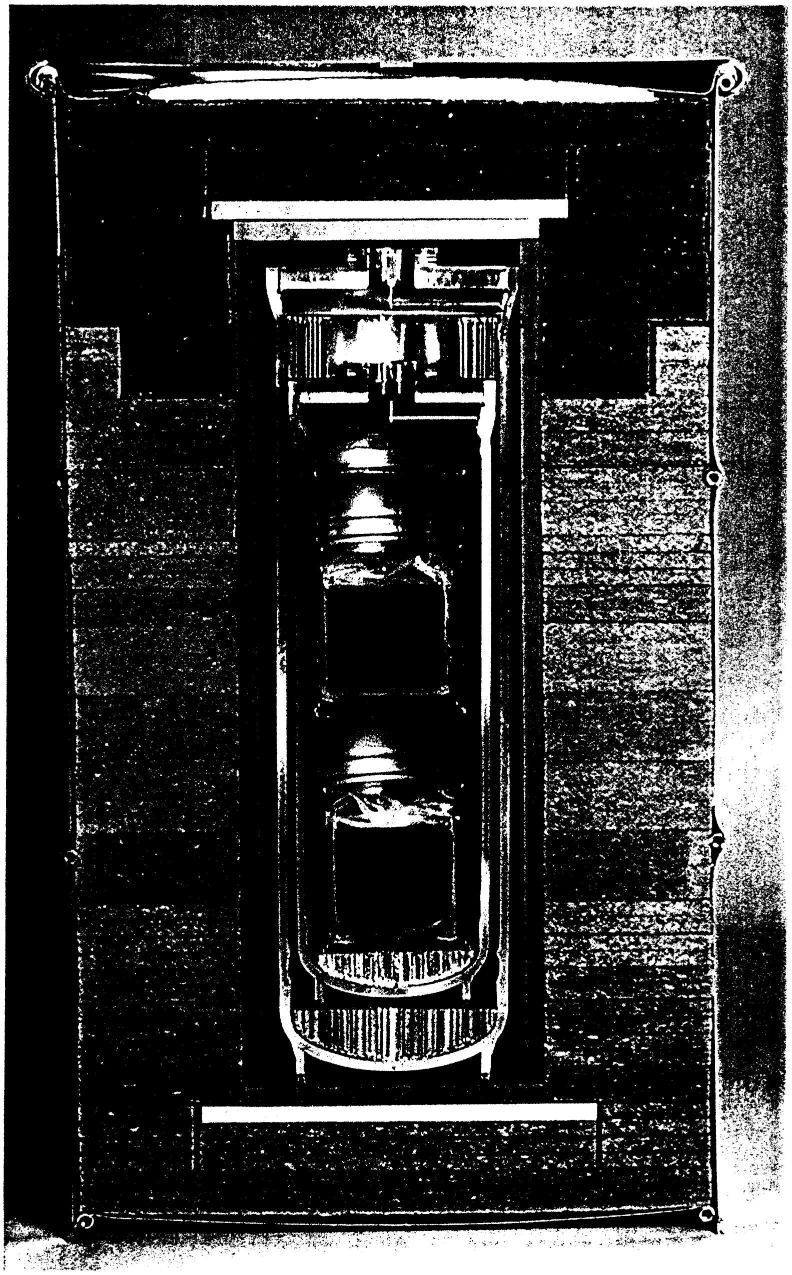
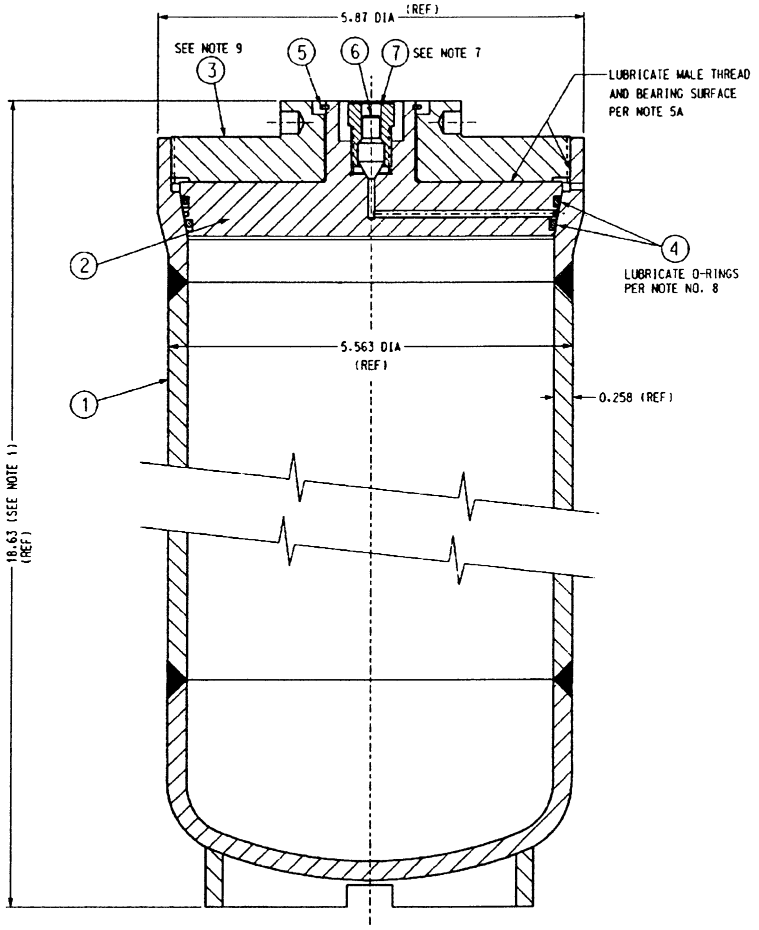


Figure 1-2



PRIMARY CONTAINMENT VESSEL (PCV) SUBASSEMBLY

REQD: 1

R-R2-F-0018-B

Figure 1-3

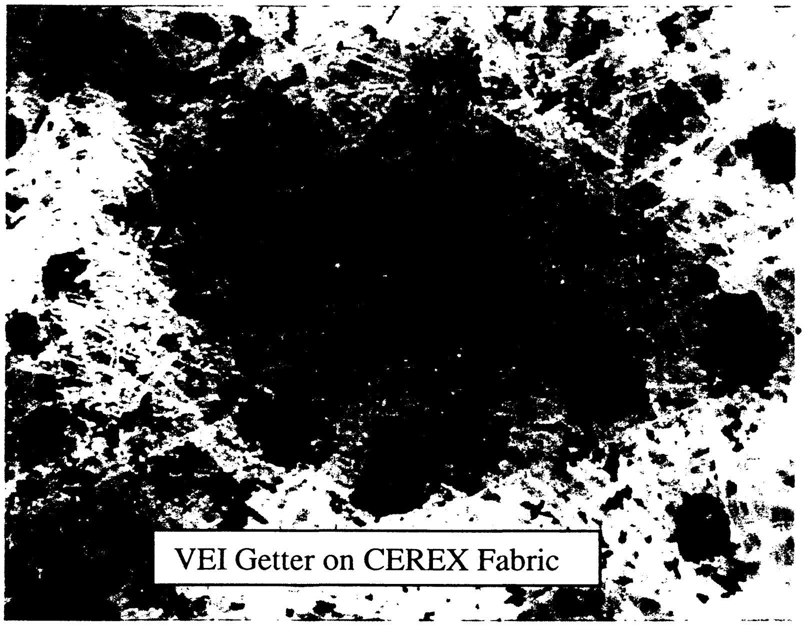


Figure 1-4

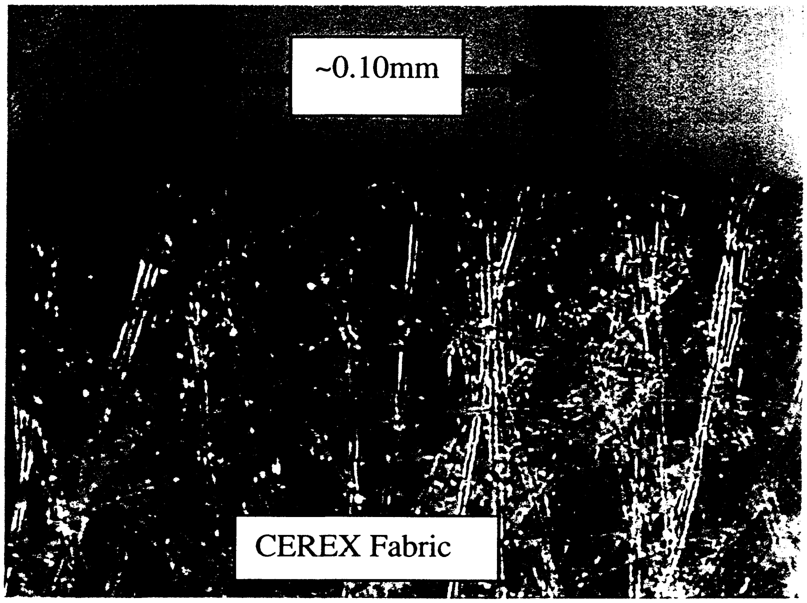
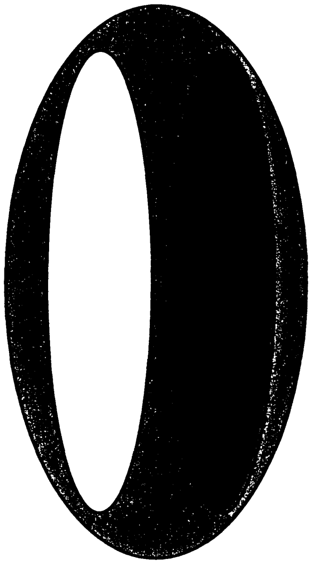


Figure 1-5

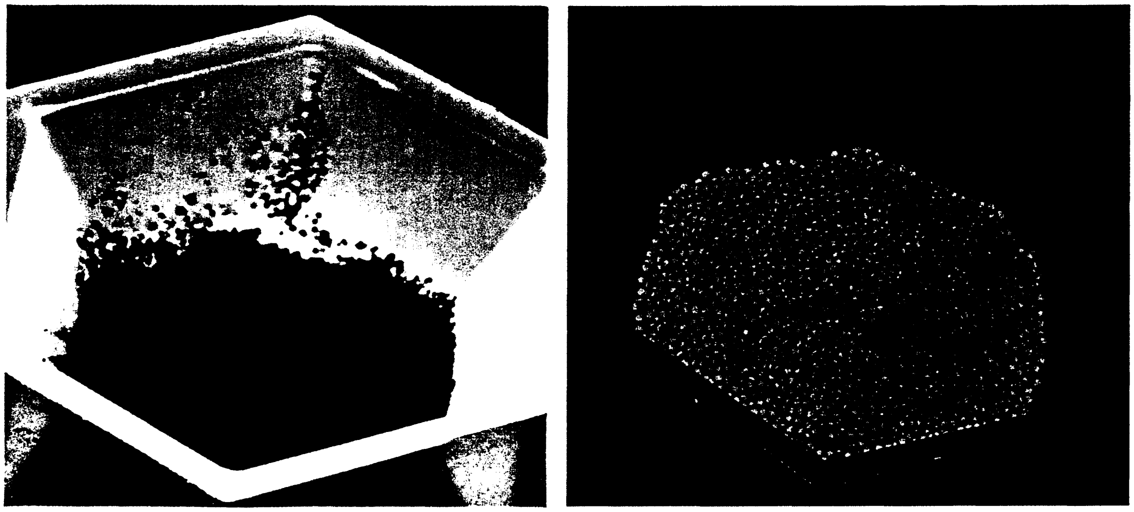
Getter Assembly



3 Primary Components

- ● VEI H_2 Getter
- ○ 4A Mol Sieve
- ◍ CEREX Fabric

Figure 1-6



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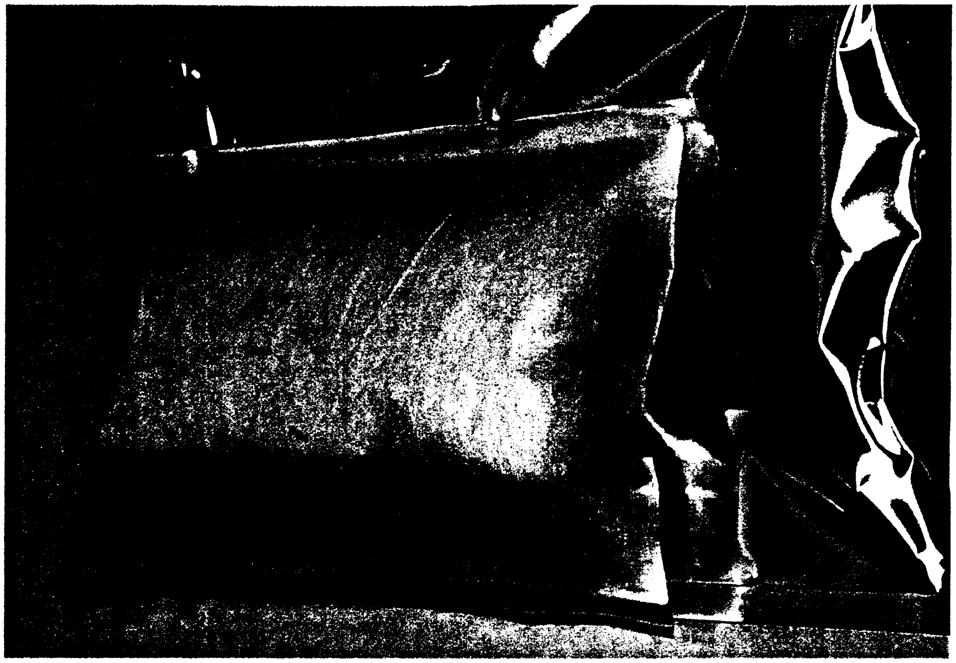

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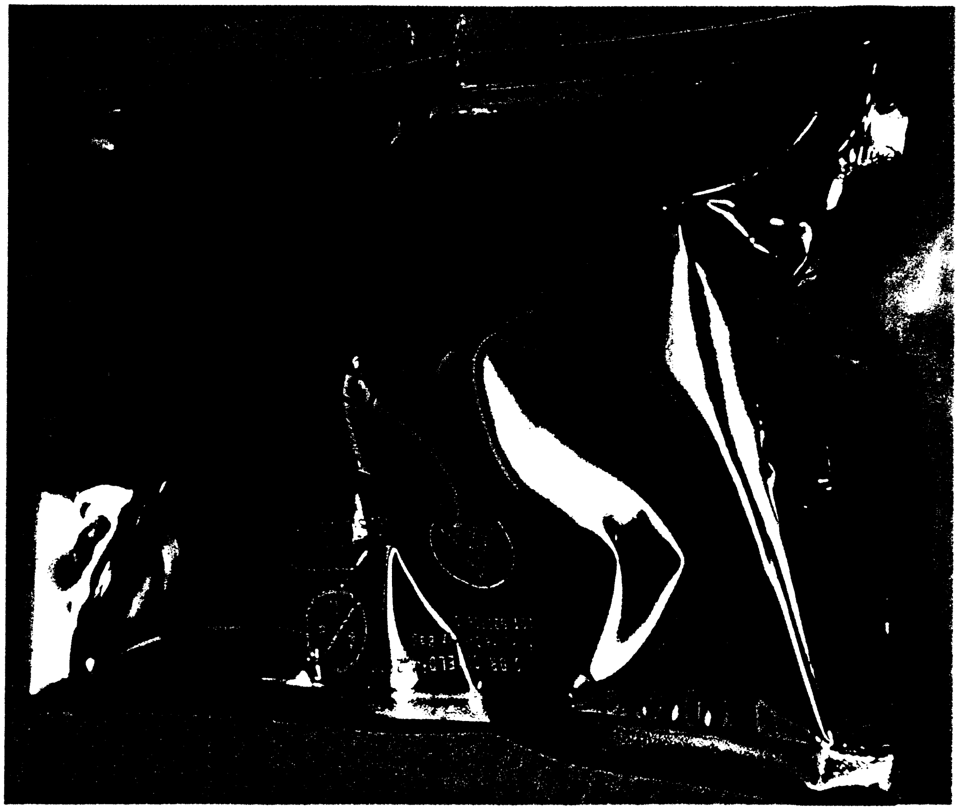

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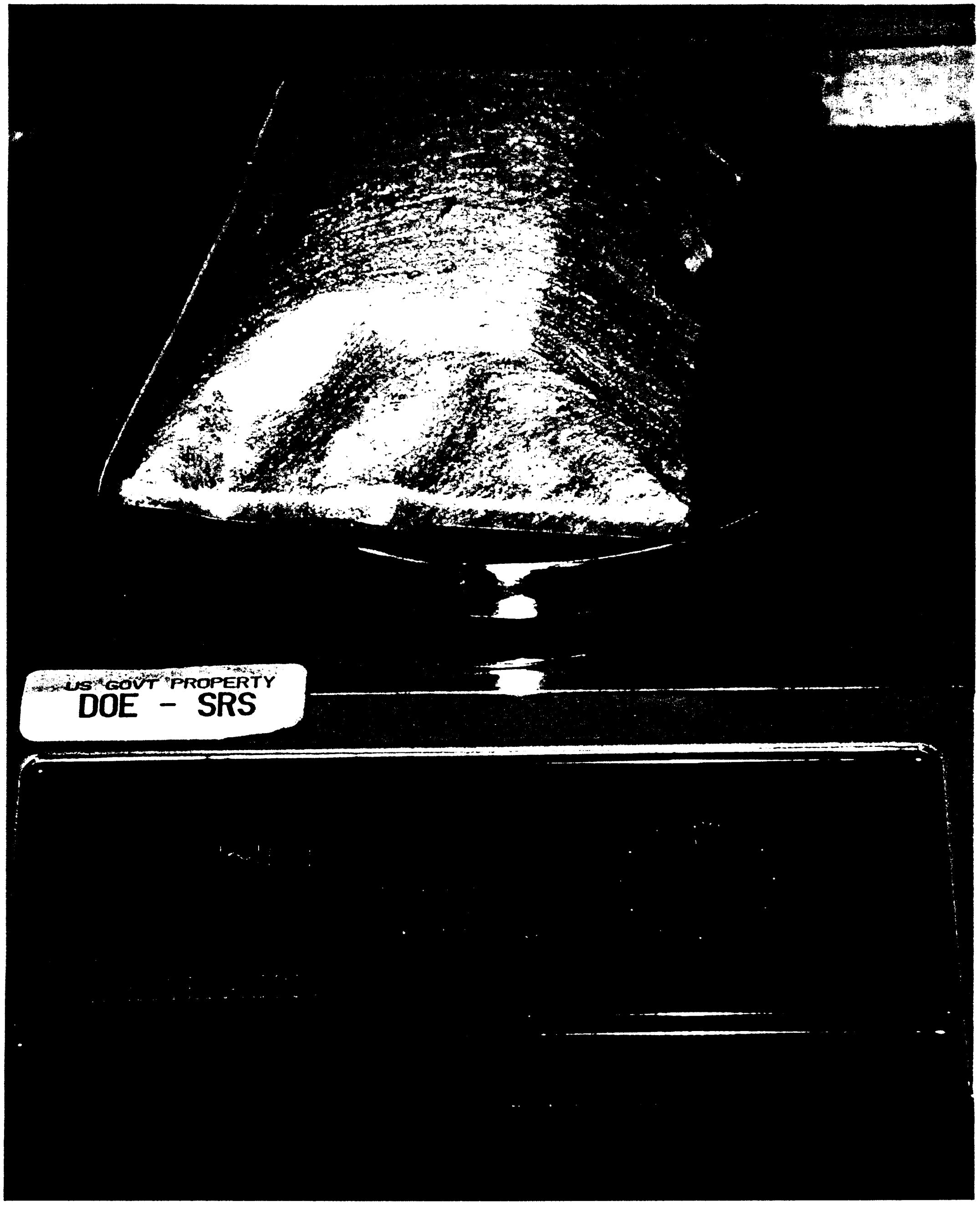

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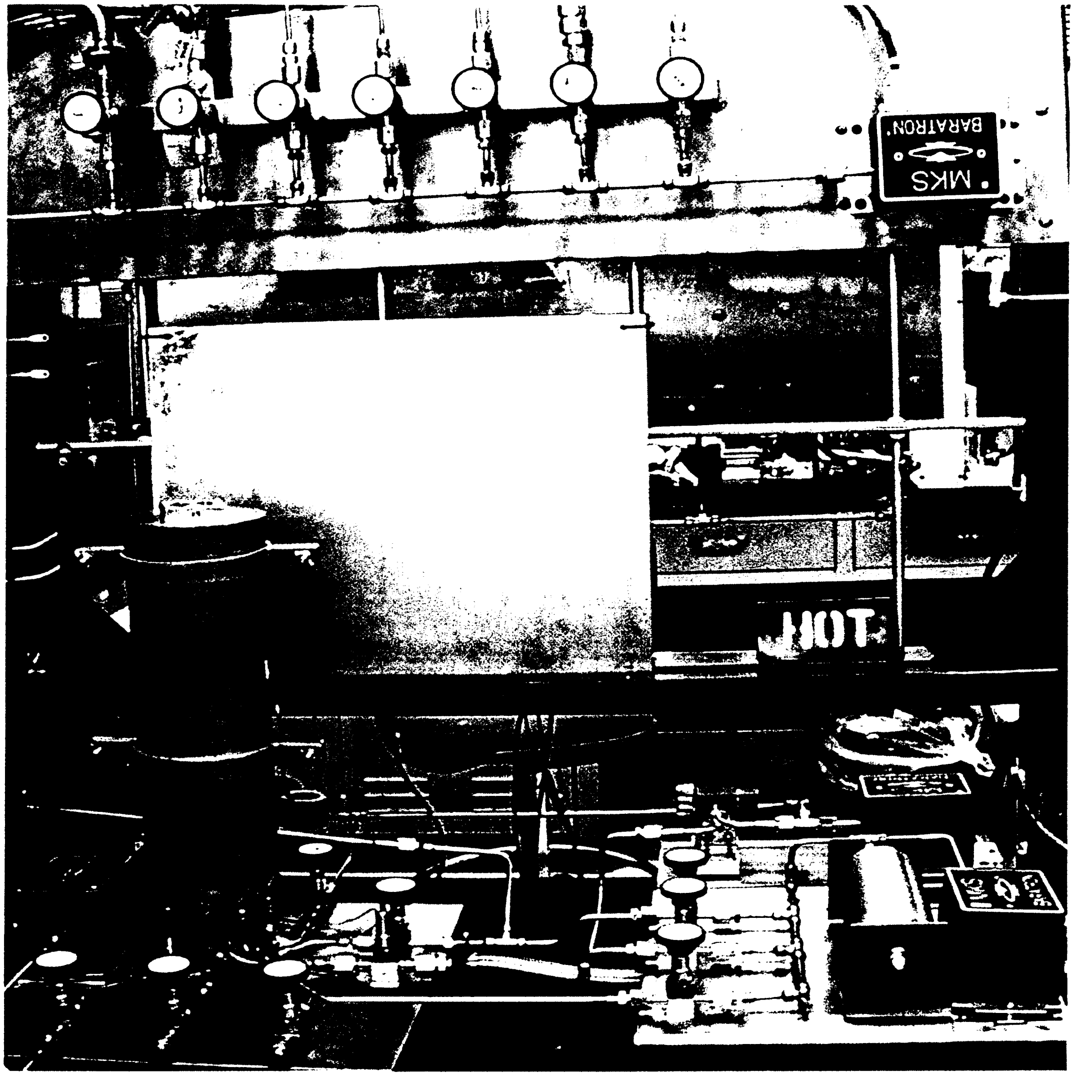

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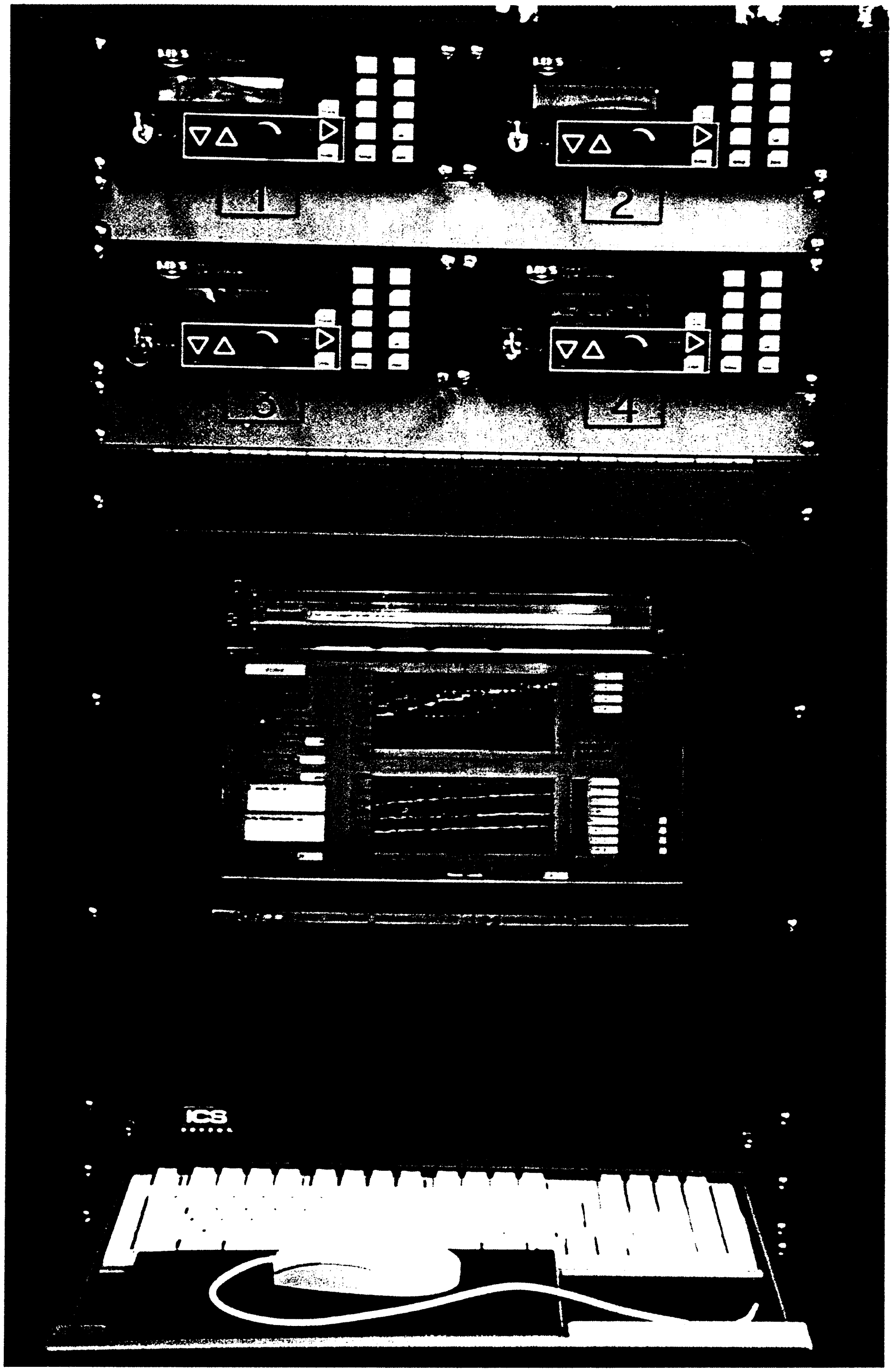

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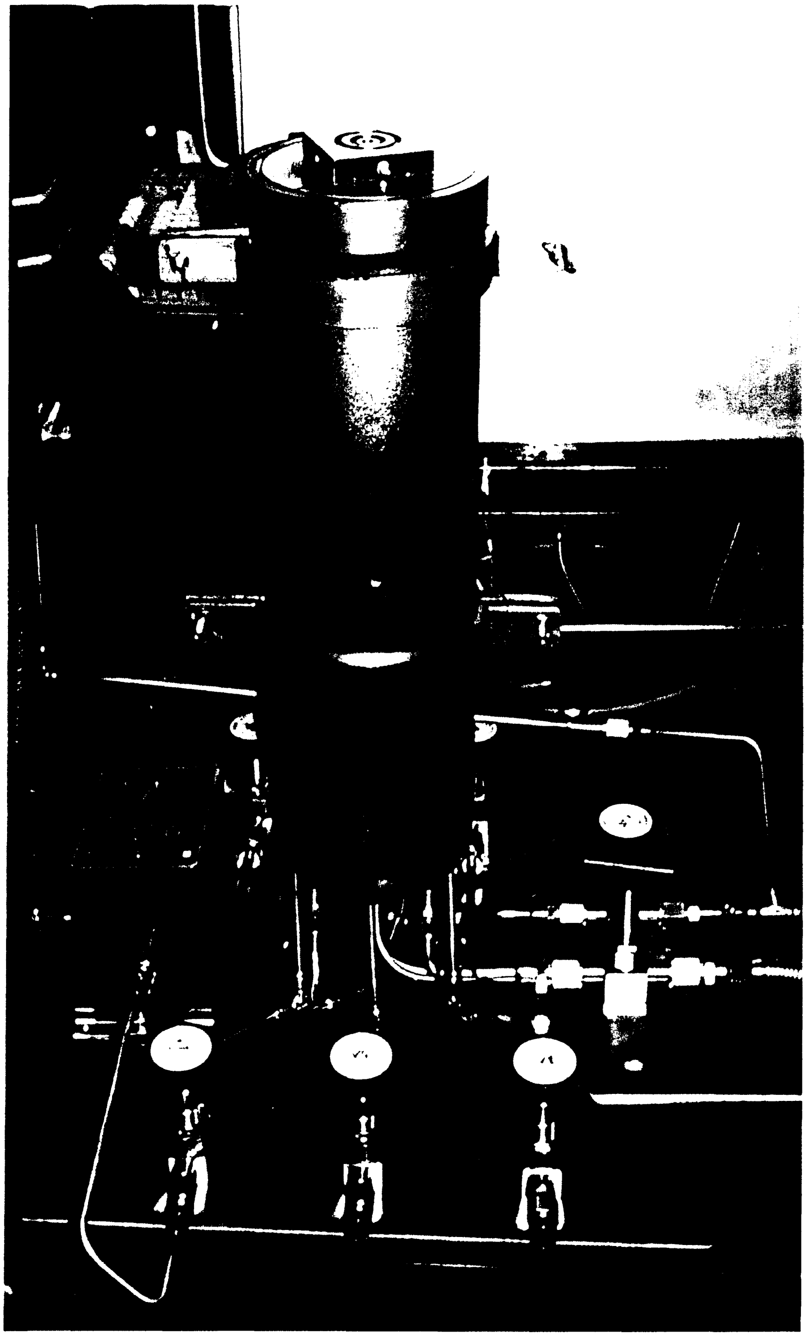

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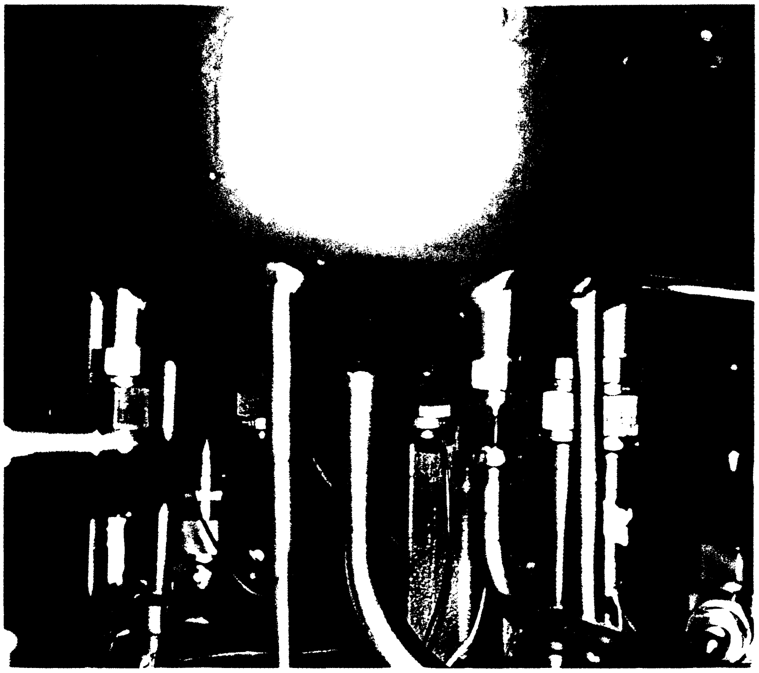
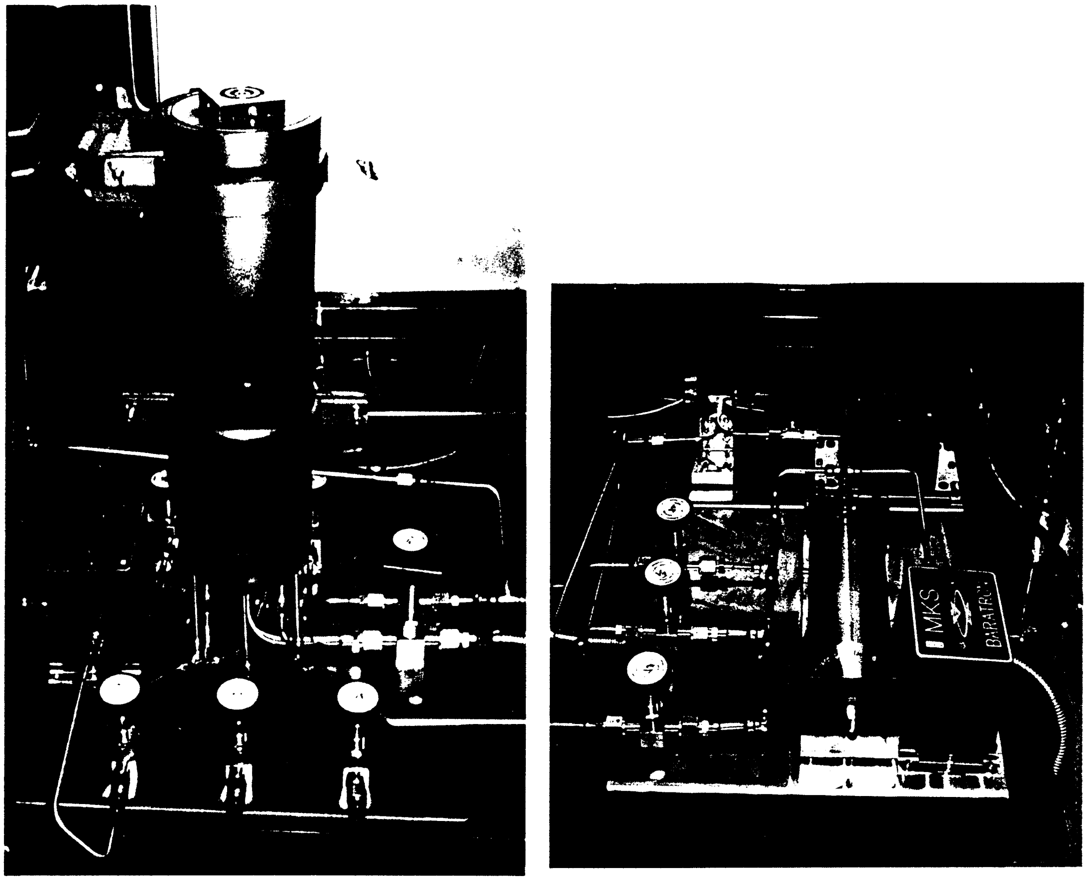


Figure 1-14

Figure 1-15



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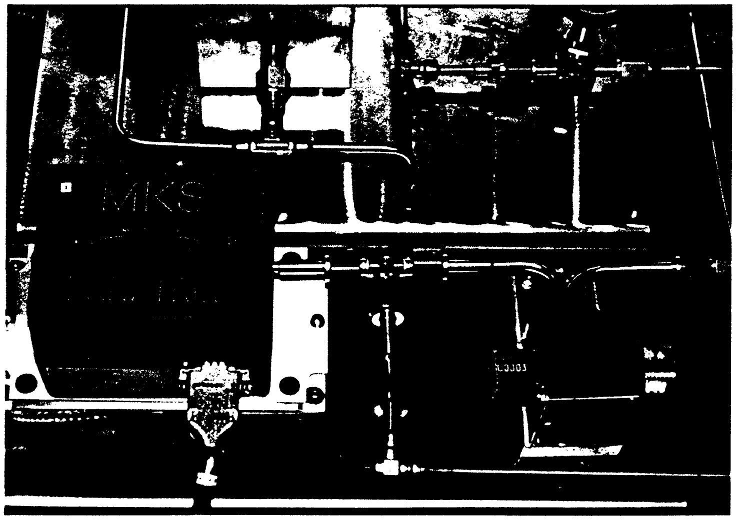


Figure 1-17

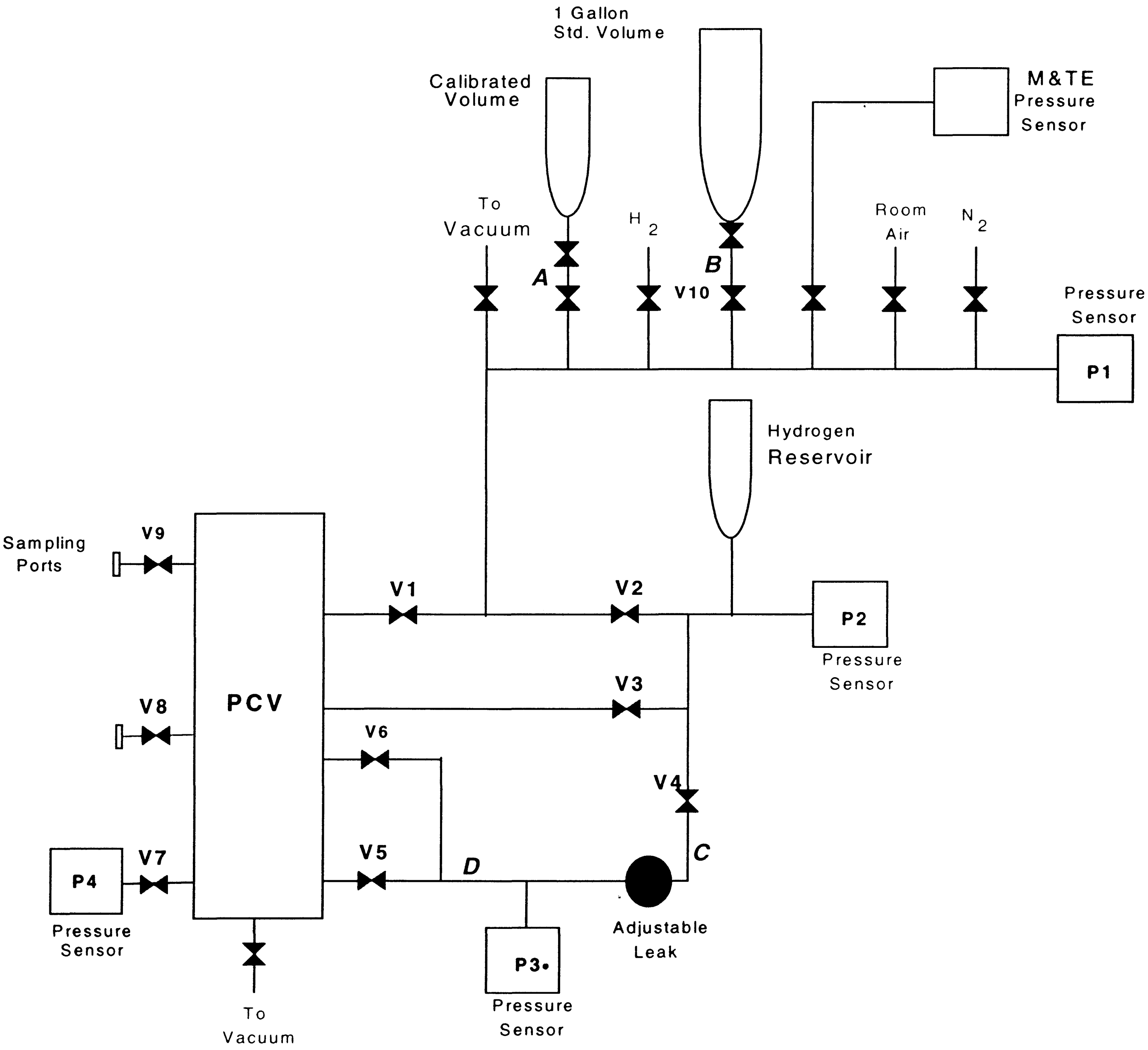
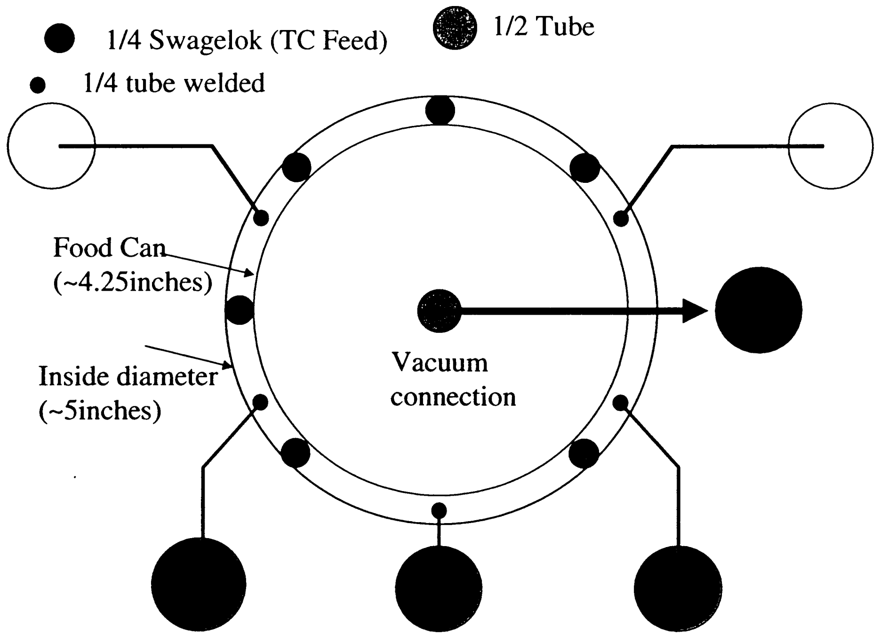

1 Gallon
Std. Volume
Calibrated
Volume
M&TE
Pressure
Sensor
To
Vacuum
H 2
Room
Air
N 2
A
B
V10
Pressure
Sensor
P1
Hydrogen
Reservoir
Sampling
Ports
V9
V1
V2
P2
Pressure
Sensor
V8
PCV
V3
V6
V4
C
P4
V7
V5
D
Pressure
Sensor
Adjustable
Leak
P3
Pressure
Sensor
To
Vacuum

Figure 1-18



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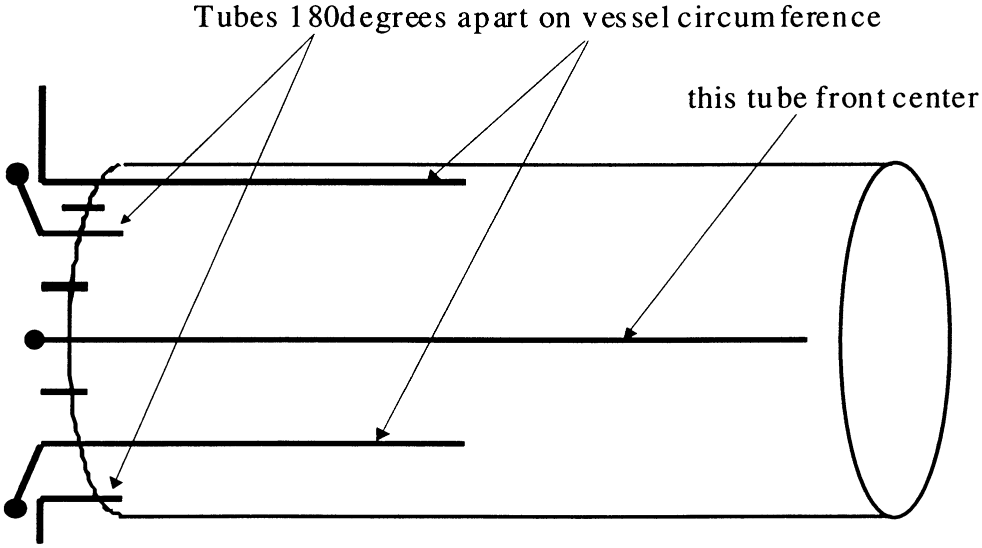

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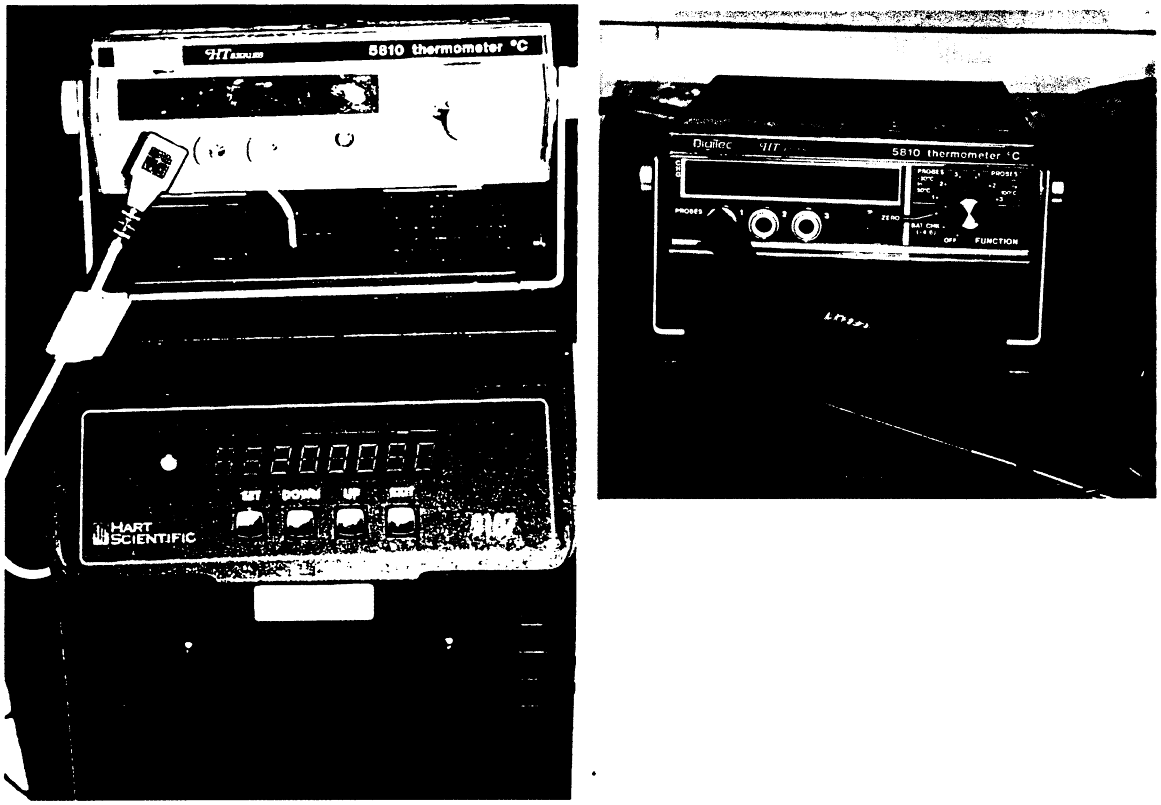


Figure 1-21

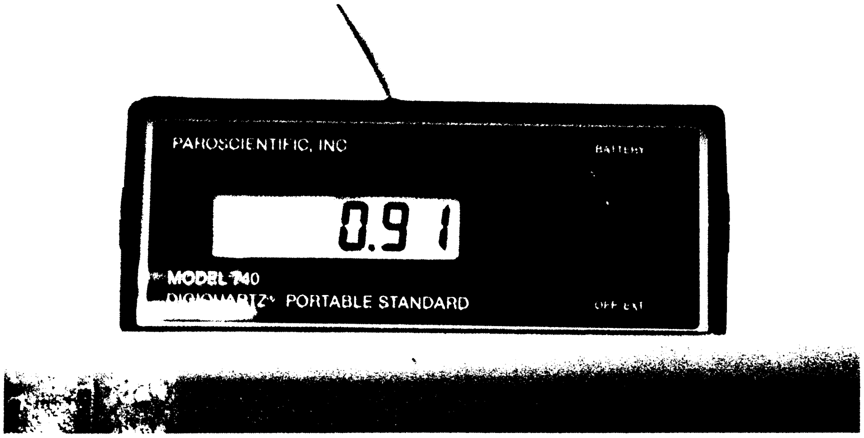


Figure 1-22

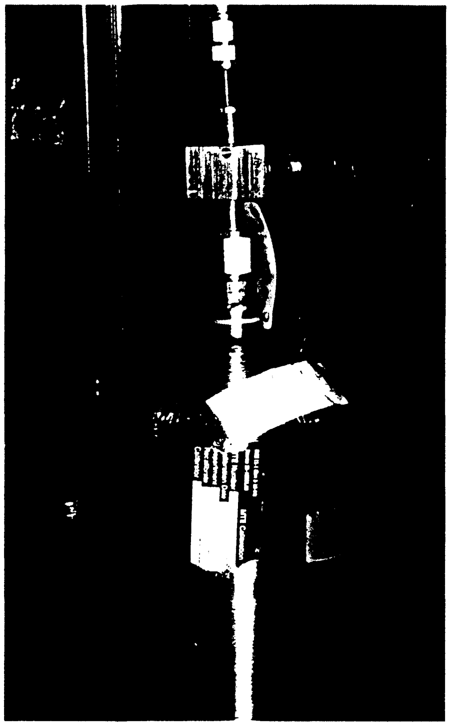


Figure 1-23

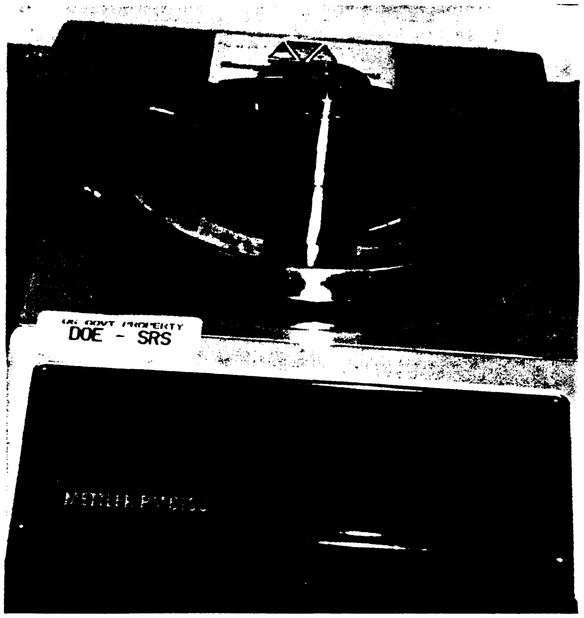
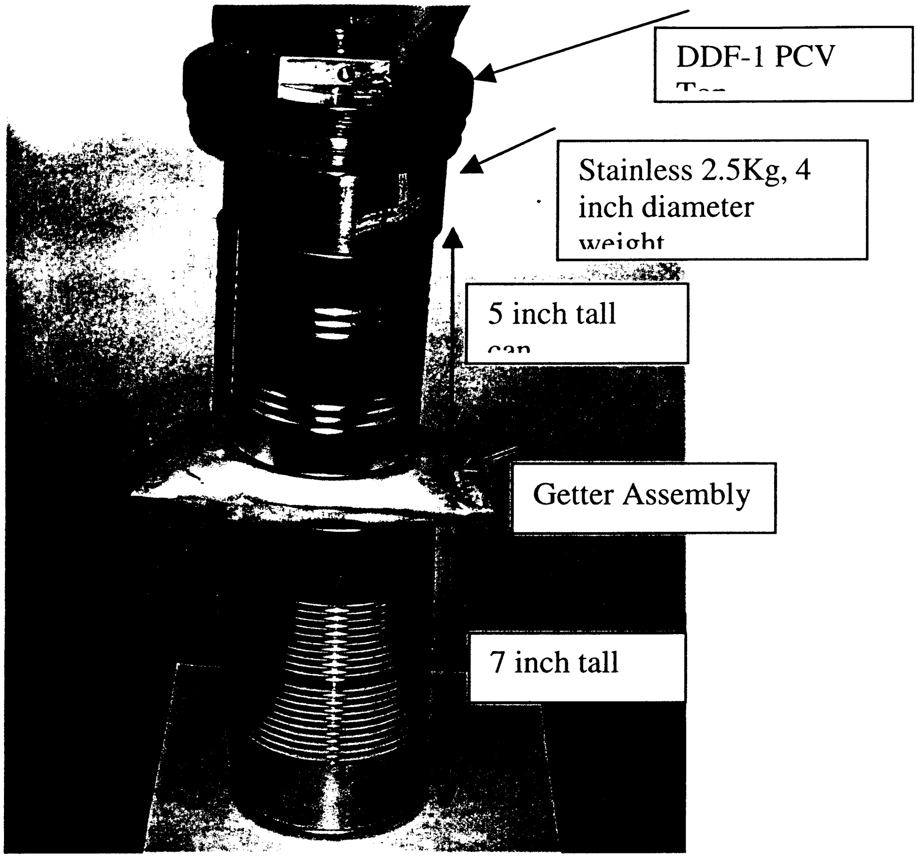


Figure 1-24



DDF-1 PCV

Stainless 2.5Kg, 4 inch diameter weight

5 inch tall can

Getter Assembly

7 inch tall

Figure 1-25

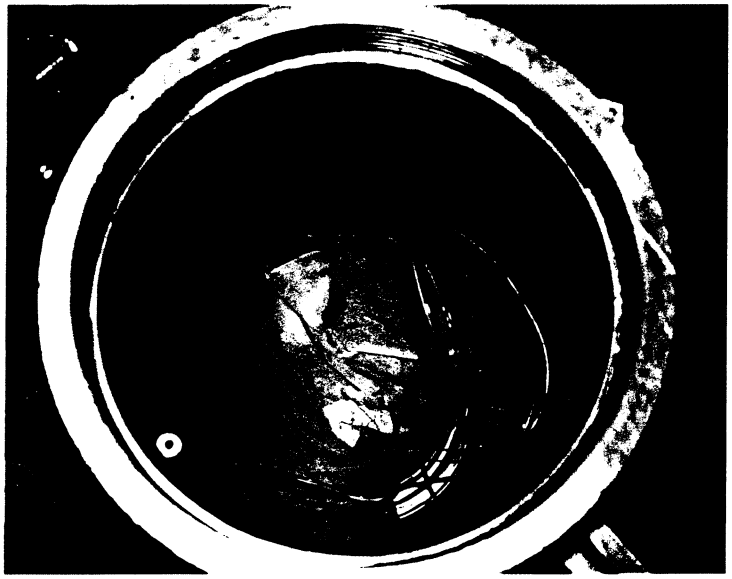


Figure 1-26

List of Vendors

List of Vendors for Hydrogen Getter Assembly Components

CEREX®Fabric Type 23, 2.0 oz./yd^2, 20" wide x 130 yards long
CEREX Advanced Fabrics supplied the fabric used to develop and build the getter assemblies used for this test. The Point of contact at CEREX is listed below.

Dan Baillie, Customer Service
CEREX Advanced Fabrics, L.P.
610 Chemstrand Road
Pensacola, FL 32533
Phone (850) 968-0100
Fax (850) 968-5229

The technical data sheet for this fabric and additional technical data provided by the manufacturer has been scanned and is included as Attachment 2.

CEREX Bags Assembly

The CEREX fabric described above was assembled manufactured into bags by a local vendor using heat sealers. The Point of Contact for this aspect of manufacturing the getter assembly is provided below:

Rian True
Carolina CoverTech
114 Shortcut Road
North Augusta, SC 29841
Phone (803) 278-4141

The assembled bags were assigned part numbers as follows:

Part Number: CCTSB1
Size: 8 inches tall by 3 inches wide
Cost: $2.00 each

Part Number: CCTMB1
Size: 9 inches tall by 4.5 inches wide
Cost: $2.00 each

Note: 250 CCTSB1 and 125 CCTMB1 were ordered on 12/5/00 and delivered on 1/20/01. This is expected to supply needed components for initial production of getter assemblies.

Hydrogen Getter

The polymer getter used as part of the getter assembly was invented by Sandia National Lab and is produced under exclusive license in the US by Vacuum Energy Incorporated (VEI). The MSDS for this product has been scanned and is included in appendix 3 of this report. Our point of contact with Vacuum Energy follows:

Bradley Phillip
Vacuum Energy Incorporated
Cleveland, OH 44128
Phone (216) 586-5459

Note: 18Kg of the VIP product was purchased in November 2000 at a cost of $750 per Kg. The purchase requisition number was AC14073. It is anticipated that VEI would be requested to repackage this product into the desired configuration at some later date (e.g. fill and heat seal CEREX bags).

Molecular Sieve

The source of molecular sieve in not viewed as critical; however, the tests reported in this document were conducted using the following product, which is supplied in a pre-dried form (i.e. at less than 2% moisture).

Multisorb Mol Sieve 4A
8x12 mesh
2mm Bead Bulk
Part Number: 00856AH02

The local point of contact for Multisorb Technologies follows:

Rod Dobson
Multisorb Technologies, Inc.
864-486-0000

The Multisorb main office is located:

325 Harlem Road
Buffalo, NY 14224
Phone: (716) 824-4091
www.multisorb.com

Note: Rod Dobson provided the sample used for the DDF-1 getter assembly test. VEI has an established working relationship with Multisorb and should be capable of packaging this 4A molecular sieve in CEREX bags similar to the getter product.

Foil Lined Bags

A high quality bag was selected for packaging the final product and maximize the getter assembly shelf life. These heat sealed bags were purchased from the following supplier:

ULINE Shipping Supply Specialists
2200 S. Lakeside Drive
Waukegan, IL 60085
Phone (800) 295-5510
www.uline.com

The product description used for these tests follows:

Model Number: S-6497
Size: 8 inches wide by 10 inches tall
Cost: $50 per 100 bags

These bags meet Mil Spec MIL-PRF-81705D, Type I and reduce transmission of vapor and moisture a factor of 15 over standard plastic bags. The Military specification for these bags requires water vapor transmission to be less than 0.02 grams per 100 square inches in 24 hours.

Manufacturer Data on CEREX Fabric

CEREX ADVANCED FABRICS, L.P.

May 31, 2000

Mr. Ron Livingston
1146 Ridgemont Drive
Aiken SC 29803

Dear Mr. Livingston:

Included in this shipment are the following roll sample(s) of our nylon spunbonded product(s):

CEREX® Type 23	2.0 oz./yd.^{2}	20" wide X 130 yards long

These rolls are being sent to you at the request of Jim Bostwick, Regional Sales Manager. If you have any questions regarding this sample, you may contact Jim at (850) 983-8584. Or, if I may assist you in any way here at the plant, please do not hesitate to call me at (800) 572-3739. Thank you for your interest in our products.

Sincerely,

Dan Baillie
Customer Service

Enclosure

cc: Jim Bostwick

610 Chemstrand Road
Pensacola, FL 32533 U.S.A.

THE NYLON ADVANTAGE™

Telephone (850) 968-0100
Facsimile (850) 968-5229

06/05/00 CEREX ADVANCED FABRICS - SHIPPING LIST PAGE 1

SOLD-TO: CEREX ADVANCED FABRICS, L.P.
610 CHEMSTRAND ROAD

CANTONMENT FL 32533

CAF ORDER: 50141

CUSTOMER PO#: SAMPLE/RON

SHIP-TO: MR.RON LIVINGSTON
1146 RIDGEMONT DRIVE

AIKEN SC 29803

ORDER QTY: 130

SHIPPER: UPS

PRINT ONLY

STYLE	LOT	LOCATION	L.METERS	NET KG	L.YARDS	NET LBS
23-200200-NN1	G37073	1-CF-00-0	119	4.1	130	9
1	ROLLS		119	4.1	130	9

TOTAL NUMBER OF ROLLS	1	
TOTAL LENGTH	119 METERS	130 YARDS
TOTAL NET WEIGHT	4.1 KG	9 LBS
TOTAL GROSS WEIGHT	5.4 KG	12 LBS

FREIGHT CHARGES__________ RATE_______ AMOUNT______________

FREIGHT CODE_______ ROUTE________ BILL OF LADING NO__________

CEREX Advanced Fabrics, Inc.

610 Chemstrand Road
Cantonment, Florida 32533 USA

Phone 1-850-968-0100
Fax 1-850-968-5229

May 31, 2000

To: Mr. Ron Livingston
Fax 1-803-725-2756

From: Tom Backus

SUBJECT: Filtration Information

Dear Ron,

It was a pleasure speaking with you today concerning a filtration application using either Cerex® or PBN-II® spunbond nylon fabrics. Under separate cover, we will be sending you a 100 yard sample of ~~1.0~~ 2 ounce per square yard (osy) Cerex® for your initial evaluation.

As discussed, we have developed summary test data on the micron rating range for our fabrics a summary of which is attached. For a reference point, on a one test basis using a Coulter Porometer, the following data were observed :

Cerex® 1.00 osy	54.5 microns
PBN-II® 1.00 osy	60.4 microns

Thanks again for your interest in our products. If you require additional information, please contact Jim Bostwick, our Regional Sales Manager, on 850-983-8584.

Best Regards,

Tom Backus

Thomas Backus
EVP, Sales & Marketing

CC: Jim Bostwick

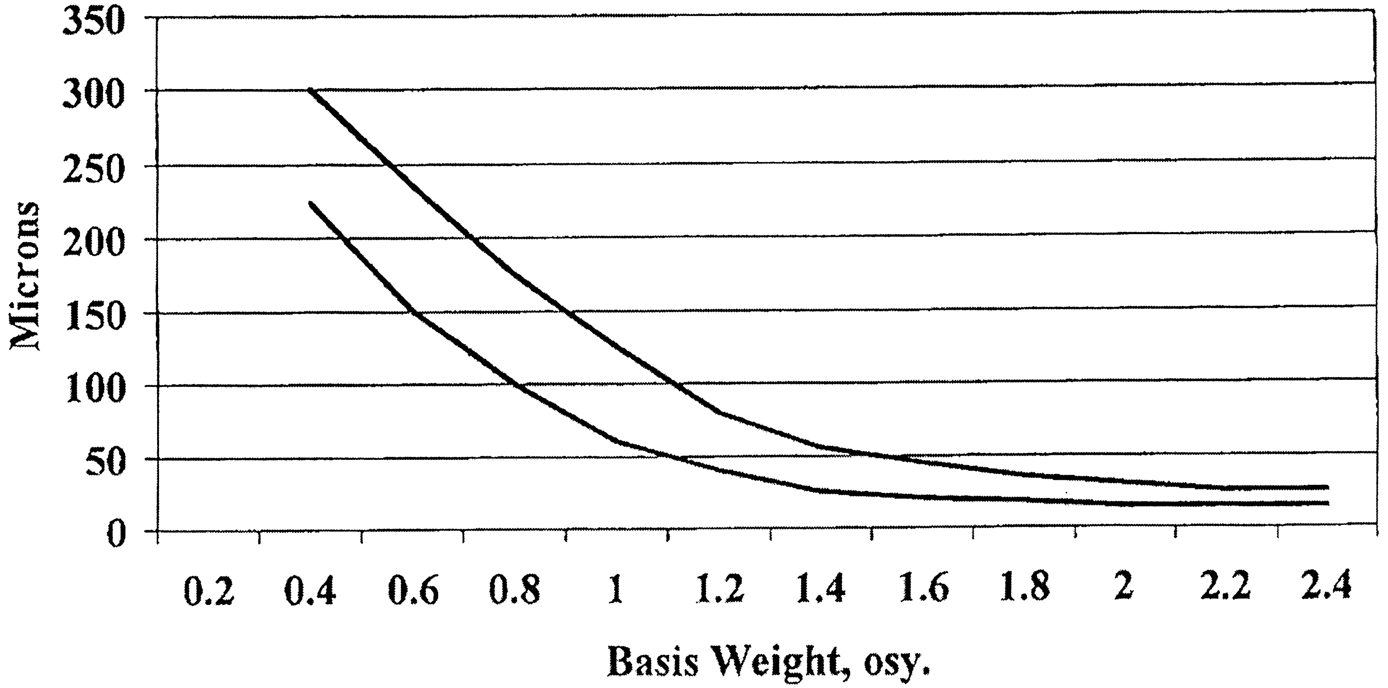
The “Micron” Rating of a Filter Medium

- There is neither a standard definition of “micron” rating, nor a standard method of determining “micron” rating
  - The rating can refer to the diameters of the pores in filter medium, or
  - It can mean the diameter of a particle retained in or on the filter medium with high efficiency
- The goal is determining the smallest diameter particle that is intercepted by the filter medium with high efficiency
- The rating of a filtration medium can be affected by changes in the fluid (gas or liquid), changes in the pressure drop, and changes in the characteristics of the particles used in the test.
- The comparative performance of different filter media can be determined only if identical test conditions are used for the media tested.

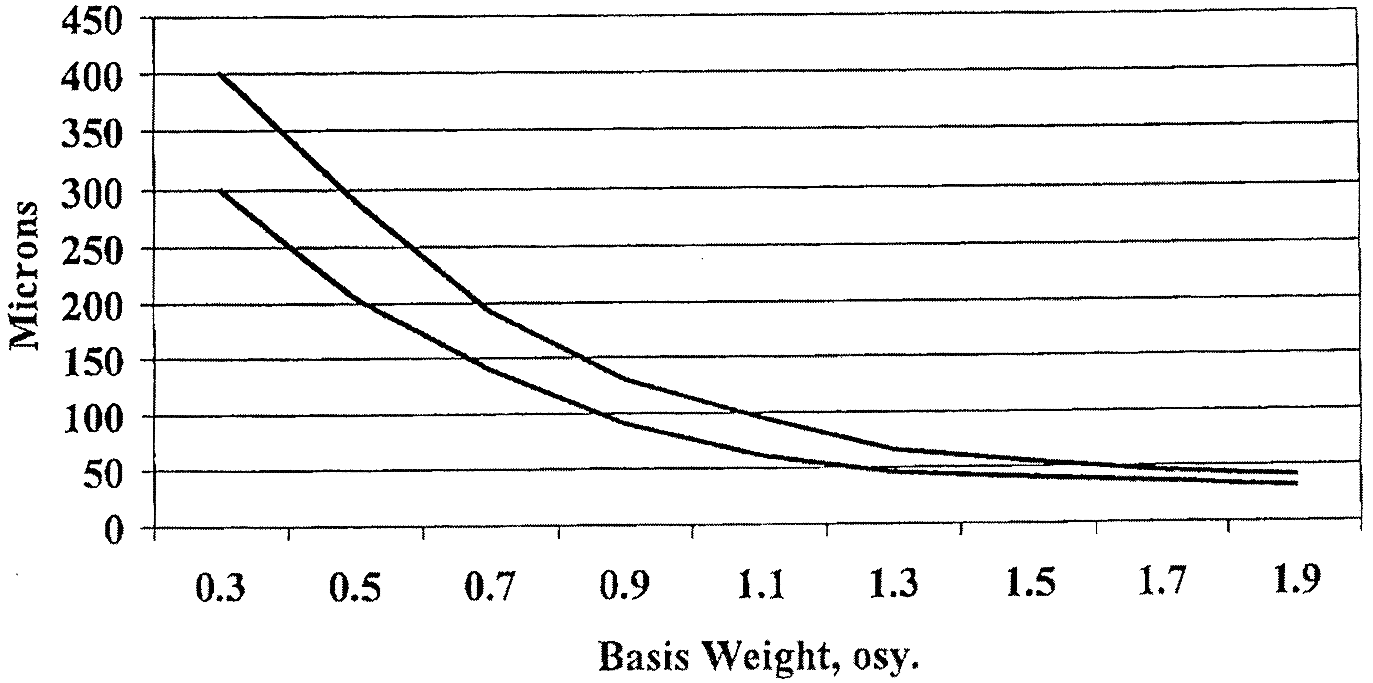
The “Micron” Rating of a Filter Medium (Cont’d)

- The following chart contains our best estimation of the micron ratings for our products as a function of fabric basis weight
  - The charts contain two curves, and the estimated micron rating for the fabric is in the area between the two curves
  - The range of micron ratings for each basis weight is intended to account for filter medium perfomance variability resulting from different fluids, pressure drops, and particle characteristics
- These figures are intended to be used <u>only</u> for general estimations of filter performance

Apparent Micron Rating of Cerex® as a Function of Basis Weight



Apparent Micron Rating of PBN-II® as a Function of Basis Weight



7/19/99

TYPICAL PHYSICAL PROPERTIES OF CEREX® FABRICS

Cerex® products are nonwoven fabrics made by spinning and autogenously bonding continuous filaments of nylon into a flat, smooth, strong, crisp fabric. Autogenous bonding is achieved by exposing the nylon web to a chemically activating gas phase that is later removed from the web. The fabric is available in weights ranging from 0.25 ounces/square yard (9 grams/square meter) to 3.0 ounces/square yard (102 grams/square meter) in widths up to 120 inches (3 meters).

Filament Geometry	Fabric Weight [1]		Average Thickness [2]		Grab Tensile Strength [3]				Trapezpoid Tear Strength [4]				Burst Strength [5]		Air Permeability [6]	
					Machine Direction		Cross Direction		Machine Direction		Cross Direction					
	osy	gsm	mil	mm	lbs	N	lbs	N	lbs	N	lbs	N	psi	kPa	cfm/ft²	m³/m²/sec
Round	0.25	8	2.6	0.07	7	31	4	18	4.5	20	2.1	9	9	62	1350	7.1
Round	0.3	10	2.6	0.07	8	36	4	18	5.0	22	2.5	11	10	69	1327	6.7
Round	0.4	14	3.0	0.08	12	53	6	27	6.7	30	3.7	16	13	90	1127	5.7
Round	0.5	17	3.3	0.08	16	71	10	44	7.3	32	4.3	19	16	110	949	4.8
Round	0.6	20	3.6	0.09	20	89	12	53	8.3	37	5.2	23	20	138	768	3.9
Round	0.7	24	3.8	0.10	25	111	15	67	9.3	41	6.0	27	24	165	664	3.4
Round	0.85	29	4.4	0.11	30	134	18	80	10.3	46	7.0	31	29	200	498	2.5
Round	1.0	34	4.8	0.12	38	165	22	98	11.2	50	7.9	35	34	234	405	2.1
Round	1.5	51	6.4	0.16	59	263	35	156	14.0	62	10.0	44	53	365	250	1.3
Round	2.0	68	7.9	0.21	78	347	48	214	22.0	98	13.0	58	71	489	163	0.8
Round**	3.0	102	9.5	0.24	123	547	87	387	32.0	142	24.0	107	110	758	98	0.5
Trilobal**	0.25	8	2.6	0.07	7	29	4	17	3.7	16	1.7	8	12	82	1385	7.0
Trilobal	0.4	14	3.4	0.09	12	52	7	31	5.1	23	3.6	16	17	117	1119	5.7
Trilobal	.045	15	3.6	0.09	13	60	8	36	6.1	27	4.4	20	18	123	1053	5.4
Trilobal**	0.5	17	3.8	0.10	16	71	10	43	6.2	28	5.3	24	22	148	875	4.5
Trilobal**	0.85	29	4.5	0.11	27	119	18	79	5.8	36	5.7	25	33	224	515	2.6
Trilobal**	1.0	34	5.0	0.13	34	149	24	107	8.8	39	7.2	32	38	260	429	2.2
Trilobal**	1.5	51	6.9	0.18	47	207	36	158	11.3	50	10.7	48	55	378	272	1.4

[1]ASTM D-3776-85; [2]ASTM D-1777-80; [3]ASTM D-5034-90; [4]ASTM D-1117-80; [5]ASTM D-3786-87; [6]ASTM D737-75

** These are tentative typical properties based on one roll sampled from one production lot

Other basis weights available upon request.

STANDARD ROLL SIZES* AND WEIGHTS OF CEREX® FABRICS**

				Regular Size						Jumbo Size					
Fabric Weight		Maximum Roll Width		Roll Length		Approximate Roll Diameter		Approximate Roll Weight		Roll Length		Approximate Roll Diameter		Approximate Roll Weight	
osy	gsm	inches	mm	yards	m	inches	cm	lbs	kg	yards	m	inches	cm	lbs	kg
0.25	8	88	234	3,000	2740	14	36	115	52	10,000	9,140	25	63	382	173
0.3	10	117	297	3,000	2740	15	38	183	83	10,000	9,140	27	68	609	276
0.4	14	120	304	3,000	2740	17	43	250	113	7,000	6,400	25	63	583	264
0.5	17	120	304	3,000	2740	19	48	313	142	7,000	6,400	28	71	729	330
0.6	20	120	304	3,000	2740	20	51	375	170	6,000	5,480	28	71	750	340
0.7	24	121	307	3,000	2740	21	53	441	200	6,000	5,480	30	76	882	400
0.85	29	122	310	3,000	2740	23	58	540	245	5,000	4,570	30	76	900	408
1.0	34	122	310	2,000	1,820	20	51	424	192	3,000	2,740	25	63	635	288
1.5	51	122	310	2,000	1,820	25	63	641	290	N/A	N/A	N/A	N/A	N/A	N/A
2.0	68	124	314	2,000	1,820	28	71	861	390	N/A	N/A	N/A	N/A	N/A	N/A
3.0	102	124	314	2,000	914	24	61	645	293	N/A	N/A	N/A	N/A	N/A	N/A

*Other roll sizes available upon request

** Roll weights are calculated and are based on nominal basis weights

CEREX ADVANCED FABRICS, L.P.

THE NYLON ADVANTAGE®

CEREX®

VIBRAtex®

PBN-II®

SPECTRALON®

CEREX --*The Nylon Advantage*®

CEREX Advanced Fabrics, L. P. is a premier producer of a wide range of specialty spunbond nylon fabrics that have served customers worldwide for over 20 years. The company is located near Pensacola, Florida, U.S.A., where it operates spunbond manufacturing lines based on Cerex's proprietary technology.

All of our fabrics exhibit excellent uniformity, plus high tensile and tear strength even at low fabric weights. Because the fabrics are made of nylon, one of the toughest and most durable manmade polymers, our fabrics have high sewn seam strength when fabricated into finished articles. The fabrics can be ultrasonically seamed. Fabrics edges are not subject to raveling. In addition, nylon bonds well with most adhesive systems, making these fabrics excellent materials for manufacturing laminate and composite systems. The fabrics have no finish and therefore require no scouring before being used.

The thermal stability of our nylon fabrics is outstanding, having melting points in the neighborhood of 500°F (260°C), with dimensional stability up to 400°F (200°C). They can be processed at temperatures of 425°F (215°C) for limited periods of time. These fabrics resist attack by many solvents, alkalis, and dilute acids used in manufacturing processes, and are resistant to biological degradation and to gamma radiation.

Nylon's chemical structure makes it receptive to a wide range of dyes, and is easier to dye than polyester fabrics and much easier to dye than polypropylene fabrics. The chemical structure also allows nylon fabrics to exhibit a significant moisture regain, resulting in greater comfort when the fabric is used in apparel. Our products do not cause skin irritation.

CEREX Advanced Fabrics offers four **product lines** to meet your needs:

- **Cerex®** products are nonwoven fabrics made by spinning and autogenously bonding continuous filaments of nylon into a flat, smooth, strong, crisp fabric. Autogenous bonding is achieved by exposing the nylon web to a chemically activating gas phase that is later removed from the web. This fabric is available in weights ranging from 0.2 ounces/square yard (7 grams/square meter) to 3.0 ounces/square yard (102 grams/ square meter) in widths up to 120 inches (3 meters).

- **VIBRAtex®** products are autogenous bonded fabrics available in custom colors. The colors are encapsulated in the fabric making the material resistant to bleach, fading, and washing out of the color. VIBRAtex® fabrics are available in the same basis weights and widths as Cerex® fabrics.

- **PBN-II®** products are nonwoven fabrics made by spinning and thermally bonding continuous filaments of nylon into drapable, conformable, textile-like fabric. Available weights range from 0.25 ounces/ square yard (8 grams/square meter) to 4.0 ounces/ square yard (136 grams/square meter) in widths up to 120 inches (3 meters).

- **Spectralon®** products are thermal bonded nonwoven nylon fabrics available in custom colors. The coloring of the fabrics is achieved by encapsulating the colors in the fibers and, therefore, is less likely to bleach, fade or wash out of the fabric. Spectralon® fabrics are available in the same basis weights and widths as PBN-II® fabrics.

All four fabrics are readily printed using the wide range of techniques, inks, dyes, and pigment formulations applicable to nylon.

Cerex®, VIBRAtex®, PBN-II® and Spectralon® fabrics are used in a variety of **applications**. Examples include:

Adhesive substrates
Apparel
Automotive
Carpet and rug components
Coated and laminate substrate
Filtration
Film reinforcement
Foam reinforcement
Home furnishings
Medical

Variables which can be designed into our fabrics include:

- Fiber size and geometry
- Softness
- Bond levels and patterns
- Customized colors

To explore unique solutions to your product requirements, please call 1-800-57 CEREX, 1-850-937-3320 or visit our website at WWW.CEREX.COM.

THE NYLON ADVANTAGE®

Composition

Our Cerex®, VIBRAtex®, PBN-II®, and Spectralon® fabrics are made of nylon, one of the toughest and most durable manmade polymers.

Strength and Uniformity

Our fabrics have high tensile, tear and burst strength, even at low fabric weight, and outstanding uniformity providing an excellent coating substrate.

Grab Strength ...is a measure of the "effective strength" of a fabric; that is, the strength of the fibers in a specific width together with the additional strength contributed by adjacent fibers. Typically, the grab strength is determined on a four-inch-wide strip of fabric. The tensile load is applied at the midpoint of the width through one-inch-wide jaw faces that are used to clamp the fabric. The breaking loads are averaged to the nearest pound for each fabric direction. *Grab strength is particularly important when Cerex Advanced Fabrics products are used to reinforce components. High grab strength means that fabric containing product will better maintain its integrity during handling and positioning, whether it is gripped mechanically or by hand.*

Tear strength ...is a measure of the force necessary to tear a fabric, expressed in pounds. In the trapezoid tear test, notched specimens are clamped in jaws and loaded until the fabric rips apart. *Tear strength is an indication of the ability to resist propagation of accidental tears or snags or deliberately-imposed perforation.*

Burst strength ...is a measure, expressed in pounds per square inch, of the ability to resist rupture by pressure. *Burst strength is important in filtration applications, measuring the ability of the fabric to withstand various air or liquid pressures that load the fabric.*

Quality

Cerex Advanced Fabrics is dedicated to providing high quality products and services and is proud to have attained **ISO 9001** certification.

Air Permeability

Our wide range of fabric weights allows the choice of fabric permeability. Air permeability is a measure of the ease with which air will pass through a fabric. It is measured in cubic feet of air passing through a square foot of fabric in a minute at a given pressure differential across the fabric. *Air permeability is of primary importance in the construction and performance of air filters.*

Chemical Resistance

Our fabrics resist attack by many solvents, alkalis, and dilute acids found in manufacturing processes.

Thermal Resistance

Our nylon fabrics have a melting point of approximately 500°F, with dimensional stability up to 400°F. They can be processed at temperatures as high as 425°F for limited periods of time.

Gamma Radiation Resistance

Nylon products are resistant to gamma radiation, maintaining significant physical properties after irradiation.

Printablility and Dyeability

Cerex® and PBN-II® fabrics are easily dyed with dyes typically used for nylon fabrics. All four fabrics are readily printed using the wide range of techniques, inks, dyes, and pigment formulations applicable to nylon fabrics.

For more information, call us at 800-57CEREX, 1-850-937-3320 or visit our website at WWW.CEREX.COM.

CEREX

"THE NYLON ADVANTAGE"®

ABS Quality Evaluations, Inc. ★ Quality Assurance Certification ★

ISO 9001

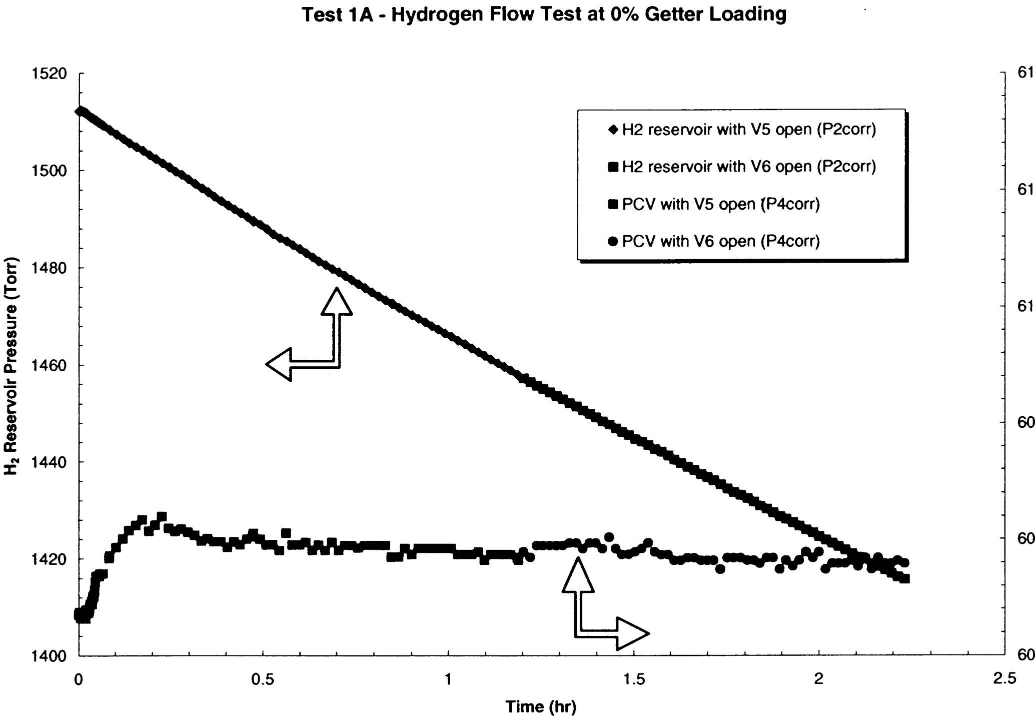
Certificate Number: 32860

Assistance in using spunbond nylon fabrics may be obtained by writing or calling:

CEREX Advanced Fabrics, L.P.
610 Chemstrand Road
Pensacola, FL 32533-6857 USA
Phone: (800) 572-3739 (Toll Free in USA)
Phone: 1-850-937-3320
Fax: 1-850-968-0688
Website: WWW.CEREX.COM

NOTICE: Nothing contained herein is to be construed as a recommendation to use any product in conflict with any patent. CEREX Advanced Fabrics, L. P., MAKES NO WARRANTIES AS TO THE MERCHANTABILITY OR FITNESS FOR A PARTICULAR PURPOSE OF ANY PRODUCTS REFERRED TO, no guarantee of satisfactory results from reliance upon information or recommendations contained herein, and disclaims all liability for any resulting loss or damage.

Rev3/98



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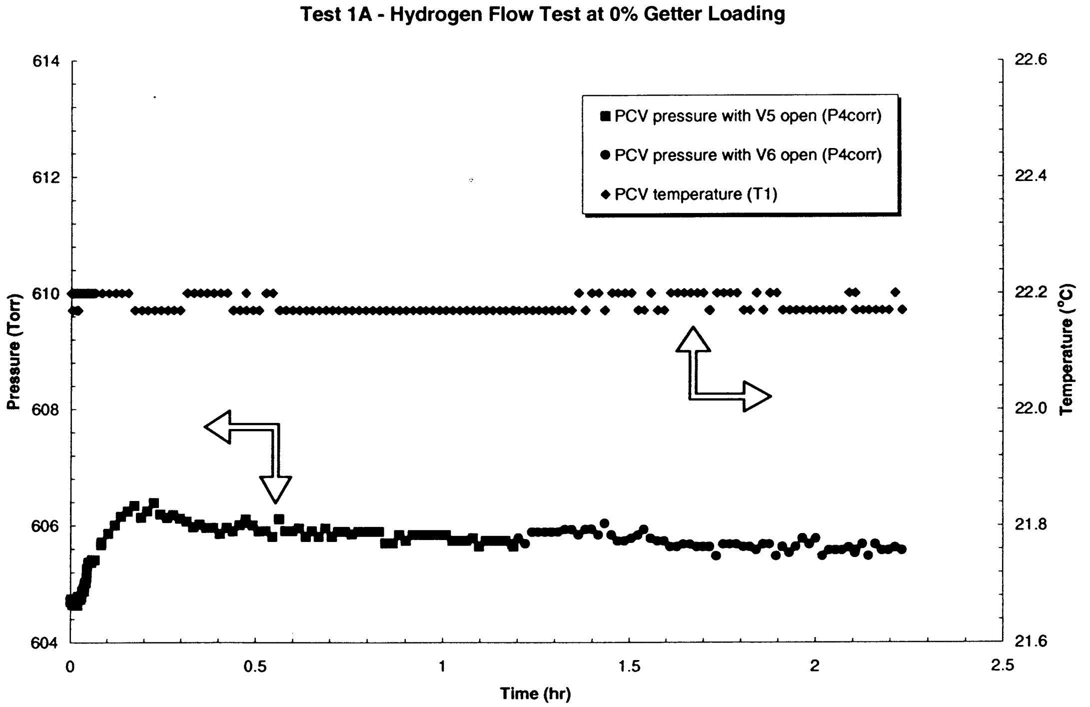

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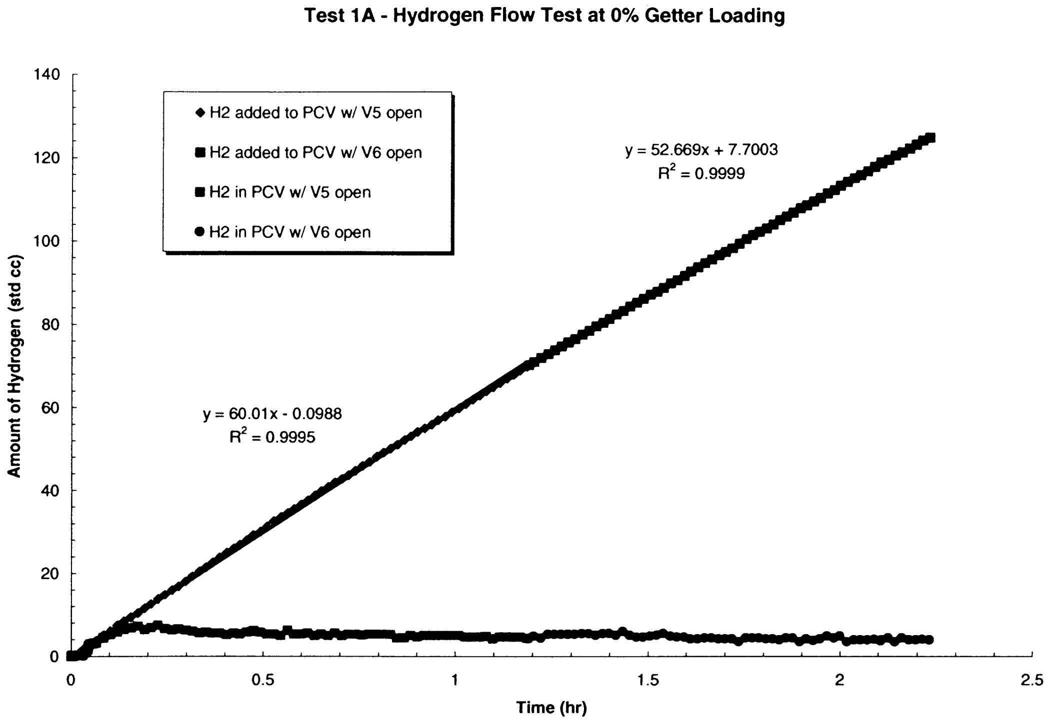

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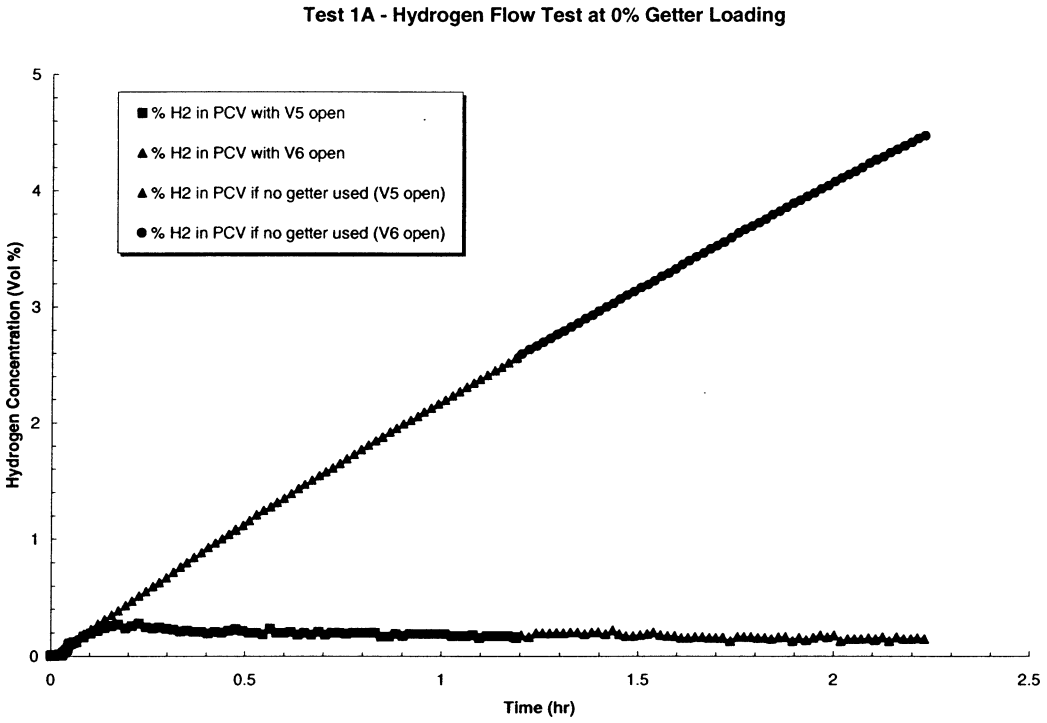

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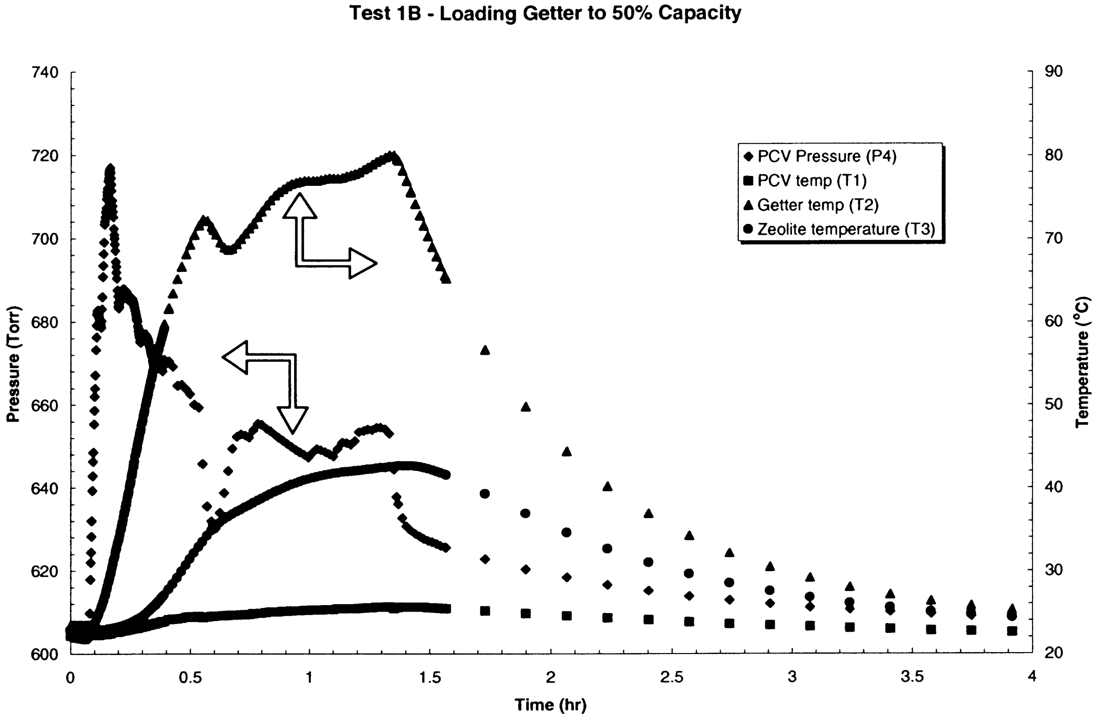

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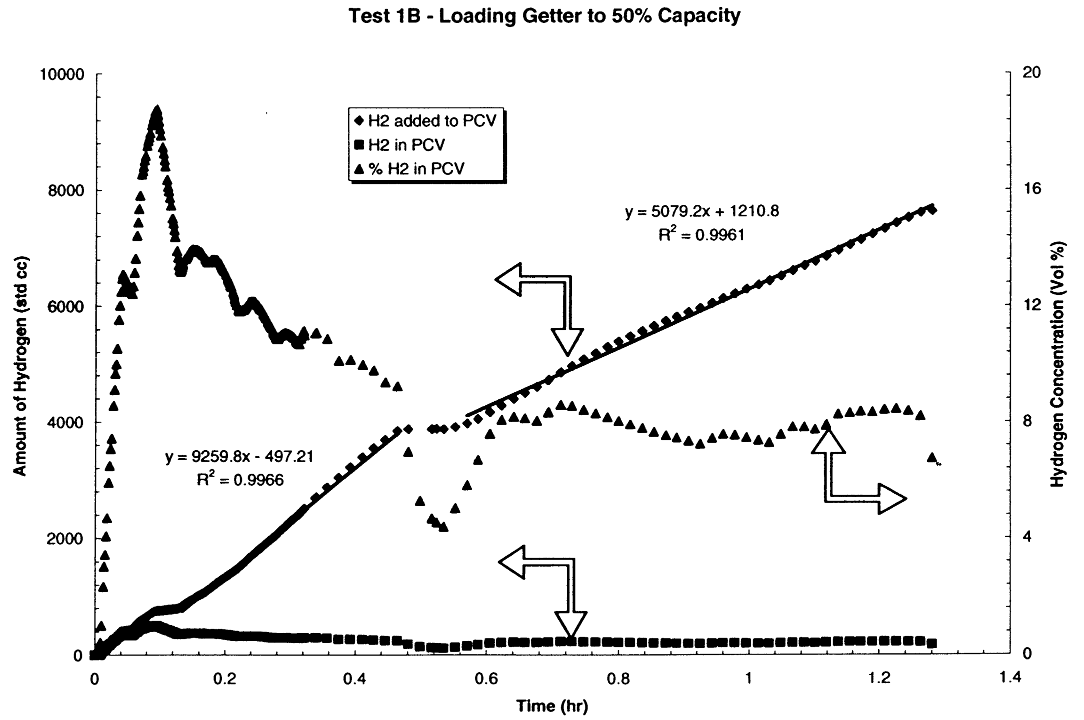

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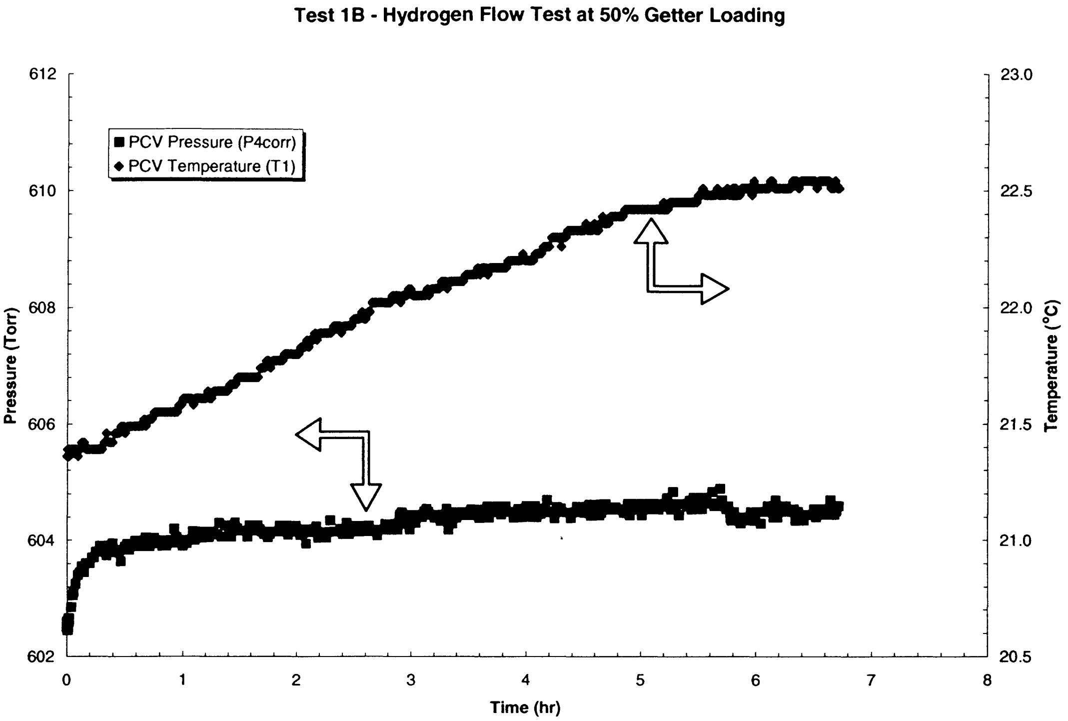

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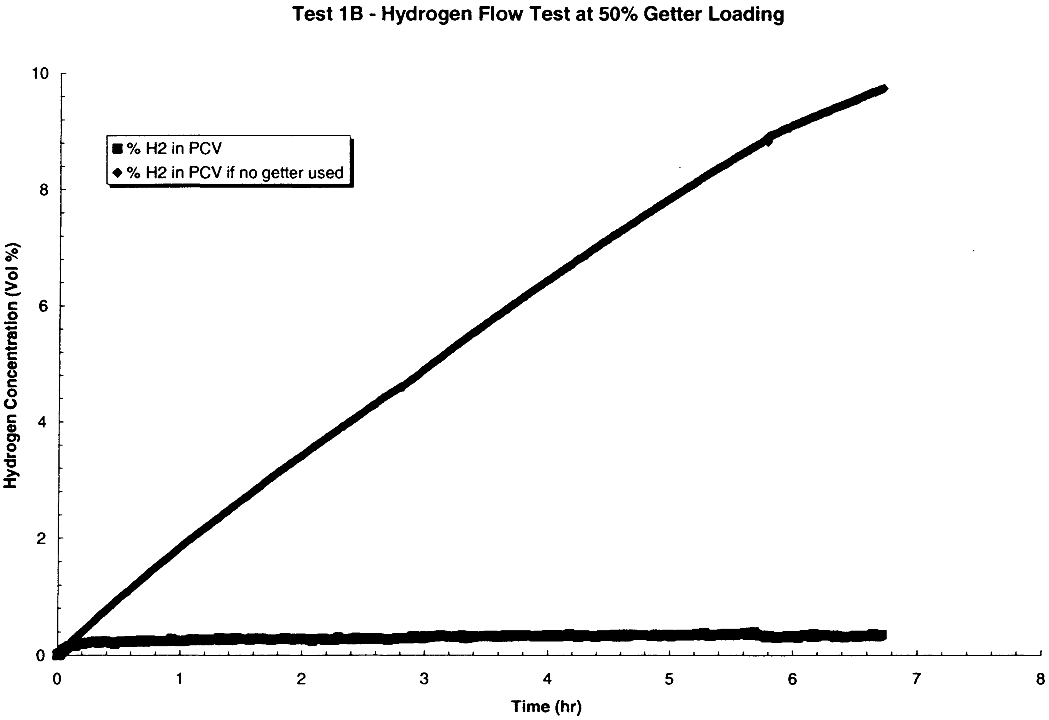

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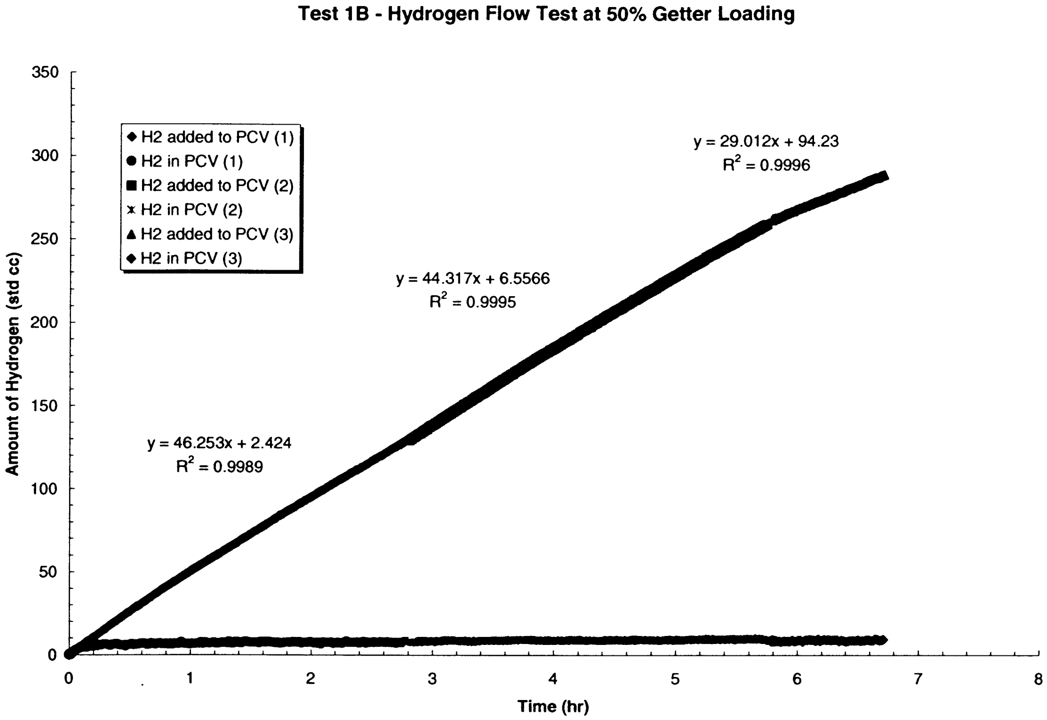

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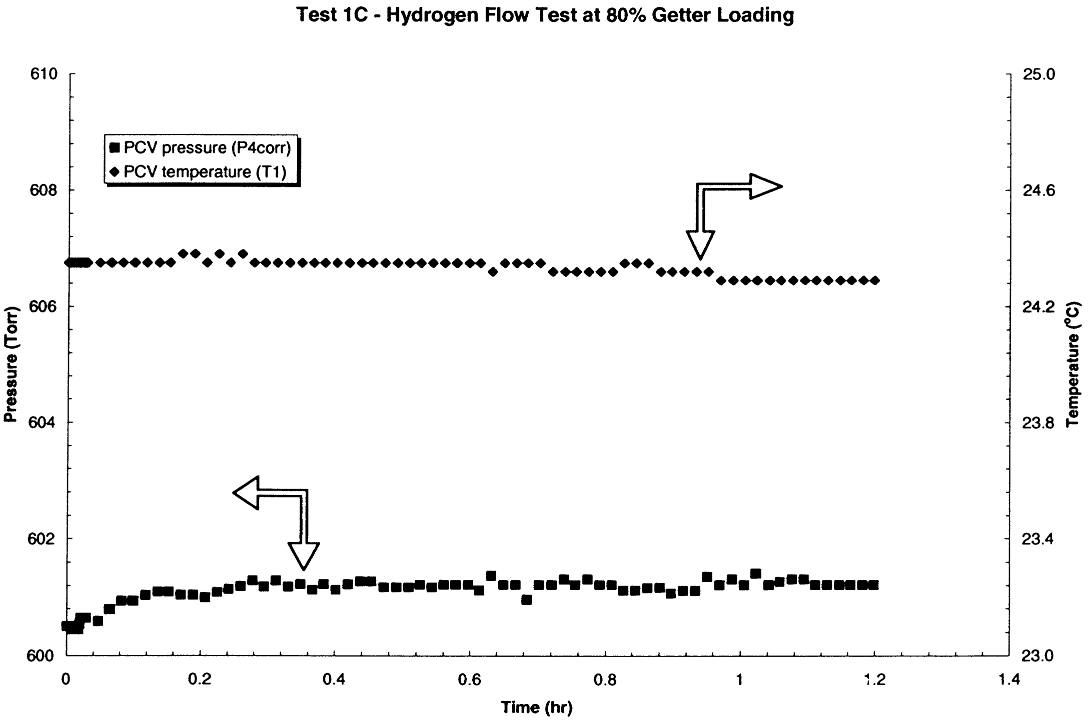

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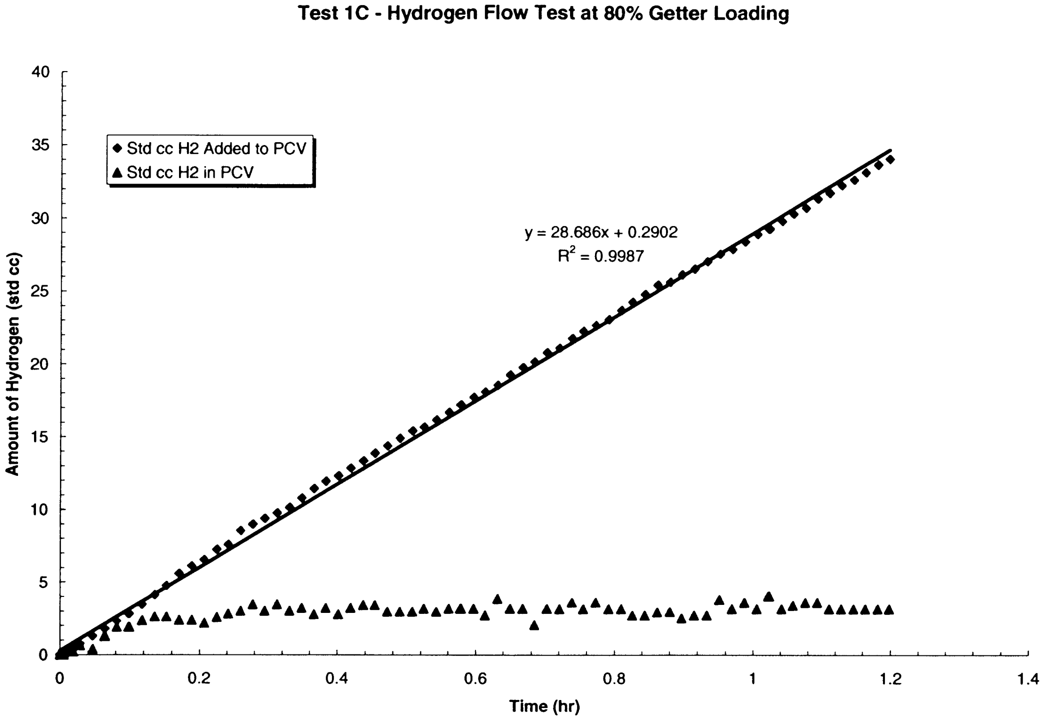

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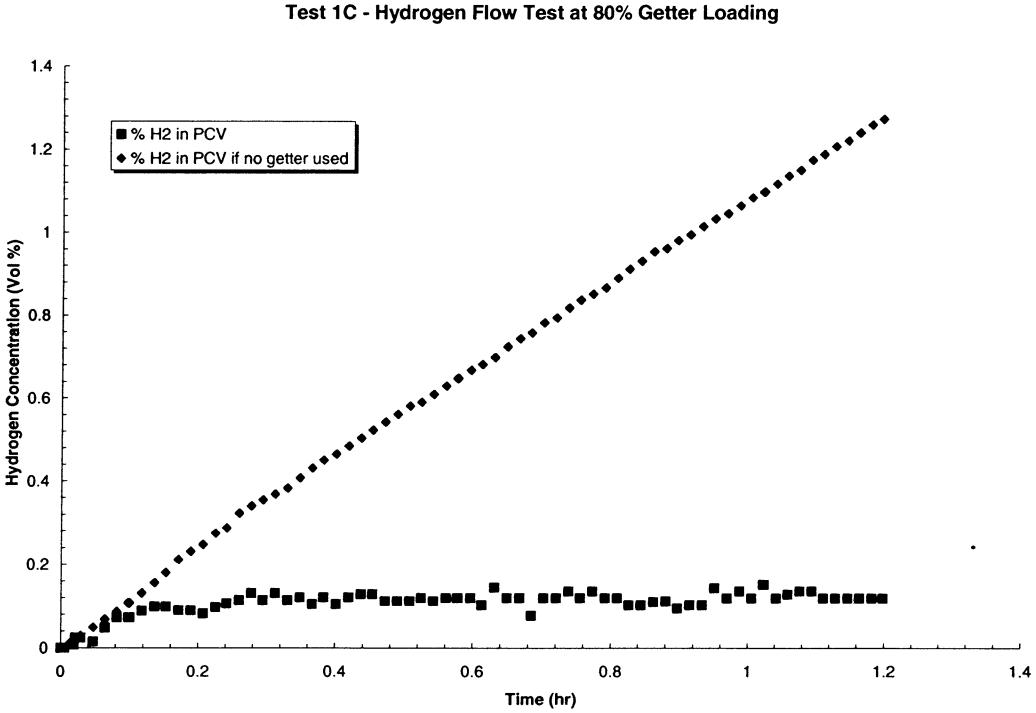

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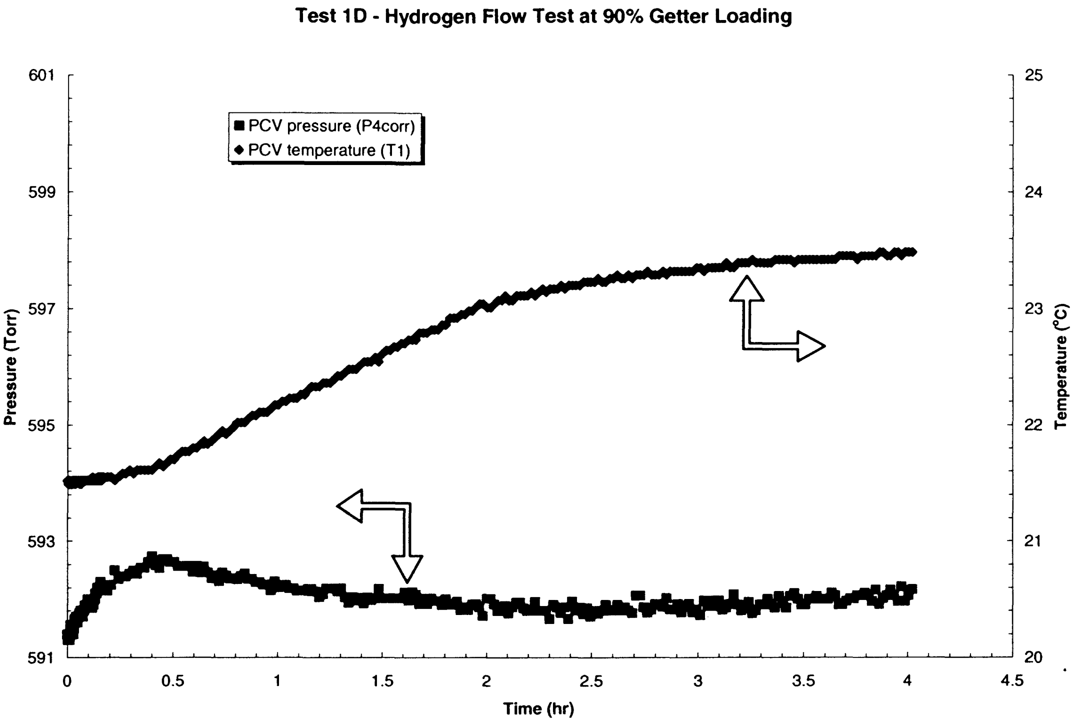

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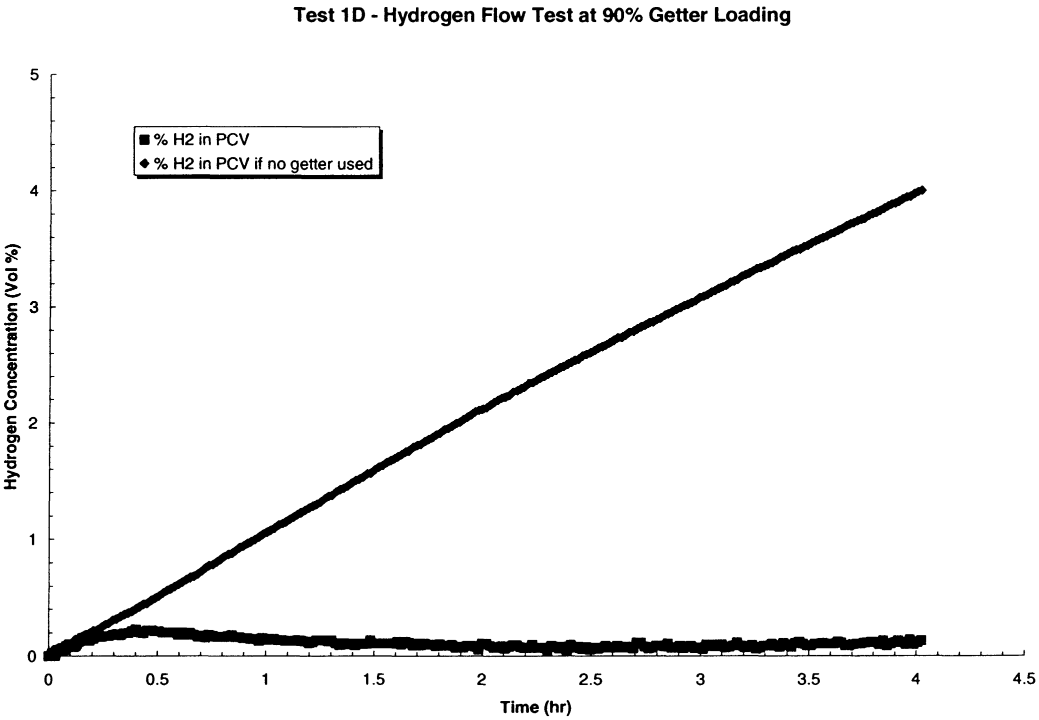

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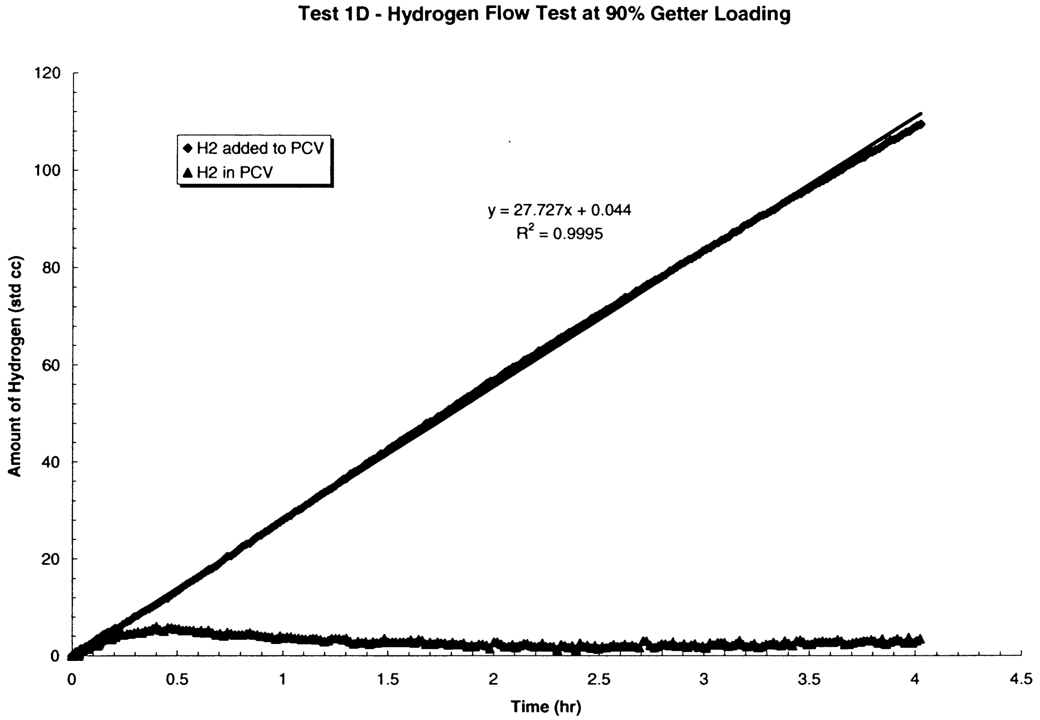

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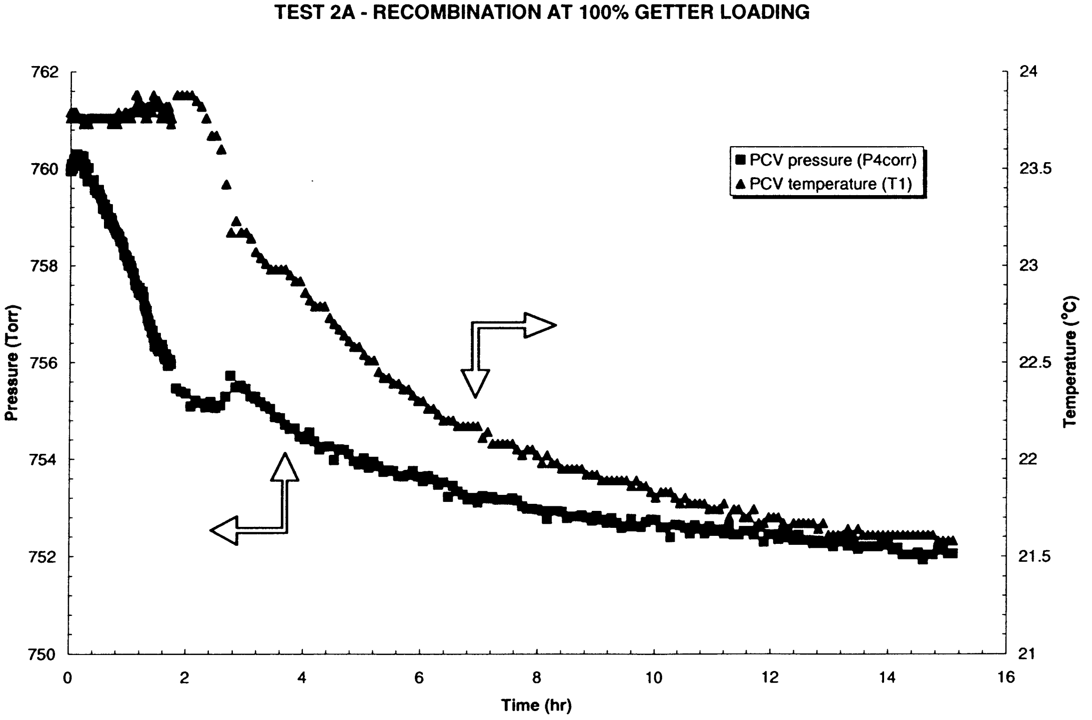

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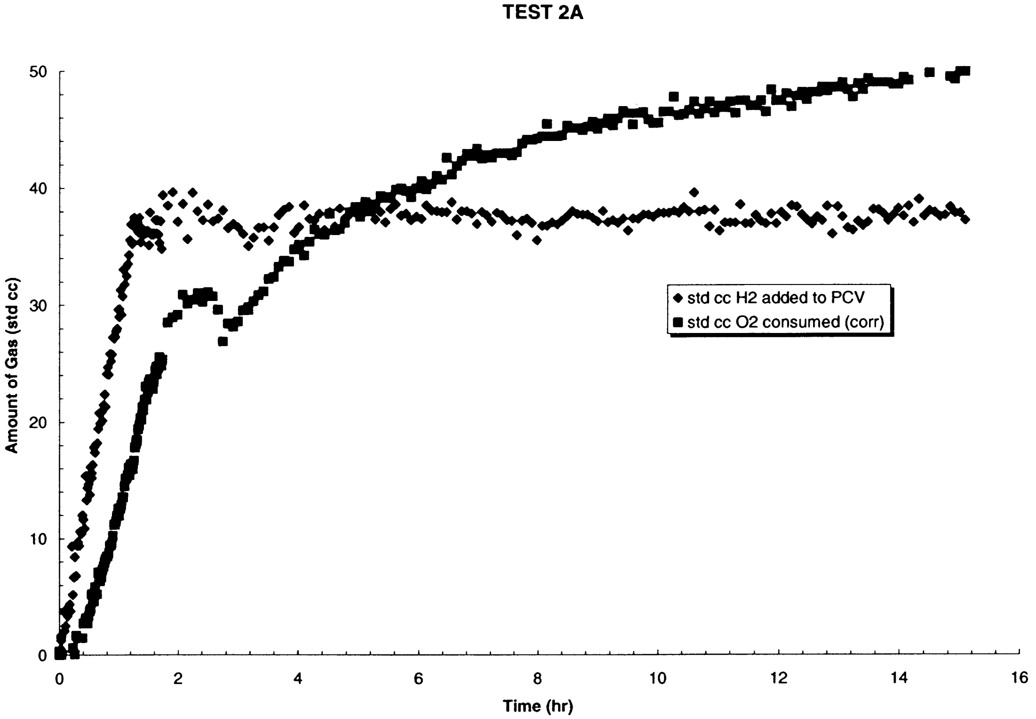

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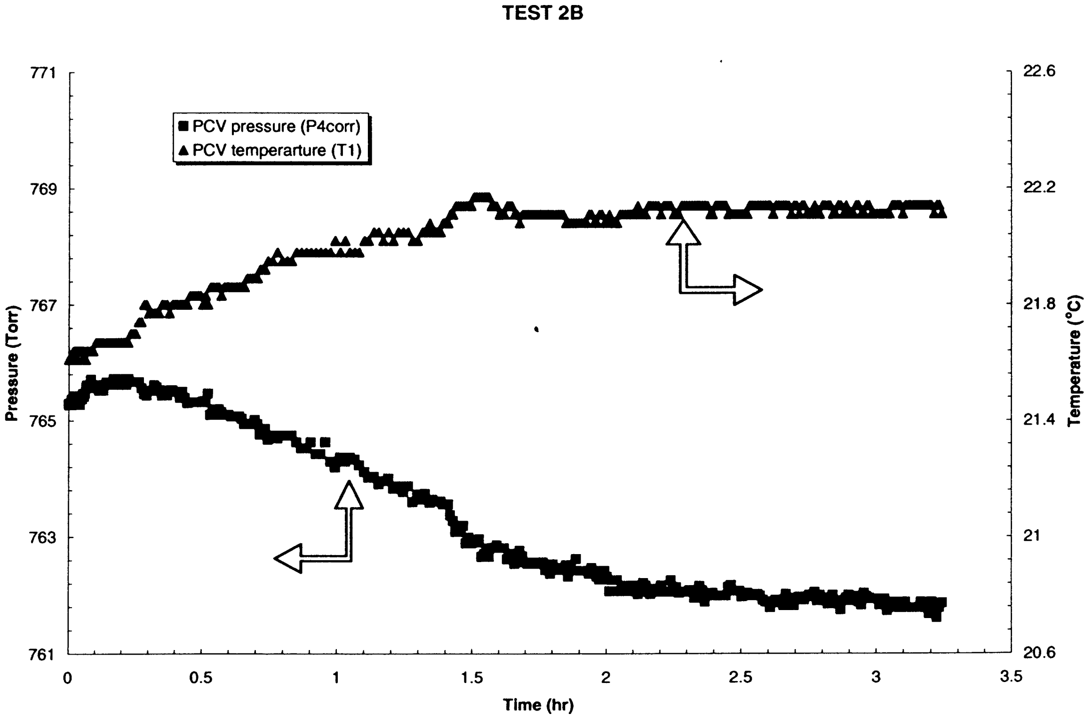

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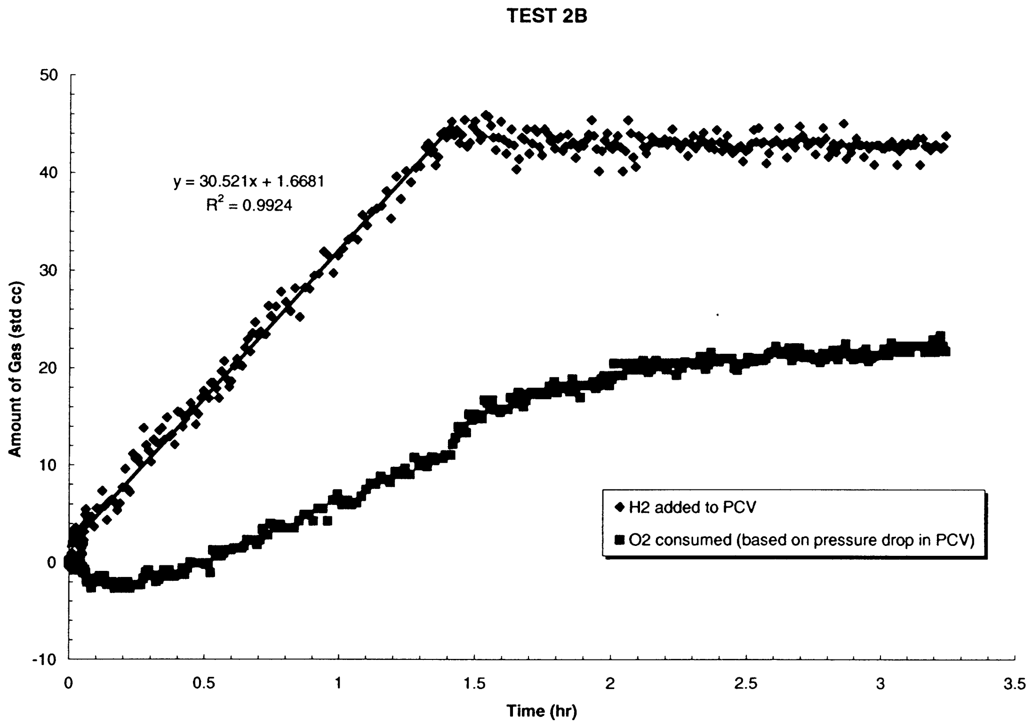

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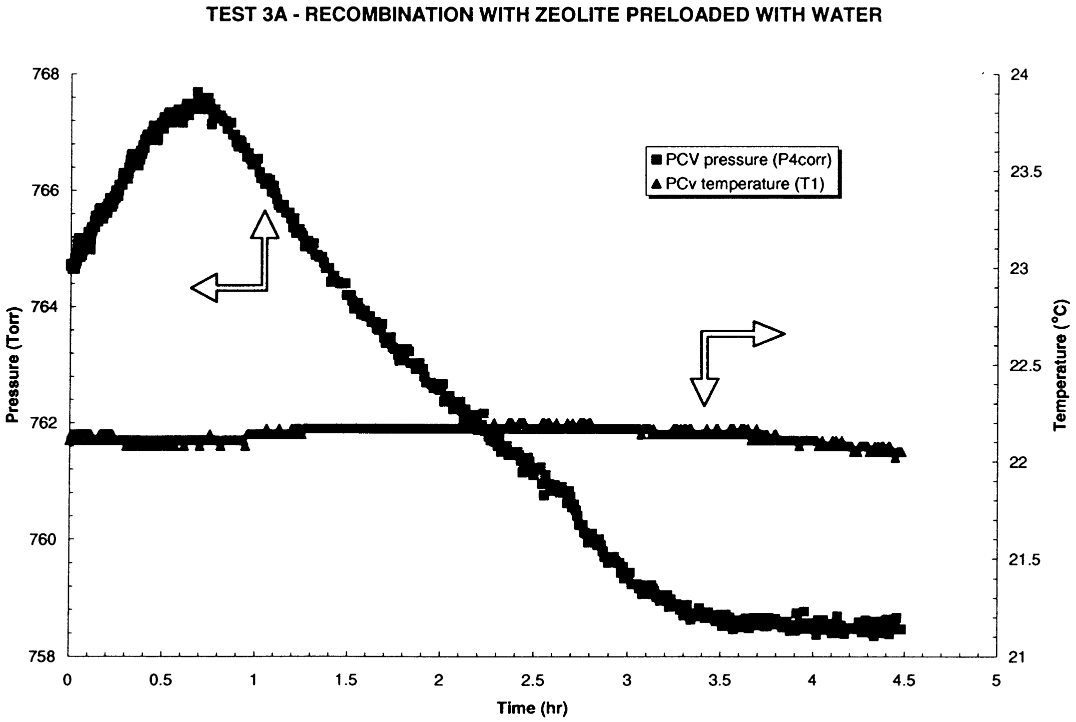

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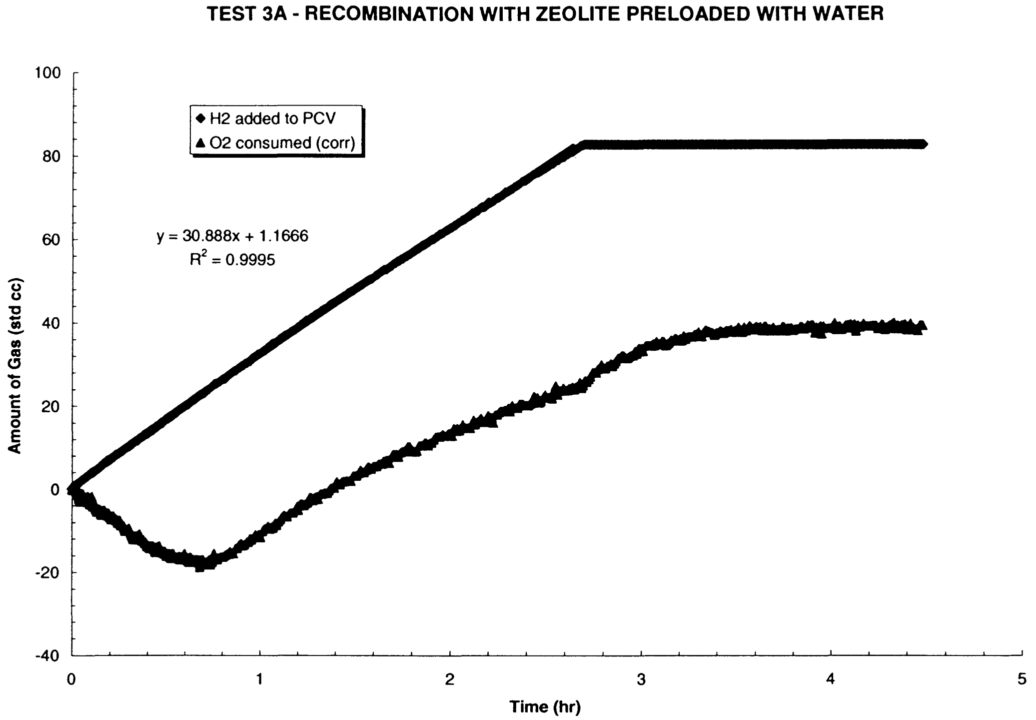

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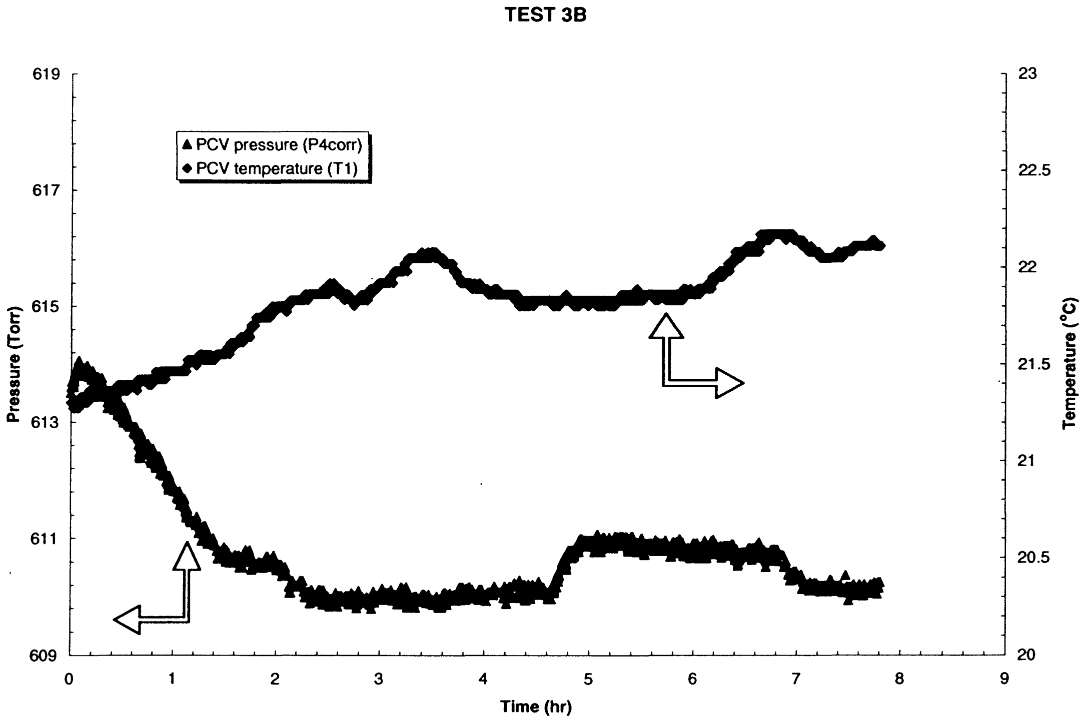

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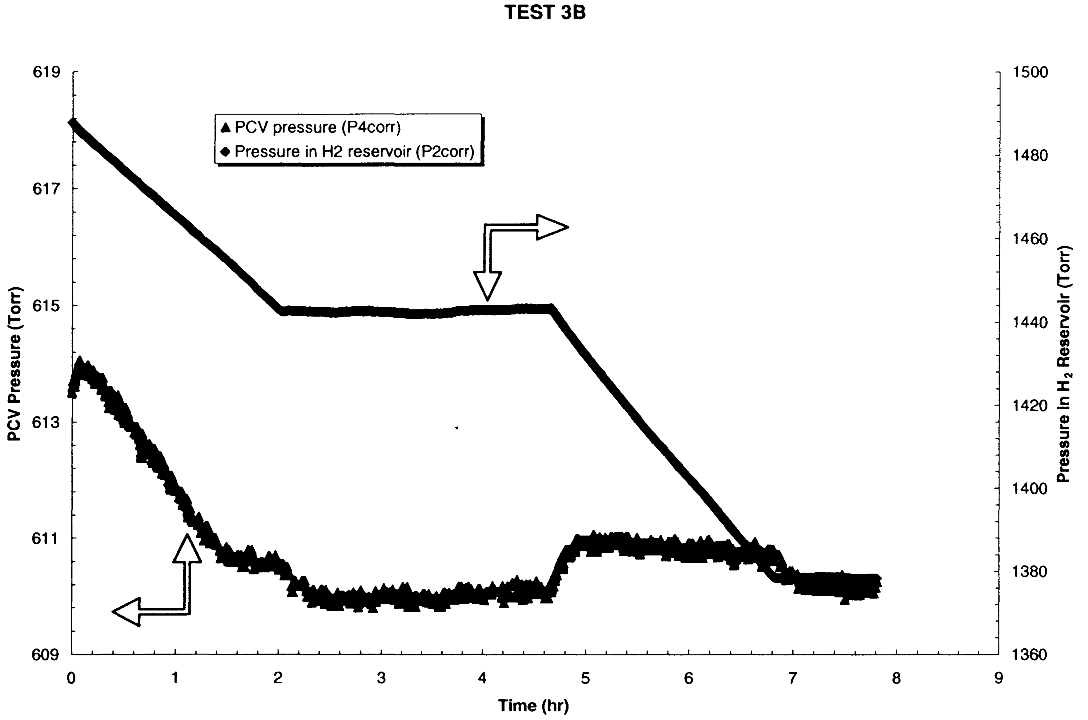

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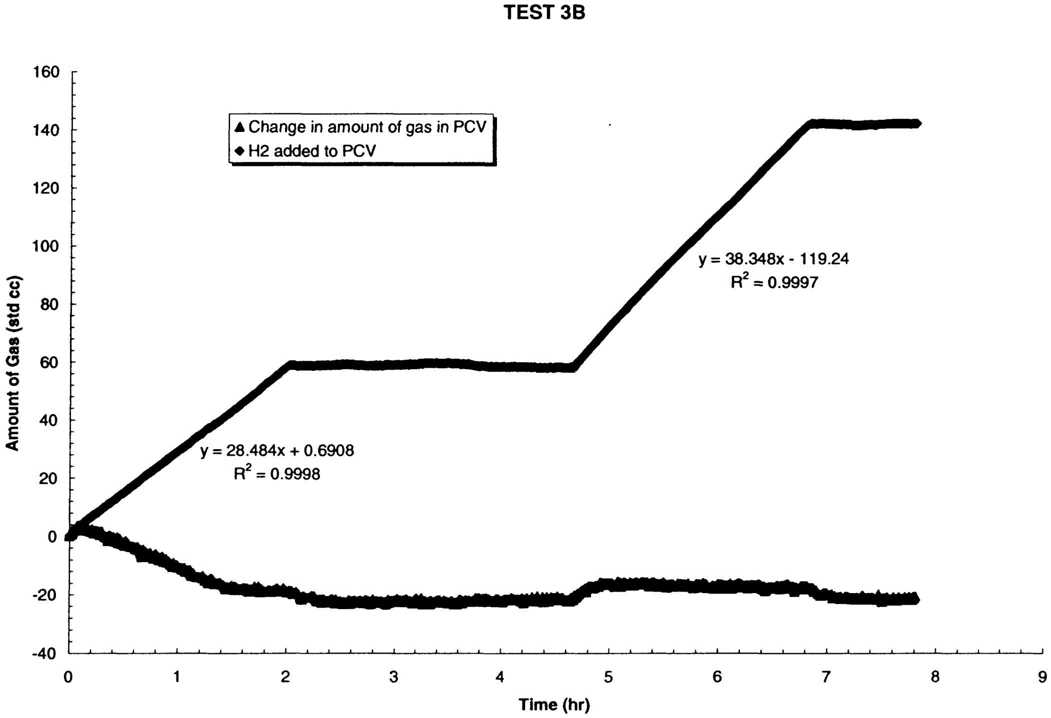

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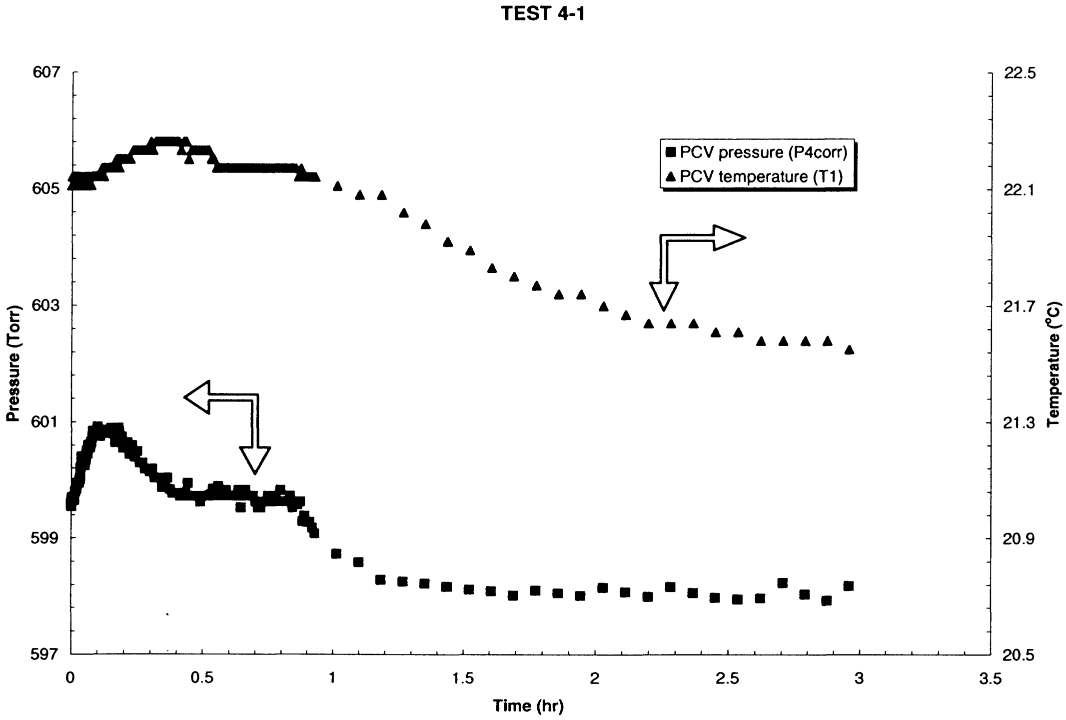

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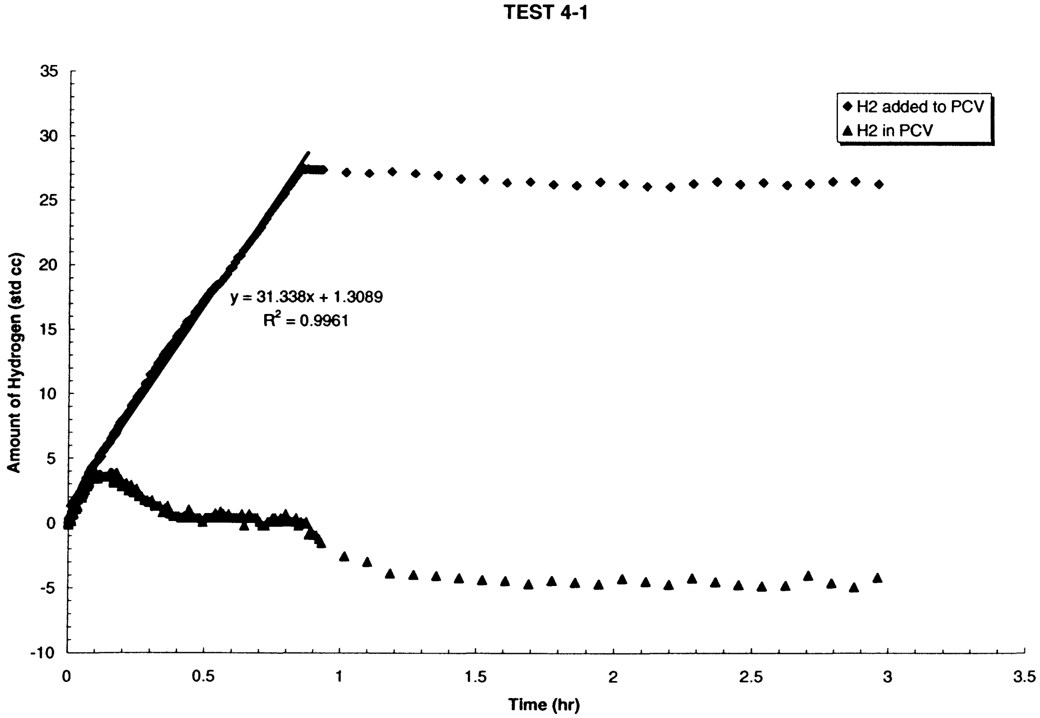

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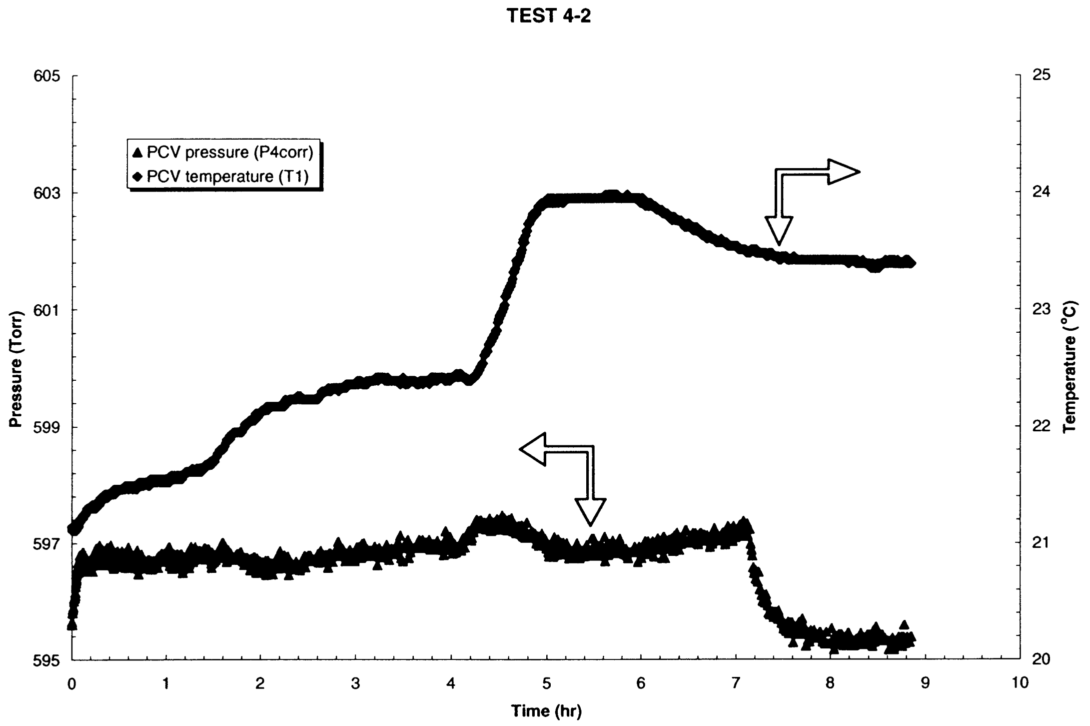

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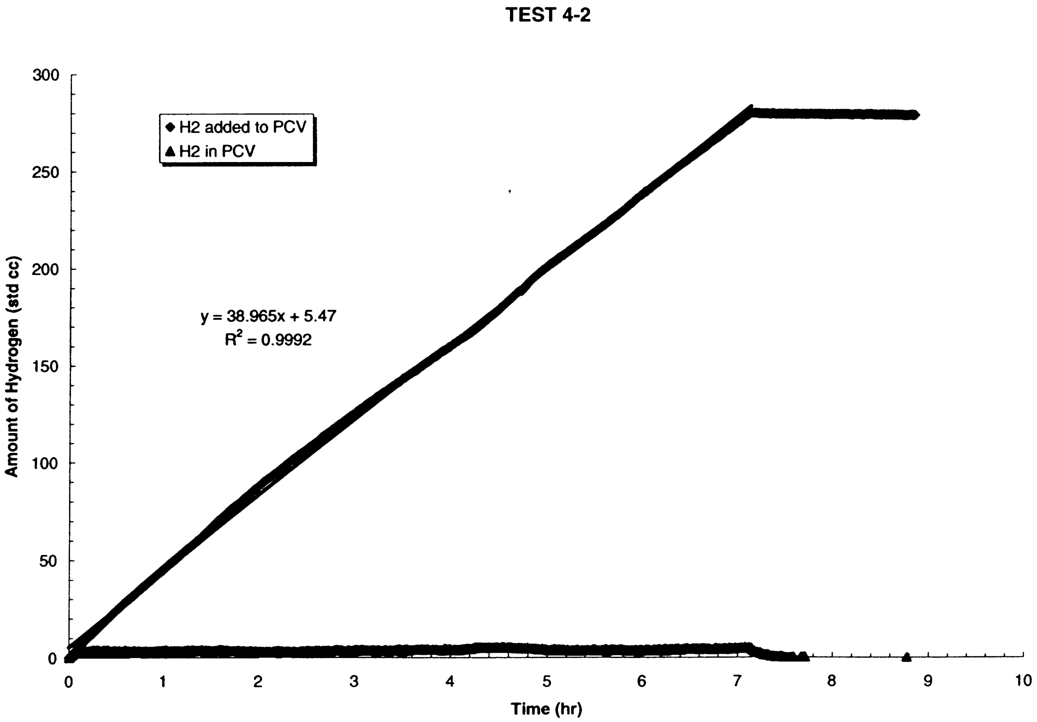

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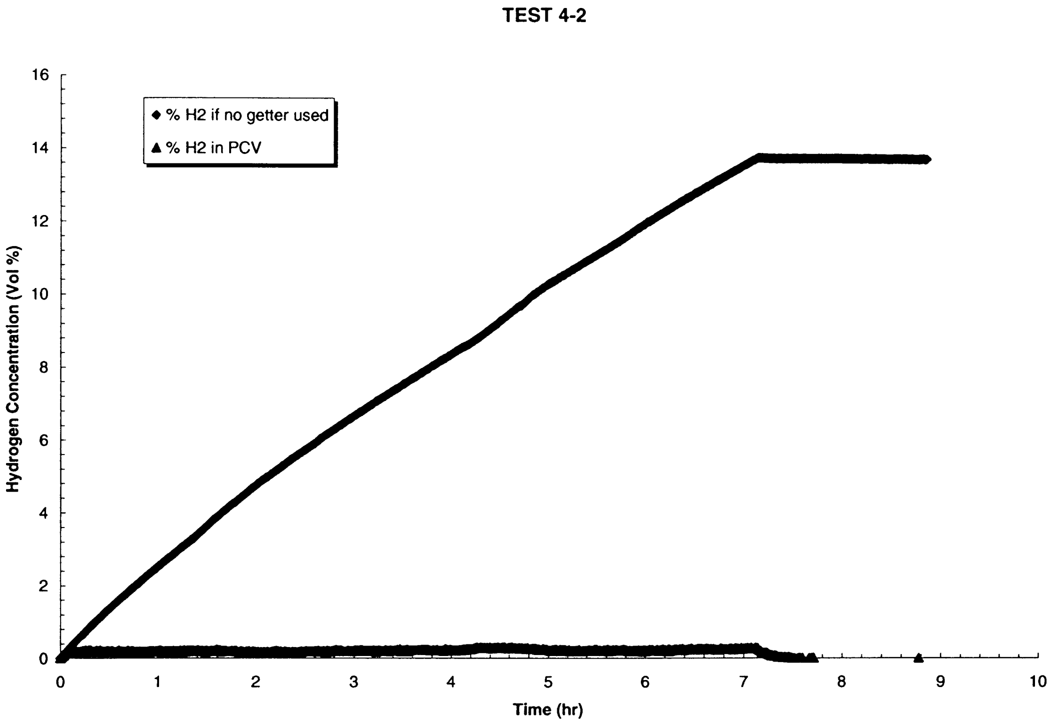

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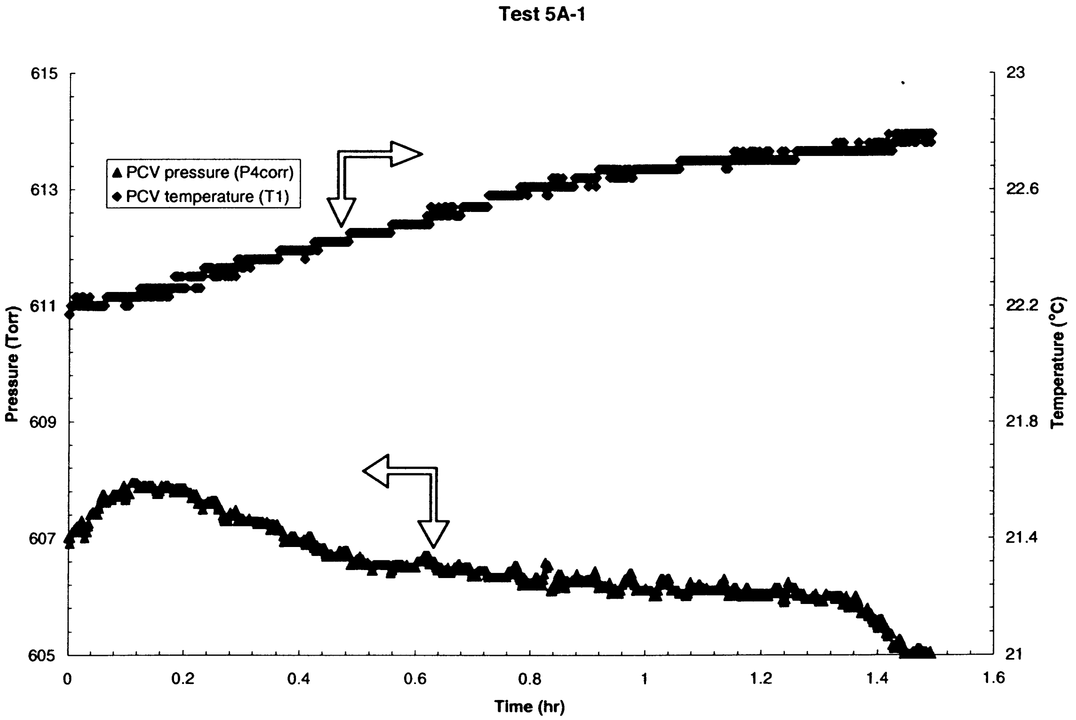

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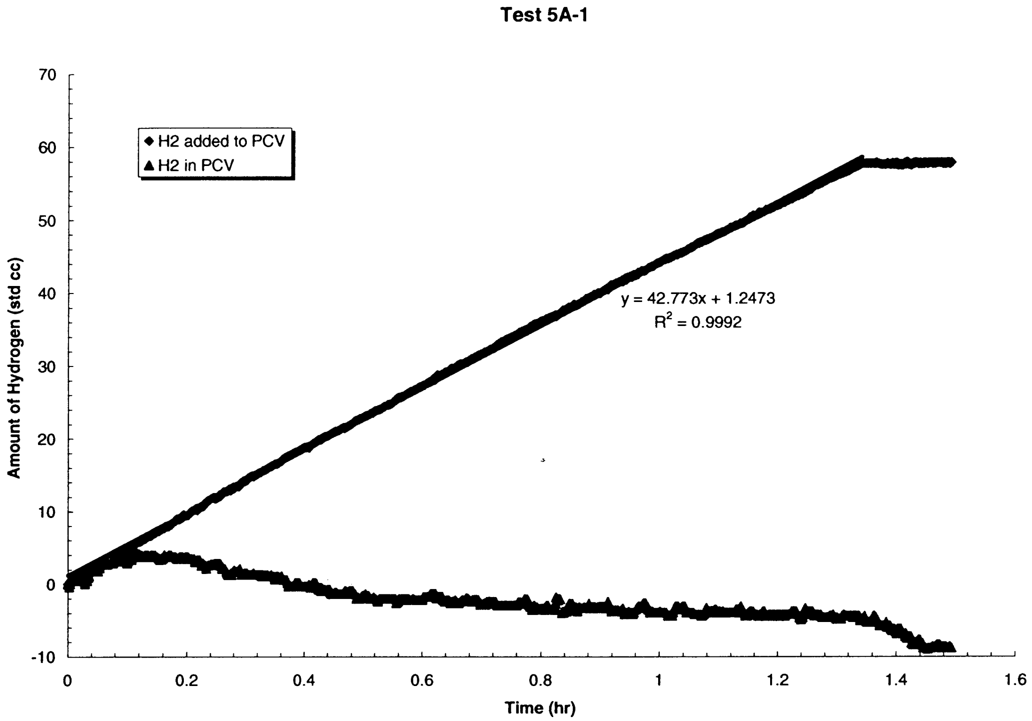

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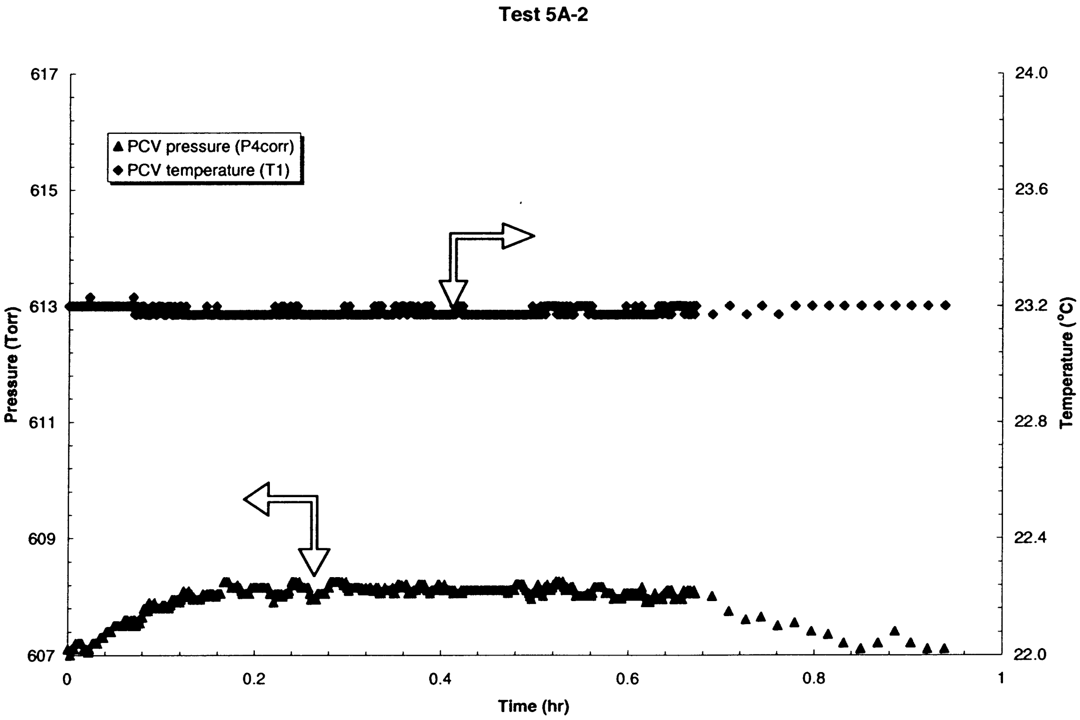

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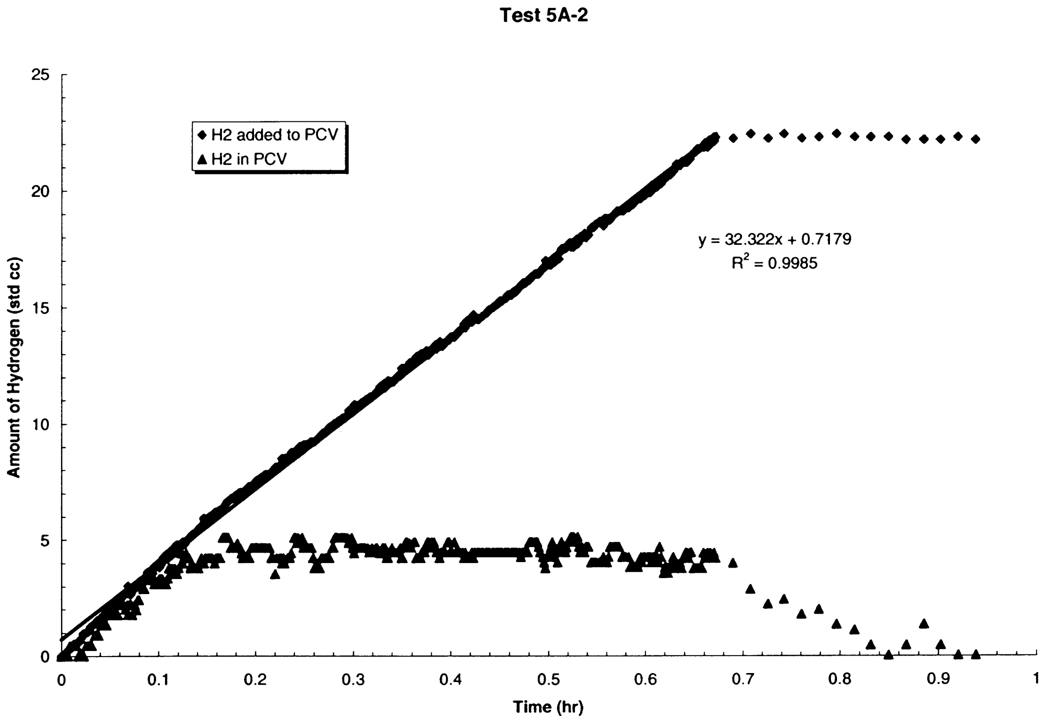

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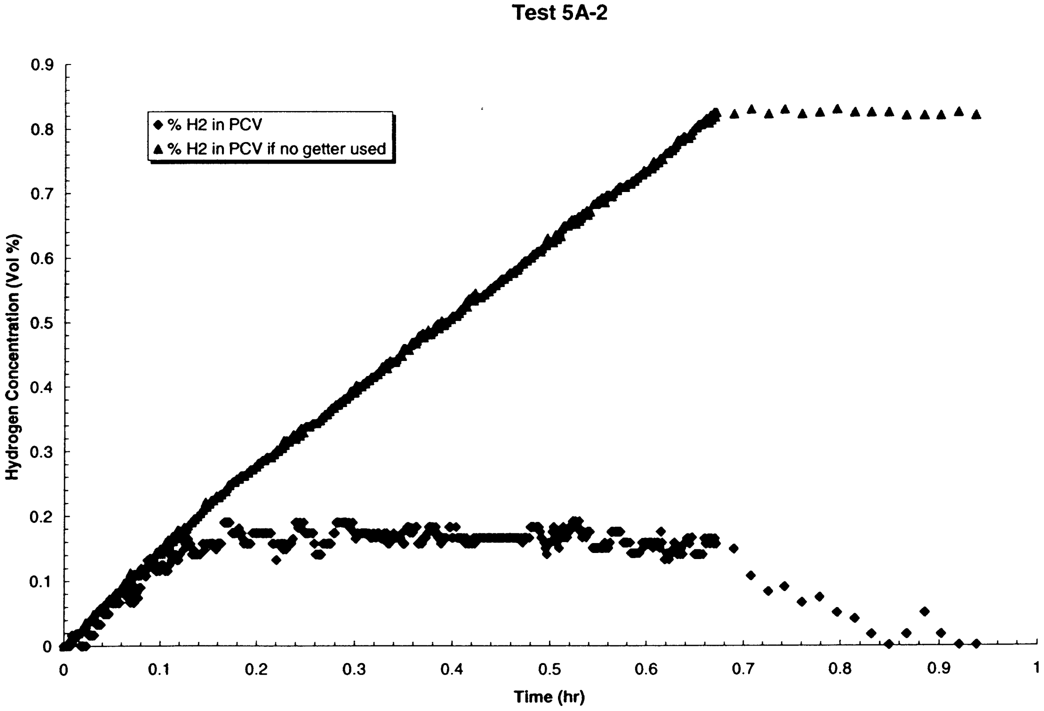

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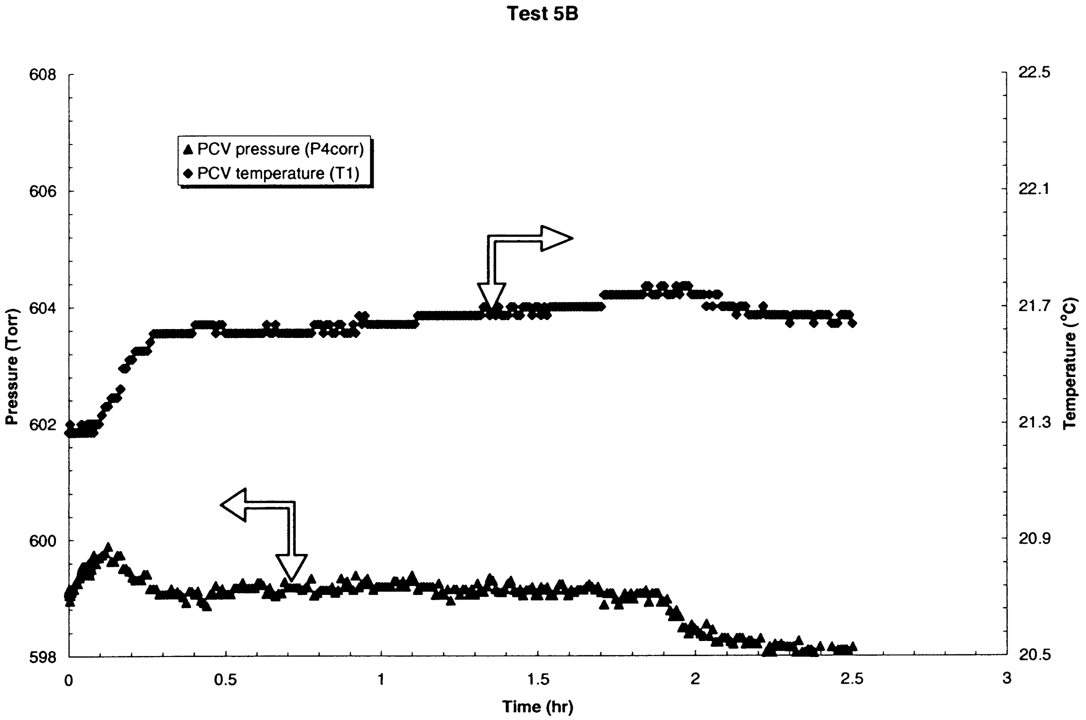

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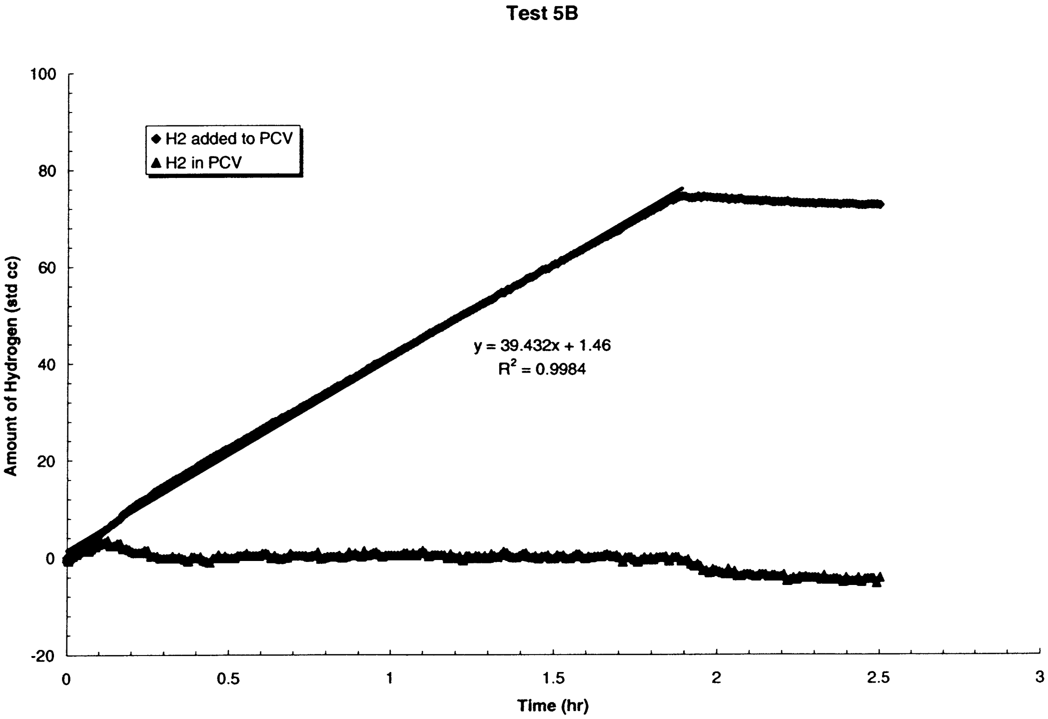

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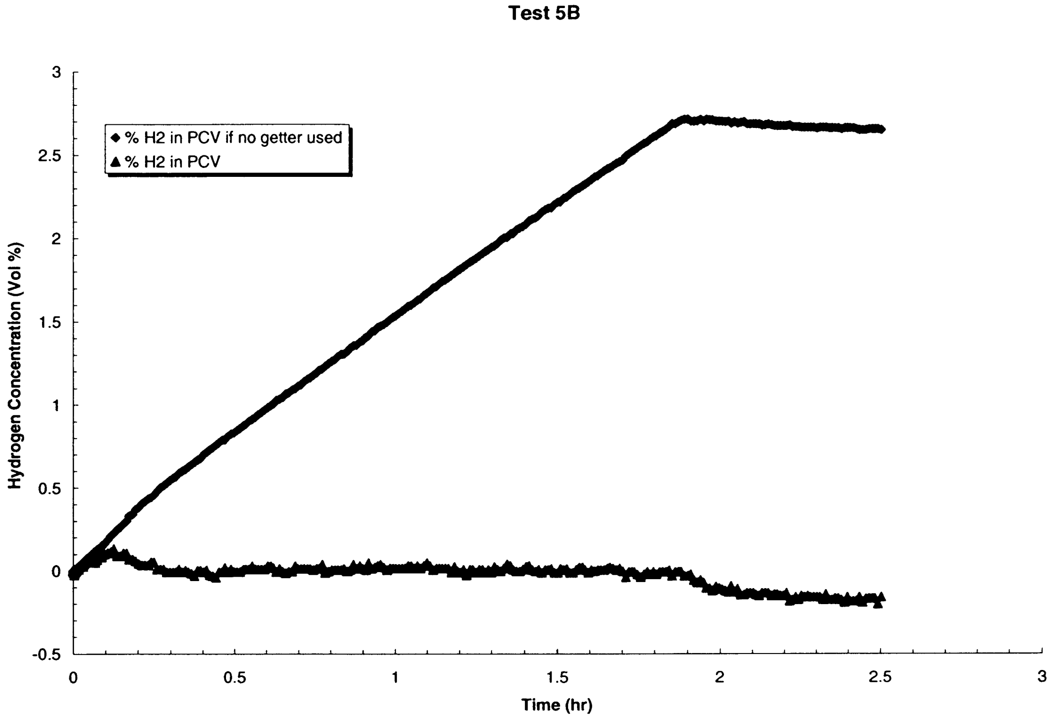

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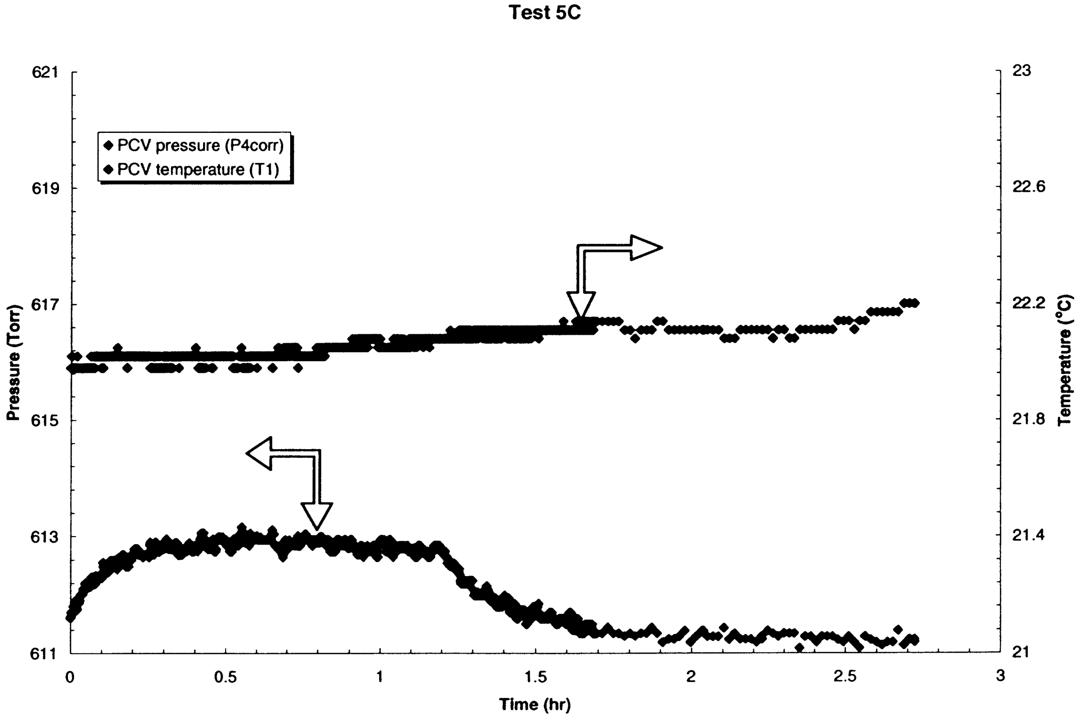

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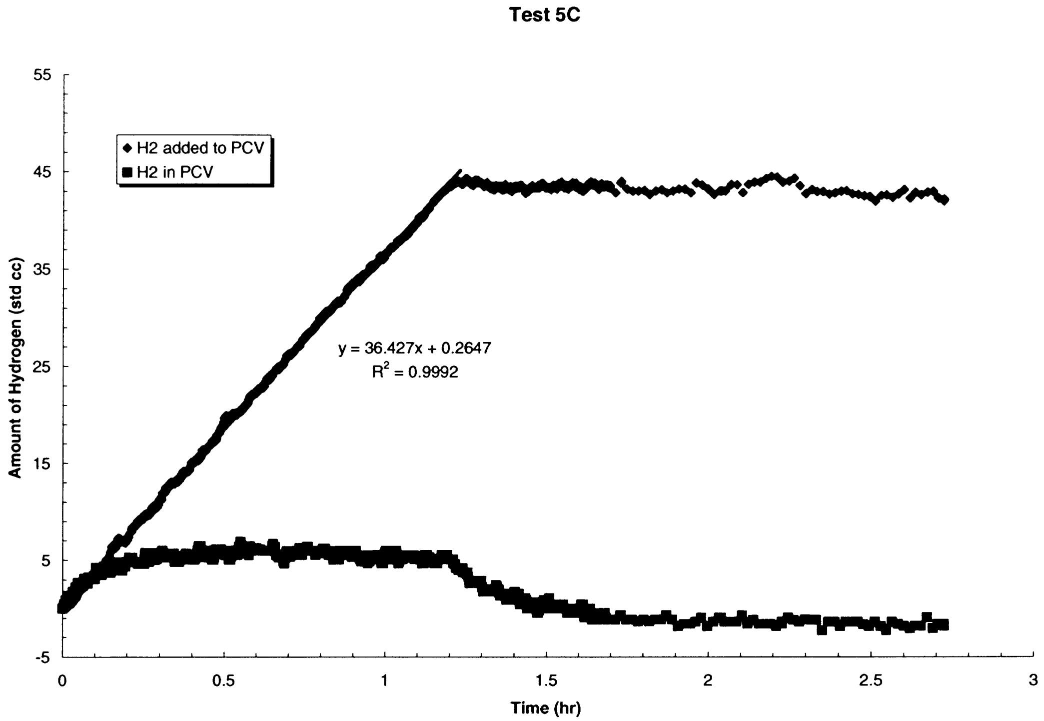

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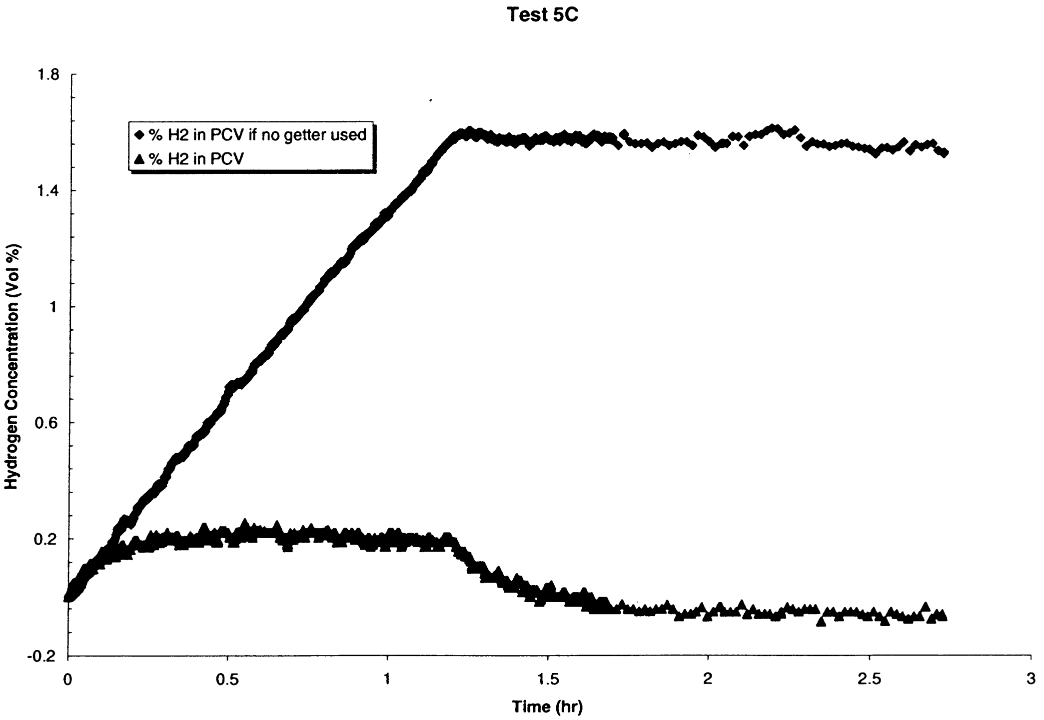

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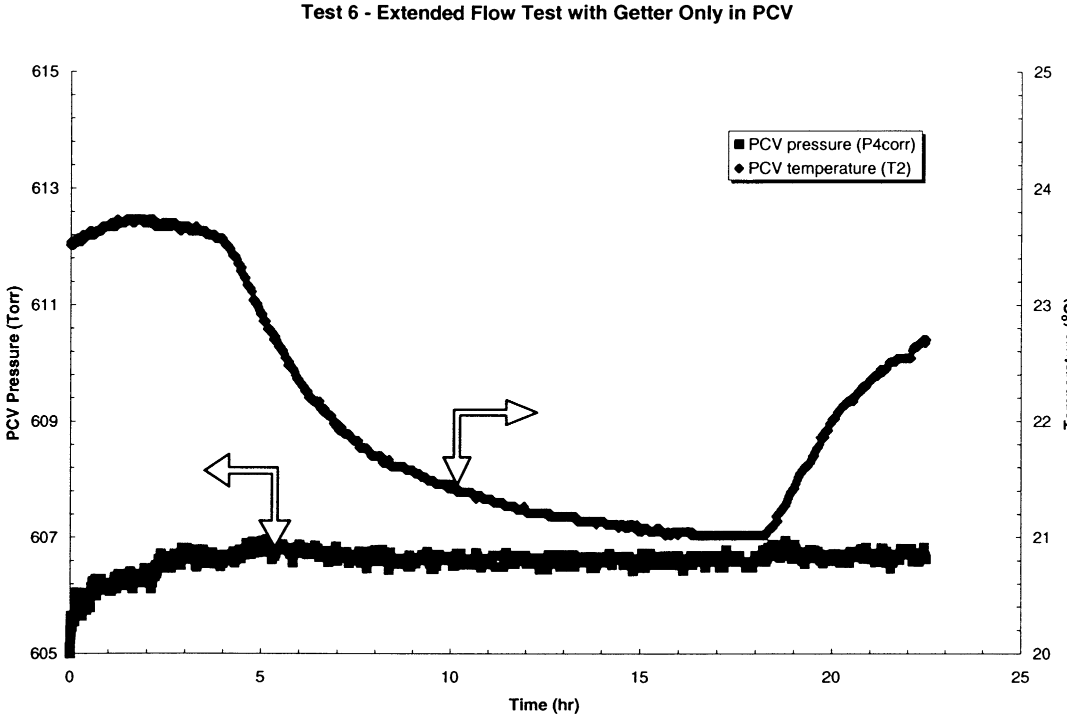

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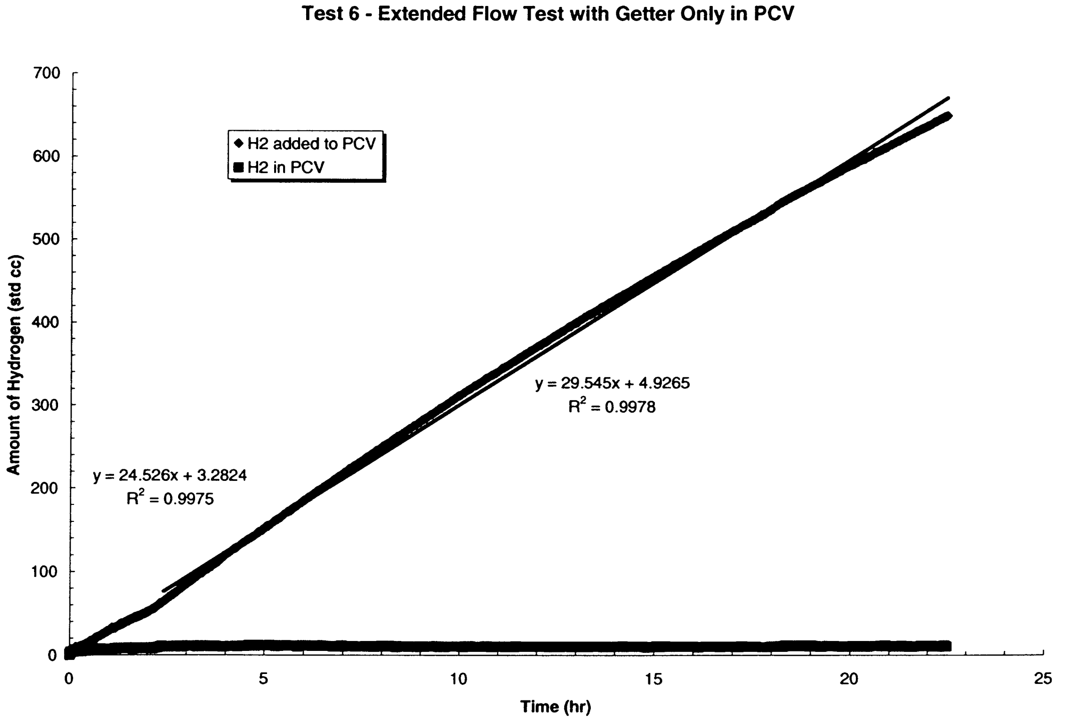

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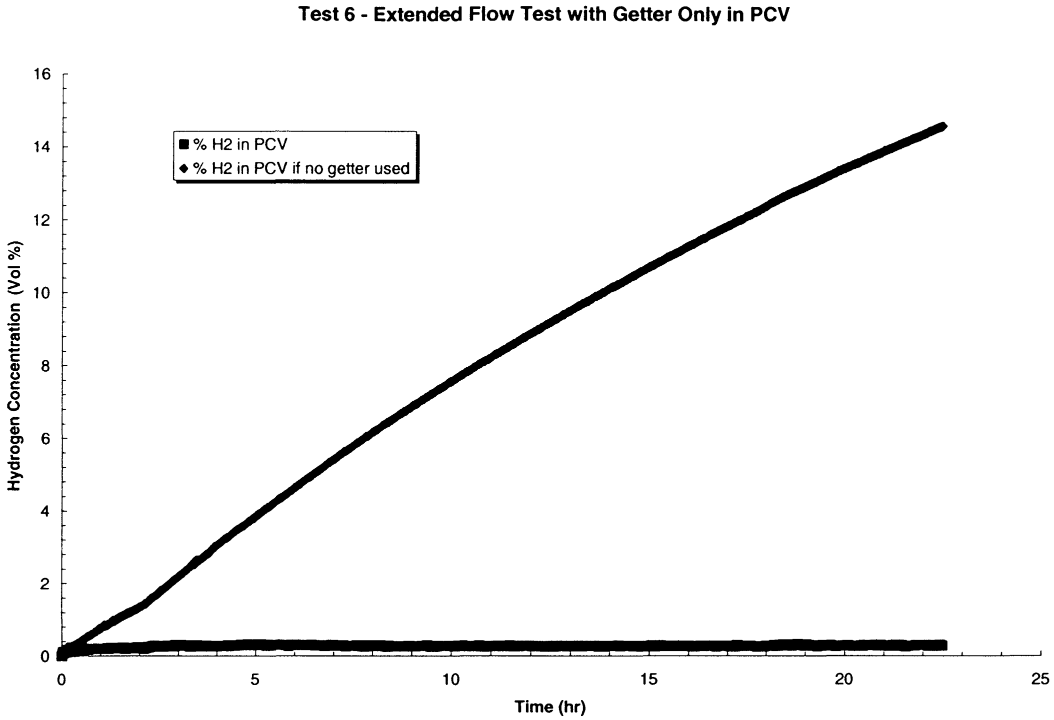

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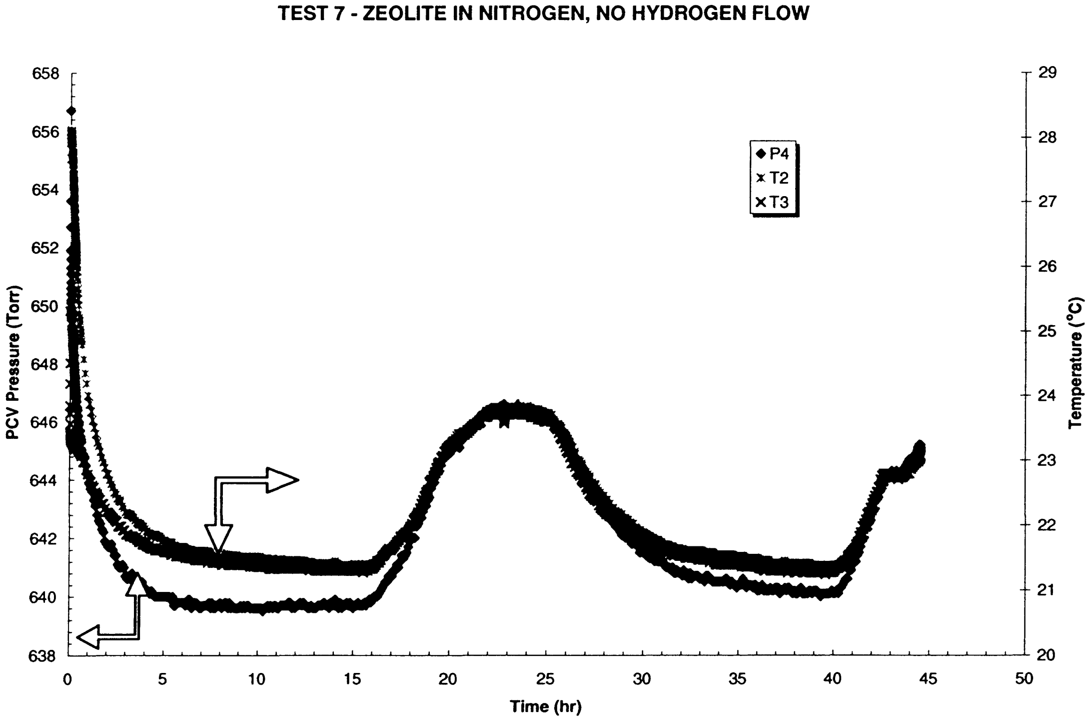

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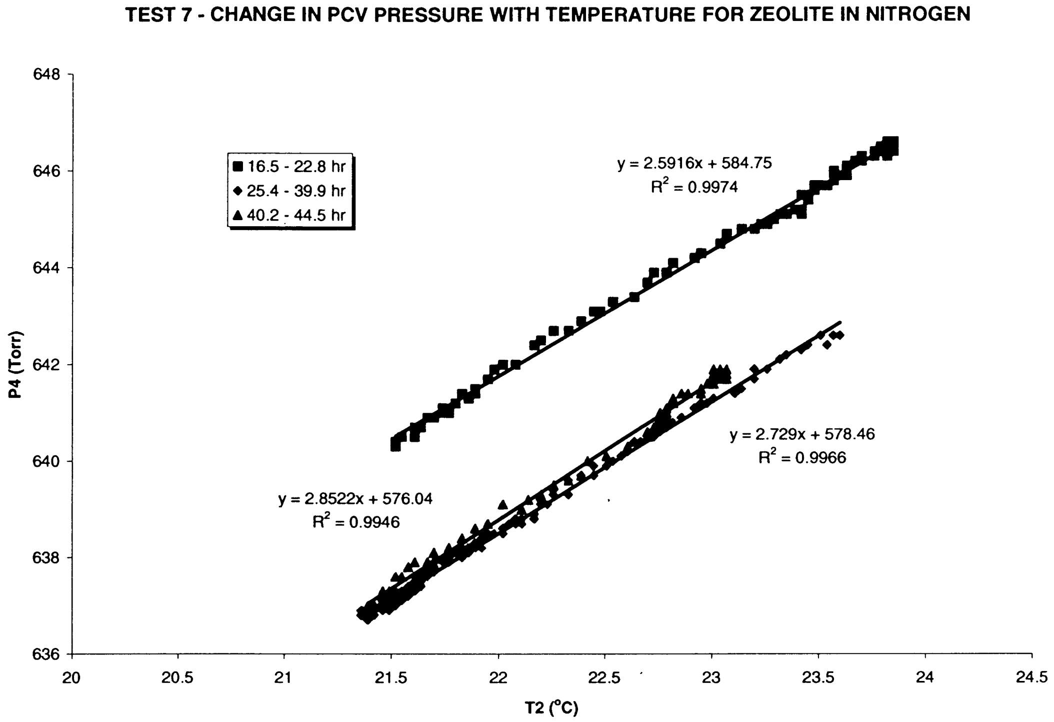

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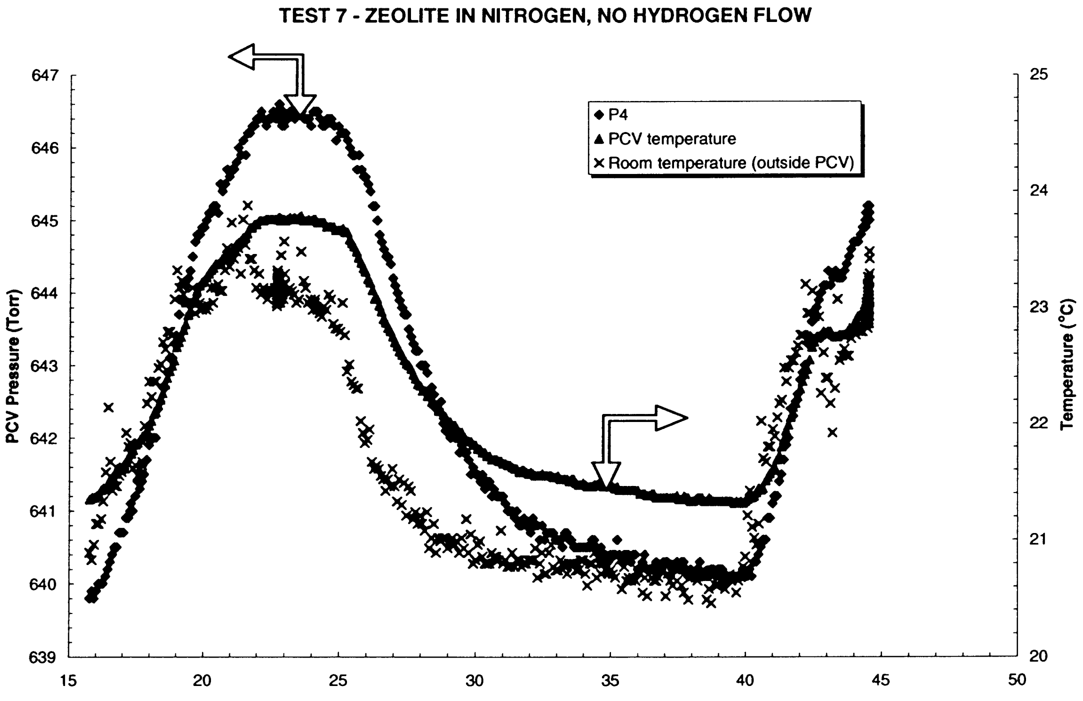

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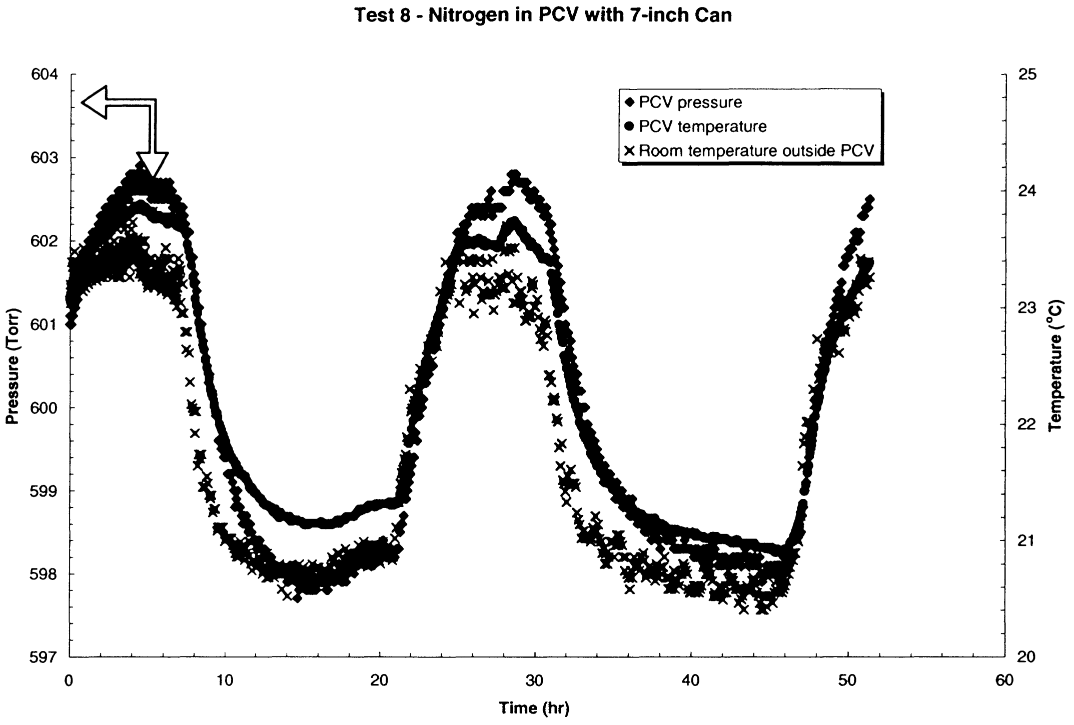

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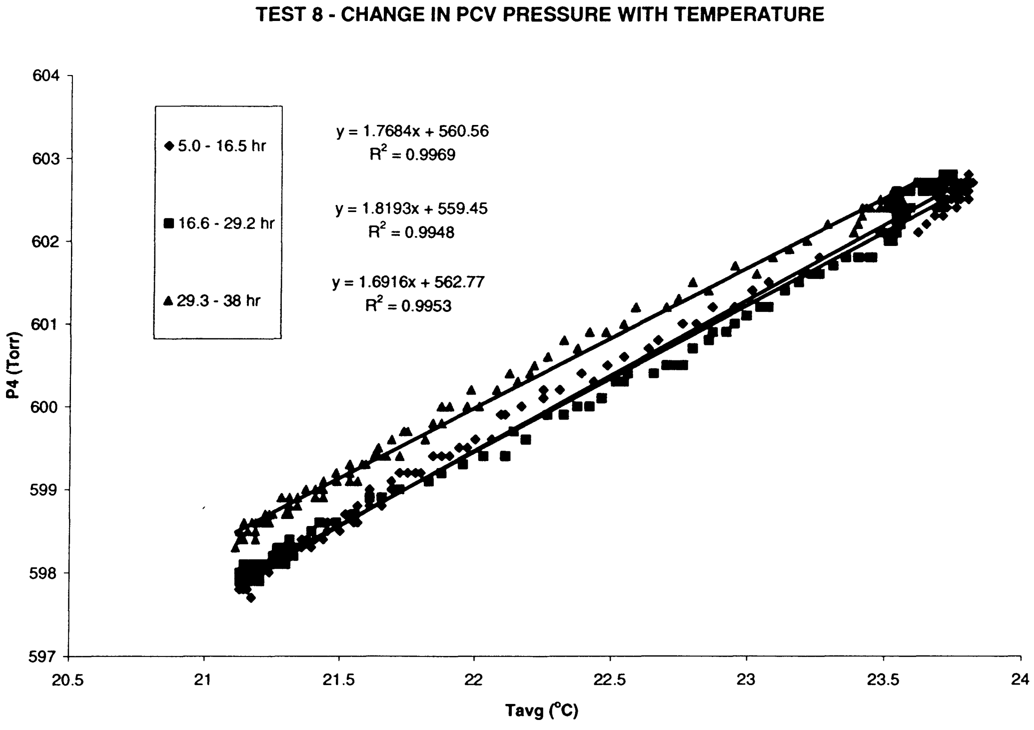

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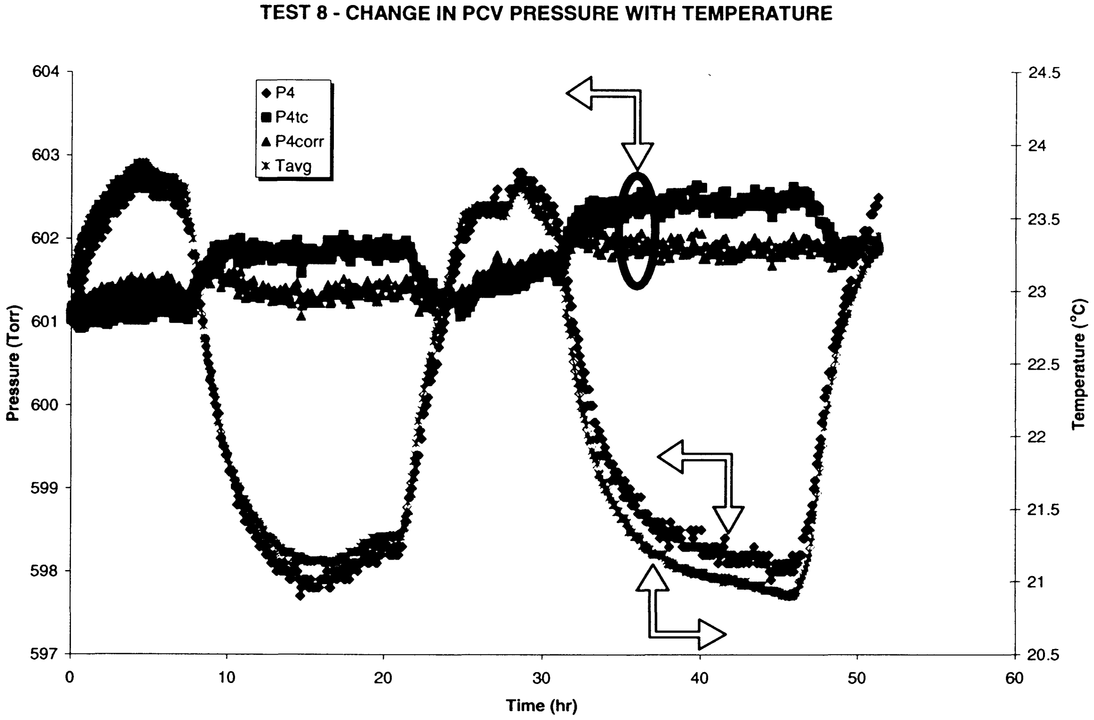

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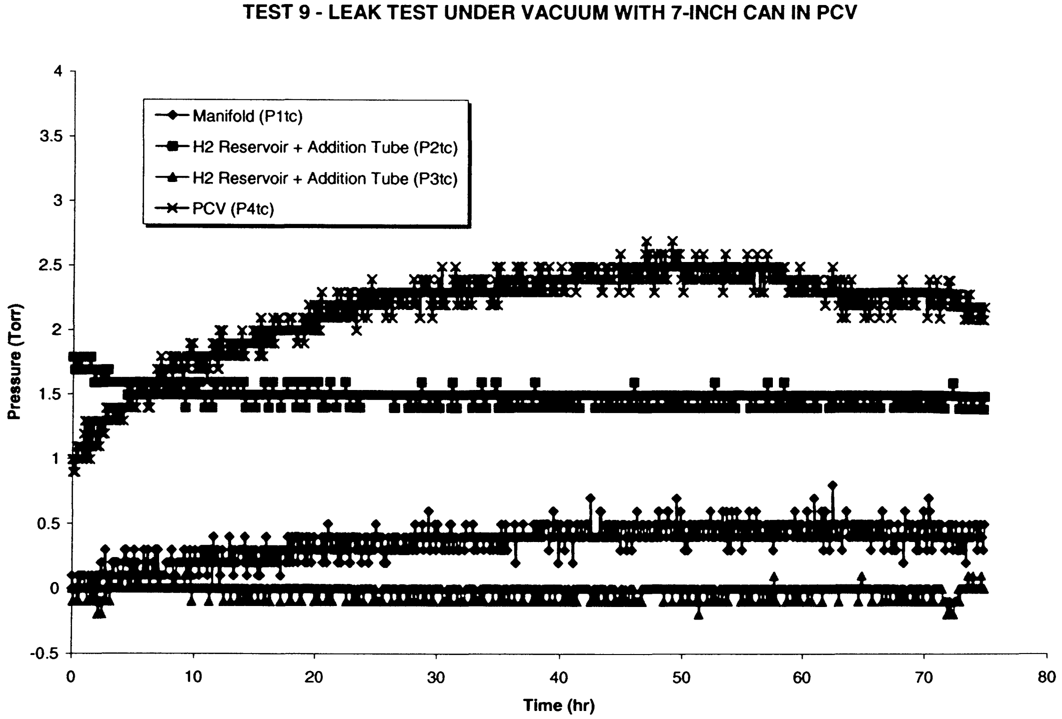

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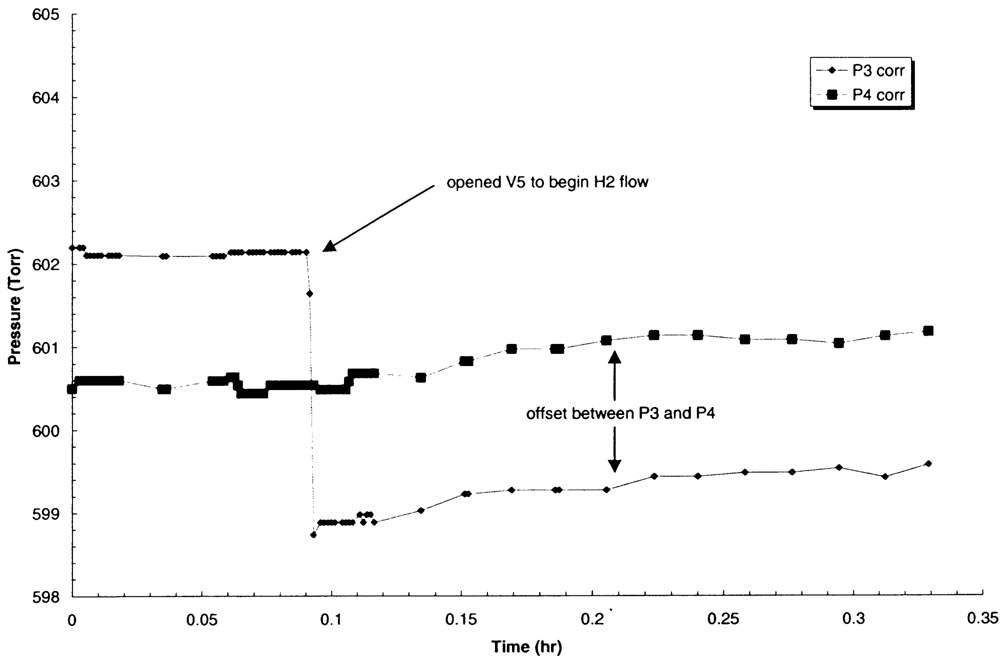


PRESSURE CHANGES ASSOCIATED WITH INITIATING HYDROGEN FLOW

Figure 4-51

Certificate of Calibration

Issued by

Savannah River Standards Laboratory

Westinghouse SRC, Building 736-A
Aiken, SC 29801

M & TE #: HTS001233 **Cal. Procedure:** NIST HANDBOOK 145
Description: CONSTANT VOL.1GAL.
Manufacturer: UNKNOWN
Model#: 304L-HDF4-1GAL
Serial#: W3B0245
Calibrated: 02/15/01
Frequency: 60 Months
Expires: 02/15/06

NIST Traceable Standard(s) used, (Expiration Date):
SL-92A,SL-347A EXP. 06/14/01

Print Specification n/a

The measurements made on **HTS001233** were obtained at 22 +/- 1.0 °C

CALIBRATION DATA FOR HTS001233

SEE ATTACHED

```
HTS001233 = 3,778.10 CC
VALVE     =       .78 CC
-----------------------
TOTAL     = 3,778.88 CC
```

THIS INSTRUMENT PASSED CALIBRATION

The estimated uncertainty of the standard(s) and measurement process at SRSL is:
SEE ATTACHED SHEETS

Metrologist: R.HUBBARD/RMG Roy Hubbard / RMG
Signature

JPC 2/15/01
Engr. Init

Note 1: This certificate shall not be reproduced except in full without the advanced written approval of the SRSL.

Note 2: The 'as received' condition of the standard, set of standards, or measurement equipment described herein was as expected, unless otherwise noted in the body of the certificate.

Issued by

Savannah River Standards Laboratory

Westinghouse SRC, Building 736-A
Aiken, SC 29801

M & TE #: 3-2124 **Cal. Procedure:** 329, REV. 0

Description: PRESS.INDICATOR, 100 PSIA

Manufacturer: PAROSCIENTIFIC

Model#: 740-100A

Serial#: 62308

Calibrated: 09/12/00

Frequency: 12 Months

Expires: 09/12/01

NIST Traceable Standard(s) used, (Expiration Date):
41773(7/06/05), SL-238(10/31/02), SL-238HR(6/18/01)

The measurements made on **3-2124** were obtained at 22 +/- 1.0 °C

CALIBRATION DATA FOR 3-2124

APPLIED PRESSURE PSIA		UUT AS FOUND PSIA	UUT AS LEFT PSIA
.00000		-.0022	SAME AS "AS FOUND" READINGS.
14.5651	(BP)	14.5602	
19.96515		19.9578	
39.92195		39.9124	
59.97970		59.9642	
79.01297		78.9992	
99.00199		98.9882	

THIS INSTRUMENT PASSED CALIBRATION

The estimated uncertainty of the standard(s) and measurement process at SRSL is:
+/- .01% RDG.

Metrologist: J.M. JEFFCOAT JM Jeffcoat
Signature

9/13/00
Engr. Init

Note 1: This certificate shall not be reproduced except in full without the advanced written approval of the SRSL.

Note 2: The 'as received' condition of the standard, set of standards, or measurement equipment described herein was as expected, unless otherwise noted in the body of the certificate.

Issued by

Savannah River Standards Laboratory

Westinghouse SRC, Building 736-A
Aiken, SC 29801

M & TE #: EA-974-1   **Cal. Procedure:** 445, REV.1
Description: TEMPERATURE PROBE
Manufacturer: DIGITEC
Model#: 5810
Serial#:
Calibrated: 12/28/00
Frequency: 12 Months
Expires: 12/28/01

NIST Traceable Standard(s) used, (Expiration Date):
SL427B(2/14/01):

The measurements made on **EA-974-1** were obtained at 23 +/- 1.0 °c

CALIBRATION DATA FOR EA-974-1

STANDARD	UUT	UUT RANGE
0.0132 C	00.18 C	-30 TO 50C
20.051	20.06	"
49.523	49.41	"
0.0132 C	00.04 C	0 TO 100 C
49.523	49.47 C	"
99.333	99.03 C	"

PROBE WITH INDICATOR PASSED CALIBRATION.

The estimated uncertainty of the standard(s) and measurement process at SRSL is:
+/- 0.025 C

The assigned uncertainty of the unit calibration is:
man.specs.with above corrections added to uut.

Metrologist: D.C. HOLLEY ______________ Signature ______ Engr. Init

Note 1: This certificate shall not be reproduced except in full without the advanced written approval of the SRSL.
Note 2: The 'as received' condition of the standard, set of standards, or measurement equipment described herein was as expected, unless otherwise noted in the body of the certificate.

Issued by

Savannah River Standards Laboratory

Westinghouse SRC, Building 736-A
Aiken, SC 29801

M & TE #: EA-974-2 **Cal. Procedure:** 445, REV. 1

Description: TEMPERATURE, PROBE

Manufacturer: DIGITEC

Model#: 5810

Serial#:

Calibrated: 12/28/00

Frequency: 12 Months

Expires: 12/28/01

NIST Traceable Standard(s) used, (Expiration Date):

SL427B(2/14/01):

The measurements made on **EA-974-2** were obtained at 23 +/- 1.0 °C

CALIBRATION DATA FOR EA-974-2

STANDARDS	UUT	UUT (RANGE)
0.010	00.25 C	-30 TO 50 C
20.051 C	20.13 "	
49.523	49.31	
0.010 C	00.04 C	0 TO 100 C
49.523	49.20 C	"
99.333	99.50 C	"

PROBE & INDICATOR PASSED CALIBRATION.

'he estimated uncertainty of the standard(s) and measurement process at SRSL is:

/- 0.025DEG.C.

he assigned uncertainty of the unit calibration is:

n.specs with the above corrections added.

rologist: D.C. HOLLEY JKHolley
Signature

CRR
Engr. Init

Note 1: This certificate shall not be reproduced except in full without the advanced written approval of the SRSL.

Note 2: The 'as received' condition of the standard, set of standards, or measurement equipment described herein was as expected, unless otherwise noted in the body of the certificate.

Certificate of Calibration

Information Only

Issued by

Savannah River Standards Laboratory

Westinghouse SRC, Building 736-A
Aiken, SC 29801

E #	ATD1-185	**Cal. Procedure**	L10.1-301,REV.1
escription	WEIGHT SET		
anufacturer	TROEMNER		
odel#	N/A		
erial#	7382		
alibrated	02/01/99		
requency	36 Months		
xpires	02/01/02		

NIST Traceable Standard(s) used, (Expiration Date):

SL-458,SL-459,SL-347A EXP.06/14/2001

Print LIOTH000C

The measurements made on **ATD1-185** were obtained at 21 +/- 1.0 °c

CALIBRATION DATA FOR ATD1-185

SEE ATTACHED SHEET

he estimated uncertainty of the standard(s) and measurement process at SRSL is:

EE ATTACHED SHEET

etrologist GEO.W.COUNTS George W. Counts AB

Signature Engr. Init

Information Only

Atd1-185 2/1/99 9:21 AM

				Metrologist	G.W.C.
				Date	2/1/99
M&TE#		**DOUBLE SUBSTITUTION METHOD**		**Number of weights**	
ATD1-185					4
ominal Weigh (grams)	Weight # or marking	True Mass (grams)	App. Mass at 8g/cc (grams)	Uncertainty (grams)	Density Used (g/cc)
******************	******************	******************	******************	******************	******************
1000		1000.03915712	1000.03871445	0.00410104	8.00
500		500.03497503	500.03476455	0.00405690	8.00
200		200.00827982	200.00820195	0.00006686	8.00
200	1	200.01190893	200.01183066	0.00006686	8.00

		COMPARISON METHOD		**Number of weights**	
					0
ominal Weigh (milligrams)	Weight # or marking	True Mass (grams)	App. Mass at 8g/cc (grams)	Uncertainty (grams)	Density Used (g/cc)
******************	******************	******************	******************	******************	******************

* markings on weights

Certificate of Calibration

Information Only

Issued by
Savannah River Standards Laboratory
Westinghouse SRC, Building 736-A
Aiken, SC 29801

M & TE #: ATD1-104 Cal. Procedure: L10.1-301,REV.1
Description: WEIGHT,50G
Manufacturer: TROEMNER
Model#: N/A
Serial#: N/A
Calibrated: 03/15/00
Frequency: 12 Months
Expires: 03/15/01

NIST Traceable Standard(s) used, (Expiration Date):
SL-459,SL-346C EXP.06/14/2001

Print Specification LIOTH000C

The measurements made on ATD1-104 were obtained at 21 +/- 1.0 ºC

CALIBRATION DATA FOR ATD1-104

SEE ATTACHED SHEET

The estimated uncertainty of the standard(s) and measurement process at SRSL is:
SEE ATTACHED SHEET

Metrologist: GEO.W.COUNTS George W. Counts Signature CPR Engr. Init

Note 1: This certificate shall not be reproduced except in full without the advanced written approval of the SRSL.

Note 2: The 'as received' condition of the standard, set of standards, or measurement equipment described herein was as expected, unless otherwise noted in the body of the certificate.

Information Only

Atd1-104 3/15/00 12:33 PM

				Metrologist	G.W.C.
				Date	3/15/00
M&TE#		DOUBLE SUBSTITUTION METHOD		Number of weights	
ATD1-104					1
Nominal Weight (grams)	Weight # or marking	True Mass (grams)	App. Mass at 8g/cc (grams)	Uncertainty (grams)	Density Used (g/cc)
**					
50		50.00067013	50.00064048	0.00011876	8.00
		COMPARISON METHOD		Number of weights	
					0
Nominal Weight (milligrams)	Weight # or marking	True Mass (grams)	App. Mass at 8g/cc (grams)	Uncertainty (grams)	Density Used (g/cc)
**					

* markings on weights

Issued by
Savannah River Standards Laboratory
Westinghouse SRC, Building 736-A
Aiken, SC 29801

M & TE #:	HTS8056	**Cal. Procedure:**	BAL-CAL
Description:	BALANCE		
Manufacturer:	METTLER		
Model#:	PM6100		
Serial#:	101374		
Calibrated:	08/17/00		
Frequency:	12 Months		
Expires:	08/17/01		

NIST Traceable Standard(s) used, (Expiration Date):
SL-346B & SL-406 EXP.10/01/2001

Print Specification N/A

The measurements made on **HTS8056** were obtained at 21 +/- 1.0 ºC

CALIBRATION DATA FOR HTS8056

SEE ATTACHED SHEET

THIS INSTRUMENT PASSED CALIBRATION

The estimated uncertainty of the standard(s) and measurement process at SRSL is:
SEE ATTACHED SHEET

Metrologist: R.M. GAY ---------- Signature ---------- Engr. Init 9/7/00

Note 1: This certificate shall not be reproduced except in full without the advanced written approval of the SRSL.

Note 2: The 'as received' condition of the standard, set of standards, or measurement equipment described herein was as expected, unless otherwise noted in the body of the certificate.

BALCAL Rev. 8.0, 5/7/98 Reference Procedure: L3.8-3000, "Balance Pipet Calibration/Certification 9/6/00

QA BALANCE CERTIFICATION
(ref. Procedure L3.8-30000,"Balance & Pipet Calibration/Certification")
Next Certification due date: 17-Aug-02

Analyst Information	
Calibrator Initials:	RMG
Calibrator ID #	Y8230
Date:	8/17/01

Calibration Weight Set Data

SL-3468	SL-406	Exp. 10/1/200
Manufacturer	METTLER	
Serial #	101375	

BALANCE INFORMATION			Nominal Wt.	App. Mass	Uncert.(mg) at 3 SD	Bal./Wt. Uncertainty Ratio @ at 3 SD
Lab / Room #	773A,C064					
MODEL#	PM6100	HighWt.	5000.0	5000.027481	1.88502	31.83
Balance ID #	HTS8056	HighWt2	1000.0	1000.006588	0.09862	608.40
QA Limit (%)	0.10	MidWt.	3000.0	3000.020082	1.23002	48.78
Sen. Limit (g)	0.0600	LowWt.	1000.0	1000.006588	0.09862	608.40
1 Bal. Maint. SD	0.0200	Sensitivity	10.0	10.00007734	12.5	4.80

20 grams minimum quantity that can be weighed to maintain QA 95% Limits.

Visual Inspection Parameters	YES	NO
Damaged		#
Level	#	
Clean	#	
Complete	#	

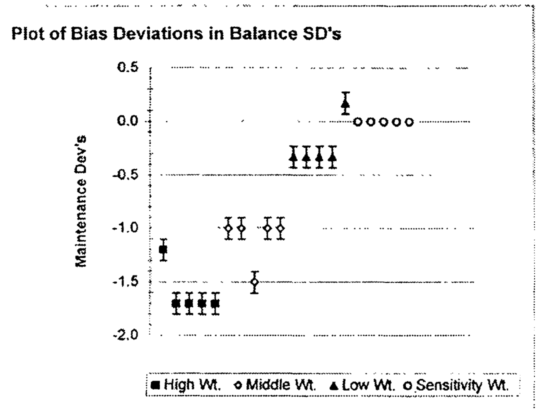
Function Tests	PASS	FAIL
Power	#	
Load	#	
Taring	#	
P/NDisplay	#	

MEASUREMENTS

High(s) Wt.	Middle Wt.	Low Wt.	Sensitivity
6,000.01	3,000.00	1,000.00	10.00
6,000.00	3,000.00	1,000.00	10.00
6,000.00	2,999.99	1,000.00	10.00
6,000.00	3,000.00	1,000.00	10.00
6,000.00	3,000.00	1,000.01	10.00

Cornerload Data (@ 50% Capacity)

Position	Reading	TSTAT
Top	3,000.00	0.10
Right	2,999.99	0.40
Bottom	2,999.99	0.40
Left	3,000.00	0.10


Plot of Bias Deviations in Balance SD's

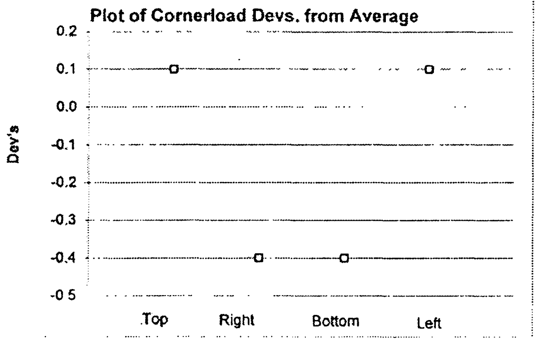
STATISTICS

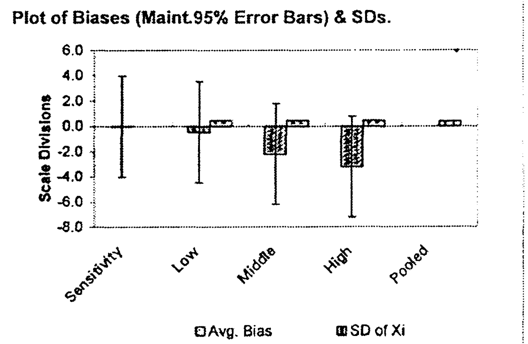
	High Wt(s).	Mid Wt.	Low Wt.	Sensitivity
AVERAGE	6000.002	2999.998	1000.002	10
Bias (abs)	-0.03206909	-0.02208218	-0.00458776	-7.734E-05
% Bias	-0.000534482	-0.00073607	-0.00045877	-0.00077339
% RSD	7.45307E-05	0.000149075	0.000447212	0
SD(abs)	0.004471841	0.004472257	0.004472127	0
Pooled SD	0.020009895	0.020004202	0.020000027	0.02042942
Calculated t	-0.886264222	-2.00285236	-3.95920345	-4.36966841

Pooled (abs), 19 df	0.00387293	Tab. Chi Sq	36.2
Tabled t, 19 df (.95)	2.09	Cal. Chi Sq	0.71248054
Tabled F, 2, 16 df (.98)	6.23	F STAT	4.8804188

CONTROL LIMITS

	Mean WT.	2 DEV'S	Low Limit	High Limit
High Wt.	6000.002	0.021944673	5999.980055	6000.02394
Middle Wt.	2999.998	0.021924342	2999.976076	3000.01992
Low Wt.	1000.002	0.021908994	999.980091	1000.02391
Sensit. Wt.	10	0.021666667	9.978333333	10.0216667


Plot of Cornerload Devs. from Average


Plot of Biases (Maint.95% Error Bars) & SDs.

ACCEPTANCE CRITERIA	
Summary:	Balance passes QA Limits
Linearity(Bias)	Balance passes
Precision (SD)	Balance passes
Corner Loading	Balance passes
Lack of Fit	Balance passes
Disposition	AS LEFT - AS FOUND

SAVANNAH RIVER TECHNOLOGY CENTER
Actinide Technology Section Work Plans
Control of MS&E for Evaluation of Hydrogen Getter
Page: 1 of 4

Effective: January 25, 2001
Expiration: January 25, 2002
Number: 1.94
Revision: 0

Approved By: Alice M. Murray Owner
Approved By: Originator
Approved By: J. Pardo 1/29/01 CQF

Title: Control of MS&E for Evaluation of Hydrogen Getter

Owner: Alice M. Murray
Originator: Ronald R. Livingston
CQF: Julio Pardo

Purpose:

This work instruction documents the actions needed to control the pressure transducers and thermocouples used on the getter test apparatus in C-064 as MS&E. The use of this work instruction is part of completing a technical baseline task described in WSRC-RP-2001-00192, Task and Quality Assurance Plan for Hydrogen Getter Testing.

Scope:

These instructions are intended as a guide for calibration and documentation of measurement equipment. These work instructions are needed to support alternate controls for the measurement equipment proposed in the identified task plan. These work instructions are required because the alternate controls will address using data acquisition software as part of the measurement system. This measurement system is described in detail as part of the task plan and related job hazard assessment (SRT-HTS-2001-00008).

Authorized Personnel:

This work instruction applies to personnel working on the referenced task being lead by Jon Duffey and Ron Livingston.

Responsibilities:

The **Actinide Laboratory Program Group** is responsible for control of measurement equipment to be used for testing of hydrogen.

Originator is responsible for assuring that a SRTC Conduct of R&D review has been completed and documented.

The User is responsible for verifying the Work Instruction has not expired prior to use.

Work Instructions:

1) Place a copy of this work instruction in the research notebook.

2) The M&TE identification number and calibration expiration date for any M&TE used as part of this work instruction shall be recorded in the research notebook. Also record calibration data on measurement system accuracy or place a copy of calibration data sheet into the research notebook.

3) Measurement values generated as part of this work instruction and used to validate the performance of pressure transducers, thermocouples, or the data acquisition system shall be recorded in the laboratory notebook.

4) See SRTC manual L1 procedure 4.19 for details on properly maintaining research notebooks.

5) No software changes are allowed to the measurement system between initial and final checks described below.

6) Initial pressure transducer checks:

   a) Install a M&TE controlled pressure transducer on the test apparatus.
   b) Evacuate the test apparatus until the M&TE pressure transducer indicates a "zero" reading.
   c) Start the data logging software to record pressure data for each of the required pressure transducers. Measurement period should be about 5 seconds.
   d) Add a comment to file indicating the "zero" M&TE pressure reading once the pressure has stabilized.
   e) Record the M&TE result for "zero" and the MKS Baratron measured value in the research notebook. Leave space for recording the value logged to file.
   f) Increase the pressure in the test apparatus to about 20% of the intended calibration pressure range. [Note: No less than 5 measurements will be used to define measurement equipment accuracy.]
   g) Add a comment to the data logging software file indicating the new pressure from the M&TE pressure transducer once the pressure has stabilized.
   h) Record the M&TE result for the new pressure and the MKS Baratron measured value in the research notebook. Leave space for recording the value logged to file.
   i) Repeat steps (f) through (h) until required measurement range has been covered.
   j) M&TE controlled pressure transducer may remain installed on the test apparatus to support periodic checks of measurement equipment, or reinstalled as needed.
   k) Maintain M&TE records (e.g. usage log) as required by M&TE owner.
   l) Review the data logging software file and record the logged value into the research notebook beside the appropriate test pressure.

7) Initial thermocouple checks:

   m) Install an M&TE controlled temperature sensor along with test apparatus thermocouples into the calibration well of a thermocouple calibrator.
   n) Set the thermocouple calibrator at the minimum value required for the test assembly calibration range.
   o) Start the data logging software to record temperature data for each of the required thermocouples. Measurement period should be about 5 seconds.
   p) Add a comment to file indicating the M&TE temperature sensor reading once the calibrator temperature has stabilized.
   q) Record the M&TE result for this measured value in the research notebook. Leave space for recording the value logged to file.
   r) Increase the temperature setting of the calibrator to about 20% of the intended calibration temperature range. [Note: No less than 5 measurements will be used to define measurement equipment accuracy.]
   s) Add a comment to the data logging software file indicating the new temperature from the M&TE temperature sensor once the temperature calibrator has stabilized.
   t) Record the M&TE result for the new temperature in the research notebook. Leave space for recording the value logged to file.
   u) Repeat steps (r) through (t) until required measurement range has been covered.
   v) M&TE controlled temperature sensor can be used to support periodic checks of thermocouple(s) at ambient temperature when appropriate.
   w) Maintain M&TE records (e.g. usage log) as required by M&TE owner.
   x) Review the data logging software file and record the logged value into the research notebook beside the appropriate test temperature.

8) Document that measurement equipment performance meets accuracy requirements for test application in the research notebook.

9) Final thermocouple and pressure transducer checks:

   a) Add statement to research notebook confirming that software has not been changed since initial check.
   b) Repeat work instruction steps 6) and 7) using pressure and temperature set points as nearly identical to set points from initial tests as reasonably achievable.
   c) Confirm that measurement equipment performance meets accuracy requirements for the test application established following initial thermocouple and pressure transducer checks.

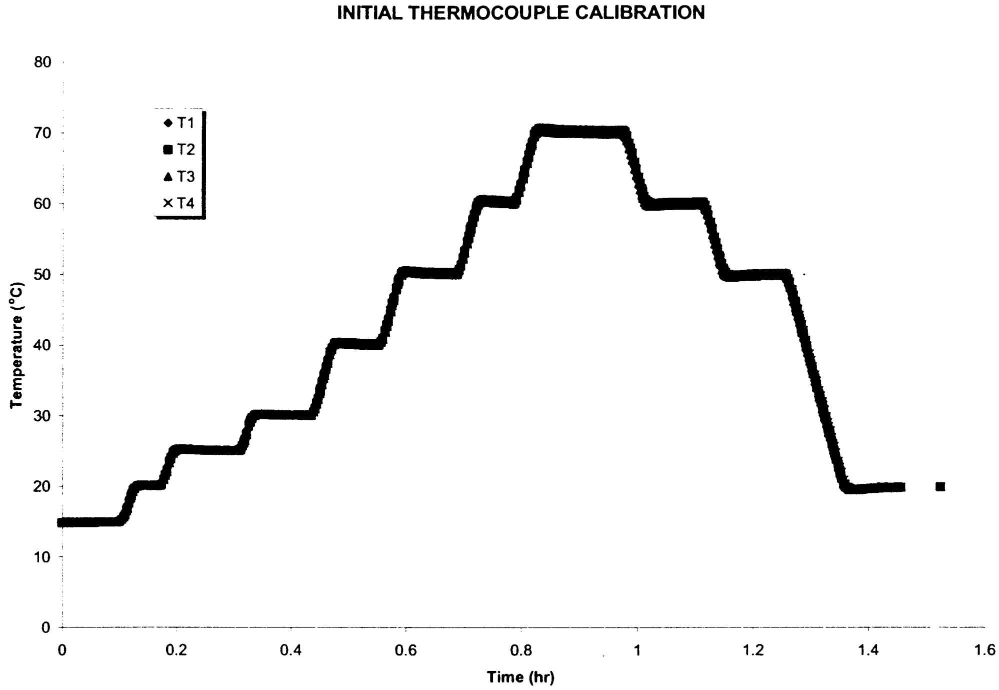


Figure 7-1

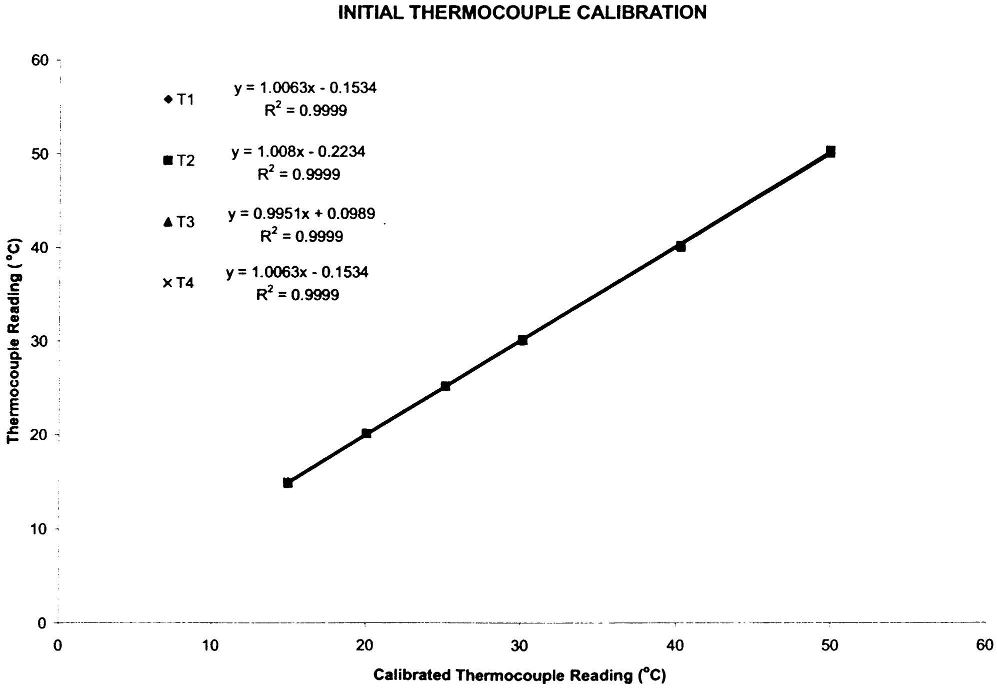
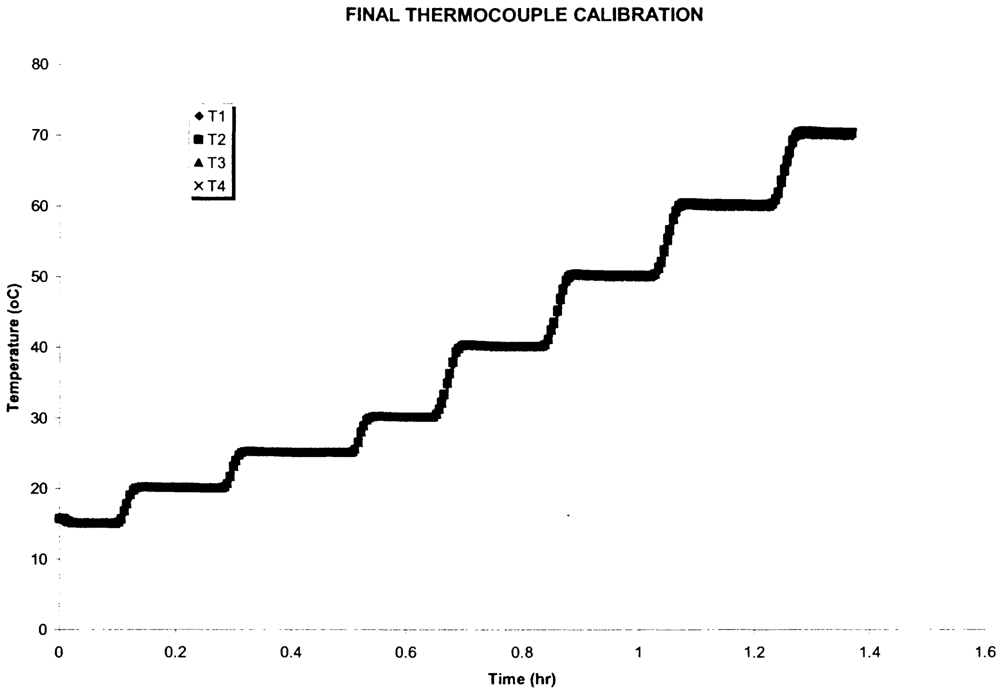
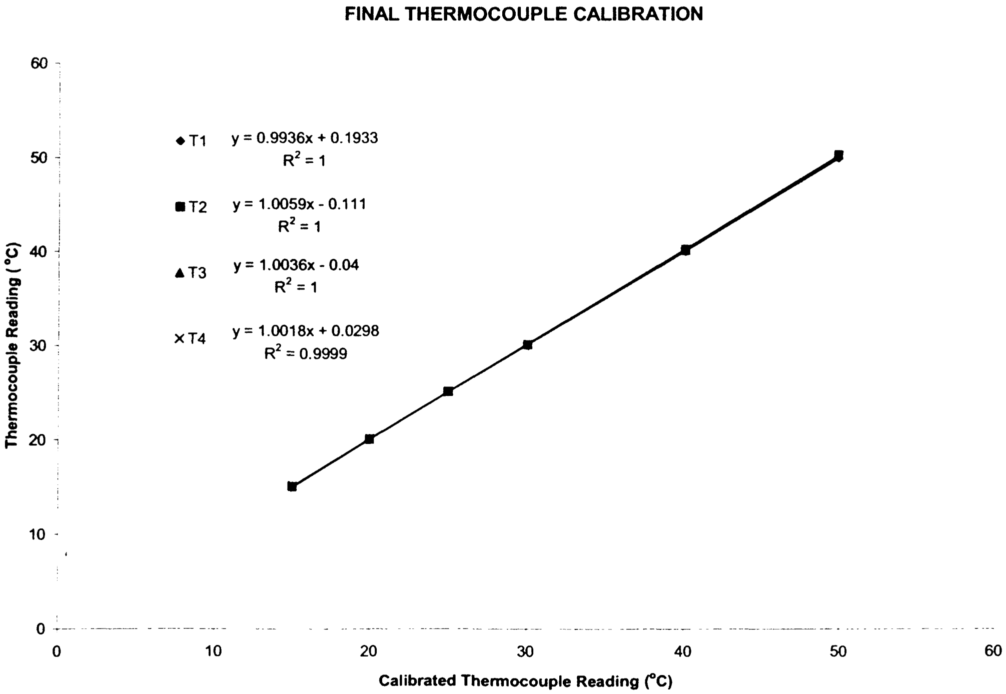


Figure 7-2



FINAL THERMOCOUPLE CALIBRATION

Figure 7-3



FINAL THERMOCOUPLE CALIBRATION

Figure 7-4

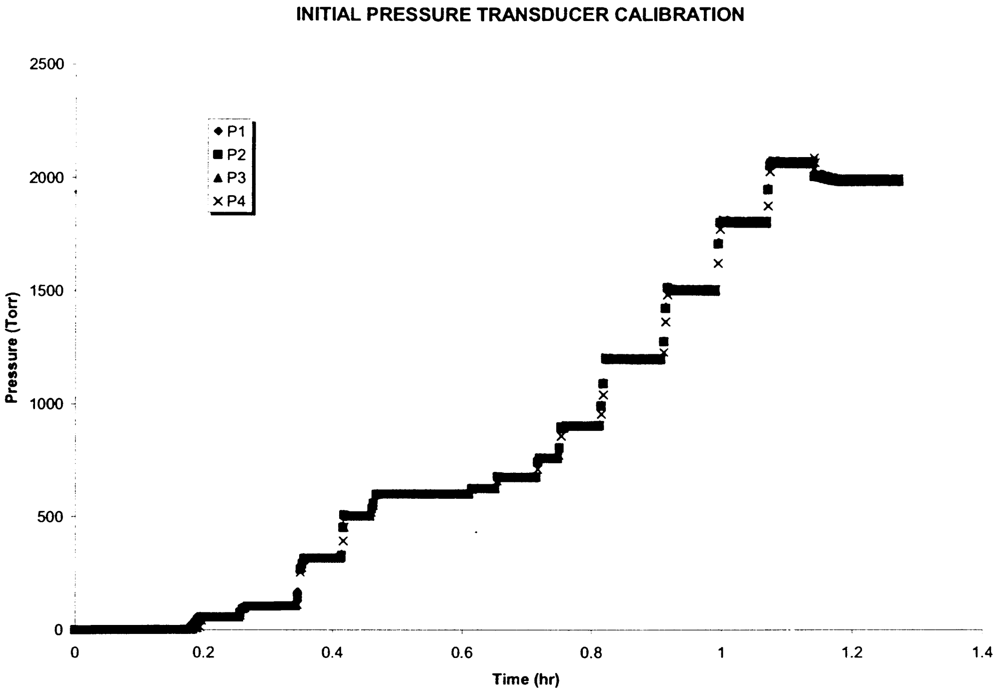


Figure 7-5

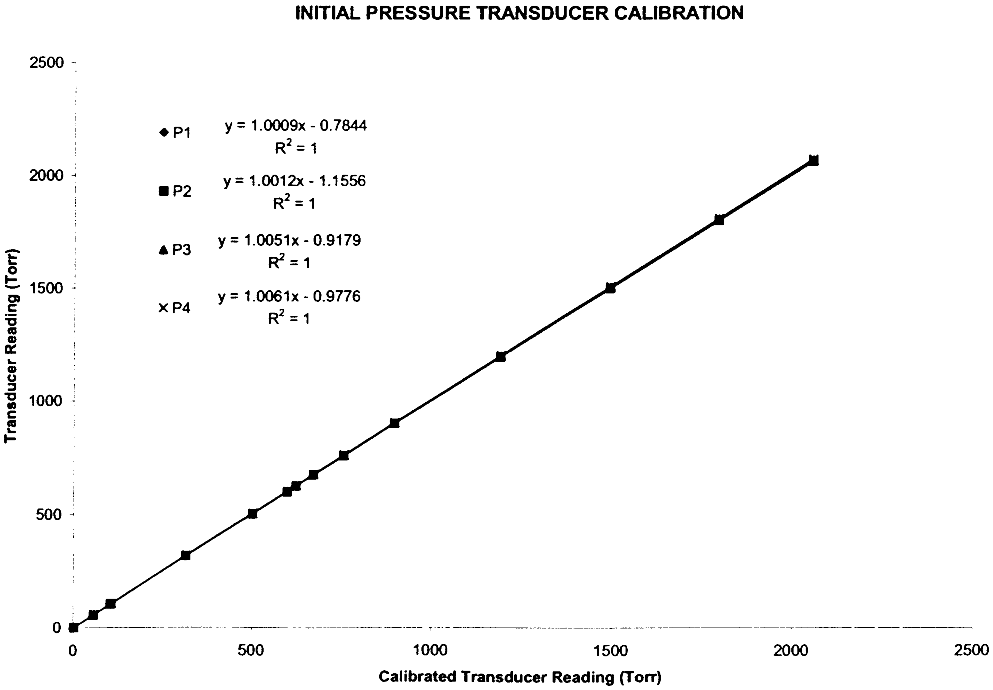


Figure 7-6

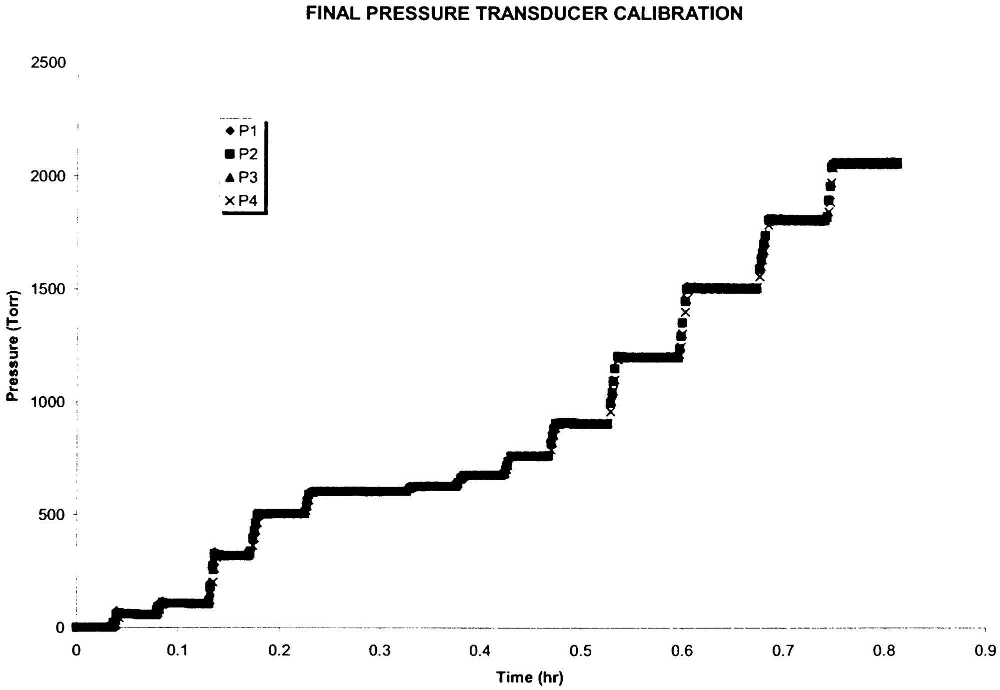


Figure 7-7

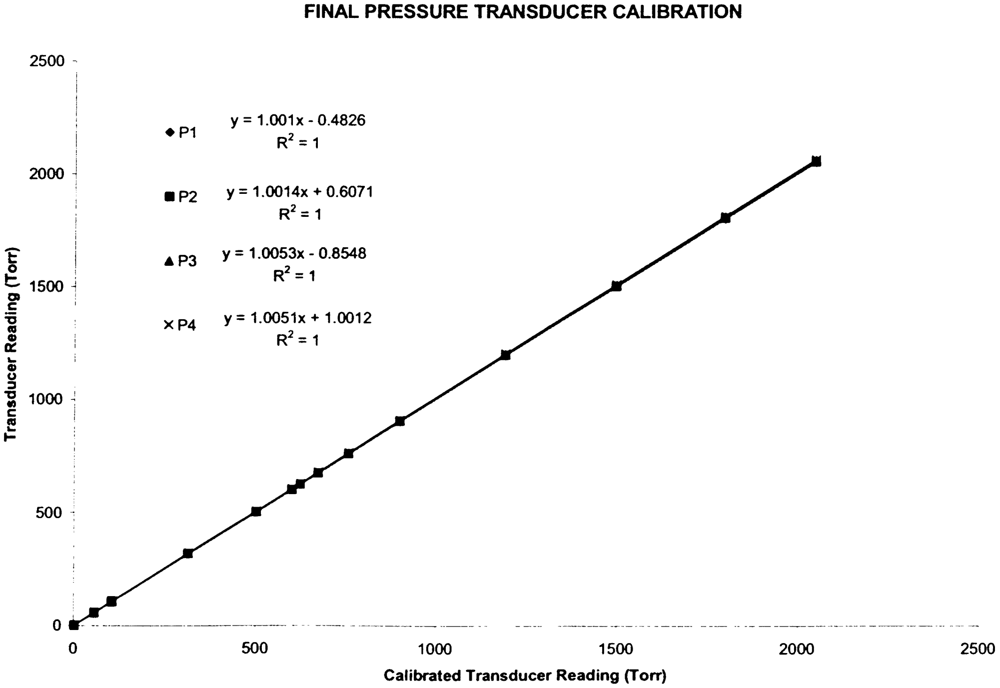


Figure 7-8

Task Technical and Quality Assurance Plan – Implementation of Hydrogen Getter in the DDF-1 Shipping Package

Document Number: WSRC-RP-2001-00192
Revision Number: 0
Preparation Date: January 25, 2001

Prepared By: Ronald R. Livingston Date: 1/25/01
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Prepared By: Jonathan M. Duffey Date: 1/25/01
Name: Jonathan M. Duffey, Senior Scientist, Actinide Laboratory Program Group

Reviewed By: T. Kurt Houghtaling Date: 1/25/01
Name: T. Kurt Houghtaling, Cognizant Technical Function, Radioactive Materials Packaging Technology Group

Reviewed By: Steve Hensel Date: 1/25/01
Name: Steve J. Hensel, DDF-1 Design Authority, FB-Line Engineering

Reviewed By: G. F. Molen Date: 01-25-2001
Name: Gary F. Molen, Cognizant Technical Function, NMSS Project Integration Department

Approved By: [illegible] for Allen L. Blancett Date: January 25, 2001
Name: Allen L. Blancett, Manager, Actinide Technology Section

Approved By: J. Stephen Bellamy Date: 1/25/01
Name: J. Steven Bellamy, Manager, Radioactive Materials Packaging Technology Group

Approved By: Tom Varallo Date: 1/25/01
Name: Thomas P. Varallo, Program Manager, NMSS Transportation Program ~~Manager~~ PV

Approved By: J. Pardo Date: 1/25/01
Name: Julio Pardo, Cognizant Quality Function, Quality Engineering Section

WSRC-RP-2001-00192
Revision 0

Task Technical and Quality Assurance Plan – Implementation of Hydrogen Getter in the DDF-1 Shipping Package

Ronald R. Livingston

Jonathan M. Duffey

Strategic Materials Technology Department
Savannah River Technology Center

January 2001

Westinghouse Savannah River Company
Savannah River Site
Aiken, SC 29808

Introduction

This document defines the testing required to validate the performance of a hydrogen getter for the DDF-1 package and describes the quality assurance measures that will be in place during execution of this task plan. The testing performed under this task plan is intended to demonstrate that the proposed getter is capable of meeting the requirements for on-site transportation of radioactive materials. The performance requirements addressed as part of this task plan include:

- Maintaining the hydrogen pressure of the DDF-1 primary containment vessel (PCV) at less than 2 psia hydrogen in the presence of air or nitrogen. This pressure limit keeps the hydrogen concentration in the PCV at less than 12 volume percent, assuming the PCV is packaged initially at 1 atmosphere pressure.

- Providing sufficient hydrogen removal rate and capacity for up to 14 standard cubic centimeters of hydrogen per hour (14 std cm^3 hr^{-1}) over a period of 30 days. This rate is based on the calculated rate of hydrogen generated from a one-watt container with a G-Value of 1.6 and 100% energy deposition into water. Consequently, this estimate is believed to be very conservative and exceeds the observed hydrogen generation rate for weapons grade PuO_2 containing about 5% moisture by a factor of nearly 10.[1]

The hydrogen getter assembly designed for this application contains a commercially available polymer hydrogen getter (Vacuum Energy, Inc.) to prevent the build-up of hydrogen gas within the Primary Containment Vessel (PCV) of the DDF-1 shipping package. This hydrogen getter has been evaluated previously as part of an extensive effort to examine the effects of temperature, pressure, poisons, radiation, and other factors on hydrogen absorption rate and capacity.[1] Results from this earlier effort will be used where needed to support development of the DDF-1 On-site Safety Assessment (OSA). However, the impact of vessel geometry and getter arrangement on hydrogen absorption rate was not addressed as part of the earlier study. Therefore it is prudent to demonstrate the performance of the current getter design prior to implementation within the DDF-1 package. As a result of this effort, we will document actual performance of the hydrogen getter under test conditions.

Hydrogen generation within the PCV will be simulated by leaking hydrogen into the PCV (either a nitrogen or air atmosphere) at or slightly above the maximum expected hydrogen generation rate of 14 std cm^3 hr^{-1} (11 Torr L hr^{-1}). In the absence of oxygen, the polymer hydrogen getter functions by an irreversible hydrogenation mechanism using a palladium catalyst. When oxygen is present, the polymer hydrogen getter can also function as a recombination catalyst. Therefore, the "getter assembly" will consist of both polymer hydrogen getter (~ 140 g) and zeolite (~ 100 g 4A molecular sieve) to adsorb any water produced from recombination. The getter and zeolite will be packaged separately but in close proximity to each other in a porous nylon fabric (CEREX®) that will contain the solid materials without restricting gas flow to the getter. The zeolite and getter are packaged separately to simplify potential recovery of the getter catalyst at a later date. Each test will use one prepackaged getter assembly, as described above, and evaluate the impact of geometry and gas composition on getter performance.

The experimental apparatus is constructed of an actual DDF-1 PCV (~ 5.2-L internal volume) modified with stainless steel tubing and fittings to allow for gas introduction, evacuation, and sampling (Figure 1). All connections to the PCV are made at the vessel bottom to minimize changes to the vessel geometry. Prior to use for hydrogen getter testing the test apparatus will be leak tested to assure any leaks are sufficiently small to have no impact on measurement accuracy. All components used in construction of this apparatus are anticipated to have leak rates less than $1x10^{-7}$ std cm^3 sec^{-1}. The pressure sensors are MKS Baratron® Series 690 sensors (0 – 5000 or 0 – 10,000 Torr). The apparatus is equipped with a Series 203 Variable Leak Valve (Granville-Phillips) to allow addition of hydrogen from a 1-L standard volume to the PCV at the desired rate to simulate hydrogen generation from within the nuclear material container. This standard volume is used for both calibration of the volume of manifold components and to supply hydrogen to the variable leak. Three separate thermocouple sensors will be positioned inside the PCV to monitor the temperature of the gas phase, getter, and zeolite. An additional thermocouple is positioned outside the PCV to monitor the temperature of the air in the laboratory.

Initial testing will consist of leaking hydrogen into a nitrogen-only atmosphere within the PCV to demonstrate that the rate of hydrogenation of the getter is sufficient to prevent the build-up of hydrogen in the PCV. The hydrogen addition rate into the PCV will be calculated from the drop in pressure in the standard volume (hydrogen supply container). This test will be conducted at increasing levels of getter loading up to approximately 80% of the theoretical hydrogenation capacity (~ 120 std cm^3 g^{-1}). At a leakage rate of 14 std cm^3 hr^{-1}, more than 30 days would be required to consume 80% of the theoretical getter capacity. To accelerate loading of the getter, testing will be alternated between two modes. The first mode will consist of leaking hydrogen into the PCV at a rate of 20 - 30 std cm^3 hr^{-1} for several hours to demonstrate the getter is capable of meeting the 14 std cm^3 hr^{-1} performance requirement. In the second test mode, the hydrogen flow rate will be increased by a factor of between 10 and 100 for a period of several hours to load the getter to some percentage (e.g., 0, 25%, 50%, 75%, and 90%) of its total capacity. These two test modes will be used to demonstrate that the getter can prevent hydrogen build-up within the PCV even when loaded to 90% of its theoretical capacity.

Following demonstration of the getter in a nitrogen-only atmosphere, performance of the getter as a recombination catalyst will be demonstrated. This test will be conducted by filling the PCV with room air, then leaking hydrogen into the PCV from the 1-L standard volume at a flow rate of 20 - 30 std cm^3 hr^{-1}. Because the getter functions primarily as a recombination catalyst, when both hydrogen and oxygen are present, a pressure drop due to oxygen consumption should be recorded as hydrogen flows into the PCV. This hydrogen addition will be continued until the pressure drop recorded in the PCV indicates that most of the oxygen has been consumed by recombination with hydrogen. Note that very little getter hydrogenation capacity should be consumed during the process of recombination. Similar to the nitrogen only test, this test will be accelerated by allowing the zeolite to adsorb slightly more water than the rated capacity (9 g) prior to executing this test. All other tests of the "getter assembly" are similar to those described here. The proposed configuration and brief description of each test are provided as part of *Task Activities.*

If the getter is not functioning properly a pressure rise due to hydrogen build-up in the PCV will be observed. This pressure rise will be used to estimate the hydrogen concentration in the PCV. This concentration estimate will be corrected for the hydrogen volume of the supply line because the volume of this tube is not accessible to the hydrogen getter.

Task Description

This task and quality assurance plan supports completion of activities requested by Steve Bellamy of the SRTC Radioactive Materials Packaging Technology Group (RMPT) under a Technical Task Request (TTR), TTR 01-RMPT-002. This TTR was initiated to support revision of the DDF-1 OSA to expand the allowed contents to include materials that liberate hydrogen gas and thereby, require the use of hydrogen getter. The Actinide Laboratory Programs Group is responsible for conducting this research and development (R&D) task. The actual test work will be conducted in laboratory space provided by the Hydrogen Technology Section. The test data generated as part of this task will be used to validate the design of the hydrogen "getter assembly" proposed for use in the DDF-1 package. The task deliverable is a technical report documenting these tests and other pertinent information on the hydrogen getter assembly. The actual performance of the getter assembly (e.g. showing that hydrogen is maintained at < 4% during testing in air) will be documented allowing the technical agency (RMPT) and design authority an opportunity to expand the safety basis used for the DDF-1. The RMPT organization, the DDF-1 design authority, and the NMSS Project Integration Department will approve the technical report. The agreed upon approach for these tests is to use an actual DDF-1 primary containment vessel to demonstrate that the "getter assembly" will meet the identified performance requirements.

Task Activities

Table I provides a list of tests that will be conducted as part of this task plan in order to validate the "getter assembly" design. Table II is a list of task activities supporting validation of the DDF-1 hydrogen getter assembly. All measurement systems used to support this task will be controlled either as Measurement and Test Equipment (M&TE) or use controls similar to Measurement Systems and Equipment (MS&E), as described by this plan. All tests and relevant supporting activities will be documented in a laboratory notebook. The required accuracy of these measurement devices has been taken into account in the assembly of the test apparatus. A discussion of measurement error will be presented in the technical report.

The pressure transducers and thermocouples installed on the test apparatus will be evaluated using suitable standards and controlled similar to MS&E using the lab notebook for all required records. A work instruction will be prepared, as described in SRTC manual L1 procedure 1.01.1, Rev. 0., to define the calibration and control of these components of the apparatus. The Cognizant Quality Function (CQF) and ATS management will approve this work instruction. The instrument controller and software used to log data from these transducers is considered a part of the MS&E system. The performance of this system will be confirmed using calibrated M&TE both before and after completion of the required tests. This evaluation will be completed at selected intervals over the entire pressure (0 to 30 PSIA) and temperature (15 to 70° C) ranges needed for completion of these tests. A comparison will be made between manually recorded values and data stored by the instrument controller to assure proper function of the measurement system. No changes to the function of this software will be allowed between calibration checks. Where possible, the M&TE devices will be incorporated into the test apparatus to allow periodic cross checks of system function (i.e. install the M&TE pressure transducer into the system manifold and make periodic comparisons of manifold pressure using each of the MS&E pressure transducer).

Table I –Tests Required to Validate Hydrogen Getter Assembly Performance

Test	Description of Test	Equipment Configuration
1	H_2 flow rate 20-30cc/hr with getter at ~0, 50 and 90% of theoretical capacity. These tests will be conducted with N_2 gas in the PCV. Pressure measured over ~2-4 hours, then getter capacity raised to next loading.	Test conducted with dummy product can(s) in PCV. Demonstrate effect of H_2 leak location (i.e. bottom or middle of vessel) on getter performance.
2	Use H_2 saturated getter assembly from test 1. Remove dummy product cans and leave PCV filled with air. Measure recombination rate of getter.	Zeolite is present to adsorb water produced during recombination reaction to prevent condensation of moisture.
3	Repeat Test 2. Replace getter assembly with one containing zeolite preloaded with water (10g) to demonstrate capability of zeolite to adsorb excess moisture.	Remove dummy product can(s) to maximize available O_2 for recombination. Recombination rate must remain at acceptable level.
4	Using a new getter assembly, repeat test 1 with getter assembly "crushed" to restrict available surface. Test needs to be conducted at only a single H_2 loading.	Same as Test 1 with heavy weight on top of "getter assembly." H_2 flow only required at bottom of PCV
5	Test 3 additional getter assemblies in the PCV for initial H_2 absorption rate to demonstrate that assemblies are produced in reproducible fashion.	Run these tests with a single PCV configuration with dummy product can(s) in place.

Mass measurements required to implement this task plan will be completed using calibrated M&TE of sufficient accuracy for the particular activity. These results will be recorded manually in the laboratory notebook. Additional measurements provided by the Analytical Development Section are expected to be of a routine nature and will be completed using existing procedures and quality assurance protocol.

TABLE II –Task Activities Supporting Validation of Hydrogen Getter Assembly

Task	Description	Comments
1	Leak Check Apparatus	Define acceptance criteria
2	P and T transducer Evaluation	Handle similar to MS&E
3	V calibration of apparatus	Define standard's traceability
4	Preparation of getter assemblies	M&TE balance
5	Confirm data acquisition performance	Use M&TE
6	Develop valve alignment checklist	From HA and JHA
7	Use M&TE periodic checks	Confirm T and P
8	Gas Sampling and Analysis	Routine ADS methods

Task Schedule

The currently scheduled completion date of all activities covered by TTR-01-RMPT-002 is April 2001. The R&D efforts covered by this task and quality assurance plan will require about two or three weeks to complete and should be finished the middle of February 2001. The technical report documenting results of this test will then be issued by the end of February 2001. Funding and schedule are reviewed on a weekly basis with the RMPT organization and other project customers. Once the tasks covered by this plan are completed, ATS will continue to support RMPT with OSA preparation as needed.

Facility Planning

The impact of this task has been reviewed using the <u>SRTC Conduct of R&D</u>[3] checklists. The required Hazard Assessment and Job Hazard Analysis[3] have been completed and reviewed by the lab custodian. No radiation exposure or radioactive material handling is required to complete this task. No toxic chemicals are used or hazardous waste generated as a result of this effort. All hydrogen getter consumed as a result of this test will be stored for eventual recycle. All equipment used in performance of this task is expected to remain in the HTS lab following completion of the testing.

No intellectual property should be generated as a result of this task. However, the technical report will be reviewed according to appropriate procedure prior to publication or release to the public. There are no known classification or security issues associated with completion of this task.

Programmatic Risk Review

Completion of this task in a timely manner is deemed important for on-site transportation of radioactive materials. Operating plans call for transfer of nuclear materials from FB-Line and 235-F Vaults for processing in the HB-Line facility. Implementation of a hydrogen getter for use in radioactive material transportation is a non-traditional approach that may be scrutinized.

No changes to the technical area procedures are required to support this task plan. All issues related to handling hydrogen gas and hydrogen safety or environmental factors have been addressed as part of the Hazard Assessment and Job Hazard Analysis.[4]

Quality Assurance Controls

Successful completion of this task may result in the implementation of the hydrogen getter assembly into the DDF-1 package. The function of this getter assembly is an integral part of the OSA required to authorize on-site transportation of radioactive materials. Consequently this R&D task is considered a technical baseline activity.

Applicable sections of the WSRC 1Q, Quality Assurance Manual, and specific implementing procedures that will be used to control the execution of this task are shown in Attachment 1, *QA Checklist*, as recommended by the Quality Assurance Manual[5].

Document Approval

This task and quality assurance plan and all resulting technical documents will be reviewed and approved by both the RMPT group and the DDF-1 design authority.

Records

- TTR-01-RMPT-0002, Technical Task Request, H2 Getter Development for DDF-1 Rev 1 OSA, Steve Bellamy, October 2000.
- WSRC-RP-2001-00192, Task Technical and Quality Assurance Plan – Implementation of Hydrogen Getter in the DDF-1 Shipping Package
- SRT-HTS-2001-00008, Hazard Assessment for 773-A, C-064: Hydrogen Getter Testing in the DDF-1 Primary Containment Vessel
- Laboratory Notebook(s)
- Technical Report(s)
- Work Instruction(s)

References

1) WSRC-TR-99-00223, Gas Generation Test Support for Transportation and Storage of Plutonium Residue Materials, Ronald R. Livingston, Westinghouse Savannah River Company, August 1999.

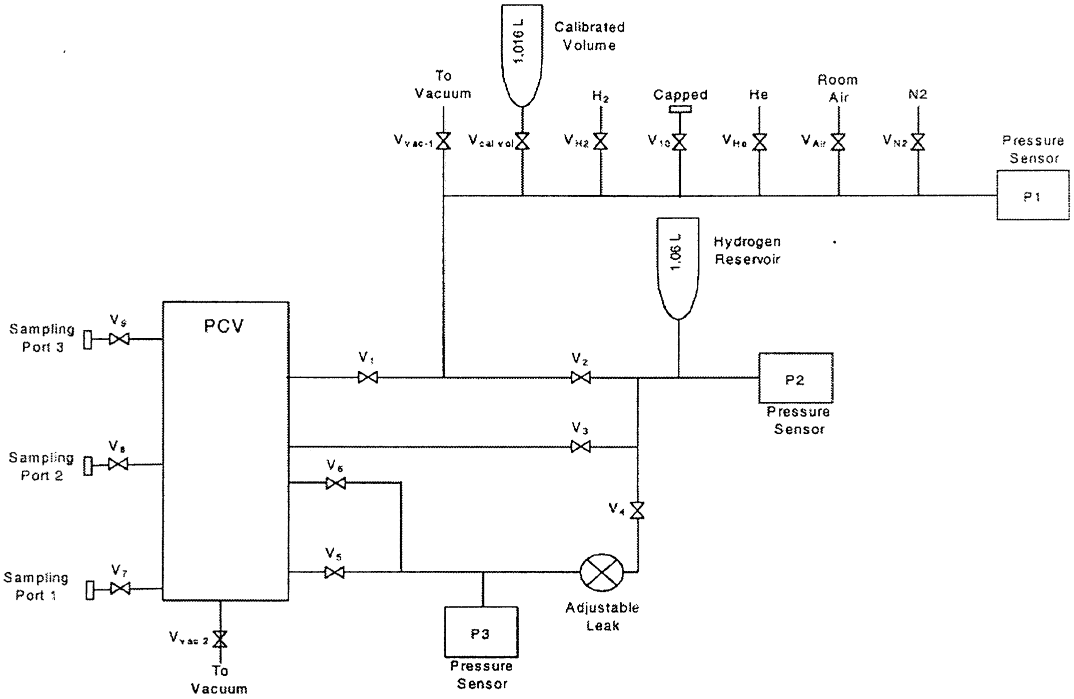
2) WSRC-RP-2000-00901, Hydrogen Getters for Use in the TRUPACT-II, J.M. Duffey and R.R. Livingston, Westinghouse Savannah River Company, October 2000.

3) WSRC-IM-97-00024, Rev. 2, "Savannah River Technology Center Conduct of Research and Development."

4) SRT-HTS-2001-00008, Hazard Assessment for 773-A, C-064: Hydrogen Getter Testing in the DDF-1 Primary Containment Vessel

5) WSRC Procedure Manual 1Q, Quality Assurance Manual, Procedure QAP 2-3, Conduct of Research and Development Activities, Rev. 3, November 1999.

Figure 1. Schematic of Test Apparatus



Attachment 1 Task QA Plan Procedure Matrix

Task Title: Gas Generation Test Support for Revision of 9975 SARP

Task Technical Plan Document No: WSRC-RP-99-00164

Listed below are the 21 sections of WSRC QA Manual 1Q and procedures used by SRTC to implement those sections. Indicate the specific procedures to be used to control the task. This checklist identifies only procedures used to control task activities performed by SRTC.

WSRC 1Q Section	SRTC Implementing Procedures	Identify Specific Procedures Applicable to Tas (Y-Yes, N-No, AR-As Required)
Organization	1Q, QAP 1-1, Organization	Y
	1Q, QAP 1-2, Stop Work	AR
	L1, 1.02, SRTC Organization	Y
Quality Assurance Program	1Q, QAP 2-1, Quality Assurance Program*	Y
	L1, 8.01, SRTC QA Program Implementation	Y
	L1, 8.02, SRTC QA Program Clarifications	Y
	1Q, QAP 2-2, Personnel Training & Qualification	Y
	L1, 1.32, Read and Sign	AR
	L1, 1.33, Facility Access Training/Employee Orientation	N
	1Q, QAP 2-3, Control of R & D Activities*	Y
	L1, 7.10, Control of Technical Work**	Y
	1Q, QAP 2-7, QA Program Requirements for Analytical Measurement Systems	Y
Design Control	1Q, QAP 3-1, Design Control	N
	L1, 7.10, Control of Technical Work	Y
Procurement Document Control	1Q, QAP 4-1, Procurement Document Control*	N
	7B, Procurement Management Manual	N
	3E, WSRC Procurement Specification Manual	N
Instructions Procedures, and Drawings	1Q, QAP 5-1, Instructions Procedures, and Drawings	N
	L1, 1.01, SRTC Procedure Administration	AR
	L1, 1.01.1, Work Instruction Administration	Y
	E7, 2.30, Drawings	N
Document Control	1Q, QAP 6-1, Document Control	Y
	L1, 1.30, Document Control	Y
Control of Purchased Items and Services	1Q, QAP 7-2, Control of Purchased Items and Services	AR
	7B, Procurement Management Manual	AR
	3E, WSRC Procurement Specification Manual	N
Identification and Control of Items	1Q, QAP 8-1, Identification and Control of Items*	N

Control of Processes	1Q, QAP 9-1, Control of Processes 1Q, QAP 9-2, Control of Nondestructive Examination 1Q, QAP 9-3, Control of Welding and Other Joining Processes 1Q, QAP 9-4, Work Control*	N N N N
Inspection	1Q, QAP 10-1, Inspection and Verification L1, 8.10, Inspection L1, 8.10.1, Independent Inspection Releases	N N N
Test Control	1Q, QAP 11-1, Test Control	N
Control of Measuring and Test Equipment	1Q, QAP 12-1, Control of Measuring and Test Equipment 1Q, QAP 12-2, Control of Installed Process Instrumentation	Y N N
Packaging, Handling, Shipping, and Storage	1Q, QAP 13-1, Packaging, Handling, Shipping, and Storage*	N
Inspection, Test, and Operating Status	1Q, QAP 14-1, Inspection, Test, and Operating Status*	N
Control of Nonconforming Items and Activities	1Q, QAP 15-1, Control of Nonconforming Items* 1Q, QAP 15-2, Control of Nonconforming Activities*	AR AR
Corrective Action System	1Q, QAP 16-1, Corrective Action System	N
Quality Assurance Records	1Q, QAP 17-1, Quality Assurance Records Management L1, 4.19, Laboratory Notebooks and Logbooks	Y Y
Audits	1Q, QAP 18-6, Quality Assurance Internal Audits 1Q, QAP 18-2, Quality Assurance Surveillance L1, 8.18.1, Surveillance 1Q, QAP 18-4, Management Assessments 12Q, SA-1, Self-Assessment by Individual Assessment Units	N N N N N
Quality Improvement	1Q, QAP 19-2, Quality Improvement*	N
Software Quality Assurance	1Q, QAP 20-1, Software Quality Assurance L1, 8.20, Software Management and Quality Assurance	N N
	1Q, QAP 21-1, Quality Assurance Requirements for the Collection and Evaluation of Environmental Data	N
Special (List As Needed)	L1, 8.21, Supplemental Quality Assurance Requirements for DOE/RW-0333P L1, Special Nuclear Material Procedures	N N

***Supplemented by a SRTC clarification in L1, 8.02, SRTC QA Program Clarifications**

**** The Task QA Plan shall identify the following information: (1) whether work is Technical Baseline or Non-Baseline, (2) whether work is R&D, Routine Service, or Engineering/Design, and (3) whether the work is for onsite or offsite customer. By identifying this information, procedure 7.10 identifies the E7 procedures applicable, as needed, for the work, and it is not necessary to list the E7 procedures in the QA plan unless there is a specific need to do so for the task.**

OSR 31-688 (Rev 11 20 97)
Stores 26 8910 00

WESTINGHOUSE SAVANNAH RIVER COMPANY
INTEROFFICE MEMORANDUM

BNFL

Date: January 23, 2001

SRT-HTS-2001-00008

To: D. D. Wilhelm, 773-A

From: R. R. Livingston, 773-A 1/23/01
J. M. Duffey, 773-A J. M. Duffey 1/23/01

Cc: L. K. Heung, 773-A
A. M. Murray, 773-A

HAZARD ASSESSMENT FOR 773-A, C-064: HYDROGEN GETTER TESTING IN THE DDF-1 PRIMARY CONTAINMENT VESSEL (PCV)

This document summarizes implementation of the Conduct of Research & Development for the Savannah River Technology Center[1] for the demonstration of a hydrogen getter in the DDF-1 primary containment vessel (PCV) in 773-A, C-064. The results showed that a Job Hazard Analysis (JHA) should be conducted.

WORK SCOPE

This work consists of a full-scale demonstration of the ability of a commercially available polymer hydrogen getter (Vacuum Energy, Inc.) to prevent the build-up of hydrogen gas within the PCV of the DDF-1 nuclear material-shipping container. Hydrogen generation within the PCV will be simulated by leaking hydrogen into the PCV (either a nitrogen or air atmosphere) at or slightly above the maximum expected hydrogen generation rate of 14 std cm^3 hr^{-1} (11 Torr L hr^{-1}). In the absence of oxygen, the polymer hydrogen getter functions by an irreversible hydrogenation mechanism using a palladium catalyst. When oxygen is present, the polymer hydrogen getter can also function as a recombination catalyst. Therefore, the "getter assembly" will consist of both polymer hydrogen getter (~ 140 g) and zeolite (~ 100 g 4A molecular sieve) to absorb any water produced from recombination. The getter and zeolite will be packaged separately but in close proximity to each other in a porous nylon fabric (CEREX™) that will contain the solid materials without restricting gas flow to the getter.

The experimental apparatus is constructed of an actual DDF-1 PCV (~ 5.2-L internal volume) modified with stainless steel tubing and fittings to allow for gas introduction, evacuation, and sampling (Figure 1). The pressure sensors are MKS Baratron® Series 690 sensors (0 – 5000 or 0 – 10,000 Torr). The apparatus is equipped with a Series 203 Variable Leak Valve (Granville-Phillips) to enable addition of hydrogen from a 1-L standard volume to the PCV at the desired rate to simulate hydrogen generation from the nuclear material container. Three separate thermocouple sensors can be positioned inside the PCV to

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[1] WSRC-IM-97-00024, Rev. 2, "Savannah River Technology Center Conduct of Research and Development."

monitor the temperature of the gas phase, getter, and molecular sieve. An additional thermocouple is positioned outside the PCV to monitor the temperature of the air in the laboratory.

Initial testing will consist of leaking hydrogen into a nitrogen-only atmosphere within the PCV to demonstrate that the rate of hydrogenation of the getter is sufficient to prevent the build-up of hydrogen in the PCV. The leakage rate of hydrogen into the PCV will be monitored by the drop in hydrogen pressure in the secondary container. This test will be conducted at increasing levels of getter loading up to approximately 80% of the theoretical hydrogenation capacity (~ 120 std cm^3 g^{-1}). At a maximum leakage rate of 14 std cm^3 hr^{-1}, more than 30 days will be required to consume 80% of the theoretical getter capacity. To accelerate loading of the getter, testing will be alternated between two modes. The first mode will consist of leaking hydrogen into the PCV at a rate of 14 - 30 std cm^3 hr^{-1} for several hours to simulate the conditions for the maximum expected hydrogen generation rate. In the second test mode, the rate of hydrogen addition will be increased by a factor of between 10 and 100 for a period of several hours to load the getter to some percentage (e.g., 25%, 50%, 75%, and 90%) of its total capacity. These two test modes will be used to demonstrate that the getter can prevent hydrogen build-up even when loaded to 90% of its theoretical capacity.

Following demonstration of the getter in a nitrogen-only atmosphere, room air will be introduced into the PCV and hydrogen will be leaked into the PCV from the 1.0-L volume at the rate of 14 - 30 std cm^3 hr^{-1}. Because the getter functions primarily as a recombination catalyst when both hydrogen and oxygen are present, a pressure drop due to oxygen consumption will be recorded as hydrogen is leaked into the PCV. This hydrogen addition will be continued until the pressure drop recorded in the PCV indicates that all of the oxygen has been consumed by recombination with hydrogen. Note that getter hydrogenation capacity will not be consumed during the process of recombination.

If the getter is not functioning properly a pressure rise due to hydrogen build-up in the PCV will be recorded. In this event, addition of hydrogen to the PCV will be stopped before the hydrogen concentration (as indicated by pressure rise) reaches the lower flammability limit of 4.0 vol %.[2] The worst-case accident scenario would consist of the formation and detonation of an explosive mixture of hydrogen in air (18 – 59 vol % H_2 in air).[2] Numerous controls are in place to prevent this accident from occurring even though the maximum calculated pressure (~ 500 psia) resulting from this accident is not sufficient to cause catastrophic failure of the test apparatus or physical harm to room occupants. These controls are:

(1) The volumes and pressures of both hydrogen and air will be controlled so that the maximum possible hydrogen concentration in air is less than 18%.
(2) Tests will be conducted only by Duffey or Livingston, and a valve alignment checklist will be used to assure proper valve positions.
(3) No known ignition sources are present (catalyst content of getter is ~ 1% in a polymer matrix - this prevents the catalyst from reaching the autoignition temperature for hydrogen in air).
(4) The hydrogen content of the apparatus is limited to a quantity easily removed by the hydrogen getter/recombiner.

If the worst case mixture of hydrogen in air is formed and ignited, the resulting pressure from deflagration in the test apparatus is estimated to increase by a factor of approximately 8 (maximum flame temperature ~ 2129 °C).[2] This resulting pressure (< 200 psia) is well below the pressure limits for this apparatus. The failure mode for this accident would be bursting of the MKS Baratrons®, which have been shown to require in excess of 1000 psig to rupture the case.[3]

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[2] DPST-82-717, "Gaseous Hydrogen Safety in a Confined Laboratory: User's Guide," L. K. Heung, 1982.
[3] WSRC-TR-93-420, "Burst Testing of Low Pressure MKS Baratron® Sensors," J. R. Wermer, August 1993.

HAZARDS ASSESSMENT

STEP 1. PRELIMINARY HAZARD ASSESSMENT LEVEL DETERMINATION

Is the activity a routine service – no.

STEP 2. HAZARDS SCREENING

IONIZING RADIATION

A. Radioactive materials – no.
B. Fissionable materials (accountable or > reportable quantity) – no.

NON-IONIZING RADIATION

A. Lasers – no.
B. Magnetic fields > 600 Gauss – no.
C. Sub-radiofrequency (< 30 kHz) electric and magnetic fields – no.
D. Microwave/radifrequencies (30 kHz – 300 GHz), electric or magnetic fields – no.
E. High intensity light and/or near-infrared radiation – no.
F. Ultraviolet radiation – no.

OTHER ENERGIES

A. Electricity (exposed energized parts > 50V) – no.
B. Maintenance or modification of equipment containing hazardous energy – **yes** (Figure 12).
C. Temperature (< 32F or > 104F) – no.
D. Vacuum and external pressure – **yes** (Figures 8, 11, & 12).
E. Internal pressure – **yes** (Figures 8, 11, & 12).

CHEMICALS/HAZARDOUS MATERIALS

A. Biological agents – no.
B. Carcinogens, mutagens, teratogens – no.
C. Corrosives – no.
D. Cryogenic gases/liquids – no.
E. Flammable/combustible gases, liquids, solids – **yes** (Figures 8, 9, 10, & 11).
F. Toxic chemicals – no.
G. Lead/Asbestos/Silica - no.
H. Oxidizers – no.
I. Explosives – no.
J. Pyrophorics – no.
K. Volatile solvents – no.
L. Compressed gases – **yes** (Figures 8, 9, 10, & 11).

HAZARDOUS MATERIALS TRANSFER AND SHIPMENT

A. Transfer (on-site) or shipment (off-site) of corrosive, flammable, oxidizer, explosive, or radioactive goods – no.

WORKSITE ENVIRONMENTAL CONDITIONS

A. Boating or work over water – no.
B. Cold or heat stress conditions – no.
C. Confined spaces, trenches, or evacuations – no.
D. Flammable atmospheres (≥ 10% lower explosive limit (LEL)) – no.
E. Oxygen deficient atmospheres (< 19.5% O_2) – no.
F. Toxic atmosphere – no.
G. Activity performed in a nuclear facility – yes (Figure 6).
H. High noise levels (> 85 dBA) – no.
I. Moving equipment – no.
J. Airborne mists, dusts, vapors – no.
K. Known or suspected hazardous waste site – no.
L. Open flame or spark – no.

ENVIRONMENTAL COMPLIANCE

A. Potential release of regulated gas or particulate to the environment – no.
B. Potential release of regulated materials to a waste disposal system – no.
C. Creation of hazardous/mixed/radioactive wastes – no.

STEP 3. PROCESS HAZARD LEVEL DETERMINATION

There were YES answers to the Hazard Screening Checklist questions. There is no potential to exceed established Process Hazard Review criteria. A Job Hazard Analysis (JHA) has been conducted (See Attachment 1).

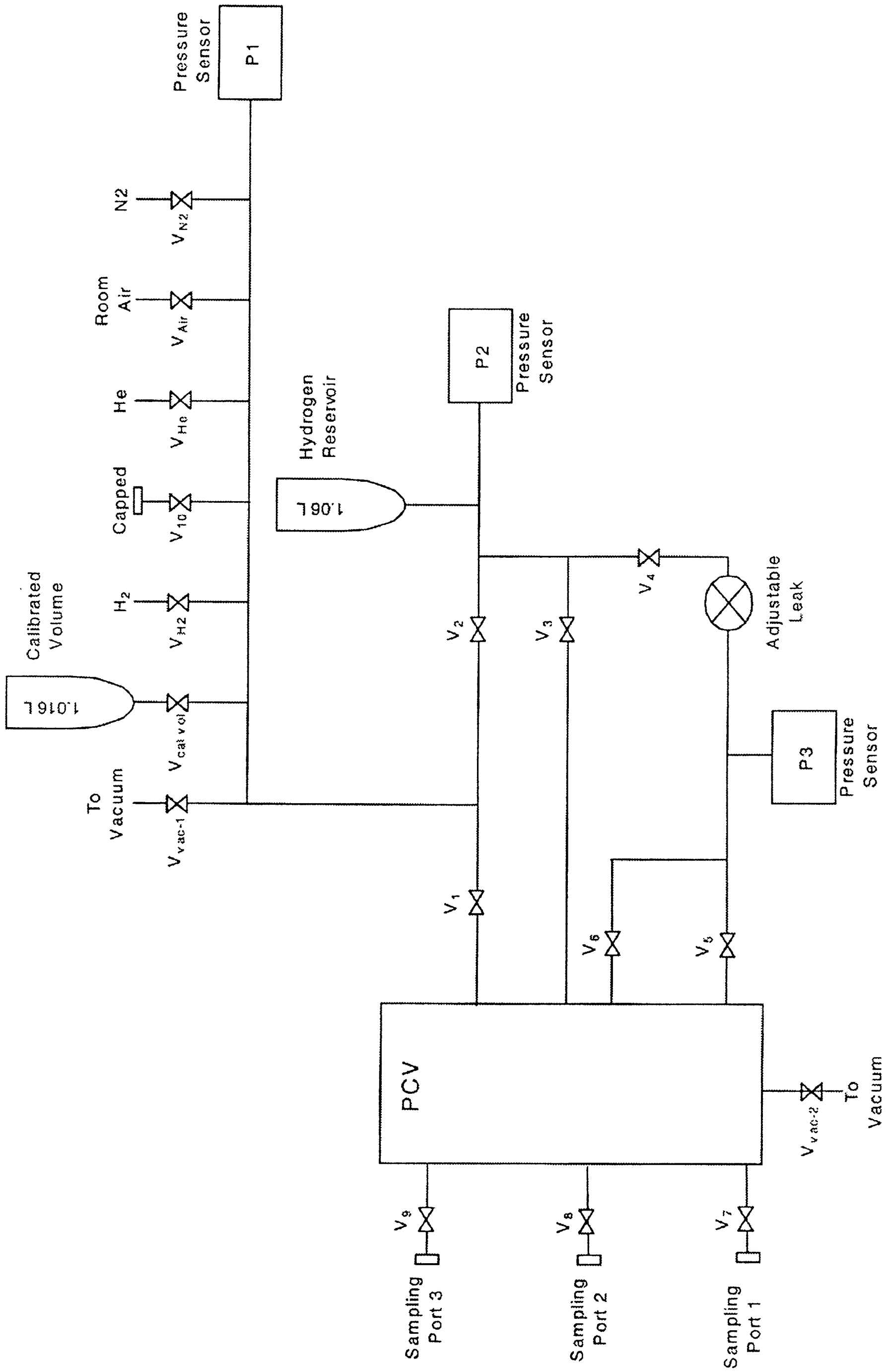

Figure 1. Schematic of test apparatus.

ATTACHMENT 1
JOB HAZARD ANALYSIS

Title:	Hydrogen Getter Testing In The DDF-1 Primary Containment Vessel (PCV)
Date:	January 11, 2001
Job Location:	773-A, C-064

A Job Hazard Analysis (JHA) was conducted by Ron Livingston (HTS, Principal Investigator), Jon Duffey (ATS) and Kit Heung (HTS, Lab Custodian) for the DDF-1 hydrogen getter testing in 773-A, C-064 Laboratory on January 11, 2001. The important job steps, potential hazards, and recommended safe practices identified as the result of the JHA are listed in Table 1.

JHA Team Members:

Position	Name	Signature	Date
Principal Investigator	R. R. Livingston		1/23/~~00~~01 RRL
Senior Scientist	J. M. Duffey	J. M. Duffey	1/23/01
Laboratory Custodian	L. K. Heung	L. K. Heung	1/23/01

Reviewed By:

Date 1/29/01
HTS Manager, D. D. Wilhelm

ATTACHMENT 1 (CONTINUED)
JOB HAZARD ANALYSIS

Table 1. Hydrogen Getter Testing in DDF-1 PCV

Key Job Steps	Hazards/Potential Hazards	Safe Practices
1. Place getter assembly in PCV and close lid on PCV.	(a) Sharp edges on PCV lid.	(a) Wear gloves when handling lid.
2. Evacuate and check system for leaks.	(a) No potential hazard.	(a) N/A
3. Add nitrogen into PCV.	(a) Hydrogen added to PCV instead of nitrogen.	(a) Use valve alignment checklist to prevent inadvertent hydrogen addition to PCV.
4. Add hydrogen into hydrogen reservoir.	(a) System leaks. (b) Hydrogen added to PCV instead of to 1.0-L volume.	(a) Check system for leaks before adding gases to apparatus. (b) Use valve alignment checklist to prevent inadvertent hydrogen addition to PCV.
5. Adjust leak valve to add hydrogen to nitrogen in PCV at desired rate.	(a) No potential hazard.	(a) N/A
6. Increase hydrogen addition rate to load getter to desired capacity.	(a) No potential hazard.	(a) N/A
7. Repeat steps 5 and 6 until test is complete.	(a) No potential hazard.	(a) N/A
8. Remove getter assembly, replace with fresh getter, and seal PCV in air.	(a) Sharp edges on PCV lid.	(a) Wear gloves when handling lid.
9. Adjust leak valve to add hydrogen to air in PCV at desired rate.	(a) Hydrogen added in excess of lower flammability limit (4% by volume).	(a) Use valve alignment checklist to prevent inadvertent hydrogen addition to PCV.
10. Take gas samples if necessary.	(a) No potential hazard.	(a) N/A

ATTACHMENT 2

VALVE ALIGNMENT CHECKLIST

Key Job Step	Sub-Steps	Check When Complete
1. Place getter assembly in PCV and close lid on PCV.	(a) Close VN2, Vair, VHe, VH2, V7, V8, V9, and V10.	
	(b) Open Vvac-1 and Vvac-2.	
	(c) Open V1, V2, V3, V4, V5, and V6 to evacuate entire system.	
	(d) When P1 - P3 read 0 Torr, close V2, V3, V4, V5, and V6.	
	(e) Close Vvac-1 and Vvac-2.	
	(f) Open Vair to equilibrate PCV with atmosphere.	
	(g) Close Vair.	
	(h) Open PCV lid, place getter assembly in PCV, and tighten lid to mark.	
	(i) Close all valves.	
2. Evacuate and check system for leaks.	(a) Open Vvac-1 and Vvac-2.	
	(b) Open V1, V2, and V5. Evacuate system at least 2 hr.	
	(c) Close Vvac-1 and Vvac-2.	
	(d) Close V1 and V2 and monitor rate of pressure rise for P1, P2, and P3.	
	(e) Close all valves.	
3. Add nitrogen into PCV.	(a) Open V1 and V5	
	(b) Add N2 to desired pressure (P3) and closed VN2.	
	(c) Close all valves.	
4. Add hydrogen into hydrogen reservoir.	(a) Ensure all valves are closed and leak is set to 00000.	
	(b) Open V2 and V4.	
	(c) Add H2 to desired pressure (P1 and P2) and close PH2.	
	(d) Close V2.	
	(e) Open Vvac-1 to evacuate H2 from manifold line.	

Key Job Step	Sub-Steps	Check When Complete
	(f) When P1 reads 0 Torr, close Vvac-1.	
5. Adjust leak valve to add hydrogen to nitrogen in PCV at desired rate.	(a) Ensure V4 and V5 are open and all other valves are closed.	
	(b) Adjust leak setting to mark (0000325)	
6. Increase hydrogen addition rate to load getter to desired capacity.	(a) Close leak valve and V5.	
	(b) Ensure V4 is open and all other valves are closed.	
	(c) Open V2.	
	(d) Add H2 to achieve desired pressure in reservoir (P1 and P2). Close VH2.	
	(e) Close V2.	
	(f) Open V5.	
	(g) Adjust leak setting to add H2 to PCV at desired rate.	
	(h) When H2 addition is complete (P2), close V5 and leak valve.	
	(i) Open V2.	
	(j) Open Vvac-1 or VH2 as necessary to achieve desired H2 pressure in reservoir (P1 and P2). Close Vvac-1 and PH2.	
	(k) Close V2.	
	(l) Open Vvac-1 to evacuate H2.	
	(m) Close Vvac-1.	
7. Repeat steps 5 and 6 until test is complete.	See 5 and 6.	
8. Remove getter assembly, replace with fresh getter, and seal PCV in air.	(a) Close all valves.	
	(b) Open Vvac-1 and Vvac-2.	
	(c) Open V2, V4, and V5.	

Key Job Step	Sub-Steps	Check When Complete
	(d) When P1, P2, and P3 read 0 Torr, close all valves.	
	(e) Open Vair and V1 to equilibrate PCV with atmosphere.	
	(f) Close V1 and Vair.	
	(g) Open Vvac-1 to evacuate air from manifold line.	
	(h) When P1 reads 0 Torr, close Vvac-1.	
	(i) Ensure all valves are closed. Remove PCV lid and remove getter assembly.	
	(j) Open V5. Place fresh getter assembly into PCV, replace PCV lid, and tighten to mark to seal.	
9. Adjust leak valve to add hydrogen to air in PCV at desired rate.	(a) Close all valves. Ensure leak is set to 00000.	
	(b) Open V2 and V4.	
	(c) Ensure P1 and P2 read 0 Torr. If necessary, open Vvac-1 to evacuate, then close Vvac-1.	
	(d) Add H2 to desired pressure (P1 and P2) and close PH2.	
	(e) Close V2.	
	(f) Open Vvac-1 to evacuate H2 from manifold line.	
	(g) When P1 reads 0 Torr, close Vvac-1.	
	(h) Ensure V4 and V5 are open and all other valves are closed.	
	(i) Adjust leak setting to mark (0000325)	
10. Take gas samples if necessary.	(a) Close all valves. Ensure leak is set to 00000.	
	(b) Attach gas-tight syringe to appropriate sampling port.	

MATERIAL SAFETY DATA SHEET

VIP HYDROGEN GETTER

Manufacturer:	Vacuum Energy, Inc.
Address:	4440 Warrensville Rd., Suite J Cleveland, Ohio 44128-2837
Telephone:	(216) 586-5459
Fax:	(216) 586-5461
Composition:	Organic Polymers consisting of one or more of the following: (isobutylene-butene co-polymer, poly 1,2-dihydro-2,2,4-trimethylquinoline, polybutadiene, styrene-butadiene copolymer) Palladium and/or Platinum dispersed metals Activated Carbon and Carbon Black
Trade Name:	VIP Hydrogen Getter
Date:	February 1, 2000
General Safety/Handling:	Materials are not dangerous. Can be stored and handled safely in air. Materials can absorb water and other active gases from the air which can later desorb under vacuum. Carbon in the materials can sorb vapors such as organic solvents, and should therefore be stored in an airtight impermeable container indoors, away from chemical storage compartments in a dry, inert gas atmosphere such as nitrogen or argon. Avoid conditions that can generate excessive dust.

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SECTION 1 - INGREDIENT INFORMATION

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Chemical Name:

Activated Carbon and Carbon Black	CAS #: 7440-44-0
Isobutylene-butene copolymer	CAS #: 9003-29-6
Palladium	CAS #: 7440-05-3
Platinum	CAS #: 7440-06-4
Poly 1,2-dihydro-2,2,4-trimethylquinoline	CAS #: 26780-96-1
Polybutadiene	CAS #: 9003-17-2
Styrene-Butadiene copolymer	CAS #: 9003-55-8

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SECTION 2 - PHYSICAL/CHEMICAL CHARACTERISTICS

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Boiling Point:	N/A
Specific Gravity (H_2O = 1):	1.1
Melting Point:	N/A
Vapor Pressure (mm Hg):	Less than 1 torr
Solubility in Water:	Less than 1%
Appearance:	Black solid
Odor:	No Odor.
Physical State:	Solid
Percent Volatiles:	None.

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SECTION 3 - REACTIVITY DATA

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Stability:
Unstable -
Stable - X
Conditions to Avoid - Avoid prolonged temperatures above 250°F

Incompatibility (Materials to Avoid): Avoid contact with strong oxidizing agents

Hazardous Decomposition or Byproducts: Combustion in insufficient oxygen can produce carbon monoxide

Hazardous Polymerization:
May Occur -
Will Not Occur - X
Conditions to Avoid -

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SECTION 4 - FIRE AND EXPLOSION HAZARD DATA

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Flash Point: Solid combustible material. Flash Point >300°F.

NFPA Hazard Classification:
Health: 0
Reactivity: 0
Flammable: 1

Extinguishing Media: CO2, foam, water. If appropriate, isolate fire and allow it to burn out.

Special Fire Fighting Procedures: Cool uninvolved drums with water from hose. Use standard procedures for combustible solids.

Unusual Fire and Explosion Hazards: None.

SECTION 5 - HEALTH HAZARD DATA

Chronic Toxicity:	Not tested for mutagenicity in the Ames test. No other applicable information has been found
Acute Toxicity:	
Oral:	No applicable data have been found
Eye:	Possible mild irritant
Skin:	Possible mild irritant
Absorption:	No applicable data have been found
Inhalation:	No applicable data have been found. Powder can form nuisance dusts. Use fullface respirator mask.
Signs and Symptoms of Overexposure:	
Eye:	Possible mild irritation
Skin:	Possible mild irritation. Possible drying of skin.
Absorption:	None expected
Ingestion:	None expected
Inhalation:	Possible mild irritation if dust is ingested. Use fullface respirator mask.
Emergency and First Aid Procedures:	On contact with skin, wash affected areas with soap and water. Practice proper hygiene, use protective gloves. On contact with eyes, flush thoroughly with water for 15 minutes. Seek medical attention if problems persist.

SECTION 6 - CONTROL MEASURES

Respiratory Protection:	No special precautions for operations that use bulk solid material. If material is sprayed with solvents, use proper source ventilation hoods, and fullface respirator with high efficiency filter media. When handling powder avoid generating dust conditions, and use fullface respirator.
Ventilation:	Local Exhaust.
Personal Protective Equipment:	Gloves, fullface respirator, and safety glasses.

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SECTION 7 - PRECAUTIONS FOR SAFE HANDLING AND USE

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Steps to be Taken in Case Material Is Released or Spilled:	Use gloves and fullface respirator when cleaning up spill. Sweep up spilled material. Place spilled material in container for disposal.
Waste Disposal Method:	Trash.
Precautions to be Taken In Handling and Storage:	Avoid unnecessary skin contact. Use protective gloves and fullface respirator when handling this material. Do not take internally. Be aware of safe handling procedures.
Other Precautions:	None.

NOTE: The data contained in this Material Safety Data Sheet relates only to the specific materials designated herein and does not relate to their use in combination with any other materials or in any process. The information herein is based on technical data that Vacuum Energy, Inc. believes to be reliable. It is intended to be used by persons with technical skill. Prior to use, users shall conduct their own investigations to determine the suitability of the information for their particular purpose, and appropriate warnings and safe handling procedures should be provided to handlers and users. Any use of this data must be determined by the user to be in accordance with Federal, State and local laws and regulations. Since conditions of use and suitability are beyond Vacuum Energy, Inc.'s control, risks of use and suitability are therefore assumed by the user, and Vacuum Energy, Inc. expressly disclaims all warranties including warranties of the merchantability and fitness for a particular purpose, express or implied, in respect to the use or suitability of the materials.

Material Safety Data Sheet

Molecular Sieve 4A

Identity (Trade Name as Used on Label)

Manufacturer	MULTISORB TECHNOLOGIES, INC. (formerly Multiform Desiccants, Inc.)	MSDS Number* :	M49
Address	325 Harlem Road Buffalo, NY 14224	CAS Number* :	
Phone Number (For Information)	716/824-8900	Date Prepared:	October 21, 1997
Emergency Phone Number	716/824-8900	Prepared By* :	G.E. McKedy

Section 1 - Material Identification and Information

Components - Chemical Name & Common Names (Hazardous Components 1% or greater, Carcinogens 0.1% or greater)	%*	OSHA PEL	ACGIH TLV	OTHER LIMITS RECOMMENDED
Sodium Oxide [Na_2O]	<30	N/A	N/A	
Magnesium Oxide [MgO]	<5	15mg/m^3 (fume, total particulate)	10mg/m^3 (Fume)	
Aluminum Oxide [AL_2O_3] (Non-fibrous)	<30	15mg/m^3 (total dust)	10mg/m^3 (total dust)	5mg/m^3 (respirable dust)
Silicon Oxide [SiO_2]	<50	15mg/m^3 (total dust) 5mg/m^3 respirable dust	10mg/m^3 (as Silicon) 3mg/m^3 respirable	5mg/m^3 (respirable dust)
Non-Hazardous Ingredients				
TOTAL	100			

Section 2 - Physical/Chemical Characteristics

Boiling Point	N/A	Specific Gravity (H_2O = 1)	2.1
Vapor Pressure (mm Hg and Temperature	N/A	Melting Point	N/A
Vapor Density (Air = 1)	N/A	Evaporation Rate (__________=1)	N/A
Solubility in Water	N/A	Water Reactive	N/A
Appearance and Odor	Product may appear as a bead, pellet, mesh cake or powder. Odorless.		

Section 3 - Fire and Explosion Hazard Data

Flash Point and Methods Used	Does not burn	Auto-Ignition Temperature	N/A	Flammability Limits in Air % by Volume	N/A	LEL	UEL
Extinguisher Media	Use media appropriate for surrounding fire.						
Special Fire Fighting Procedures	Used material may contain products of a hazardous nature. The user of this product must identify the hazards of the retained material and inform the fire fighters of these hazards.						
Unusual Fire and Explosion Hazards	When exposed to water, molecular sieve can get quite hot. When first wetted it can heat to the boiling point of water. Flooding with water will reduce the temperature to safe limits.						

Section 4 - Reactivity Hazard Data

STABILITY ☐Stable ☐Unstable	Conditions To Avoid	The addition of water without flooding can cause a rise in temperature from the heat of adsorption, and contact with skin might result in burns.
Incompatibility (Materials to Avoid)	Sudden contact with high concentration of chemicals having high heats due to adsorption.	
Hazardous Decomposition Products	does not readily decompose. Adsorbed materials such as hydrocarbons will be more easily given off.	
HAZARDOUS POLYMERIZATION ☐May Occur	Conditions To Avoid	None.

*Optional

☐Will Not Occur

Section 5 - Health Hazard Data

PRIMARY ROUTES OF ENTRY	☐ Inhalation ☐Skin Absorption	☐Ingestion ☐Not Hazardous	CARCINOGEN LISTED IN	☐ NTP ☐IARC Monograph	☐OSHA ☐Not Listed
HEALTH HAZARDS	Acute	May cause eye, skin and mucous membrane irritation.			
	Chronic	Prolonged inhalation may cause lung damage			
Signs and Symptoms of Exposure	Eye skin and mucous membrane irritation.				
Medical Conditions Generally Aggravated by Exposure	Breathing of dust may aggravate asthma and inflammatory of fibrotic pulmonary disease.				

EMERGENCY FIRST AID PROCEDURES - Seek medical assistance for further treatment, observation and support if necessary.	
Eye Contact	Flush eyes with water for 15 minutes and contact physician.
Skin Contact	Wash affected area with soap and water.
Inhalation	Remove to fresh air and get medical assistance if necessary.
Ingestion	If ingested in large quantities, drink (2) glasses of water. Contact physician for permission to induce vomiting. This product gets hot as it adsorbs water. Burns to moist body tissues can result if contact is prolonged. No other evidence of adverse effects from available information.

Section 6 - Control and Protective Measures

Respiratory Protection (Specify Type)	Use NIOSH approved respirator when PEL might be exceeded.		
Protective Gloves	Recommended	Eye Protection	Safety goggles with side shields.
VENTILATION TO BE USED	☐Local Exhaust N/A	☐Mechanical (General) N/A	☐Special N/A
	☐Other (Specify) N/A		
Other Protective Clothing and Equipment	N/A		
Hygienic Work Practices	Avoid raising dust. Avoid contact with skin, eyes and clothing.		

Section 7 - Precautions for Safe Handling and Use/Leak Procedures

Steps to be Taken if Material is Spilled Or Released	Sweep the spill area. Collect and place the spilled material in a waste disposal container. Avoid raising dust.
Waste Disposal Methods	Discard any product, residue, disposal container or liner in an environmentally acceptable manner in full compliance with federal, state and local regulations.
Precautions to be Taken In Handling and Storage	Avoid breathing dust. Use adequate ventilation. Wash after handling.
Other Precautions and/or Special Hazards	Use good personal hygiene