TREATMENT TANK OFF-GAS TESTING FOR
THE ENHANCED CHEMICAL CLEANING
PROCESS

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ACRONYMS

ASTM - American Society of Testing and Materials
ECC – Enhanced Chemical Cleaning
GC – Gas Chromatograph
LPR – Linear Polarization Resistance
M&TE – Materials and Testing Equipment
UNS – Unified Numbering System
1.0 Executive Summary

The purpose of this activity was to provide a bounding estimate of the volume of hydrogen gas generated during Enhanced Chemical Cleaning (ECC) of residual sludge remaining in a Type I or Type II treatment tank as well as to provide results independent of the sludge volume in the waste tank to be cleaned. Previous testing to support Chemical Cleaning was based on a 20:1 oxalic acid to sludge ratio [1]. Hydrogen gas evolution is the primary safety concern.

Sealed vessel coupon tests were performed to estimate the hydrogen generation rate due to corrosion of carbon steel by 2.5 wt.% oxalic acid. These tests determined the maximum instantaneous hydrogen generation rate, the rate at which the generation rate decays, and the total hydrogen generated. These values were quantified based on a small scale methodology similar to the one described in WSRC-STI-2007-00209, Rev. 0 [1]. The measured rates support identified Safety Class functions [2].

The tests were performed with ASTM A285 Grade C carbon steel coupons. Bounding conditions were determined for the solution environment. The oxalic acid concentration was 2.5 wt.% and the test temperature was 75 °C. The test solution was agitated and contained no sludge simulant. Duplicate tests were performed and showed excellent reproducibility for the hydrogen generation rate and total hydrogen generated. The results showed that the hydrogen generation rate was initially high, but decayed rapidly within a couple of days.

A statistical model was developed to predict the instantaneous hydrogen generation rate as a function of exposure time by combining both sets of data. An upper bound on the maximum hydrogen generation rate was determined from the upper 95% confidence limit. The upper bound confidence limit for the hydrogen generation rate is represented by the following equation.

\[ \ln (G_v) = -8.22 - 0.0584 \cdot t + 0.0002 \cdot t^2 \]

This equation should be utilized to estimate the instantaneous hydrogen generation rate per unit surface area, \( G_v \), at a given time, \( t \). The units for \( G_v \) and \( t \) are \( \text{ft}^3/\text{ft}^2/\text{min} \) and hours, respectively.

The total volume of hydrogen gas generated during the test was calculated from the model equation. An upper bound on the total gas generated was determined from the upper 95% confidence limit. The upper bound limit on the total hydrogen generated during the 163 hour test was 0.332 \( \text{ft}^3/\text{ft}^2 \).
The maximum instantaneous hydrogen generation rate for this scenario is greater than that previously measured in the 8 wt.% oxalic acid tests [1] due to both the absence of sludge in the test (i.e., greater than 20:1 ratio of acid to sludge) and the use of polished coupons (vs. mill scale coupons). However, due to passivation of the carbon steel surface, the corrosion rate decays by an order of magnitude within the first three days of exposure such that the instantaneous hydrogen generation rates are less than that previously measure in the 8 wt.% oxalic acid tests. While the results of these tests are bounding, the conditions used in this study may not be representative of the ECC flowsheet, and the applicability of these results to the flowsheet should be evaluated for the following reasons:

- The absence of sludge results in higher instantaneous hydrogen generation rates than when the sludge is present.
- Polished coupons do not represent the condition of the carbon steel interior of the tank, which are covered with mill scale. Based on lower instantaneous corrosion rates measured on mill scale coupons exposed to oxalic acid, lower instantaneous hydrogen generation rates are expected for the tank interior than measured on the polished coupons.

Corrosion rates were determined from the coupon tests and also calculated from the measured hydrogen generation rates. Excellent agreement was achieved between the time averaged corrosion rate calculated from the hydrogen generation rates and the corrosion rates determined from the coupon tests. The corrosion rates were on the order of 18 to 28 mpy. Good agreement was also observed between the maximum instantaneous corrosion rate as calculated from the hydrogen generation rate and the corrosion rate determined by previous electrochemical tests [3].

2.0 Background

As a part of chemical cleaning, oxalic acid is added to the treatment tank to dissolve and break up the residual sludge heel that remains after bulk sludge removal is complete [1]. However, the acid also corrodes the carbon steel tank wall and cooling coils. If sludge has dissolved into the oxalic acid, little or no hydrogen evolution is anticipated due to corrosion of the carbon steel or other chemical reactions. Various corrosion tests, including those at 1 wt%, 2.5 wt.% and 8 wt% [3, 4], show that when the sludge simulant is present, the electrochemical potential shifts toward more oxidizing values and therefore reduces the likelihood of hydrogen generation.

On the other hand, if the oxalic acid has little interaction with the sludge, hydrogen gas, could conceivably evolve at cathodic areas due to the corrosion of the carbon steel. Scenarios where hydrogen evolution could occur during ECC include the initial filling of the tank prior to agitation and near the end of the process when there is little or no sludge present. The purpose of this activity was to provide an estimate of the volume of gas generated during ECC of the residual sludge remaining in a Type I or Type II treatment tank.
A test condition that was expected to result in the highest instantaneous corrosion rate was selected. An upper bound oxalic acid concentration of 2.5 wt.% was utilized for the tests. The oxalic acid concentration of the ECC process will nominally be 2 wt.%. No sludge was present in the test to address the scenarios where the tank is essentially empty and oxalic acid is added back either inadvertently or for a final rinse. Hydrogen evolution may occur at these acidic, reducing conditions.

Previous electrochemical tests indicated that the highest corrosion rates in 2.5 wt.% oxalic acid were observed at 75 °C in an agitated solution (i.e., approximately 300 mpy) [3]. However, corrosion rates measured on 30 day coupon tests at the same test conditions indicated that the corrosion rate is on the order of 20 mpy [3]. This result indicates that after an initially high corrosion rate, the surface is passivated and the corrosion rates decrease dramatically. Based on this observation, the hydrogen generation rates measured initially will be quite high, and will decay significantly as the corrosion rate decreased.

The tests reported in this document determined the maximum instantaneous hydrogen generation rate, the rate at which the generation rate decays, and the total hydrogen generated. The generation rates for hydrogen and total gas were quantified based on a small scale methodology similar to the one described in WSRC-STI-2007-00209, Rev. 0 [1]. The measured rates support identified Safety Class functions [2]. Quality assurance measures for this testing were identified in the task technical and quality assurance plan [5].

3.0 Experimental

3.1 Test Material

The material tested was ASTM A285, Grade C carbon steel (UNS K02200). This material has similar chemical and physical properties as the Type I and II waste storage tanks that will be the focus of the initial chemical cleaning operations. The chemical composition and the mechanical properties (see Tables 1 and 2) of the as-received coupons were vendor certified. The dimensions of each coupon were measured with digital calipers to the nearest 0.025 mm (or 0.001 inches). The coupons were weighed on an analytical balance to the nearest 0.0001 grams. Table 3 shows the dimensions, surface area, and weight of the two coupons that were tested.

The initial surface condition of the coupons was a 600 grit polished finish. The polished coupons provide a uniform, reproducible surface finish ideal for studying reactions between the steel and the environment.
Table 1. Chemical Composition (Wt %) of A285 Grade C, Carbon Steel

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr</th>
<th>Si</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.18</td>
<td>0.75</td>
<td>0.011</td>
<td>0.008</td>
<td>0.03</td>
<td>0.03</td>
<td>0.06</td>
<td>-</td>
<td>balance</td>
</tr>
</tbody>
</table>

Table 2. Mechanical Properties of A285 Grade C, Carbon Steel

<table>
<thead>
<tr>
<th>Property</th>
<th>Value (ksi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength</td>
<td>48</td>
</tr>
<tr>
<td>Tensile Strength</td>
<td>67</td>
</tr>
<tr>
<td>% Elongation</td>
<td>31 (2 inch)</td>
</tr>
</tbody>
</table>

Table 3. Initial Dimensions, Surface Areas and Weights of Corrosion Coupons

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Length (cm)</th>
<th>Width (cm)</th>
<th>Thickness (cm)</th>
<th>Surface Area (cm²)a</th>
<th>Initial Weight (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>2.534</td>
<td>2.178</td>
<td>0.587</td>
<td>16.212</td>
<td>23.778</td>
</tr>
<tr>
<td>9</td>
<td>2.224</td>
<td>2.227</td>
<td>0.614</td>
<td>15.019</td>
<td>22.488</td>
</tr>
</tbody>
</table>

a – Surface area includes correction for the hole in coupon which was 0.476 cm in diameter.

3.2 Test Solution

The 2.5 wt.% oxalic acid solution was prepared with reagent grade C₂H₂O₄•2H₂O. The solution was prepared by adding 35 g of the reagent to 1 liter of distilled water. The solution temperature during the test was 75 ± 5 ºC.

3.3 Test Set-up

The tests were performed in the stainless steel container shown in Figure 1. Two of these vessels were used for the tests. A copper gasket was utilized to seal the test vessel. Nominally the vessel had a three inch interior diameter and a two inch internal height. Each vessel was equipped with a 100 psig rupture disk as shown in Figure 1a.

The set up of the coupon test is shown in Figure 1c. A glass insert was utilized to contain the oxalic acid and the coupon. The insert was equipped with a glass hanger for the coupon. A small sheet of Teflon™ was also hung to shield the stirring bar from the carbon steel coupon. Agitation was achieved with the magnetic stirring bar on a stirring hot plate. The volume of oxalic acid and the vapor space volume is shown in Table 4. The vapor space volume was determined from the vapor space in the vessel, the volume of the tubing, and the volume within the pressure transducer that was utilized for the test.
Table 4. Liquid and Vapor Space Volumes that Were Utilized for the Tests.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Liquid Volume (ml)</th>
<th>Vapor Volume</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>93</td>
<td>143</td>
</tr>
<tr>
<td>9</td>
<td>93</td>
<td>137</td>
</tr>
</tbody>
</table>

The complete system is shown in Figure 2. The test vessels were placed in a bed of sand on a hot plate with stirring capability. The sand was utilized to moderate the temperature of the vessel during the test. Type E thermocouples were used to monitor the container temperature. The pressure in each container was measured with a Rosemount™ Model 1151DP pressure transducer (see Figure 3). The transducers were calibrated with a range of 0 to 150 inches of water (0 to 5.4 psig). The temperature and pressure were monitored with LabVIEW™ 7.1 (National Instruments) software.

Figure 1. Sealed vessel utilized for off-gas tests (a) vessel with rupture disk assembly, (b) interior of vessel, and (c) vessel with coupon and solution.
Figure 2. System utilized to perform off-gas tests.

Figure 3. Transducers used to monitor pressure.
Samples were obtained manually during the test. The stainless steel tubing included a port whereby samples could be withdrawn from the closed system periodically via a syringe. Sufficient sample was withdrawn to perform duplicate analyses with a gas chromatograph (GC). A MTI Model M200 Micro GC™ gas chromatograph was used to analyze for hydrogen. EZChrom™ version 4.5 software, developed by Agilent, operated and provided output from the GC. Prior to testing, the GC system was checked with prepared calibration gases.

3.4 Test Procedure

This procedure was developed to obtain an “instantaneous” hydrogen generation rate. A volume of gas was captured in the system over a given time interval. The system was then sampled to determine the concentration of hydrogen present. The system was then purged and the process was initiated again to determine another instantaneous hydrogen generation rate. The following steps outline this process and were taken once the system had achieved equilibrium at 75 ºC.

1) Isolate the system by closing all valves, except those between the test vessel and the pressure transducer.
2) Monitor the pressure and temperature for time intervals between 2 to 20 hours (see Figure 4). If the pressure exceeded 150 inches of water, a sample would be obtained and the system purged.
3) Use a syringe to obtain a gas sample from the port. Analyze the sample in the GC for hydrogen.
4) Vent the system to the atmosphere by opening a valve on the manifold.
5) Isolate test vessel from the system. Evacuate the stainless steel tubing to remove residual hydrogen from the system.
6) Vent the test vessel and the system to the atmosphere by opening a valve on the manifold.
7) Repeat steps 5 and 6 twice.
8) Return to step 1 to gather the next data point.

3.5 Post-Test Characterization of Coupons

At the completion of the test, the coupons were removed from the test vessel for visual examination. During this examination, the form of corrosion on each coupon was identified (e.g., general) and differences in the corrosion products were noted. Photographs were taken to document these results. ASTM standard practices were followed to determine the general corrosion rate [6]. The corrosion products were removed from the sample by a two step process. First, loose corrosion products were removed using a wire brush. The coupons were then immersed in Clarke’s solution (i.e., an inhibited HCl acid solution) to remove the final corrosion products. After removal of the corrosion products, the coupons were weighed on an M&TE calibrated balance to determine the resultant weight loss.
The corrosion rate, in mils per year (mpy), is related to the weight loss of the coupon by the following equation:

\[ \text{Corrosion Rate} = \frac{3.45 \times 10^6}{A \times T \times \rho} w \]

where \( w \) is the weight loss in grams, \( A \) is the area in cm\(^2\), \( T \) is the exposure time in hours, and \( \rho \) is the density in g/cm\(^3\).

Figure 4. Example of pressure and temperature transient measured during the test. The dashed line is the temperature and is shown on the y-axis on the right. The solid line with the trend line is the pressure transient and is shown on the y-axis on the left. Data was obtained between approximately 8:00 pm on June 14, 2011 through 8:00 am on June 15, 2011.

3.6 Data Analysis

Direct and indirect methods were utilized to measure the hydrogen generation rate. The direct method involved obtaining a measurement of the hydrogen concentration via the GC. Two gas samples were obtained from each test and the results for each time interval are reported in the Appendix. The hydrogen generation rate may be calculated given the
concentration, the volume of the system, and the time interval over which the hydrogen had accumulated in the system.

The indirect method involved the pressure measurements that were made during the test. The primary purpose of the pressure measurements was to ensure that the system did not over-pressurize. Secondarily, the pressure is related to the volume of gas generated via the ideal gas law. The pressure, $P$, in the vessel is expressed as:

$$ P = \frac{n R T}{V} \quad \text{Equation 2} $$

where $n$ is the number of moles, $R$ is the gas constant (of 0.0821 mole-l/atm-K), $T$ is the temperature in degrees K, and $V$ is the volume in liters of the vessel, tubing and pressure transducer. For this test, $T$ and $V$ are constant and therefore the first derivative of Equation 2 with respect to time is:

$$ \frac{dP}{dt} = \frac{RT}{V} \cdot \frac{dn}{dt} \quad \text{Equation 3} $$

In these tests, the only assumed change in the number of moles is due to the generation of hydrogen gas. Re-arranging Equation 3 gives the following equation for the molar hydrogen generation rate.

$$ \frac{dn}{dt} = \frac{V}{RT} \frac{dP}{dt} \quad \text{equation 4} $$

The pressure as a function of time was recorded in an EXCEL™ spreadsheet by the LabView software. The response was linear as a function of time in each vessel for nearly all the tests. The EXCEL™ program was used to determine the slope of the line, or $dP/dt$.

**4.0 Results and Discussion**

Figure 5 shows the instantaneous hydrogen generation rate per unit area measured for the duplicate tests. The maximum generation rate occurred between 2.5 to 5 hours after the beginning of the test for both samples. The maximum generation rate ranged between $7.1 \times 10^{-5}$ to $8.6 \times 10^{-5}$ ft$^3$/ft$^2$/min for samples 9 and 7, respectively. Within 48 hours after the test began, the hydrogen generation rate had decayed by an order of magnitude from the maximum value. At the end of the week the rate had decreased even further to approximately one to two orders of magnitude less than the initial maximum rate.
As mentioned above, two methods were utilized to measure the volume of hydrogen generated during the tests. Figure 6 shows a comparison of the direct, by volume change, and the indirect, by pressure change, methods utilizing the data from the test with sample 9. The first observation is that fewer data points were gathered with the indirect method. The changes in pressure measured were very small, typically less than 10 inches of water (i.e., approximately 0.3 psig). As a result, changes in the ambient conditions are believed to have influenced the pressure gauge readings at these low values. Thus, the pressure gauge was not sensitive enough in these tests to provide sufficient resolution at these low pressures. In previous tests, when other gases such as carbon dioxide were evolved, these gauges performed well [2]. Nevertheless, the hydrogen generation rate calculated from this method does indicate the same decaying trend with time as the rates measured by the direct method.
These results indicate that the hydrogen generation rates from samples 7 and 9 were virtually identical (see Figure 5). A statistical analysis was performed to evaluate the two data sets and determine an equation that would predict the maximum generation rate and the rate at which the generation rate decays. Confidence intervals (95% level) were also determined to assess the uncertainty in the hydrogen generation rates.

To perform the analysis a log transformation of the hydrogen generation rates was performed. (Note: For this analysis the first data point obtained after 2.5 hours of testing was neglected. The generation rate was low during this time due to a number of factors such as a transient temperature and the time necessary to establish an equilibrium between the sample and the solution.) The data were then input into the JMP™ statistical package to determine the best fit regression model and the confidence interval (see Figure 7). The best fit model as shown by the middle curve was a quadratic expression.

\[
\ln (G_v) = -9.65 - 0.0361 \, t + 0.00011 \, (t - 68.5)^2
\]

where \( G_v \) is the volumetric hydrogen generation rate per unit area in \( \text{ft}^3/\text{ft}^2/\text{minute} \) and \( t \) is the time in hours. The \( R^2 \) for this model was 0.96. The model predicts a high initial hydrogen generation rate followed by a rapid decay to a low constant generation rate that is one to two orders of magnitude less than the initial generation rate. Given that the maximum generation rate was observed five hours after the initiation of the test, the equation would predict a hydrogen generation rate of \( 8.38 \times 10^{-5} \, \text{ft}^3/\text{ft}^2/\text{minute} \). After two
and five days the hydrogen generation rate decreased to $1.2 \times 10^{-5}$ and $1.1 \times 10^{-6}$ ft$^3$/ft$^2$/minute, respectively.

The upper and lower 95% confidence intervals are shown around the polynomial fit. An upper bound on the maximum hydrogen generation rate was determined from the upper 95% confidence limit. The curve that represents the 95% upper confidences limit may be fit with a spline technique. The equation for this line is:

$$\ln (G_v) = -8.22 - 0.0584 t + 0.0002 t^2$$  \hspace{1cm} \text{Equation 6}

The upper bound limit on the maximum instantaneous generation rate at 5 hours was $2.03 \times 10^{-4}$ ft$^3$/ft$^2$/minute. After two and five days the upper bound limit decayed to $2.6 \times 10^{-5}$ and $4.4 \times 10^{-6}$ ft$^3$/ft$^2$/minute, respectively.

![Figure 7](image-url)

**Figure 7.** Model for prediction of the volumetric hydrogen generation rate as a function of time. Red squares represent the test data from sample 7 and blue crosses represent the test data from sample 9.

The total volume of hydrogen gas generated during the test may be calculated by integrating the model equation with respect to time. The total hydrogen generated as a function of time is shown in Table 5. Approximately 86% of the total gas that was generated during the 1 week interval was generated during the first two days of the test. The upper bound for the total gas generated was determined by integrating beneath the curve for the upper 95% confidence interval. As shown in Table 5, the upper bound limit is approximately 2.4 times greater than that predicted by the model.
Table 5. Total Volume of Hydrogen Gas Per Unit Area Generated During the Test

<table>
<thead>
<tr>
<th>Time (hours)</th>
<th>Total Volume of Hydrogen from Model Equation (ft³/ft²)</th>
<th>Total Volume of Hydrogen Upper 95% Confidence Interval (ft³/ft²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>0.029</td>
<td>0.071</td>
</tr>
<tr>
<td>48</td>
<td>0.121</td>
<td>0.279</td>
</tr>
<tr>
<td>120</td>
<td>0.139</td>
<td>0.323</td>
</tr>
<tr>
<td>160</td>
<td>0.140</td>
<td>0.332</td>
</tr>
</tbody>
</table>

The moles of hydrogen generated are related to the volume of hydrogen generated by the ideal gas law. Furthermore, in the case of iron in an acidic reducing environment, the number of moles of hydrogen generated is equal to the number of moles of iron corroded [7]. A relationship between the hydrogen generation rate and the corrosion rate was previously derived [8]:

\[
\text{Corrosion Rate} = \frac{G_r}{S_A} \left(\frac{1}{3.8 \times 10^{-5}}\right) \quad \text{Equation 7}
\]

where \(G_r\) is the hydrogen generation rate in moles/hr, \(S_A\) is the surface area in ft², and the corrosion rate is in mpy. The instantaneous corrosion rate as a function of time was calculated based on the instantaneous hydrogen generation rates and the surface area of each sample. Figure 8 shows that the maximum corrosion rate also occurred between 2.5 and 5 hours after the initiation of the test and ranged between 135 and 162 mpy for samples 9 and 7, respectively. The corrosion rates also decay by an order of magnitude after 48 hours. This decay in the corrosion rate clearly indicates that passivation occurs at this relatively high temperature.

These corrosion rates were approximately a factor of 2 less than the corrosion rates measured previously by the linear polarization resistance (LPR) technique [3]. Duplicate LPR tests on carbon steel samples resulted in corrosion rates of 264 and 312 mpy. However, the LPR test was conducted approximately 2 hours after the sample was exposed and lasted approximately 10 minutes. On the other hand, these corrosion rates were calculated based on measurements that were taken over a 2.5 hour time period. Thus, given the decay of the corrosion rate with time the lower corrosion rate obtained for the longer test time interval is not surprising that the initial instantaneous corrosion rate is lower for the hydrogen generation tests than the LPR tests.
Similar to the hydrogen generation rate, both sets of corrosion rate data may be combined again. The JMP™ statistical analysis package was utilized to determine the best fit line through the corrosion rate data. Figure 9 shows that a quadratic equation fits both sets of data quite well. The equation for the corrosion rate is:

\[
\ln \text{(Corrosion Rate)} = 4.82 - 0.0367 \cdot t + 0.00012 \cdot (t-68.5)^2
\]  

Equation 8

where the corrosion rate is in mpy and \( t \) is the time in hours. The \( R^2 \) for this model was 0.96. The model predicts a high initial corrosion rate followed by a rapid decay to a low constant generation rate that is one to two orders of magnitude less than the initial generation rate. Given that the highest corrosion rate was observed five hours after the initiation of the test, the equation would predict a maximum corrosion rate of 167 mpy. After two and five days the corrosion rate decreased to 22.4 and 2 mpy, respectively.

The upper and lower 95% confidence intervals are shown around the polynomial fit. An upper bound on the maximum hydrogen generation rate was determined from the upper 95% confidence limit. The upper bound limit on the maximum instantaneous corrosion rate at 5 hours was 353 mpy. Thus, the corrosion rate estimated by the LPR test was within this confidence interval. After two and five days the upper bound limit has decayed to 45 and 3 mpy, respectively.
The time averaged corrosion rate was calculated by integrating Equation 8 over the time interval of the test, approximately 160 hours, and then dividing by the time interval. The time averaged corrosion rate as a function of time is shown in Table 4. In addition, the 95% upper and lower confidence interval on the corrosion rate is shown. The time averaged corrosion rate after 160 hours may be compared to the corrosion rates measured from weight loss measurements on the samples used for these studies.

Table 6. Time averaged corrosion rate at various times during the test. Upper and lower 95% confidence intervals are also shown.

<table>
<thead>
<tr>
<th>Time (hours)</th>
<th>Time Averaged Corrosion Rate from Model Equation (mpy)</th>
<th>Time Averaged Corrosion Rate Upper 95% Confidence Interval (mpy)</th>
<th>Time Averaged Corrosion Rate Lower 95% Confidence Interval (mpy)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>191</td>
<td>404</td>
<td>104</td>
</tr>
<tr>
<td>48</td>
<td>82.3</td>
<td>172</td>
<td>43.8</td>
</tr>
<tr>
<td>120</td>
<td>37.6</td>
<td>77.4</td>
<td>19.6</td>
</tr>
<tr>
<td>160</td>
<td>28.5</td>
<td>58.5</td>
<td>14.9</td>
</tr>
</tbody>
</table>

The coupons that were tested during these off-gas studies are shown in Figure 10. Both coupons had a dark ferrous oxalate film on the surface similar to that observed in previous coupon tests [3]. The yellow line of ferrous oxalate precipitate on the coupon
could be indicative of a stagnant liquid level. However, the dark ferrous oxalate film was also apparent above this line suggesting that during the test that the whole sample had been exposed. The yellow line may have formed after the test, when the solution was no longer being agitated and while the solution cooled down prior to opening the vessel.

Figure 10. Photographs of post-test coupons (a) sample 7 and (b) sample 9.

The general corrosion rate was calculated based on the weight loss of the sample as shown by Equation 1. The weight loss, exposure time, and general corrosion rates for each sample are shown in Table 7. The general corrosion rates were 17 and 18.5 mpy for samples 9 and 7, respectively. These rates are in good agreement with the 30 day coupon tests that were performed previously [3] and are within the 95% confidence interval for the corrosion rates calculated from the hydrogen generation rates (see Table 6). The slight differences observed are likely due to the error in the assumed model fit for the corrosion rates that were calculated from the hydrogen generation rates.

Table 7. Weight Loss, Exposure Time and General Corrosion Rates for each Sample.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Weight Loss (g)</th>
<th>Exposure Time (hours)</th>
<th>Corrosion Rate (mils/yr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>0.1116</td>
<td>163.25</td>
<td>18.5</td>
</tr>
<tr>
<td>9</td>
<td>0.0947</td>
<td>163.25</td>
<td>17</td>
</tr>
</tbody>
</table>

5.0 Conclusions

Sealed vessel coupon tests were performed to estimate the hydrogen generation rate due to corrosion of carbon steel by 2.5 wt.% oxalic acid. These tests determined the maximum instantaneous hydrogen generation rate, the rate at which the generation rate decays, and the total hydrogen generated. These values were quantified based on a small scale methodology similar to the one described in WSRC-STI-2007-00209, Rev. 0 [1]. The measured rates support identified Safety Class functions [2].
The tests were performed with ASTM A285 Grade C carbon steel coupons. Bounding conditions were determined for the solution environment. The oxalic acid concentration was 2.5 wt.% and the test temperature was 75 °C. The test solution was agitated and contained no sludge simulant. Duplicate tests were performed and showed excellent reproducibility for the hydrogen generation rate and total hydrogen generated. The results showed that the hydrogen generation rate was initially high, but decayed rapidly within a couple of days.

A statistical model was developed to predict the instantaneous hydrogen generation rate as a function of exposure time by combining both sets of data. An upper bound on the maximum hydrogen generation rate was determined from the upper 95% confidence limit. The upper bound confidence limit for the hydrogen generation rate is represented by the following equation.

\[ \ln (G_v) = -8.22 - 0.0584 t + 0.0002 t^2 \]

This equation should be utilized to estimate the instantaneous hydrogen generation rate per unit surface area, \( G_v \), at a given time, \( t \). The units for \( G_v \) and \( t \) are ft\(^3\)/ft\(^2\)/min and hours, respectively.

The total volume of hydrogen gas generated during the test was calculated from the model equation. An upper bound on the total gas generated was determined from the upper 95% confidence limit. The upper bound limit on the total hydrogen generated during the 163 hour test was 0.332 ft\(^3\)/ft\(^2\).

The maximum instantaneous hydrogen generation rate for this scenario is greater than that previously measured in the 8 wt.% oxalic acid tests [1] due to both the absence of sludge in the test (i.e., greater than 20:1 ratio of acid to sludge) and the use of polished coupons (vs. mill scale coupons). However, due to passivation of the carbon steel surface, the corrosion rate decays by more than two orders of magnitude within the first three days of exposure such that the instantaneous hydrogen generation rates are less than that previously measure in the 8 wt.% oxalic acid tests. While the results of these tests are bounding, the conditions used in this study may not be representative of the ECC flowsheet, and the applicability of these results to the flowsheet should be evaluated for the following reasons:

- The absence of sludge results in higher instantaneous hydrogen generation rates than when the sludge is present.
- Polished coupons do not represent the condition of the carbon steel interior of the tank, which are covered with mill scale. Based on lower instantaneous corrosion rates measured on mill scale coupons exposed to oxalic acid, lower instantaneous hydrogen generation rates are expected for the tank interior than measured on the polished coupons.

Corrosion rates were determined from the coupon tests and also calculated from the measured hydrogen generation rates. Excellent agreement was achieved between the
time averaged corrosion rate calculated from the hydrogen generation rates and the corrosion rates determined from the coupon tests. The corrosion rates were on the order of 18 to 28 mpy. Good agreement was also observed between the maximum instantaneous corrosion rate as calculated from the hydrogen generation rate and the corrosion rate determined by previous electrochemical tests [3].

6.0 Records and Quality Assurance

All records for the tests were maintained in laboratory notebook SRNL-NB-2011-00054. M&TE equipment that were utilized are recorded in the notebook. The equipment includes: thermocouples, pressure transducers, balance, and digital caliper. The mill certificate for the carbon steel samples is also shown here. Copies of the results of the GC analysis for samples 7 and 9 are shown in the appendix of this report.

7.0 Acknowledgements

The author recognizes the invaluable assistance provided by G.D. Creech in the set-up and performance of these tests. Special thanks also to H. T. Sessions for providing technical assistance with the gas chromatograph and the laboratory facility in the Center for Hydrogen Research. T. K. Williamson and L. T. Smith also provided necessary assistance by preparing the test vessel and solutions. K. R. Hicks and K. J. Kalbaugh were responsible for preparation and cleaning of the carbon steel coupons. The author also recognizes the contributions of Dr. J. I. Mickalonis, who provided insights into the test results, and Dr. S. P. Harris, who provided the statistical analysis of the data.

8.0 References

GC Data for Sample 7
External Standard Report

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<th>Max</th>
<th>Mean</th>
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<tbody>
<tr>
<td>Hydrogen</td>
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Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
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<tr>
<td>Hydrogen</td>
<td>0.098</td>
<td>%</td>
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Sample 7-1a
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Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
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Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 48 seconds

7-1a
### External Standard Report

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**Column Temperature:** 65 C  
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Sample 7-2b  
Low
External Standard Report

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<th>%SD</th>
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</thead>
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Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
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Inject Time: 10 milliseconds
Run Time: 45 seconds
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Instrument Gain: LOW  
Sample Time:  2 seconds  
Inject Time: 10 milliseconds  
Run Time:  45 seconds
External Standard Report

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Carrier Gas: He
Column Head Pressure: 23.8 psi
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Instrument Gain: LOW
Sample Time: 2 seconds
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Sample 7-3a
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Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Injection Time: 10 milliseconds
Run Time: 45 seconds

Page A-12
External Standard Report

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Carrier Gas: He
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Column Temperature: 65 C
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Sample 9-4
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Column Temperature: 65 C
Instrument Gain: LOW
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Inject Time: 10 milliseconds
Run Time: 45 seconds
## External Standard Report

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Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
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Channel: A
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Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
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Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
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<tbody>
<tr>
<td>Hydrogen</td>
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File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 16:32:42
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Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 4-5
7-5
6/14/11
Channel: A
Current Time: Jun 14, 2011  16:33:55
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011  16:32:42
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Column Type: MS-5A 4m
Carrier Gas: He
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Column Temperature: 65 C
Instrument Gain: LOW
Sample Time:  2 seconds
Inject Time:  10 milliseconds
Run Time:  45 seconds
External Standard Report

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Data file creation time: Jun 14, 2011 16:35:54
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Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample

7.5x

6/14
Channel: A
Current Time: Jun 14, 2011 16:37:01
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Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

7-5a
External Standard Report

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<tr>
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Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-6
Channel: A
Current Time: Jun 14, 2011 19:52:52
Method: c:\mti\ezchrom\200\methods\bk51.
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Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
**External Standard Report**

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Run Time: 45 seconds
# External Standard Report

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Sample 7-7

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Column Type: MS-5A 4m  
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Instrument Gain: LOW  
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Inject Time: 10 milliseconds  
Run Time: 45 seconds
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Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

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</thead>
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<tr>
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<td>µl</td>
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<td>10.203</td>
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Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds

Sample 7-7a
Current Time: Jun 15, 2011 08:02:42
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 08:01:41
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
### External Standard Report

<table>
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<tr>
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<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
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<tbody>
<tr>
<td>Hydrogen</td>
<td>0.147</td>
<td>%</td>
<td>5.700</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.514</td>
<td>189.971</td>
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**Channel:** A  
**Current Time:** Jun 15, 2011 13:36:57  
**Method:** c:\mti\ezchrom\200\methods\bh51  
**File:** c:\mti\ezchrom\200\chrom\01090815.1  
**Data file creation time:** Jun 15, 2011 13:35:55  
**Instrument ID:** 180122  
**Column Type:** MS-5A 4m  
**Carrier Gas:** He  
**Column Head Pressure:** 23.8 psi  
**Column Temperature:** 65 C  
**Instrument Gain:** LOW  
**Sample Time:** 2 seconds  
**Inject Time:** 10 milliseconds  
**Run Time:** 45 seconds
Channel: A
Method: c:\mti\ezchrom\200\methods\bh51
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 13:35:55
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-8
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<tr>
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<td>0.147</td>
<td>%</td>
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<td>-1.000</td>
<td>10.203</td>
<td>0.510</td>
<td>190.793</td>
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</tbody>
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Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 13:38:43
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Sample 1-8a

Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 13:38:43
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time:  2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

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<tbody>
<tr>
<td>Hydrogen</td>
<td>0.063</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.495</td>
<td>194.045</td>
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</tbody>
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Channel: A
Current Time: Jun 15, 2011 17:09:52
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 17:09:47
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Channel: A
Current Time: Jun 15, 2011 17:09:54
Method: c:\mti\ezchrom\200\methods\bh51
File: c:\mti\ezchrom\200\chrom\0109015.1
Data file creation time: Jun 15, 2011 17:08:47
Instrument ID: 190122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
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<tbody>
<tr>
<td>Hydrogen</td>
<td>0.062</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.491</td>
<td>195.016</td>
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</table>

Sample 7-9a

Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 17:12:07
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Channel: A
Current Time: Jun 15, 2011 17:13:17
Method: C:\mti\ezchrom\200\methods\bh51.
File: C:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 17:12:07
Instrument ID: 120122
Column Type: MS-SA 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Internal Standard Report

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</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.033</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.479</td>
<td>197.907</td>
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</table>

:Channel: A
:Current Time: Jun 15, 2011 20:29:45
:Method: C:\mti\ezchrom\200\methods\bh51.
:File: C:\mti\ezchrom\200\chrom\01090815.1
:Data file creation time: Jun 15, 2011 20:27:40
:Instrument ID: 180122
:Column Type: MS-5A 4m
:Carrier Gas: He
:Column Head Pressure: 23.8 psi
:Column Temperature: 65 C
:Instrument Gain: LOW
:Sample Time: 2 seconds
:Inject Time: 10 milliseconds
:Run Time: 45 seconds

Sample 7-10
Channel: A
Current Time: Jun 15, 2011 20:29:52
Method: c:\mti\ezchrom\200\methods\bh51.m
Data file creation time: Jun 15, 2011 20:27:40
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

7-10
External Standard Report

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</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.032</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.475</td>
<td>198.918</td>
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</table>

Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 20:42:25
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-10a
Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 20:42:25
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Page A-40
## External Standard Report

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</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.168</td>
<td>%</td>
<td>5.710</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.464</td>
<td>201.565</td>
</tr>
</tbody>
</table>

Sample: 7-11

**Channel A**

- **Current Time:** Jun 16, 2011 08:08:23
- **Method:** c:\mti\ezchrom\200\methods\bh51.m
- **Data file creation time:** Jun 16, 2011 08:07:02
- **Instrument ID:** 180122
- **Column Type:** MS-5A 4m
- **Carrier Gas:** He
- **Column Head Pressure:** 23.8 psi
- **Column Temperature:** 65 C
- **Instrument Gain:** LOW
- **Sample Time:** 2 seconds
- **Inject Time:** 10 milliseconds
- **Run Time:** 45 seconds
External Standard Report

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</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.169</td>
<td>%</td>
<td>5.710</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.460</td>
<td>202.249</td>
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Sample 7-11a

Channel: A
Current Time: Jun 16, 2011 08:11:11
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 08:10:11
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Channel: A
Current Time: Jun 16, 2011 08:11:12
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 08:10:11
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

$\theta = \alpha$
### External Standard Report

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<tr>
<td>Hydrogen</td>
<td>0.031</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.450</td>
<td>204.947</td>
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**Sample 7-12**
Channel: A
Current Time: Jun 16, 2011 11:35:53
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 11:34:21
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
<table>
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<th>Mean</th>
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</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.031</td>
<td>%</td>
<td>5.68</td>
<td>-1.00</td>
<td>10.20</td>
<td>0.446</td>
<td>205.91</td>
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Sample 7-12a

Detector Sensitivity

Medium
Channel: A
Current Time: Jun 16, 2011 11:38:36
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 11:37:37
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
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<th>Mean</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.054</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.436</td>
<td>208.693</td>
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</table>

Channel: A
Current Time: Jun 16, 2011 16:59:50
Method: \c:\ti\ezchrom\200\methods\bh51.m
File: \c:\ti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 16:58:30
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-13
Channel: A
Current Time: Jun 16, 2011 16:59:52
Method: c:\mti\ezchrom\200\methods\bh51
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 16:58:30
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Page A-50
External Standard Report

<table>
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<tbody>
<tr>
<td>Hydrogen</td>
<td>0.054</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.433</td>
<td>209.582</td>
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Channel: A
Current Time: Jun 16, 2011 17:02:46
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 17:01:44
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-13a
Channel: A
Current Time: Jun 16, 2011 17:02:49
Method: C:\mti\ezchrom\200\methods\bh51.
File : C:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 17:01:44
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

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<tbody>
<tr>
<td>Hydrogen</td>
<td>0.093</td>
<td>%</td>
<td>5.700</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.424</td>
<td>212.172</td>
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Channel: A
Current Time: Jun 17, 2011 07:57:35
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 17, 2011 07:56:29
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-14
Channel: A
Current Time: Jun 17, 2011 07:57:30
Method: c:\mti\ezchrom\200\methods\bh51.m
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 17, 2011 07:56:29
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
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<th>Mean</th>
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<tbody>
<tr>
<td>Hydrogen</td>
<td>0.093</td>
<td>%</td>
<td>5.700</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.421</td>
<td>212.949</td>
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</tbody>
</table>

Channel: A
Current Time: Jun 17, 2011 08:01:44
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 17, 2011 08:00:21
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Sample 7.14a
External Standard Report

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<th>Max</th>
<th>Mean</th>
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</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.045</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.412</td>
<td>215.536</td>
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Channel: A
Current Time: Jun 17, 2011 16:36:05
Method: c:\mti\ezchrom\200\methods\bh51
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 17, 2011 16:34:52
Instrument ID: 180122
Column Type: MS-SA 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 °C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-15

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External Standard Report

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<tr>
<td>Hydrogen</td>
<td>0.045</td>
<td>%</td>
<td>5.67</td>
<td>-1.00</td>
<td>10.203</td>
<td>0.409</td>
<td>216.403</td>
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Channel: A
Current Time: Jun 17, 2011 16:39:51
Method: C:\mti\ezchrom\200\methods\bh51.m
File: C:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 17, 2011 16:38:18
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65°C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

7-15a
Channel: A
Current Time: Jun 17, 2011 16:39:54
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 17, 2011 16:38:18
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

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<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.528</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.401</td>
<td>218.489</td>
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Channel: A
Current Time: Jun 18, 2011 09:17:06
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 09:15:04
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MD
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-16
Channel: A
Current Time: Jun 18, 2011 09:17:10
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 09:15:04
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%DD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.527</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.400</td>
<td>218.328</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 18, 2011 09:20:33
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 09:19:19
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-16a
External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
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<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.050</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.397</td>
<td>219.152</td>
</tr>
</tbody>
</table>

Channel: A  
Current Time: Jun 18, 2011  09:25:45  
Method: C:\mti\ezchrom\200\methods\bh51.  
File : C:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 18, 2011  09:23:39  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time:  2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds

Sample 7-16 b  
Shows the effect of gain.
Channel: A
Current Time: Jun 18, 2011 09:25:50
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 09:23:39
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: Low
Sample Time: 2 Seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Page A-66
External Standard Report

<table>
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<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.232</td>
<td>%</td>
<td>5.670</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.389</td>
<td>221.020</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 18, 2011 17:02:08
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 17:00:58
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-17
Channel: A
Current Time: Jun 18, 2011 17:02:11
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 17:00:58
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
<table>
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<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.231</td>
<td>%</td>
<td>5.670</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.387</td>
<td>221.416</td>
</tr>
</tbody>
</table>

Sample 7-17a
External Standard Report

<table>
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<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.380</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.382</td>
<td>222.394</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 19, 2011 14:59:19
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 19, 2011 14:58:11
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-19

Page A-71
Channel: A
Current Time: Jun 19, 2011 14:59:25
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 19, 2011 14:58:11
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
## External Standard Report

<table>
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<th>Mean</th>
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<tbody>
<tr>
<td>Hydrogen</td>
<td>0.379</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.381</td>
<td>222.471</td>
</tr>
</tbody>
</table>

- Channel: A
- Current Time: Jun 19, 2011 15:02:35
- Method: C:\mti\ezchrom\200\methods\bh51.
- File: C:\mti\ezchrom\200\chrom\01090815.1
- Data file creation time: Jun 19, 2011 15:01:30
- Instrument ID: 180122
- Column Type: MS-5A 4m
- Carrier Gas: He
- Column Head Pressure: 23.8 psi
- Column Temperature: 65 C
- Instrument Gain: MED
- Sample Time: 2 seconds
- Inject Time: 10 milliseconds
- Run Time: 45 seconds

Sample 7-18a
Channel: A
Current Time: Jun 19, 2011  15:02:38
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 19, 2011  15:01:30
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time:  2 seconds
Inject Time:  10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
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<tr>
<th>Name</th>
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<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.233</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.375</td>
<td>223.614</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 20, 2011 07:35:59
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 20, 2011 07:34:51
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-19

Page A-75
Current Time: Jun 20, 2011 07:36:02
Method: c:\mti\ezchrom\200\methods\bh51
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 20, 2011 07:34:51
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
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<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.233</td>
<td>%</td>
<td>5.69</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.374</td>
<td>223.972</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 20, 2011 07:39:27
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 20, 2011 07:37:59
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 7-19a
Channel: A
Current Time: Jun 20, 2011 07:39:29
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 20, 2011 07:37:59
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
GC Data for Sample 9
External Standard Report

<table>
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<tr>
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<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.009</td>
<td>%</td>
<td>5.670</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.688</td>
<td>163.207</td>
</tr>
</tbody>
</table>

Channel: A
Method: \c:\mti\ezchrom\200\methods\bh51.
File: \c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 13, 2011 16:54:53
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-1
Channel: A
Current Time: Jun 13, 2011 16:56:39
Method: C:\mti\ezchrom\200\methods\bh51.
File: C:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 13, 2011 16:54:53
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
<table>
<thead>
<tr>
<th>Name</th>
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<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.096</td>
<td>%</td>
<td>5.670</td>
<td>1.000</td>
<td>10.203</td>
<td>0.679</td>
<td>164.513</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 13, 2011 17:00:51
Method: c:\mti\ezchrom\200\methods\bh51
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 13, 2011 16:59:49
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Channel: A
Current Time: Jun 13, 2011 17:00:54
Method: C:\mti\ezchrom\200\methods\bh51.
File: C:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 13, 2011 16:59:49
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.369</td>
<td>%</td>
<td>5.730</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.624</td>
<td>172.557</td>
</tr>
</tbody>
</table>

Channel: A
Method: C:\mti\ezchrom\200\methods\bh51.
File: C:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 13, 2011 19:52:21
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65°C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.369</td>
<td>%</td>
<td>5.730</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.618</td>
<td>173.176</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 13, 2011 19:56:06
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 13, 2011 19:55:09
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 69 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-29
Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 13, 2011 19:55:09
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

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<table>
<thead>
<tr>
<th>Name</th>
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<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.196</td>
<td>%</td>
<td>5.710</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.601</td>
<td>175.874</td>
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</tbody>
</table>

Channel: A  
Current Time: Jun 14, 2011 09:29:22  
Method: c:\mti\ezchrom\200\methods\bh51  
File: c:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 14, 2011 09:28:26  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds  

Sample 9-3
Channel: A
Current Time: Jun 14, 2011 09:29:25
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 09:28:26
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
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<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.196</td>
<td>%</td>
<td>5.710</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.595</td>
<td>176.788</td>
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</tbody>
</table>

Channel: A
Current Time: Jun 14, 2011 09:32:31
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 09:31:29
Instrument ID: 1B0122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-3

Page A-90
Channel: A
Current Time: Jun 14, 2011  09:32:34
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011  09:31:29
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time:  2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

Name  Amount  Units  RT  Min  Max  Mean  %SD
Hydrogen  0.313  %  5.690  -1.000  10.203  0.579  179.200

Channel: A
Current Time: Jun 14, 2011  13:35:50
Method: c:\mti\ezchrom\200\methods\bh51.
File   : c:\mti\ezchrom\200\chrom\01090816.1
Data file creation time: Jun 14, 2011  13:34:33
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time:  2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample  9-4
Channel: A
Current Time: Jun 14, 2011 13:35:54
Method: c:\mti\ezchrom\200\methods\bh51
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 13:34:32
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Page A-93
### External Standard Report

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<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.311</td>
<td>%</td>
<td>5.720</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.575</td>
<td>179.835</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 14, 2011 13:40:05
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 13:38:56
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-42

Page A-94
Channel: A
Current Time: Jun 14, 2011 13:40:06
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 13:38:56
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
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<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.000</td>
<td>%</td>
<td>5.650</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.552</td>
<td>184.438</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 14, 2011 16:43:08
Method: C:\mti\ezchrom\200\methods\bh51.
File: C:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 16:41:50
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-5
Bad sample
Channel: A
Current Time: Jun 14, 2011 16:43:10
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 16:41:50
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instruments Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Bad Sample
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.243</td>
<td>%</td>
<td>5.710</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.547</td>
<td>185.155</td>
</tr>
</tbody>
</table>

Channel: A  
Current Time: Jun 14, 2011 16:47:15  
Method: c:\mti\ezchrom\200\methods\bh51.1  
File: c:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 14, 2011 16:45:27  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds
Channel: A
Current Time: Jun 14, 2011 16:47:18
Method: C:\MTI\EzChrom\200\Methods\bh51.
File: C:\MTI\EzChrom\200\Chrom\01090815.1
Data file creation time: Jun 14, 2011 16:45:27
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: Low
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

Name          Amount Units  RT  Min  Max  Mean  %SD
Hydrogen  0.302 %  5.720  -1.000  10.203  0.531  187.437

Channel: A
Current Time: Jun 14, 2011  20:00:56
Method: \c:\mti\ezchrom\200\methods\bh51.
File : \c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011  19:59:51
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time:  2 seconds
Inject Time:  10 milliseconds
Run Time:  45 seconds

Sample 9-6
Only one
Sample
Channel: A
Current Time: Jun 14, 2011 20:00:58
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 14, 2011 19:59:51
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Injekt Time: 10 milliseconds
Run Time: 45 seconds

Page A-101
External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.325</td>
<td>%</td>
<td>9.740</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.528</td>
<td>187.943</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 15, 2011 07:51:57
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 07:50:53
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Channel: A
Current Time: Jun 15, 2011 07:51:59
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 07:50:53
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.324</td>
<td>%</td>
<td>5.740</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.524</td>
<td>188.443</td>
</tr>
</tbody>
</table>

Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 07:54:33
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.9 psi
Column Temperature: 69 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-7a

Page A-104
External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.127</td>
<td>%</td>
<td>5.700</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.505</td>
<td>191.652</td>
</tr>
</tbody>
</table>

Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 13:42:23
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-8
Channel: A
Method: c:\mti\ezchrom\200\methods\bh51
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 13:42:23
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-8
### External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
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<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.123</td>
<td>%</td>
<td>5.700</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.499</td>
<td>193.070</td>
</tr>
</tbody>
</table>

Channel: A  
Method: c:\mti\ezchrom\200\methods\bh51  
File : c:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 15, 2011 13:46:25  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds  

Note: Not much sample  

---

Page A-108
Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 13:46:25
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-89
### External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.073</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.487</td>
<td>195.958</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 15, 2011 17:18:22
Method: C:\mti\ezchrom\200\methods\bh51.
File: C:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 17:17:21
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-9
## External Standard Report

<table>
<thead>
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<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.072</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.483</td>
<td>196.892</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 15, 2011 17:20:57
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 17:19:56
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-9a

Page A-112
Channel: A
Current Time: Jun 15, 2011 17:20:59
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\0109015.1
Data file creation time: Jun 15, 2011 17:19:56
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

Name       Amount  Units  RT   Min   Max   Mean   %SD
----------  -------  ----  ----  -----  -----  -----  ----
Hydrogen    0.043    %  5.600  -1.000 10.203  0.471  199.899

Channel: A
Method: c:\mti\ezchrom\200\methods\bh51.
File   : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011  20:45:43
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time:  2 seconds
Inject Time:  10 milliseconds
Run Time:  45 seconds

Sample 9-10

Page A-114
Channel: A
Current Time: Jun 15, 2011 20:46:56
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 20:45:43
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 °C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.043</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.467</td>
<td>200.874</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 15, 2011 20:50:32
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 20:49:20
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-10a
Channel: A
Current Time: Jun 15, 2011 20:50:35
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 15, 2011 20:49:20
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Injection Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
## External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.082</td>
<td>%</td>
<td>5.700</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.457</td>
<td>203.117</td>
</tr>
</tbody>
</table>

Channel: A  
Current Time: Jun 16, 2011 08:15:17  
Method: c:\mti\ezchrom\200\methods\bh51.  
File: c:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 16, 2011 08:14:15  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds
Current Time: Jun 16, 2011 08:15:18
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090015.1
Data file creation time: Jun 16, 2011 08:14:15
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.081</td>
<td>%</td>
<td>5.700</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.453</td>
<td>203.979</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 16, 2011 08:17:56
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\010908151
Data file creation time: Jun 16, 2011 08:16:56
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-11a
# External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.036</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.443</td>
<td>206.857</td>
</tr>
</tbody>
</table>

Channel: A  
Current Time: Jun 16, 2011 11:42:06  
Method: c:\mti\ezchrom\200\methods\bh51  
File : c:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 16, 2011 11:40:58  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds

Sample 9-12

Detector Sensitivity: [Signature]

Page A-122
Channel: A
Current Time: Jun 16, 2011 11:42:07
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 11:40:58
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Page 123
# External Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.036</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.439</td>
<td>207.798</td>
</tr>
</tbody>
</table>

Channel: A  
Current Time: Jun 16, 2011 11:44:33  
Method: c:\mti\ezchrom\200\methods\bh51  
File : c:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 16, 2011 11:43:32  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds  

Sample 9-12a  

Page A-124
Channel: A
Current Time: Jun 16, 2011 11:44:34
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 11:43:32
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Page A-125
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.044</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.430</td>
<td>210.488</td>
</tr>
</tbody>
</table>

Channel: A

Current Time: Jun 16, 2011 17:06:31

Method: c:\mti\ezchrom\200\methods\bh51.

File: c:\mti\ezchrom\200\chrom\0109015.1

Data file creation time: Jun 16, 2011 17:04:47

Instrument ID: 180122

Column Type: MS-5A 4m

Carrier Gas: He

Column Head Pressure: 23.8 psi

Column Temperature: 65 C

Instrument Gain: LOW

Sample Time: 2 seconds

Inject Time: 10 milliseconds

Run Time: 45 seconds
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.044</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.427</td>
<td>211.389</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 16, 2011 17:09:46
Method: c:\mti\ezchrom\200\methods\bh51.m
Data file creation time: Jun 16, 2011 17:08:34
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.9 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-13a

Page A-128
Channel: A
Current Time: Jun 16, 2011 17:09:49
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 16, 2011 17:08:34
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
# External Standard Report

<table>
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<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.054</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.418</td>
<td>213.809</td>
</tr>
</tbody>
</table>

Current Time: Jun 17, 2011 08:06:33
Method: c:\mti\ezchrom\200\methods\bhs1.py
ata file creation time: Jun 17, 2011 08:04:16
strument ID: 180122
olumn Type: MS-5A 4m
rier Gas: He
olumn Head Pressure: 23.8 psi
olumn Temperature: 65 C
strument Gain: LOW
ample Time: 2 seconds
ject Time: 10 milliseconds
un Time: 45 seconds

Sample 9-14
### External Standard Report

<table>
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<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.054</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.415</td>
<td>214.655</td>
</tr>
</tbody>
</table>

Channel: A  
Current Time: Jun 17, 2011 08:09:48  
Method: c:\mti\ezchrom\200\methods\bh51.  
File: c:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 17, 2011 08:08:52  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: LOW  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds

Sample 9-149
Sample 9-14a
Internal Standard Report

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.035</td>
<td>%</td>
<td>5.670</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.406</td>
<td>217.289</td>
</tr>
</tbody>
</table>

Current Time: Jun 17, 2011 16:43:23
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Actual file creation time: Jun 17, 2011 16:42:20
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Injection Time: 2 seconds
Injection Time: 10 milliseconds
Injection Time: 45 seconds

Sample 9-15
Amplitude
5.000
4.500
4.000
3.500
3.000
2.500
2.000
1.500
1.000
0.500

Time (sec.)
4.25 4.75 5.25 5.75 6.25 6.75 7.25 7.75 8.25

Channel: A
Current Time: Jun 17, 2011 16:43:27
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 17, 2011 16:42:20
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Table:

<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.035</td>
<td>%</td>
<td>5.670</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.403</td>
<td>218.170</td>
</tr>
</tbody>
</table>

Sample 9-15a

Current Time: Jun 17, 2011 16:46:45
Method: c:\mti\ezchrom\200\methods\bh51.exe
File: c:\mti\ezchrom\200\chrom\01090815.1&
Texture creation time: Jun 17, 2011 16:45:36
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Channel: A
Current Time: Jun 17, 2011 16:46:47
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 17, 2011 16:45:36
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: LOW
Sample Time: 2 seconds
Injext Time: 10 milliseconds
Run Time: 45 seconds
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.300</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.393</td>
<td>220.357</td>
</tr>
</tbody>
</table>

Sample 9-16

Current Time: Jun 18, 2011 09:33:24
Method: c:\mtl\ezchrom\200\methods\bh51..
File: c:\mtl\ezchrom\200\chrom\01090815.1
File creation time: Jun 18, 2011 09:32:12
Instrument ID: 150122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Injection Time: 2 seconds
Injection Time: 10 milliseconds
Injection Time: 45 seconds
Channel: A
Current Time: Jun 18, 2011 09:33:26
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Log file creation time: Jun 18, 2011 09:32:12
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Amplifier Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

\( y = 16 \)
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.299</td>
<td>%</td>
<td>5.680</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.391</td>
<td>220.620</td>
</tr>
</tbody>
</table>

Sample 9-16a

Current Time: Jun 18, 2011 09:36:31
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 09:35:16
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain (MED)
Sample Time: 2 seconds
Inject Time: 10 milliseconds
run Time: 45 seconds
channel: A
Current Time: Jun 18, 2011 09:36:33
Method: c:\mti\ezchrom\200\methods\bh51.
File : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 09:35:16
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time:  2 seconds
Inj ect Time: 10 milliseconds
Run Time: 45 seconds

Page A-141
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.203</td>
<td>%</td>
<td>5.670</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.385</td>
<td>221.968</td>
</tr>
</tbody>
</table>

Sample: 9-17
Channel: A
Current Time: Jun 18, 2011 17:09:19
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 17:07:56
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.204</td>
<td>%</td>
<td>5.670</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.383</td>
<td>222.315</td>
</tr>
</tbody>
</table>

Channel: A  
Current Time: Jun 18, 2011 17:12:10  
Method: c:\mti\ezchrom\200\methods\bh51.  
File: c:\mti\ezchrom\200\chrom\01090815.1  
Data file creation time: Jun 18, 2011 17:11:07  
Instrument ID: 180122  
Column Type: MS-5A 4m  
Carrier Gas: He  
Column Head Pressure: 23.8 psi  
Column Temperature: 65 C  
Instrument Gain: MED  
Sample Time: 2 seconds  
Inject Time: 10 milliseconds  
Run Time: 45 seconds
Channel: A
Current Time: Jun 18, 2011 17:12:13
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 18, 2011 17:11:07
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.223</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.379</td>
<td>222.363</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 19, 2011 15:06:29
Method: c:\mti\ezchrom\200\methods\bh51.m
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 19, 2011 15:05:05
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 °C
Instrument Gain: MBG
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds

Sample 9-18
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>$SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.223</td>
<td>%</td>
<td>5.00</td>
<td>1.000</td>
<td>10.203</td>
<td>0.377</td>
<td>223.251</td>
</tr>
</tbody>
</table>

**Sample 9-18a**
Name | Amount | Units | RT | Min | Max | Mean | %SD
---|---|---|---|---|---|---|---
Hydrogen | 0.092 | % | 5.690 | -1.000 | 10.203 | 0.372 | 224.633

Sample 9-19

Page A-150
Channel: A
Current Time: Jun 20, 2011  07:44:02
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chron\01090815.1
Data file creation time: Jun 20, 2011  07:42:50
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
<table>
<thead>
<tr>
<th>Name</th>
<th>Amount</th>
<th>Units</th>
<th>RT</th>
<th>Min</th>
<th>Max</th>
<th>Mean</th>
<th>%SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen</td>
<td>0.091</td>
<td>%</td>
<td>5.690</td>
<td>-1.000</td>
<td>10.203</td>
<td>0.370</td>
<td>225.290</td>
</tr>
</tbody>
</table>

Channel: A
Current Time: Jun 20, 2011 07:46:57
Method: c:\mti\ezchrom\200\methods\bh51.
File: c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 20, 2011 07:45:50
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time: 2 seconds
Inject Time: 10 milliseconds
Run Time: 45 seconds
Channel: A
Current Time: Jun 20, 2011  07:47:00
Method: c:\mti\ezchrom\200\methods\bh51
File  : c:\mti\ezchrom\200\chrom\01090815.1
Data file creation time: Jun 20, 2011  07:45:50
Instrument ID: 180122
Column Type: MS-5A 4m
Carrier Gas: He
Column Head Pressure: 23.8 psi
Column Temperature: 65 C
Instrument Gain: MED
Sample Time:  2 seconds
Inject Time:  10 milliseconds
Run Time:  45 seconds