Executive Summary

The overall objective of this two phase program is to investigate the use of dry carbon-based sorbents for mercury control. This information is important to the utility industry in anticipation of pending regulations. During Phase I, a bench-scale field test device that can be configured as an electrostatic precipitator, a pulse-jet baghouse, or a reverse-gas baghouse has been designed, built and integrated with an existing pilot-scale facility at PSCO’s Comanche Station. Up to three candidate sorbents will be injected into the flue gas stream upstream of the test device to and mercury concentration measurements will be made to determine the mercury removal efficiency for each sorbent. During the Phase II effort, component integration for the most promising dry sorbent technology shall be tested at the 5000 acfm pilot-scale.

An extensive work plan has been developed for the project. Three sorbents will be selected for evaluation at the facility through investigation, presentation, and discussion among team members: PSCO, EPRI, ADA, and DOE. The selected sorbents will be tested in the five primary bench-scale configurations: pulse-jet baghouse, TOXECON, reverse-gas baghouse, electrostatic precipitator, and an ESP or fabric filter with no Comanche ash in the flue gas stream. In the EPRI TOXECON system, mercury sorbents will be injected downstream of a primary particulate control device, and collected in a pulse-jet baghouse operated at air-to-cloth ratios of 12 to 16 ft/min, thus separating the mercury and sorbent from the captured flyash.
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the no-ash configuration, an external flyash sample will be injected into a clean gas stream to investigate possible variations in sorbent effectiveness in the presence of different ashes. The use of an existing test facility, a versatile design for the test fixture, and installation of a continuous mercury analyzer will allow for the completion of this ambitious test plan.

The primary activity during the quarter was testing in the ESP configuration. Significant effort was focused on the continuous mercury analyzer with the goal of establishing stable and reliable data acquisition from the instrument. Unfortunately, at the very low baseline mercury levels the analyzer is not currently proving reliable data despite a number of changes and improvements to the data analysis algorithms. Several test conditions with the ESP configuration were completed with data collection via iodated carbon sorbent traps (modified MESA method). The data from the MESA tests is limited to a few grab samples with additional data. Preliminary indications are that up to 60% mercury removal can be obtained by operating at low flue gas temperatures. As only limited data is available at this time, conclusions will require additional data.

The project is approximately two months behind the original schedule. The delay was caused by the additional time required to improve the mercury analyzer performance. Performance of the analyzer is not sufficient for data reporting. Work will continue on further improvements in the next quarter. It may be impossible to obtain reliable data at the very low mercury levels at this site. A decision will be made on further work with the analyzer or to continue the test program using the MESA sampling and analysis technique. The ESP work will be completed in early November and testing will continue with the pulse jet configuration.

Activities During Reporting Period

Task 2. Design, Fabrication, and Installation of laboratory-scale test facility

Some small sub-assemblies specific to the pulse-jet and reverse-gas configurations of the test facility remain to be fabricated. These sub-assemblies include the pulse pipes and pressure reservoir for the pulse-jet and the reverse-gas valving sub-assembly. No work was performed on these in September due to delays in Task 3 testing. Fabrication should be completed in October.
Task 3. Field Evaluations of Sorbents for Mercury Control

The lab-scale test facility was designed and fabricated to permit significant control over the operating conditions during evaluations. In addition to changing the particulate control configurations, the test plan identifies tests over a range of operating parameters such as duct temperature, flue gas moisture content, in-duct sorbent residence time, and flue gas mercury concentration. Sorbent effectiveness will be evaluated for the temperature ranges: 1) 190-210 °F (expected cold weather baseline at Comanche), 2) 225-275 °F (expected warm weather baseline), and 3) 300-325 °F. Duct cooling will be achieved by two methods: 1) spray cooling with water (increased moisture content), and 2) dry cooling with a heat exchanger. The sorbent injection ports are located for in-duct sorbent residence times of 0.75 to 1.5 seconds to evaluate the effect of residence time on sorbent effectiveness.

ESP operation

The particulate control module (PCM) was brought on-line configured as an ESP on July 7, 1996. Initial carbon injection was begun July 26. Carbon injection in the ESP is scheduled through the first week in November. ESP testing was extended through November 1 to allow time to complete FGD and AC-1 activated carbon testing at various temperatures and injection rates. Data will be collected with the mercury analyzer and with a modified MESA method until the analyzer is providing representative mercury data. The modified MESA method consists of a quartz probe with a glass wool insert to collect particulate followed by two iodated carbon traps to collect vapor phase mercury.

After several weeks of operation, the ESP began intermittently powering down. Comanche Station burns a Powder River Basin coal, which often causes problems with back corona on full-scale ESPs. It is expected that back corona was causing the operational difficulties at Comanche. The ESP T/R controls were set for intermittent energization which successfully quenches the back corona in the pilot. No problems with ESP operation were experienced after the change in the controls.

Preliminary Mercury Removal Results - MESA method

Preliminary Mercury Speciation Adsorption (MESA) results from Darco FGD (Norit) and AC-1 activated carbon testing at temperatures from 209 to 253 °F in the ESP configuration are shown on Figure 1 and in Table 1. All data shown except the inlet measurements on Test ID 1, 3
and 5 were conducted with the modified MESA method described above. Tests 1, 3 and 5 were conducted with full MESA trains. Figure 1 presents the inlet and outlet mercury measurements with the ESP inlet temperature and carbon injection rate noted. Initially, it was hoped that the inlet mercury concentration would remain stable during a test day, making it possible to conduct more than one outlet measurement for each inlet measurement. The variability in the inlet measurements shown on the graph makes this impractical. Some early outlet data is presented without a corresponding inlet measurement, as it was taken before initial inlet sample results were available.

![Graph showing inlet and outlet mercury measurements](image)

**Figure 1. Preliminary Results: MESA method.**

The ability of Comanche flyash to adsorb mercury at the temperatures tested is also indicated in figure 1. Samples were collected for three baseline test pairs (tests 2, 7, and 8). Two tests resulted in 25 to 30% mercury removal. The third test had a very low mercury inlet and related to a large negative removal. While no sampling abnormalities were noted, it is believed the inlet data is in error. If the inlet concentration is erroneous and an inlet of 5.4 μg/Nm³ (the average of the inlet vapor concentrations for the six other tests) is assumed, the calculated Hg removal rate is a much more reasonable 20%.
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<tr>
<th>Test ID</th>
<th>Sorbent</th>
<th>Date &amp; Test#</th>
<th>Host T ofF</th>
<th>ESP in T ofF</th>
<th>load MW</th>
<th>av flow acfm</th>
<th>Inj Rate g/min</th>
<th>Inj Ratio</th>
<th>Inlet ELEM $\mu g/Nm^3$</th>
<th>Inlet Part $\mu g/Nm^3$</th>
<th>Inlet Hg(II) $\mu g/Nm^3$</th>
<th>Outlet vapor $\mu g/Nm^3$</th>
<th>Outlet* Part $\mu g/Nm^3$</th>
<th>% Hg Removal Total</th>
<th>% Hg Removal Vapor</th>
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<td>2.37</td>
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<tr>
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<td>Norit</td>
<td>8-27-O1/11</td>
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<td>235</td>
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<td>317</td>
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<td>0.001</td>
<td>64</td>
<td>62</td>
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</tbody>
</table>

* Particulate measurements are collected non-isokinetically and may not be representative of the particulate mercury concentration in the duct.
The percent mercury removal for the three available data points with similar carbon injection rates are plotted in Figure 2. This plot shows a possible correlation of increasing mercury removal to decreasing flue gas temperature, however, with only limited data it is not possible to make a sound conclusion at this time. The measured mercury removal of 48 to 62\% does agree well with the predicted removals using mass transfer limits at these carbon injection rates.

![Figure 2. Hg removal with increasing collection temperature.](image)

One area of concern with the MESA method at Comanche is accounting for the particulate fraction. Samples are collected non-isokinetically (at 90° to the flow). The flyash entering the MESA train and collecting on the glass wool plug is much less than representative of the flue gas concentration and is dependent on parameters such as flyash size distribution and gas velocities. Calculations will be made to predict the fraction of the flyash that is likely collected in the MESA train to help interpret the data collected to date. It may be possible to compare mercury concentration of the ash collected in the pilot hopper to the mercury concentration of the ash collected on the glass wool plug filter to increase the confidence of the particulate mercury concentration during future baseline testing.

Since a true total mercury (vapor + particulate) measurement is not made with the MESA, change in the particulate mercury fraction due to factors other than sorbent injection will confuse
the interpretation of the data. During testing at Comanche, the flue gas is often cooled or heated to a target temperature for a particular test condition. In tests 1-10, the inlet sample was collected upstream of heating/cooling the fluegas supplied to the pilot. In samples 11-29 (data to be reported in the 4th quarter report), inlet samples were collected downstream of the heating/cooling section. If the temperature of the flyash is modified, it is possible that the ash is adsorbing or desorbing mercury. Thus, the vapor phase mercury measured downstream of heating/cooling may be different than what was measured upstream of heating/cooling. The ash may not be in equilibrium with the flue gas with respect to the phase distribution of the mercury when it reaches the downstream inlet sampling location. If a representative total measurement can be obtained, errors due to non-isokinetic flyash sampling could be minimized. Options include isokinetic sampling through a filter, which would require passing the sampled flue gas through a possible adsorbent (flyash or carbon), or a cyclone to collect a representative flyash sample. Both of these are options would require either a very small diameter nozzle (flow through the MESA is 0.5 l/min and the velocity in the nozzle must match the velocity in the duct to be isokinetic) or a secondary sampling system to draw a more reasonable flow through the nozzle with a slipstream going to the MESA train. Another option would be to use a larger diameter iodated carbon trap to maintain the face velocity through the carbon while increasing the overall sample flowrate to a reasonable isokinetic sampling range. Further investigation into sampling variations will be completed next month.

**Mercury Analyzer and Analyzer Sampling System**

Significant effort has been undertaken to acquire stable and accurate mercury measurements with the continuous mercury analyzer. This effort was placed at high priority to maximize the information that could be gained from the limited testing time available for this project. With manual measurements, discrete data samples are taken during each test condition. Several discrete data points must be collected before a trend may emerge and the trend may be masked by one or two data points that are anomalies. A real-time analyzer would allow monitoring a test case over time, minimizing the often confusing influence of anomalies and allowing conditions such as changes in boiler load or host duct temperature to be evaluated.

The analyzer has undergone several software and hardware changes to improve resolution. Its operation has been carefully studied and the response of the analyzer to mercury and SO₂ is now better understood. A calibration routine has been developed and a sampling system has been fabricated to allow calibration through the sampling hotlines or directly to the analyzer. In addition, the analyzer was moved in to a temperature controlled trailer the third and
fourth weeks in September in an attempt to improve analyzer performance by minimizing temperature drift effects. This move was made after encouraging analyzer data was collected the first week in September. The following sections summarize theory behind analyzer operation, engineering issues associated with continuous stable operation, and concerns with bringing a representative gas sample from the stack to the analyzer.

**Mercury Analyzer Fundamentals - Theory**

Measuring trace gas concentration continuously at levels of less than one part per billion is a complicated matter. ADA’s approach is based on the Zeeman effect on the UV emission spectrum of natural mercury. Figure 1 presents this effect pictorially. The mercury lamp shown in the figure emits light at 2537 Å with subpeaks of several isotopes closely grouped. If the lamp is placed in a magnetic field, the spins of outer shell electrons will align, causing the emission profile to change. The new profile adds two “winglets” spaced a fraction of an angstrom on either side of the main grouping and orthogonally polarized to the main emission lines.

![Figure 1. Emission profile of natural mercury and mercury in a magnetic field.](image)

Figure 3. Zeeman splitting: Emission profile of natural mercury and mercury in a magnetic field.

The absorption profile of mercury is superimposed over the emission profile in Figure 3. Elemental mercury vapor absorbs the \( \pi \) wavelength but does not significantly absorb the \( \sigma^+ \) or \( \sigma^- \) wavelengths. Because of this difference in the absorption of the \( \pi \) and \( \sigma \) wavelengths, the mercury concentration can be determined even in the presence of interferents that are broad-band absorbers in the same region, such as \( \text{SO}_2 \).

It is difficult to directly differentiate the light attenuation with polarization. Therefore, a waveplate (sectioned Polaroid filter) in the light path is used to separate the different polarization of light into discrete light pulses. The light then passes though a measurement absorption cell in the sample gas flowpath, and finally to a photodetector.
The photodetector responds to differing light intensities with a varying output voltage signal. The response of a photodetector in the mercury analyzer is represented on the graph in Figure 4, showing photodetector millivolt response over time. The first third of the graph represents the response with zero gas in the measurement cell. The intensity of the s and p polarization of the light create a waveform with distinct maximum, minimum and average millivolt values. When mercury is present in the measurement cell, the intensity of the p polarization is directly attenuated, causing the minimum millivolt signal from the photodetector to decrease. Thus, the difference between maximum and minimum is greater and the average is slightly lower. The difference between maximum and minimum light intensities can be considered for discussion purposes as the mercury signal strength. In the presence of SO2, both p and s polarization are attenuated, decreasing the mercury signal strength and the average light intensity.

![Graph](image)

**Figure 4.** Ideal response of photodetector.

**Figure 5** is a graph of mercury signal strength versus average light intensity for various concentrations of SO2 measured in the laboratory. The mercury signal strength is seen to be a quite linear function of average light intensity for this broad band absorber. This allows the correction of the mercury signal based on a known SO2 concentration.
Figure 6 is a graph of mercury signal strength plotted against average light intensity for varying concentrations of mercury. Four concentrations of mercury were passed through the measurement cell with and without SO2. The lines representing each mercury concentration have nearly identical slopes and are spaced linearly with mercury concentration.

Figures 5 and 6 define the algorithm used in the computer for this analyzer to calculate mercury concentration. This calculation is graphically summarized in Figure 7. A three point calibration (zero gas, SO2 span gas, mercury span gas) is used to define the slope of the SO2 line and the spacing of the mercury lines. These points are identified in Figure 7 and represent the points referred to in Figure 4.
Noise, temperature and other non-ideal effects

The theory behind the analyzer operation is well known. However, when this theory is applied to an instrument designed to measure concentrations less than 1 ppb, engineering details become tremendously important. The effect of changes in the temperature of the environment seems to be the most critical in the prototype ADA instrument.

The analyzer was recently moved into a “temperature-controlled trailer” in anticipation that this would stabilize the signals, reduce the noise and improve resolution of the instrument. Although the overall temperature range in the test trailer was much smaller than in the previous location, the frequency of the change was much faster. Figure 8 shows changes in the mercury signal strength, average light intensity, detector temperature and mercury lamp temperature over a continuous 40 hour period. The sawtooth pattern in the detector temperature profile from hours 25 to 35 and 45 to 60 reflect temperature fluctuations caused by a room air conditioner cycling on and off during the day. The air conditioner shuts off at night. The signal strength and average light profiles follow the temperature profile. These fluctuations represent 10 to 100 μg/Nm³ swings in the calculated mercury concentration. This data was collected while sampling actual flue gas from Comanche and the true fluctuations in mercury concentration were probably less than 3 μg/Nm³.

In an attempt to minimize the temperature fluctuations, the air conditioner is now shut off during analyzer operation. In addition, the lamp temperature and the detector temperature are now controlled independently. This decreases the frequency of room temperature cycling to 24 hours and holds two critical components of the system relatively stable.

Mathematical corrections for temperature drift are used with the analyzer at Comanche. Zero gas is periodically passed through the measurement cell. The average light and mercury signal strength are compared with the previous zero values. The real-time measurements are then corrected to the slope of the drift curve. This is not ideal because the slope of the drift line over the previous drift period is used to predict the subsequent drift slope. However, the technique gives a reasonable representation if instrument temperatures are not changing quickly.

Attempts have been made to calculate the mercury signal strength and average light intensity as a function of temperature to mathematically correct for temperature in real-time. However, it has been difficult to select which temperature to use in the calculations. When the detector temperature is held constant and the lamp temperature is allowed to drift, the signals drift. This would indicate a change in light intensity from the lamp. If the lamp temperature is
held constant and the detector is allowed to drift, the signals still indicate some drift. This would imply a change in the responsivity of the detector. When attempts are made to hold both constant, slight fluctuations in temperature remain due to null bands in the temperature controllers and a temperature effect is still noted. Figure 9 presents three plots including short term drift of the average light intensity and mercury signal strength for hours 37-47 from Figure
8, and the corresponding trace of lamp temperature versus time for this period. Three time periods are highlighted: a constant lamp temperature segment from hour 37 to 38.2 (light triangles), and increasing temperature period from hour 38.2 to hour 42.3 (gray squares), and a decreasing temperature period from hour 42.3 to hour 45.5 (black diamonds). The average light and mercury signals in the top two graphs show a hysteresis associated with increasing or decreasing temperature. This characteristic of the analyzer needs to be examined further before a real-time mathematical correction can be developed.

Figure 9. Short term temperature drift of mercury signal strength and average light intensity.
Electrical noise on the detector signals has proven to be another complex problem with the analyzer. Several modifications have been made to the analyzer since it arrived at Comanche. These include assuring that the power to the analyzer was properly grounded and that the analyzer cabinet was also grounded. Signal cables have been shielded, ac motors have been replaced with dc motors, and a line conditioner is used to assure that power to the computer, demodulator and photodetectors is “clean”. The signal to noise ratio for the instrument at a level of mercury of interest at coal plants (< 10 μg/Nm³) is currently quite low. Figure 10 presents data from a recent zero and span check of the analyzer conducted after the analyzer was moved into the test trailer. The data presented is 5 sample averages of 2 second data. The mercury signal strength is plotted for elemental and total measurement cells with 6.57 μg/Nm³ elemental mercury (from a permeation tube calibration source) in the gas cells. The noise is similar from each photodetector; however, the signal is lower from the total cell. This is expected from the ideal gas law and Beer’s Law (the temperature of the total measurement cell is 900 °F higher than the elemental cell so that a smaller mass of sample gas is present in the cell).

![Figure 10](image_url)

Figure 10. Zero and Span (6.57 μg/Nm³ mercury) for elemental and total mercury signal strength.
The current noise level is higher than that experienced before the analyzer was moved into the test trailer. Two possible explanations for this include noise pickup from the nearby Comanche ID fan which may have been shielded by the previous enclosure, or mechanical vibrations that are affecting the instrument. The analyzer was previously installed on a concrete pad. Changes are continuing to be made to improve shielding and grounding in attempts to reduce the noise.

Analyzer Sampling and Calibration System

A critical component in assuring that an analyzer is capable of providing meaningful mercury measurements is to confirm that a representative gas sample is passed through the analyzer. This may sound obvious, but the engineering is not trivial and studies are continuing to determine the optimal material and gas flowrates required in a transport assembly to bring flue gas from the duct to the analyzer. A system was fabricated for this program with a special sampling filter, valve boxes and controls to allow automatic calibration through the sampling lines and sampling filter and to allow automatic switching between inlet and outlet sampling lines.

A sketch of the stack sampling filter is shown in Figure 11. An oversized buttonhook nozzle turned out of the flow (non-isokinetic sampling) is used to minimize the particulate included in the flue gas sample. This is followed by a ceramic or glass filter held at 300 °F to remove any remaining particulate at a temperature where little adsorption of vapor-phase mercury is expected. It is possible to calibrate through the filter to determine if any mercury is being adsorbed by particulate on the filter or by the filter material itself, or to calibrate without passing through the filter. A second option would have been to install a cyclone followed by a filter.

![Figure 11. Analyzer stack sampling filter.](image-url)
The sampling lines used for this program are 1/4" diameter heated Teflon tubes. Teflon is used for mercury permeation tubes and it would follow that Teflon may absorb mercury during sampling. However, it is hoped that any mass transfer to the Teflon approaches an equilibrium state after some period time of constant flow and that the sampling lines adequately transport the flue gas to the analyzer without changing the mercury concentration in the gas. A constant flow of flue gas is pulled through both the inlet and outlet sample lines. Just before entering the mercury analyzer, one sample stream is vented and one enters the analyzer. Two additional 1/4" Teflon lines are heat-traced with the sample lines to transport calibration gas from the analyzer to the sampling filter. One line is exposed only to zero gas and the second line is exposed to mercury span gas. Calibrations through the sampling hot-lines are referred to as “overboard” calibrations (“onboard” for a calibration directly to the analyzer). The confidence in the ability of the hot-line to transport mercury laden flue gas is enhanced when an overboard calibration closely matches an onboard calibration, which is the case for the tests at Comanche Station.

Non-elemental mercury may be more likely to “stick” to the Teflon lines. Early sampling data (see Table 1) indicates that Comanche flue gas contains mostly elemental mercury, thus calibrations at Comanche were conducted only with elemental mercury. Teflon may pose problems for other locations with higher fractions of non-elemental mercury.

**Analyzer Results**

Most of the data collected with the analyzer at Comanche to date has been used to understand and improve analyzer performance. Much of the data collected before September is biased due to pressure fluctuations in the analyzer during measurement. Most of the data collected after the analyzer was moved into a trailer is noisy and it is difficult to confidently glean quantitative data. Figure 12 presents elemental mercury data collected on September 5 (before the move) during injection of Norit carbon at 0.55 g/min. The graph shows data from the automatic zero and span (every 15 minutes) and inlet and outlet mercury measurements. The mercury span gas concentration was 6.57 µg/Nm³.

![Figure 12. Mercury analyzer data.](image-url)
Each data point is a 5 sample average of 2 second samples. The graph indicates some temperature drift. The graph also indicates an outlet elemental mercury concentration of roughly 2.5 μg/Nm³ and 50% mercury removal, which is similar to the MESA results.

While many improvements have been made to the analyzer, the data obtained still has significant drift. The analyzer is not working well enough to provide data of high confidence. Work will continue on improving analyzer performance next quarter, but it may be necessary to discontinue analyzer work and continue the project using MESA analysis.

Task 4. Waste Characterization

The EPA classification of the collected sorbent and flyash mixture is of great concern in the use of sorbent technologies for the removal of mercury from flue gas streams. If the combined sorbent-flyash product collected in the particulate collector hopper remains in a nonhazardous category, it can be handled and disposed of using methods currently employed to dispose of flyash. Samples collected and analyzed during ESP testing at Comanche indicate that the sorbent-flyash material is nonhazardous.

The TCLP (toxicity characteristic leaching procedure) from two samples collected during carbon injection upstream of the pilot ESP at Comanche Station are shown in Table 2. These results show that all 8 RCRA elements of concern are well below regulatory limits. Sample A was collected while injecting 0.47 g/min Norit carbon and sample B was collected while injecting 0.05 g/min Norit carbon. Samples will be collected for TCLP analysis during testing with each particulate control device.

Table 2. TCLP Summary Report

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<th>Element</th>
<th>Sample A</th>
<th>Sample B</th>
<th>Regulatory Levels (mg/L)</th>
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Task 6. Management and Reporting

The project is behind schedule by approximately two months due to the complications experienced with the mercury analyzer. A decision will be made in early November to continue analyzer sampling or to proceed with the program using MESA and other methods. A decision to use the analyzer will be made based on its ability to measure total mercury at a concentration useful to the program.

Activities Scheduled for Next Quarter (October 1 - December 31, 1996)

Task 3. Field Evaluations of Sorbents for Mercury Control

Carbon injection in the ESP is scheduled through the first week in November. Several more Norit and AC-1 tests are scheduled to give data in the three temperature ranges of interest with various injection ratios. The PCM will then be configured as a pulse-jet baghouse. The pulse jet will be operated in baseline operation for a number of weeks to condition the bags. Carbon injection testing will continue though the end of the quarter after determining the best method for mercury measurement.

Task 6. Management and Reporting

Contract Concerns

The project is approximately two months behind schedule. If a decision is made to discontinue analyzer sampling, the number of test completed at different operating conditions will be reduced as a longer duration will be required to collect the appropriate MESA samples. This will allow the project to be completed within the schedule time and within the approved budget.