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I am submitting herewith a thesis written by Lisa N. Urman entitled "The Investigation of a Tin Oxide-Based MEMS Sensor for Measurement of Nitrogen Oxides in Aircraft Engine Exhaust." I have examined the final paper copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Aerospace Engineering.

Dr. Frank Collins, Major Professor

We have read this thesis and
recommend its acceptance:

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Vice Provost and
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The Investigation of a Tin Oxide-Based MEMS Sensor for Measurement of Nitrogen Oxides in Aircraft Engine Exhaust

A Thesis
Presented for the
Master of Science Degree
University of Tennessee, Knoxville

Lisa Urman
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DEDICATION

This thesis is dedicated to my family. To my parents, Dr. Charles and Pamela Goetzke, thank you for providing me such a strong foundation and for the many years you've so selflessly given all that you could. To Laura and Donovan, my brother and sister, thank you for your love and humor. To my mothers-, fathers-, brother-, and sister-in-law, Nanette and Bruce Berglund, Mike and Sarah Urman, Jeramee Prichard, and Tanya Urman, your love and support were greatly appreciated. To my best friend and husband, Joe Urman, thank you for your unbounded love, encouragement and understanding.

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ABSTRACT

An investigation was conducted of the performance of a tin oxide-based, MicroElectro Mechanical Sensor for making measurements of NOx levels on gas turbine engine exhaust. This sensor was provided by Makel Engineering, Inc., as a prototype device for evaluation. In this thesis, the MEMS device is described together with its supporting equipment. Tests, and calibrations were performed and evaluations made on the MEMS sensor, and are discussed in three parts: laboratory calibration and evaluation, off-engine installation and performance, and on-engine installation and performance.

The MEMS device was first tested in a laboratory environment where the temperature and pressure of the gas sampled were at room conditions. NO was provided to the sensor from calibrated gas bottles. The warm up and signal drift characteristics of the MEMS device were recorded and analyzed. The sensitivities of the device to NO levels in the air were recorded and corrected for drift. The output of the MEMS sensor is scaled by an input electronic gain factor called the Data Acquisition Code (DAC). The effect of the DAC on NO level measurements was recorded and analyzed.

After the laboratory calibration and evaluation phase was completed, the MEMS gas emission measurement system was transported to Middle Tennessee State University Airport where an operable jet engine was available for use. The jet engine was a Pratt-Whitney JT-12, 3000lbf thrust class engine that had been modified to permit exhaust gas sensors to be placed inside the engine exhaust duct. In the first series of testing, the sensor was mounted to a heated valve box and was supplied engine exhaust gases through

heated gas lines by use of a vacuum pump. In the second series of testing, the sensor was mounted to the JT-12 exhaust probe, and measured a direct sample of exhaust gas.

Results from the testing of the MEMS sensor include its sensitivity to NOx and CO levels in the engine exhaust. Analysis of the data, taking the laboratory calibrations of the MEMS device into consideration, allowed the MEMS NO levels to be predicted by correcting for the engine exhaust CO levels.

Conclusions of the MEMS study were:

1. The sensor responds to nitrogen oxides and carbon monoxide with an accuracy of 93%.
2. Warm-up times, of approximately 70 minutes, are comparable to other types of gas analysis systems.
3. Recovery times were too long, taking 11 minutes for the no flow case and 3 minutes for the flow case.
4. The resolution of the MEMS sensor was approximately 103 counts per 1ppm NOx.
5. Due to drift, there is about 3ppm NOx difference in measurements on any given day.
6. Many improvements in development are required for this device to be used for actual emission monitoring, such as faster recovery times, more durable packaging, and more selective sensitivity.

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LIST OF ACRONYMS

F/A	Fuel to Air Ratio
MEMS	MicroElecto Mechanical Systems
NASA	National Aeronautics and Space Administration
MTSU	Middle Tennessee State University
MEI	Makel Engineering, Inc.
MGA	MultiGas Analyzer
DAC	Data Acquisition Code
AEDC	Arnold Engineering Development Center

1.0 INTRODUCTION

Due to society's increased concern for air quality control, coupled with an increased use in aircraft, there arose a necessity to detect, monitor, and control emissions from jet engines. Like automobiles, aircraft engine emissions are regulated. While aircraft emissions contribute to only 1% of atmospheric pollution, the Federal Aviation Administration regulates aircraft emissions because they are injected into the upper troposphere causing an increase in ozone by about 20%.[1]

For a jet exhaust with a temperature and pressure of 815°F and 15psi, respectively, a typical exhaust gas from a modern gas turbine engine includes the greenhouse gases carbon dioxide and water vapor, as well as NOx, SOx, and soot. The NOx species is a major contributor to an increase in atmospheric ozone which causes the surface of the Earth to warm.[1]

There have been many tests conducted to measure emissions of engines, most commonly in the automotive industry. Emission testing of aircraft engines is difficult and expensive. There are several reasons for the cost to run these tests, including the costs of the use of a test facility, the fuel, and engine maintenance. Another problem is that emissions testing equipment must withstand the harsh conditions associated with running jet engines. A challenge also lies in the lack of feedback of information to the engine control system. A sensor that measures the engine fuel to air ratio (F/A), and signals the engine controller to run the engine at optimal conditions for reducing NOx emissions is desirable for both emissions testing and engine control optimization.

The necessary criteria of such a sensor are as follows: the sensor must be small in order to place it on board an engine; it must be cost effective, durable, and have a short

warm up time; it must have rapid response times, sensitivity and selectivity to certain gases; and it must not require constant calibration. One candidate is a MEMS chemical sensor.

There are many different types of MEMS chemical gas sensors. NASA is currently using this microfabrication technology to develop H_2, C_xH_y, NO_x, CO, O_2, and CO_2 sensors.[2] MEMS-type sensors are generally based on one of three techniques: the Schottky diode; the resistor; and the electrochemical cell. The Schottky diode is made of a metal in contact with a semiconductor. The metal is chosen to be sensitive to the species that is measured. For instance, if one wants to measure hydrogen, palladium is used for the metal.

For Pd-SiO_2-Si Schottky diodes, hydrogen dissociates on the Pd surface and diffuses to the $PdSiO_2$ interface, affecting the electronic properties of the MOS system and resulting in an exponential response of the diode current to hydrogen. This exponential response has a higher sensitivity at low concentrations and decreasing sensitivity at higher concentrations as the sensor saturates.[2]

To have a greater sensitivity at higher concentrations, a resistive sensor is used. The other type of sensor is based on an electrochemical cell, used for oxygen detection. These electrochemical cells generally use ZrO_2

…as a solid electrolyte and platinum as the anode and cathode. The anode is exposed to a reference gas, usually air, while the cathode is exposed to the gas to be detected. ZrO_2 becomes an ionic conductor of oxygen at temperatures of 600°C and above. This means that the electrochemical potential of the cell can be used to measure the ambient oxygen concentration at high temperatures.[2]

With these specifics in mind, Makel Engineering, Inc. designed and fabricated a MicroElectro Mechanical System (MEMS) chemical sensor. This type of sensor has been used successfully in automobiles to monitor oxygen.[3] Other applications of this type of sensor include H_2 leak detection on the space shuttle, more accurate fire safety monitoring devices, and engine emission monitoring devices to reduce emissions.[2]

For our goals, however, a NOx sensor is needed. The sensor was chosen for its small size, about as large as a dime, and it appears rugged enough to be placed directly on-board a jet engine. Its extreme durability to the harsh environments of operating engines results from its fabrication using SiC.[2] The sensor is also very cost effective. Tests were conducted by applying this sensor to an operating JT-12 turbojet engine to determine whether other requirements have been met with this sensor. Table 1 presents the list of tests, approaches, and success criteria. (All figures and tables are located in the Appendices)

This thesis is a presentation of the tests conducted and results acquired for the Pt004 MEMS sensor constructed of glass and silicon created by Makel Engineering, Inc. This study represents an attempt to evaluate the performance of a tin oxide MEMS chemical gas sensor for making gas turbine engine exhaust measurements. Tests were conducted both in a lab and on site using a JT-12 engine.

2.0 SENSOR DESCRIPTION

The Pt004 sensor is a tin oxide (SnO_2) based sensor that was developed and supplied by Makel Engineering, Inc. (MEI) for measuring the oxides of nitrogen concentration in gas turbine exhaust gas measurements. MEI also provided technical support for the sensor during testing. A photo of the sensor is shown in Figure 1. Figure 2 shows a schematic of the sensor package and Figure 3 shows a schematic of the sensor.

It is desirable that gas species measurements take place *in situ*; the sensor must be able to operate in hot exhaust gas environments. Also, because weight and size are important factors concerning aircraft, the sensor must be both light and small so that multiple sensors can be placed on it. Furthermore, due to the demand of multiple sensors, low cost and excellent durability are a necessity. The sensor should have a fast response time and warm up time, as well as good sensitivity, and selectivity.[3]

To satisfy these requirements, MEI designed, developed, and fabricated a MEMS-type chemical sensor. The MEI sensor, constructed of glass and silicon which are bonded together, is approximately 300μm wide, and 250μm in height. The sensor also has built into it a temperature detector, heater, and gas-sensing element placed over a diaphragm minimizing the "thermal mass of the sensing area."[2] See Figure 3 for a typical SnO_2 sensor. NOx and CO are detected by measuring the conductivity of a thin film of nanocrystalline SnO_2. To meet the selectivity and sensitivity requirements for different chemical species, the SnO_2 is usually doped.[2]

Detection and sensitivity are accomplished by firing a SnO_2 gel, which yields a nanocrystalline SnO_2 film whose conductivity is measured across microscopically small platinum conductors that are arranged like interdigitated fingers.[1] Changes in

conductivity of the SnO_2 film across the interdigitated Pt electrodes are measured and calibrated to CO and NOx concentration in the gas above the film. The detection of NO_2 has been demonstrated down to the 5-ppm level at 360°C, where the highest level of sensitivity exists with a very stable response.[2]

Doping the SnO_2 film can improve both the selectivity of the sensor to a specific gas as well as improve sensor stability. For example, the inclusion of nanoparticulates of platinum (Pt) into the film has been shown to improve sensitivity of the sensor to CO, while the inclusion of SiO_2 has been shown to significantly decrease the grain growth of SnO_2.[2]

SnO_2 is a semi conductor material that reacts with oxidizing and reducing gases. The oxidizing gases cause the sensor resistivity to increase, while reducing gases lower the sensor resistivity and restore the initial resistivity of the sensor.

The reactive gases alter the bonding of oxygen adsorbates on the surface of tin oxide grains, changing the conductivity of the tin oxide from that in air. The generic response of the resistive material to oxidizing and reducing species limits the selectivity of these devices.[2]

For this reason, the sensor has a difficult time differentiating between CO and NOx, and a single sensor is not effective for measuring NOx if both species are present, unless a special calibration procedure has been used to develop the data reduction techniques. These procedures were developed in part of the present study as will be described later.

Along with the Pt004 sensor, Makel Engineering, Inc. provided the general software to operate the sensor. The software allowed many choices concerning the settings of the sensor. One can choose sensor temperature, time for data acquisition, as

well as measurement recording range or data acquisition code (DAC) setting, which controls the measurement range of the sensor. The software works by relating the NOx concentration in ppm to counts. These counts are recorded, by the software, on a graph vs. time plot and are also recorded in a spreadsheet file for further analysis.

In order to provide a better sensor description, sensor testing must be performed. The first priority is to characterize the sensor, beginning with warm up time. Knowing the warm up time is inherent to having a successful test. The most important characterization is the calibration. For this sensor, the number of counts must be related to the level of NOx present. Following these tests is the determination of the sensor measurement range, time constant, recovery time, performance at varying oxygen levels, performance at varying sensor temperatures, durability against harsh environment, and longevity. Knowing all of these characteristics will provide information that determines for which applications the sensor will be useful, as well as information that will aid in determining the sensor weaknesses. This information can then be used to improve the sensor.

3.0 AEDC LAB TESTS AND RESULTS

3.1 Sensor Configuration and Set Up

The sensor, powered with 7.5volts, was normally set up with a temperature of 225°C and a DAC setting between 0 and 1000. These configurations were values suggested by MEI. The gas applied to the sensor was kept at a pressure of approximately 25psi with a flow rate of about 4 liters/minute. For the tests conducted for this thesis, with the exception of *in-situ* tests done at Middle Tennessee State University (MTSU), gas applied to the sensor was always at room temperature. Data was recorded by use of MEI software specifically designed for the MEMS sensor. The software recorded the sensor NOx counts and sensor temperature versus time. The calibration tests typically ran for approximately two hours.

A device called a gas divider controlled the rates of flow of NOx and air that were mixed to form the test gas. The gas divider was used to get the correct NOx concentration for each test, which was verified by also measuring NO and NO_2, which sum to yield NOx, with a MultiGas Analyzer (MGA). This Fourier Transform Infrared MGA was used due to its minimal support infrastructure along with its low operating costs and fast response times. The MGA can simultaneously measure many species including NO, NO_2, CO, and CO_2. The MGA

...contains a 200-cc, 5.11m effective optical path length multipass gas cell, operated at 300°F. The small volume of the gas cell is convenient for fast response data requirements. At 10-1/min flow, 99-percent gas exchange is achieved in 4 sec. The MGA sampling rate is variable, allowing for higher resolution measurements with longer scan times.[4]

3.2 Warm Up and Health Monitoring Parameters

The warm up time is defined as the amount of time required for the sensor null level to stabilize. During the warm up time, the counts rise with time without the presence of NOx. After the warm up time, the counts read is considered the base level, where there is no NOx present. It is imperative to know the warm up time in order to acquire accurate data. If one were to acquire the data prior to null level stabilization, it would include not only the relationship between NOx counts and NOxppm, but also an increasing number of counts due to warm up.

For the first test, the sensor was placed in the apparatus shown in Figure 4. The data for this test can be seen in Figure 5. The second test was conducted in the same manner, with one exception. The sensor in this test was left in open air to rule out any possible negative effects of a closed volume (see Figure 6.). It was thought, at one point, that thermal NOx, which is NOx created due to an increase in temperature, may be contributing to the continuous rise of counts with time. Noticing that the trend lines for both tests are very similar demonstrates that the rise in counts is not dependent on the volume of the apparatus, or on the creation of thermal NOx in the sensor. This makes sense, considering that thermal NOx is not significant in air temperatures below 2800°F.[16]

Many more warm up tests were conducted and are presented on Figure 7. This graph shows that while the trend lines vary considerably at the beginning of the scan, they are very similar 3500 seconds into the scan. It can also be seen that at about 4000 seconds that while the slopes do not appear to go to zero, they do decrease considerably. The remaining slope can be considered drift, which will be explained shortly. The warm

up time of the sensor is thereby concluded to be approximately 70 minutes at approximately 90% response.

3.3 Calibration

In jet engine exhaust, there are many different gases emitted including N_2, O_2, CO_2, CO, NOx, SOx, particulates, and others. Their concentrations depend on engine throttle settings or turbine inlet temperature. The MEI sensor that is being evaluated is sensitive not only to NOx, but also to CO concentration. The sensor counts rise with an increase of NOx, while the counts fall with an increase in CO. Because both NOx and CO are present in engine emissions, it is essential that the relationships between counts and NOx and CO levels be determined.

First, in the lab, the sensor was warmed up for approximately 70 minutes. Then, using the gas divider, the NOx concentration was slowly increased and measurements of the corresponding counts determined a relationship between counts and NOx concentration. For this test, the sensor was placed in the apparatus shown in Figure 8 and the DAC setting set to 500 to allow a higher measurement range. Figure 9 shows a photo of the lab installation at AEDC. A gas divider and a needle valve were used to select the concentration of NOx, which was then measured by the MGA. The counts signal from the MEMS sensor were then correlated with the NOxppm readings from the MGA, and graphed to find analytical relationships between MEMS sensor counts and NOx levels for different DAC setting levels. This experiment was repeated for DAC settings of 0 and 750.

For a DAC of 0,

$$Counts = 155.95*(NOxppm) + 786.28$$

For a DAC of 500,

$$Counts = 96.8*(NOxppm) + 767$$

For a DAC of 750,

$$Counts = 55.8*(NOxppm) + 523$$

These averaged to a linear slope of approximately 103 counts/NOxppm. These results are shown on Figures 10, 11, and12, respectively.

Assuming one chooses one DAC code for all testing purposes, a more specific calibration can be made. By combining many calibration curves at a DAC setting of 500, an average calibration was determined. As shown in Figure 13, there is a 100counts to 1 NOxppm relationship, after accounting for the y-intercept, with a precision of approximately 7%.

After the testing was moved from the lab to an *in-situ* test at MTSU, with the third test on-board the engine, the sensor packaging failed. Some of the wires to the electronics broke. After getting it back following repairs, it did not work as it had previously. Due to sensor failure, the relationship between CO levels and sensor counts was no longer feasible in the lab environment.

3.4 Drift

In general, the counts recorded by the sensor will continuously rise slightly even without an increase in NOx. This is called drift and is caused by SnO_2 grain boundary changes, which results in changes in sensor output as well as a reduction in sensor sensitivity.

In order to quantify drift, calibration data must be analyzed. To calculate drift, counts vs. time was measured after warm up by taking a measurement of counts and time at 0ppm, which gave the base for measuring drift. The NOx was then increased and measurements were taken again. Then dropping back to 0ppm NOx and measuring counts and time once more, comparisons were made of both measurements of counts and times at 0ppm NOx. The drift was determined by calculating the difference in counts at the base level over time. This process was repeated many times to get an accurate drift calculation. This process was repeated approximately 5 times per test and 4 tests were compiled to acquire the drift. From the graph in Figure 14, a drift of approximately 615 counts/hour or 10 counts/minute was calculated. A drift translates to roughly 6ppm NOx/hour.

Using this information, one can compare calibration schemes for NOx with or without the drift correction.

Without drift correction: $NOx = Counts \times CalSlope + CalIntercept$

Using data from Figure 25: $NOx = .01 \times Counts - 4.81$

If Counts is 2600, NOx is 21.2ppm

With drift correction:

$$NOx = (Counts - [\Delta Time * Drift]) * CalSlope + CalIntercept$$

Using data from Figure 13 and 14:

$$NOx = (2600 - [.467 * 615]) * .01 - 4.81$$

If counts is 2600, NOx is 18.3ppm

Taking into account drift, results in a 2.9ppm difference, which is also verified by Figure 25. This is a large enough difference that it is imperative that drift be taken into account during data analysis that relies on NOx calibration of the sensor.

3.5 Performance at Varying Chip Temperature Set Points

The sensor can be set to operate at any temperature within a range of temperatures. Because changing sensor temperatures alters the number of counts measured, the relationship between sensor temperature and counts must be identified.

The experiment took place in the apparatus shown in Figure 8. First, the sensor warmed up for approximately 70 minutes. The NOx concentration applied thereafter, 20ppm, remained constant throughout the test while the sensor temperature increased in 25-degree increments beginning with 225°C and ending with 375°C. The first test was done with a DAC setting of 500 and the second of 0. After plotting counts vs. sensor temperature, the relationships found were:

$$Counts = -44*(Temperature) + 16565 \quad \text{for a DAC of 500 and}$$

$$Counts = -37*(Temperature) + 15617 \quad \text{for a DAC of 0}$$

As shown on Figures 15 and 16, an increase in temperature causes a decrease in counts, hence a decrease in sensor sensitivity to NOx levels.

3.6 Performance at Varying Oxygen Levels

While testing for calibration of NOx, it was observed that the sensor appeared to take inaccurate data at higher levels of NOx. The sensor output also varied depending on the DAC setting. Because it was mentioned in discussion with MEI, that the sensor

required a certain amount of oxygen to function correctly, and the fact that increasing NOx levels decreased O_2 levels, it was decided to test the sensor performance at varying oxygen levels and constant NOx concentration.

For these tests, the sensor was placed in the apparatus shown in Figure 17. The sensor was allowed to warm up for approximately 1.5 hours. Following the warm up, both the air and NOx were introduced to the sensor. For the first test, the gas divider was set at 20%. This means that there was a mixture of 20% from the container supplying NOx and 80% from the container supplying air. Because the NOx bottle had a concentration of about 93ppm NO, the NOx level for this test was about 18ppm NO. The standard instrument used to measure this was the MGA. The goal was to keep this number constant while performing a test using separate N_2 and air supplies to change the N_2/O_2 rate of the dilution gas. This was accomplished by varying the settings of the corresponding N_2/air supply bottle needle valves. As was N_2 increased and the amount of O_2 decreased in the air mixed with the NOx, counts were recorded to determine the point at which the decrease in O_2 affected the sensor performance. An O_2 analyzer measured the actual O_2 concentration and the N_2 concentration was calculated.

Because drift exists, the counts rose throughout the test until the O_2 level tapered enough to affect the sensor performance. At this point, the counts began to decrease due to lack of oxygen. This test was repeated with differing NOx concentrations and using different DAC settings, though these alterations proved unrelated to oxygen level required to affect the MEMS sensor output. The minimum percentage of O_2 required in the gas to affect sensor output is shown in Table 2 and Figure 18, to be approximately 12%.

3.7 Measurement Range

In order to determine proper applications for the sensor, the measurement range for NOx must be known. It is also important to know the measurement range to ensure accuracy during testing and data analysis. Applying 500ppm NOx to this sensor, for example, would saturate it (saturate its output) and give inaccurate data. This is known because initially a bottle of NO with a concentration of 500ppm was used. It was quickly changed out in favor of a 91ppm NO bottle due to the near-immediate saturation of the sensor, and the extended time required to bring the sensor back to its base reading.

The MEMS sensor has the option of being set to many different DAC settings, which control the measurement range of the sensor. A few of these DAC settings were tested, specifically DACs of 0, 500, and 750. At each of these settings, using a gas divider, a needle valve, and a bottle of 91.3ppm of NOx, the NOx concentration in the sample gas was increased slowly and the corresponding sensor counts were recorded. While the sensor measures NOxppm correctly, a linear relationship between NOxppm and counts exists. The measurement range peaks at the point where this relationship ceases to be linear.

At a DAC setting of zero, a measurement range of 0-17ppm was found. For this entire test, there was a linear relationship and 17ppm of NOx was the peak because at ~4000 counts, the sensor saturates and increasing the NOx any more would have saturated the sensor. This relationship can be seen on Figure 10. Likewise, at a DAC setting of 500, the measurement range determined was 0-30ppm, as can be seen on Figure 11. For the DAC setting of 750, Figure 12 shows a measurement range of 0-40ppm.

3.8 Recovery Time

Recovery time is defined as the time required for the sensor to return to a nominal base level or null after sensing a high NOx concentration. Knowing the recovery time is essential in determining the frequency with which one should take data. If, for example, a NOx concentration were applied to the sensor before it had a chance to recover, the sensor counts would be higher and without adequate time for counts to stabilize, it would not correctly represent the concentration of NOx. Not allowing for recovery would cause the data to be inaccurate.

Recovery time testing was performed using the apparatus shown in Figure 4. The sensor was allowed to warm up for 70 minutes and then a NOx concentration was applied, which saturated the sensor. After saturating the sensor, the needle valve supplying the NOx was closed to allow the sensor counts to return to null. After graphing the counts vs. time, which can be seen on Figure 19, the recovery time was determined by taking the average of the two recovery times. The recovery time was found to be approximately 11 minutes for the case where flow was no longer passing over the sensor. This was a test of the worst-case recovery time. A real-case test was performed at MTSU and is explained in the next section.

4.0 UTILIZATION OF SENSOR ON A JET ENGINE

4.1 Performance on a Jet Engine

The sensor was taken to the Middle Tennessee State University Airport for testing on a JT-12 aircraft gas turbine engine, which was provided by the MTSU Aerospace Department. The JT-12 is a 3,000-lbf-thrust class engine and typically produces emission levels of 3.2 percent CO_2 ,1050 ppm CO, 16 ppm NOx, and a smoke number of 26 at 45% engine power. [4]

The JT-12 has four engine mounted probes that were inside the engine at the turbine exit and fed an external manifold on which the MEMS sensor was mounted. This assured that the sensor received a representative exhaust sample, which was directly comparable to the ganged-probe sample collected at the nozzle exit. The gas-sampling probes are shown in Figures 20 and 21. The MEMS sensor, attached to the manifold exit, had a vacuum pump attached to the end of the sensor apparatus, which drew an accurate sample of engine exhaust gas across the sensor. A schematic is shown in Figure 22.

This was an extremely important part of the sensor testing because there are many other factors that affect the sensor operation when installed on a jet engine, that are not seen in a lab. For example, in the lab, the influence of CO on the sensor output was not measured. Also there was no heated gas supplied to the sensor in the lab, whereas the emissions from an engine are at approximately 375°F. In this way, *in-situ* testing shows the influences that affect the MEMS sensor that have not been seen by testing in the lab, and give perspective on the sensor testing that remains. Another major reason that testing on an engine is so important is because it is the best way to test the sensor in a harsh environment typical of its intended applications.

For the first few tests, the MEMS sensor was mounted onto a heated valve box, as shown in Figure 23, with heated gas lines running to an MGA. At each engine power level, the MEMS sensor counts were measured and compared to the data collected by the MGA. After the results showed that the sensor responded well to the engine NOx emissions, it was decided that in the next test the sensor would be placed on-board the engine.

The sensor was directly attached on-board the bottom of the engine, as shown in Figure 24, with a vacuum pump attached to the apparatus to ensure a good sample flow. The sensor was set to a temperature of 225°C with a DAC setting of 0. There was also an MGA measuring the engine emissions as a check for the sensor. A graph showing a comparison between NOx measured by the MGA and MEMS sensor is shown in Figure 25. After the sensor completed its warm up, the engine was powered up to 45%, which was the baseline power level. The engine was increased in peak power in 5% increments up to 75% peak power, before returning to 45% power. NOxppm was measured and recorded with corresponding power levels. After the test, a graph of NOxppm vs. MEMS sensor counts was made.

When this graph was compared with a similar graph based on lab data, a substantial difference was seen; the counts at MTSU were much lower than those from lab testing. This difference was accredited to the presence of CO in the exhaust gas supplied to the sensor during the engine emission test because while an increase in NOx causes the counts to increase, a rise in CO causes the counts to decrease. Because there existed the presence of CO in the exhaust, the number of counts per ppm was lower than it had been in the lab. The DAC setting, which is usually set to 500 in the lab, had to be

changed to 0 for most of the MTSU tests to lower the measurement range. Because the MGA also measured the CO emissions from the engine, a correction had to be determined for the MTSU NOx data for the presence of CO according to both the sensor data and the MGA measurements. This data can be seen on Figure 26, and the calculations that determined the correction for CO levels are shown in Appendix C.

The temperature of the MEMS sensor for the tests at MTSU of the sensor was set at 225°C or 437°F. The gas temperature was not higher than this, as shown in Figure 27, so, according to MEI, it did not affect the measurements. If the temperature of the gas exceeds the temperature of the sensor, the sensor temperature becomes that of the gas and this causes a difference in the data recorded, as shown by lab tests that altered sensor temperature.

Depletion of oxygen was not a concern for these tests. An MGA monitored oxygen levels and there was greater than 12% oxygen present for each increasing power step of the engine. Oxygen present for engine power steps is shown in figure 28.

The sensor recovery time for the MTSU tests was substantially lower than that in the lab. Recovery time at MTSU was approximately 200 seconds or 3 minutes 20 seconds. This was a result of a couple factors that differed from lab conditions. The factor that had the greatest effect on reducing recovery time was the presence of CO, which depresses the counts. Also, a vacuum pump was attached to the sensor apparatus, which brought clean air across the sensor, whereas in the lab, the NOx exited the apparatus without the use of a vacuum pump, which caused it to take longer.

4.2 Sensor Time Constant

The time constant, as defined in equation (1), is the speed of response of the sensor. It is important to know the time constant to ensure that data is not taken too quickly. If data is taken faster than the time constant, it will not be accurate.

This test was conducted with the sensor on-board the engine. There was also a gas sample line connected to an MGA to record NOx and CO levels. The engine was initially running at 45% power, and then increased by 5% increments up to 75% power, while returning to 45% between each increase. The data for this is shown on Figure 29.

To find the time constant, this equation was used:

$$[\text{Counts}(t) - \text{Counts}(\infty)]/[\text{Counts}(0) - \text{Counts}(\infty)] = e^{-t/\tau} \quad (1)$$

Where t is recovery time and τ is the time constant.

A calculation for the time constant for 55% peak engine power follows:

Let $t_{1/2}$ be the time for $[\text{Counts}(t) - \text{Counts}(\infty)]/[\text{Counts}(0) - \text{Counts}(\infty)] = ½$

$$t_{1/2} = 50\text{sec}$$

$$\tau = -t_{1/2}/\ln(1/2) = -50/-0.693 = 72.2\text{sec}$$

This calculation was repeated for each power step and an average time constant of approximately 75 seconds was found.

4.3 Durability and Longevity

The sensor functioned for approximately 200 hours in a year, including three tests on-board the jet engine at MTSU. While there were a few problems with the electronics

packaging, the sensor did not start to malfunction until after the third MTSU test, where it was exposed for the third time to the harsh environment of jet engine exhaust.

The Pt004 sensor and electronics cost about $3000 ($400 for sensor and $2600 for electronics and software). Assuming that the electronics and software are very durable and last through multiple sensors, the sensor costs are estimated at $400 per year. For 5 years, the sensor budget would be approximately $2,000. Comparing this to a standard NOx gas analyzer, which costs approximately $12,000 every 5 years, one would save about $10,000 using this MEMS sensor.

5.0 SUMMARY AND CONCLUSIONS

5.1 Summary of Results

The first tests revealed a warm-up time of seventy minutes. While this may be too long for the flight application, because it should take no longer than a few minutes to warm up and be ready for flight, the sensor would be very useful in other testing applications, where fast warm up time is not required. In fact, 70 minutes is average for a gas sampling system. Along with a slow warm up time, there is approximately 6ppm/hour of drift associated with the sensor. Even without a presence of nitrogen oxides, the counts continue to rise, which could cause inaccurate data or require continuous calibration of the sensor. It was also determined, by applying different oxygen concentrations, that the sensor requires at least twelve percent oxygen in order to perform correctly. This isn't a big problem because most of the measurements will be conducted at cruising speed or at 45% power, where there is enough oxygen present. Because the sensor software allowed for different DAC settings, the sensor was able to measure a range of 0-40ppm of nitrogen oxides. Also, because the application of the sensor would be typically measuring NOx at 45% power of the engine, the DAC setting would not require alteration during flight.

One of the biggest problems with the sensor is its calibration. The calibration has an approximate error of 7% due to the inconsistency of the sensor measurement. First, the warm up each day is different from the previous day. While the warm-up time is usually about the same, the number of counts that increased during that time is inconsistent. Even after warm-up, the number of counts corresponding to a concentration

of NOx applied is not the same from one day to the next. This is due in large part to drift. It would not be practical for one to have to calibrate the sensor prior to each flight.

The recovery time, which is measured at within 10% of null level, is much too long as well. At a recovery time of nearly 11 minutes for the no-flow condition, and ~3 minutes for the flow condition, it is consistent with the time constant calculations. Using a value of .1 for [Counts(t) – Counts(∞)]/[Counts(0) – Counts(∞)] and 75sec for τ, gives a recovery time (t) of 173sec or 2.9 minutes. With a recovery time of 3 minutes, this sensor would not be able to take data quickly enough to measure emission levels, with the goal of altering power settings to run at optimal emissions. It would, however, do well to measure the average NOx emissions about a steady state operating point as with aircraft engines at cruise conditions.

The sensor measurements for a certain concentration of nitrogen oxide change with differing temperature set points. Most of the testing was done at 225°C. This should not be a problem once each temperature setting is tested and calibrated. It also is not an issue as long as the gas temperature does not exceed the sensor temperature setting.

When mounted to the jet engine, the sensor functioned well, considering its limitations. The counts rose linearly with the increase in engine power and nitrogen oxides concentration. However, *in-situ* calibration is required so that some alternate measurement system, such as an MGA would be needed near where the sensor would be located. Alternatively, an improvement in drift of the sensor is required.

5.2 Conclusions

This MEMS sensor is a good starting point for its objective. There exists a relationship between its counts and the concentration of nitrogen oxides. However, this relationship is difficult to determine with any consistency. Also, the sensor does not react quickly enough to take decent data on-board a jet engine in order to alter power settings to optimize emissions. As it stands, the sensor could be a useful and inexpensive development tool. With continued development, testing, and software development for interpreting sensor output, the sensor shows potential for monitoring jet engine emissions.

One of the greatest features of the MEMS sensor is its size and its cost. The Pt004 sensor and electronics cost about $3000. Assuming that the electronics last through multiple sensors, the sensor costs are estimated at $400 per year. For 5 years, the sensor budget would be approximately $2,000. Comparing this to a standard NOx gas analyzer, which costs approximately $12,000 every 5 years, one would save about $10,000 using this MEMS sensor.

REFERENCES

REFERENCES

1. J. Penner, D. Lister, D. Griggs, D. Dokken, M. McFarland, Aviation and the Global Atmosphere, Cambridge University, 1999.
2. G.W. Hunter, C-C Liu, and D.B. Makel, The MEMS Handbook, CRC Press LLC, 2002.
3. J.H. Visser and R.E. Soltis, "Automotive Exhaust Gas Sensing Systems," *IEEE Transactions on Instrumentation and Measurement*, Vol. 50, No. 6, December 2001.
4. Lee S. Langston and George Opdyke, Jr., "Introduction to Gas Turbine Engines for Non-Engineers," *Global Gas Turbine News*, Vol. 37: 1997.
5. SAE International Committee E31, Aerospace Recommended Practice 1256.
6. D. Marren, P. Kenny, J. Markham, P. Jalbert, R. Moyers, D. Gardner, "The Application of FT-IR Spectroscopy to Turbine Engine Exhaust Measurement", AIAA 2000-2211, 21st AIAA Advanced Measurement Technology and Ground Testing Conference, June 2000.
7. Asad M. Madni and Lawrence A. Wan, "Microelectromechanical Systems (MEMS): An Overview of Current State-of-the-Art," *IEEE*, 1998.
8. D.G. Gardner, V.A. Zaccardi, P.A. Jalbert, and M.D. Bryant, "Reducing the Cost of Aircraft Engine Emission Measurements," Jacobs Sverdrup AEDC Group.
9. Brian Kinkade, "Bringing Nondispersive IR Spectroscopic Gas Sensors to the Mass Market," http://www.sensormag.com/articles/0900/83/main.shtml, August 2000.

10. Gary Hunter, Liang-Yu Chen, and Phillip G. Neudeck, "Development of High Temperature Gas Sensor Technology," 42nd Turbo-Expo '97, June 2-3, 1997.
11. Brian Kinkade, James Daly, and Edward Johnson, "MEMS Device for Mass Market Gas and Chemical Sensors," Ion Optics, Inc.
12. J.T. Woestman and E.M. Logothetis, "Controlling Automotive Emissions," American Institute of Physics, 1995.
13. Dae-Sik Lee, Jun-Woo Lim, Sang-Mun Lee, Jeung-Soo Huh, and Duk-Dong Lee, "Fabrication and Characterization of Micro-Gas Sensor for Nitrogen Oxides Gas Detection," Elsevier Sciences, 2000.
14. Asad M. Madni and Lawrence A. Wan, "Microelectromechanical Systems (MEMS): An Overview of Current State-of-the-Art," *IEEE*, 1998.
15. Rajeshuni Ramesham, Reza Ghaffarian, and Namsoo P. Kim, "Reliability Issues of COTS MEMS for Aerospace Applications," *SPIE* Vol. 3880, September 1999.
16. http://www.mindspring.com/~tfmcgowan/presentation/nox.ppt

APPENDICES

APPENDIX A

FIGURES

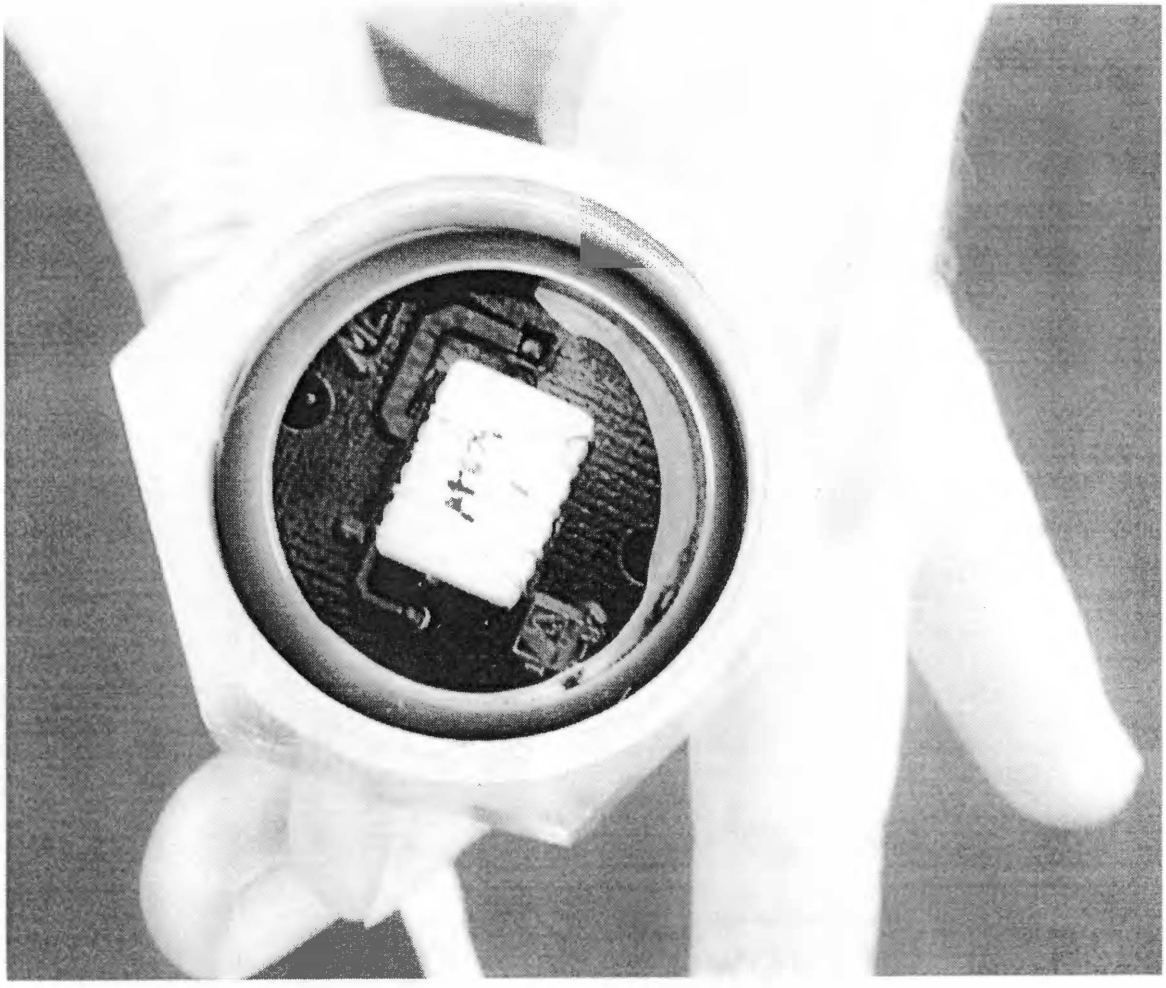


Figure 1. MEMS Sensor

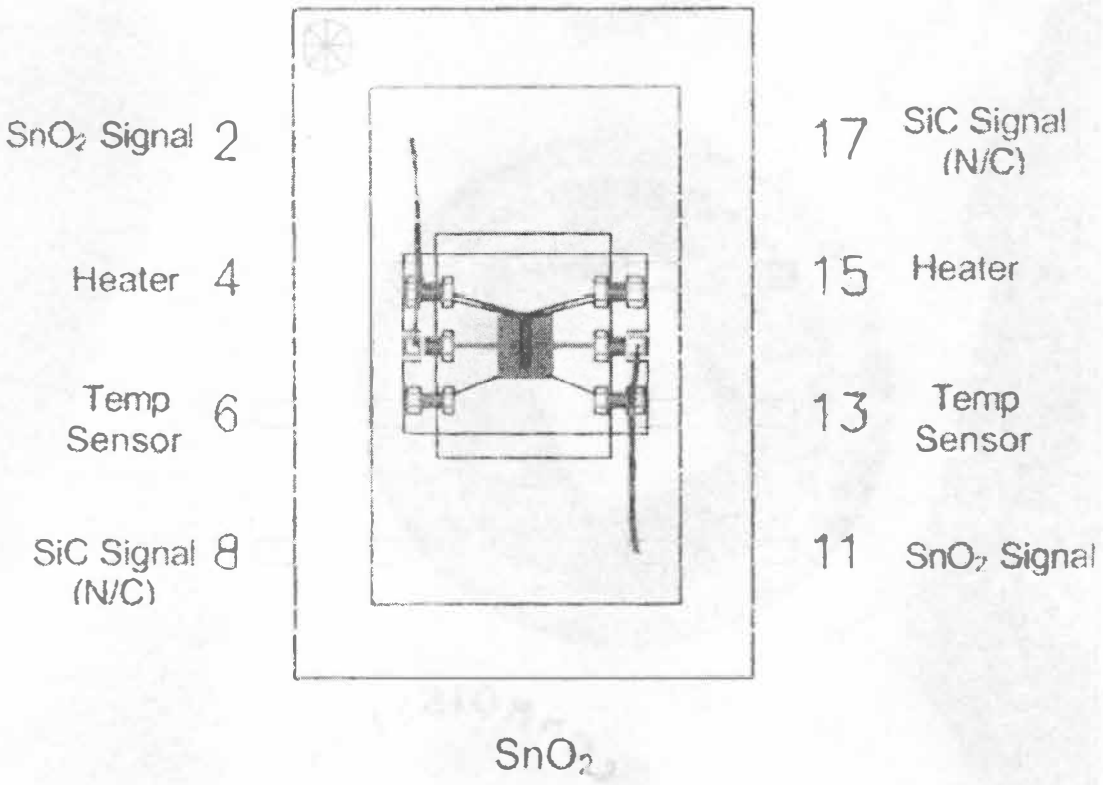


Figure 2. Sensor Package for SnO_2 Sensor Elements

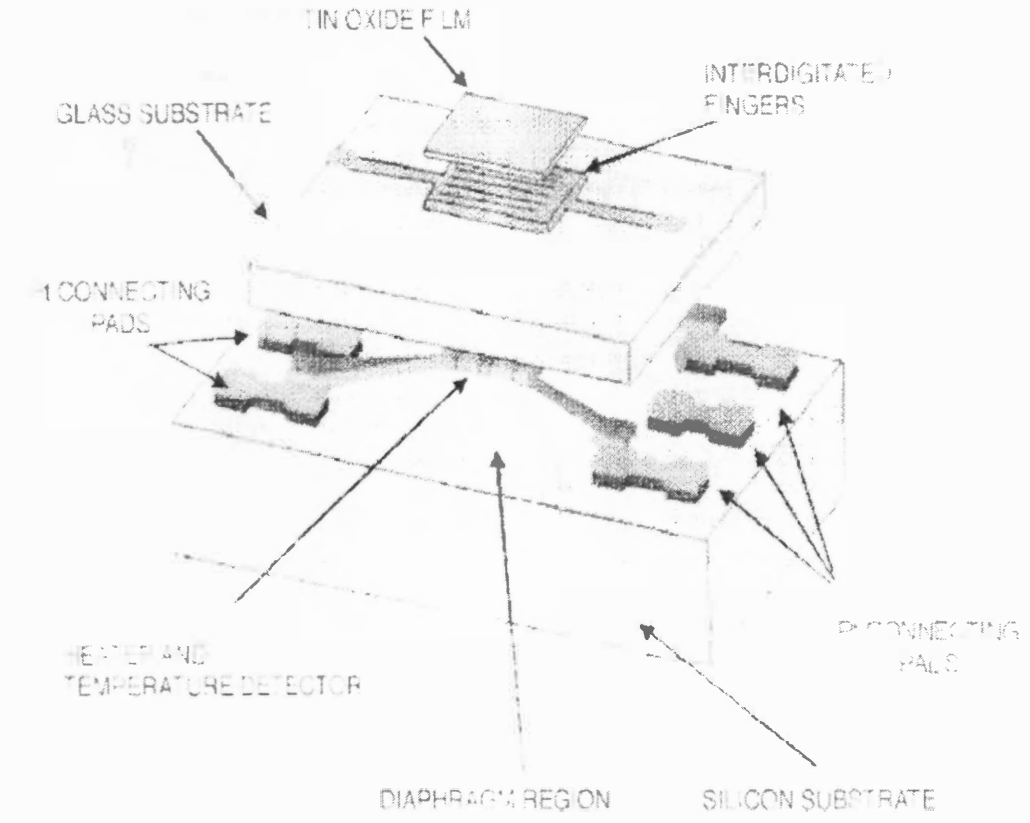


Figure 3. Structure of SnO_2-Based Sensor on a Si Substrate [2]

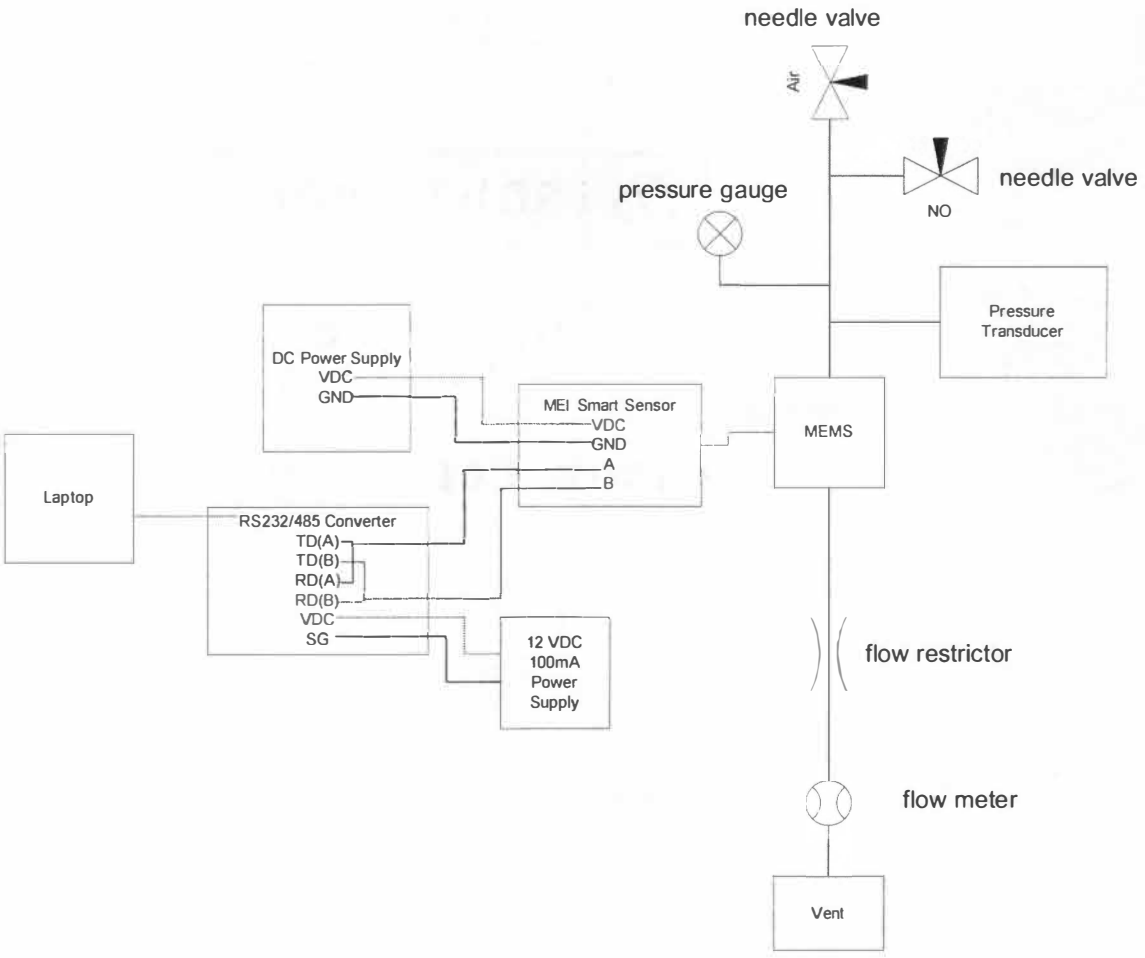


Figure 4. Apparatus Used for Sensor Warm Up Tests in the Lab

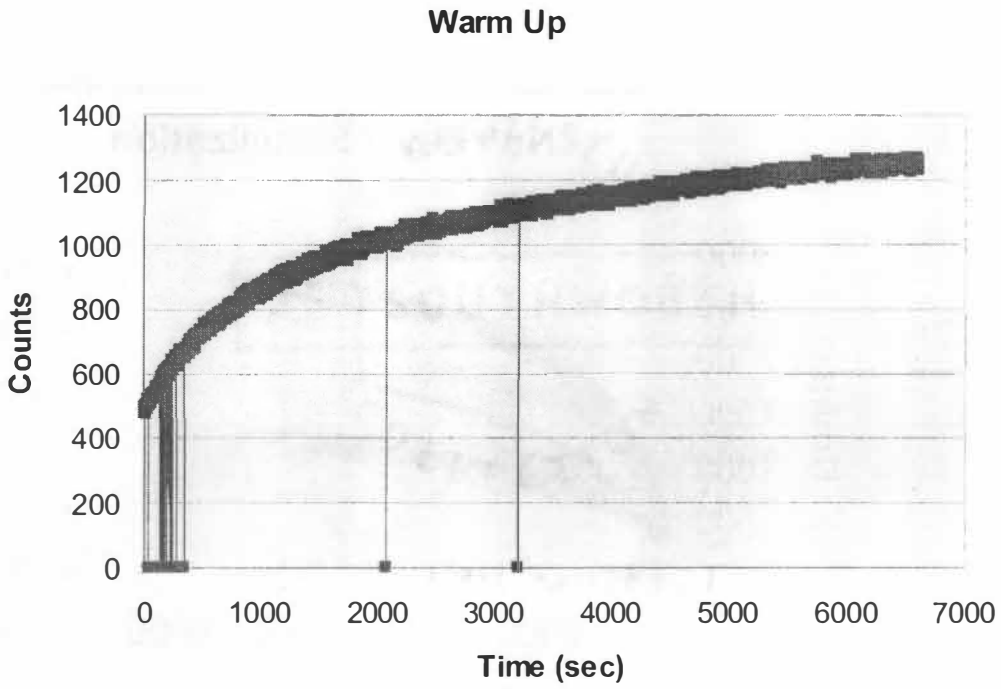


Figure 5. Warm-Up

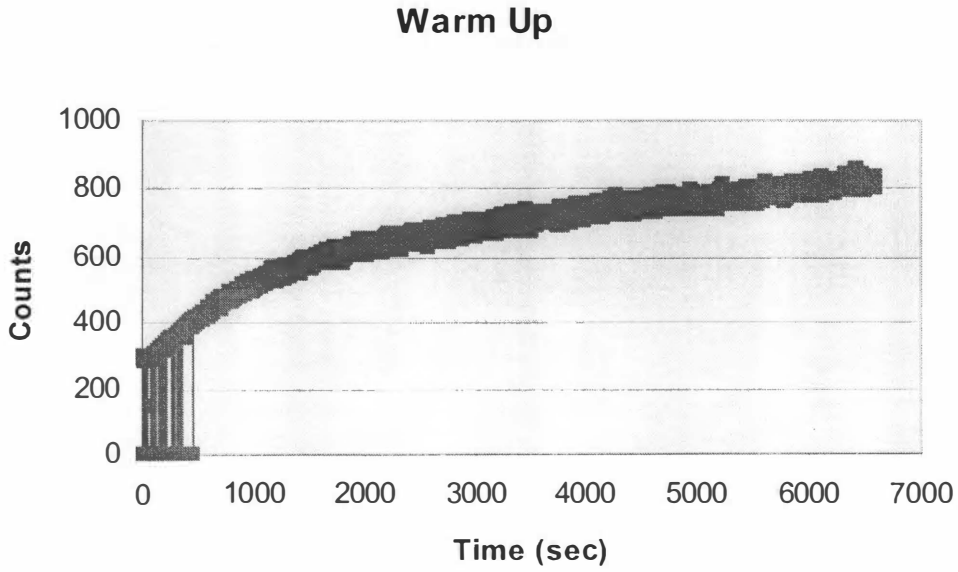


Figure 6. Closed Volume Warm-Up

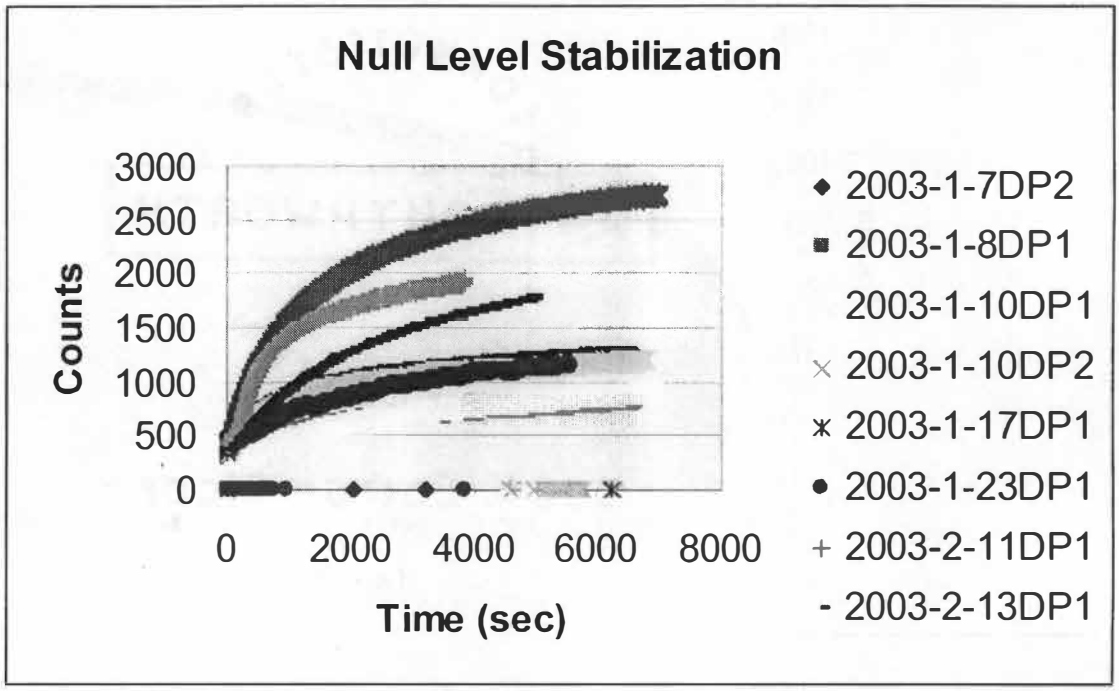


Figure 7. Warm-Up Times

All at DAC=0. Legend names are dates of each test. For example, 2003-1-7DP2 is a test on January 7, 2003 data point #2.

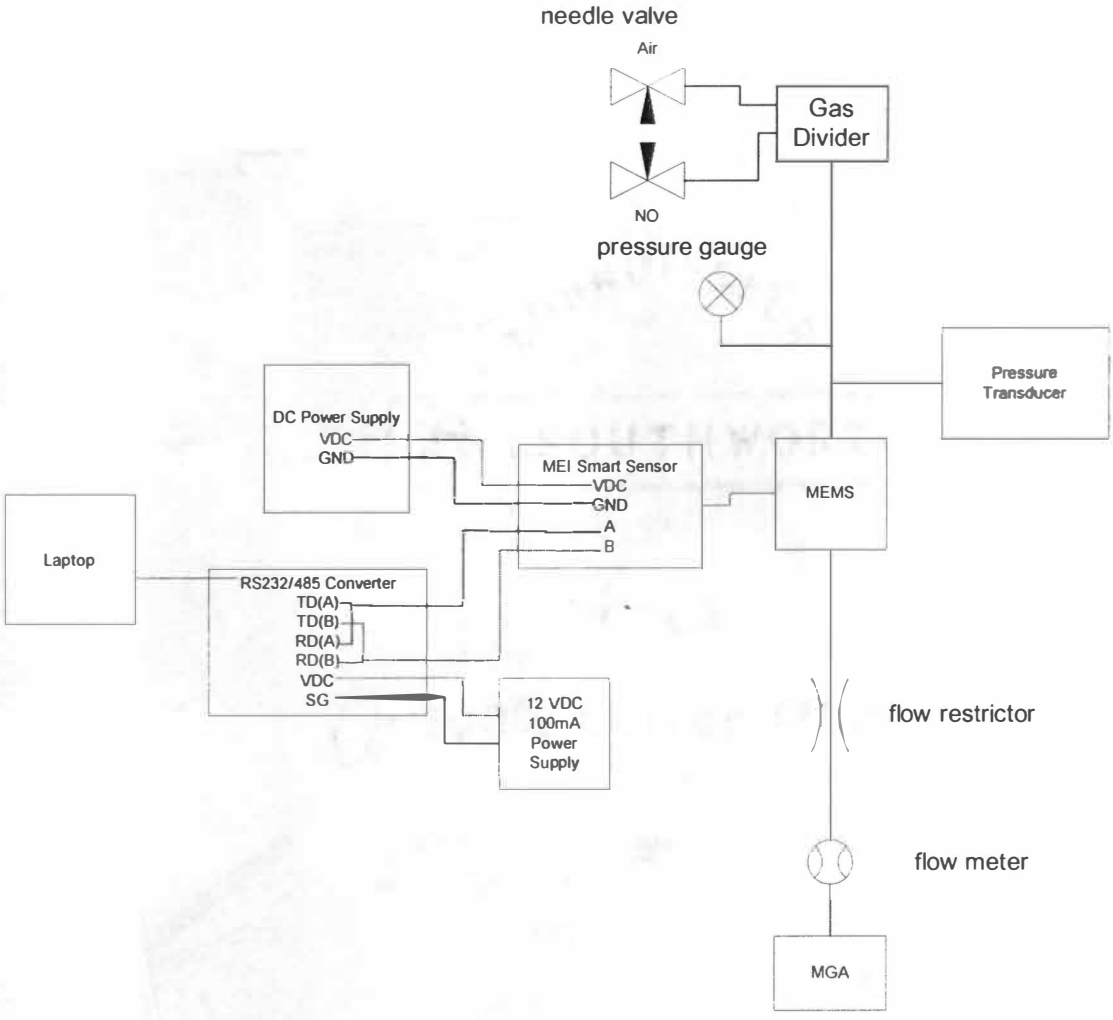


Figure 8. Apparatus Used for Sensor Calibration and Temperature Set Points Tests

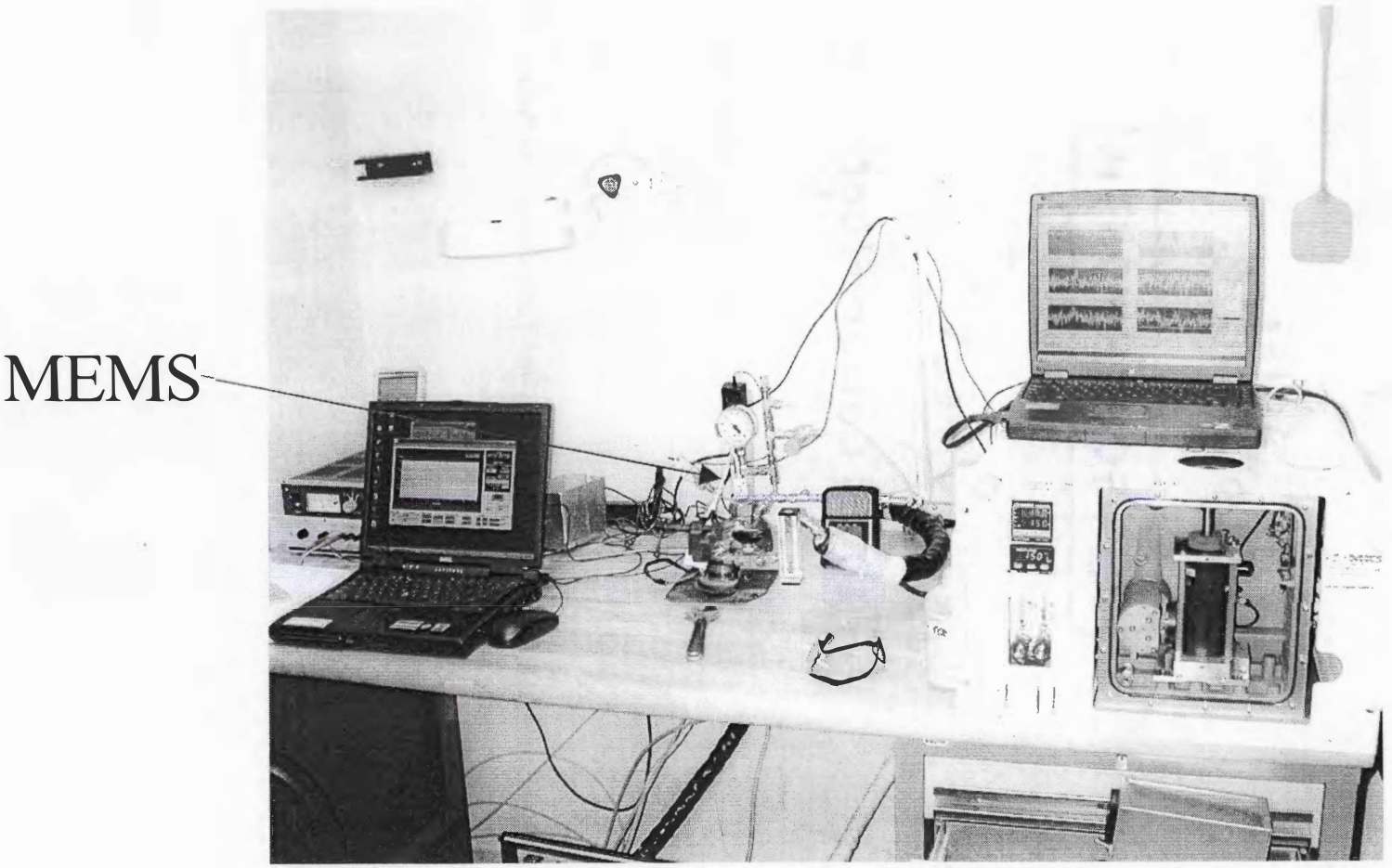
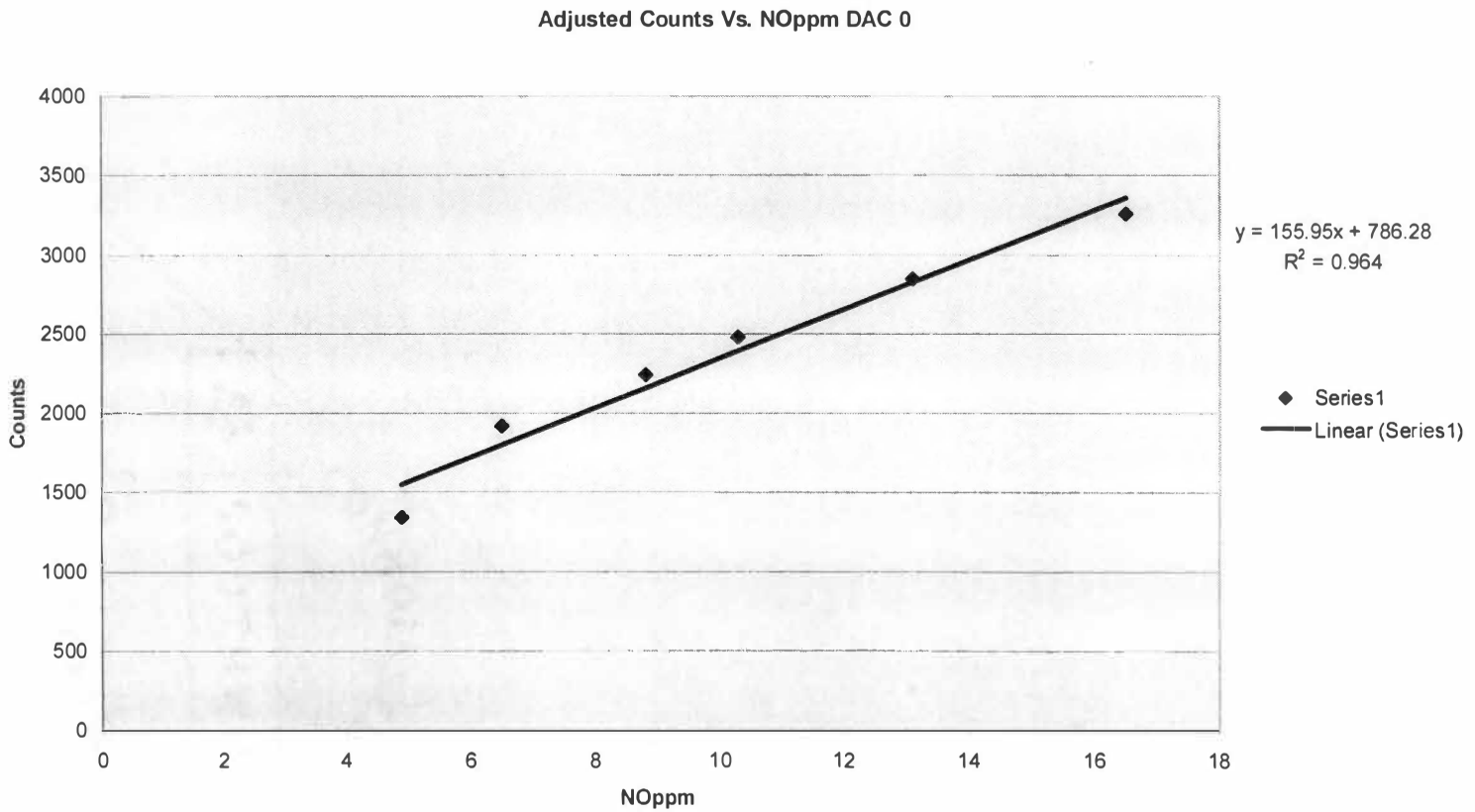
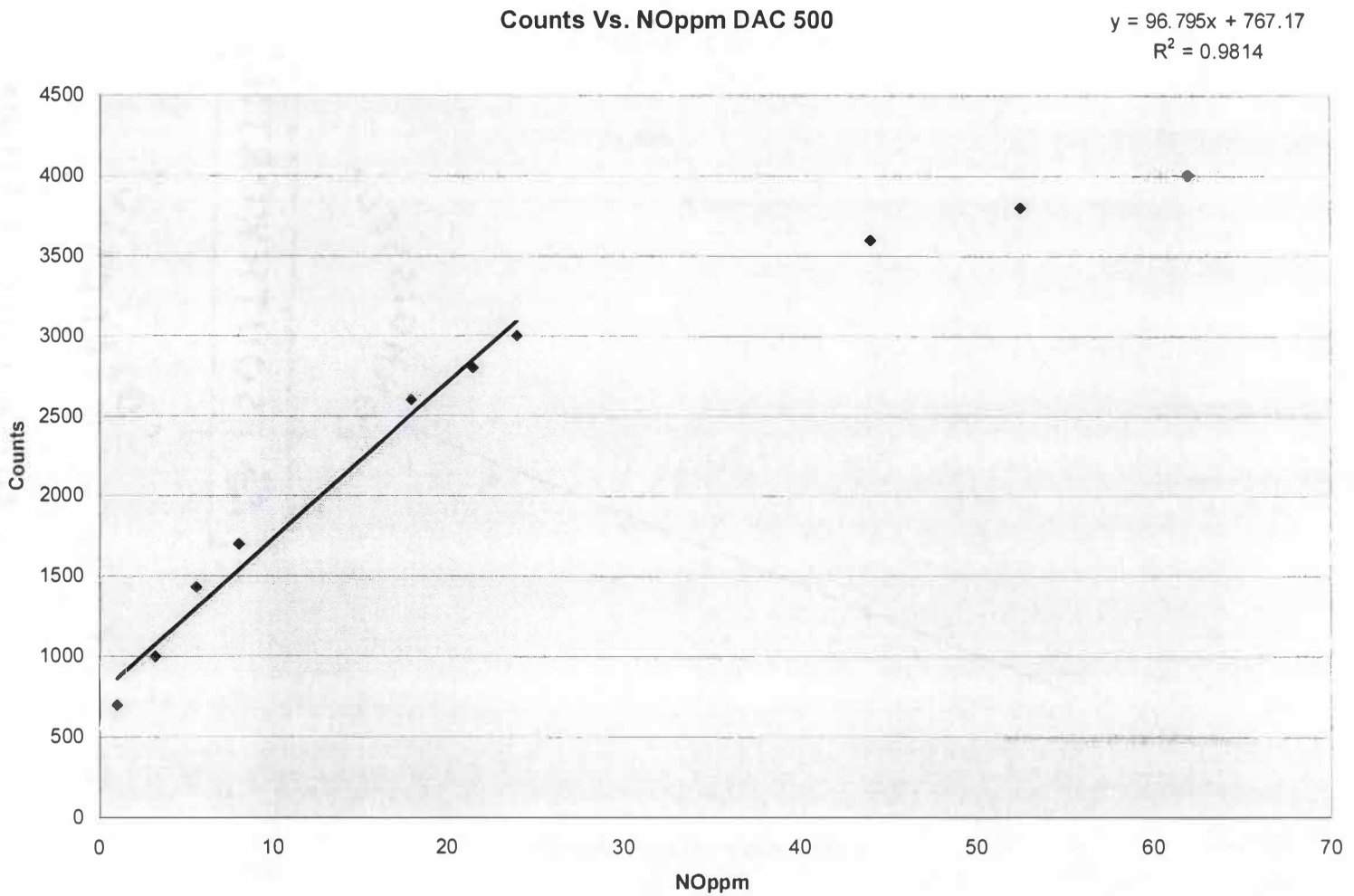


Figure 9. Lab Installation From Left: Power Supply, Laptop/MEI Software, MEMS Apparatus, MGA

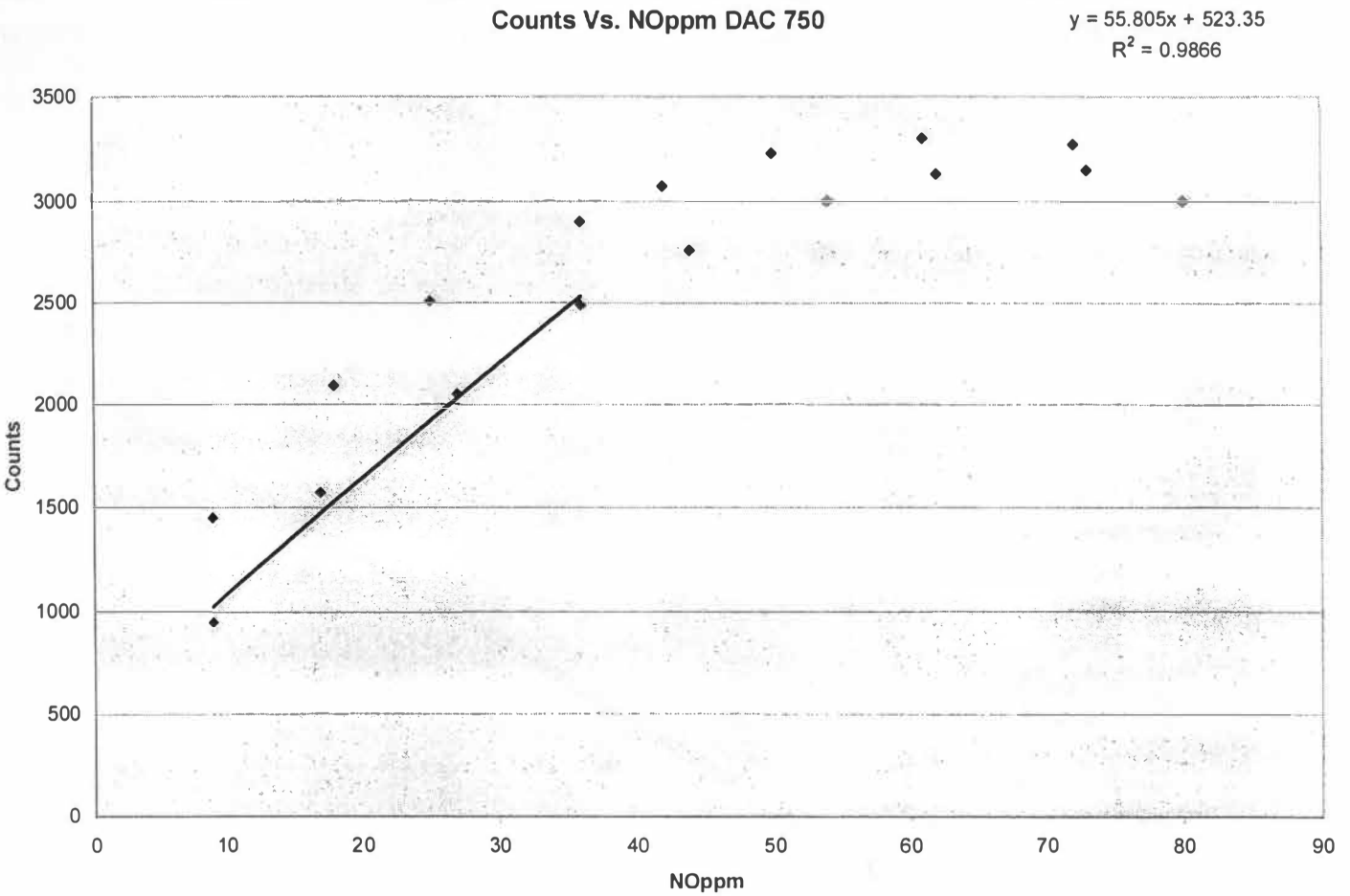


2003-4-21dp1

Figure 10. DAC 0 Calibration



2003-1-27dp3
Figure 11. DAC 500 Calibration



2003-1-29dp3

Figure 12. DAC 750 Calibration

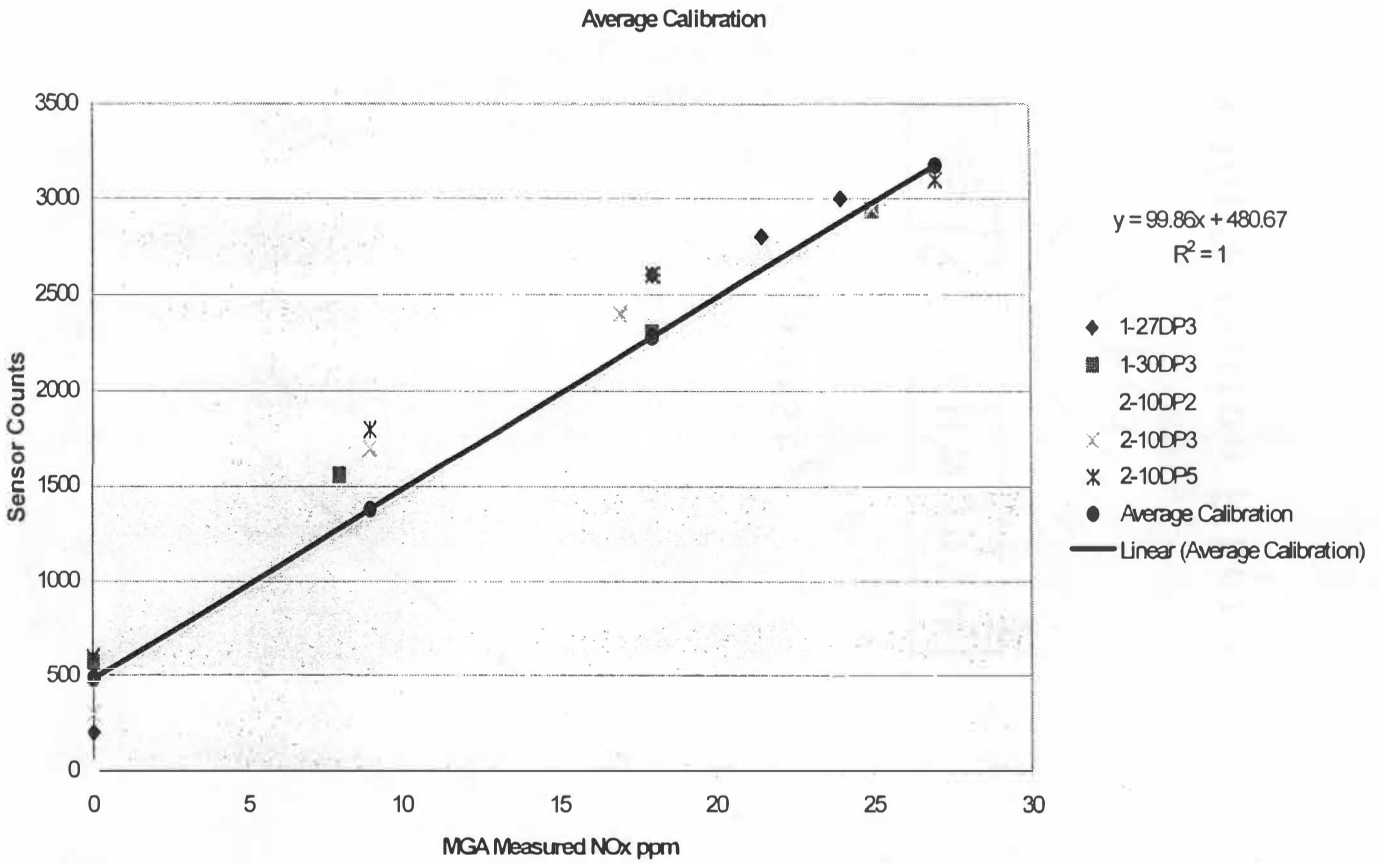


Figure 13. Average Calibration at DAC 500

Legend names represent test dates for each test. For example, 1-27DP3 was a test on January 27 for data point #3.

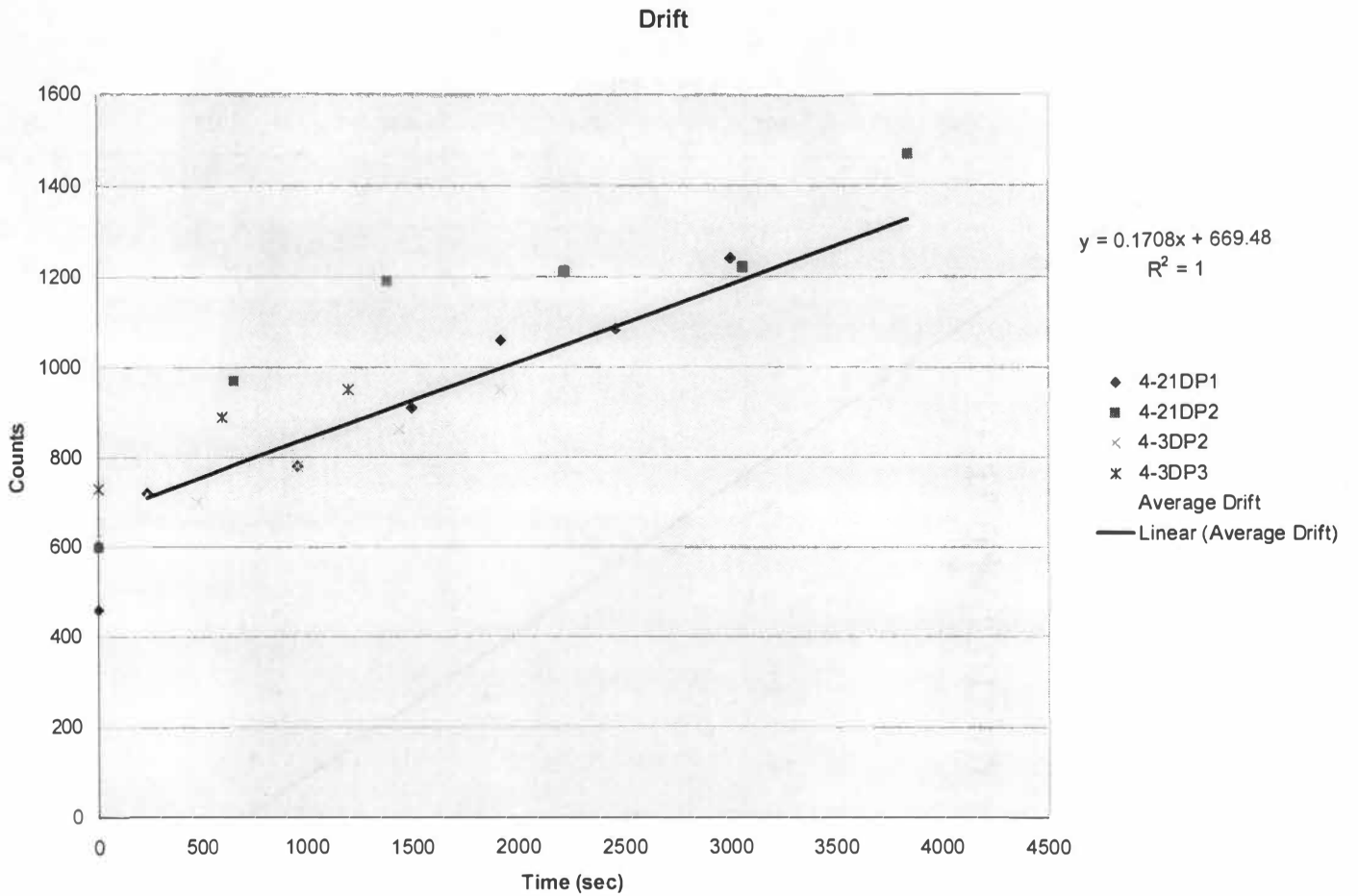


Figure 14. Drift

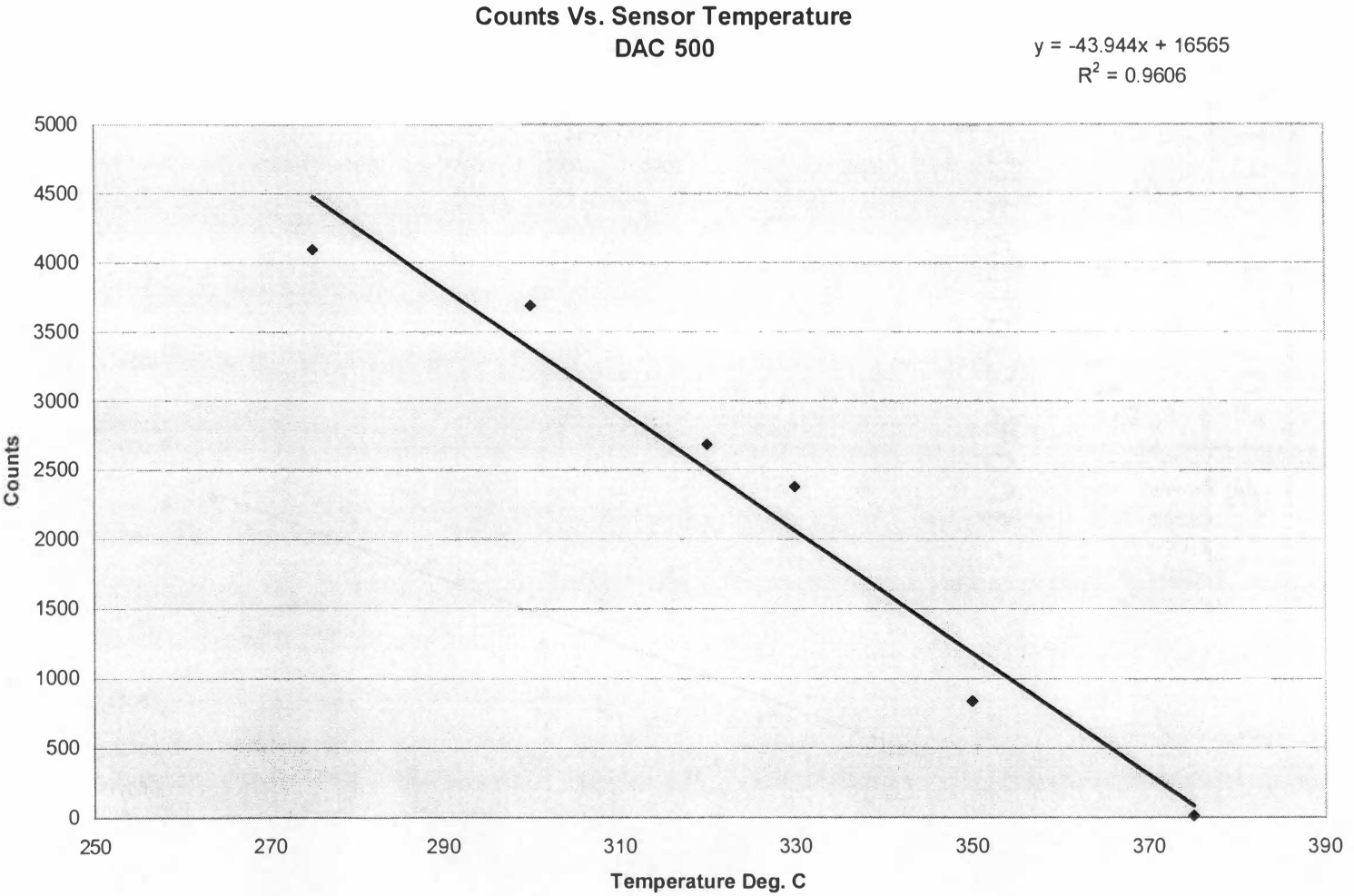


Figure 15. DAC 500 Counts Vs. Sensor Temperature Set Points

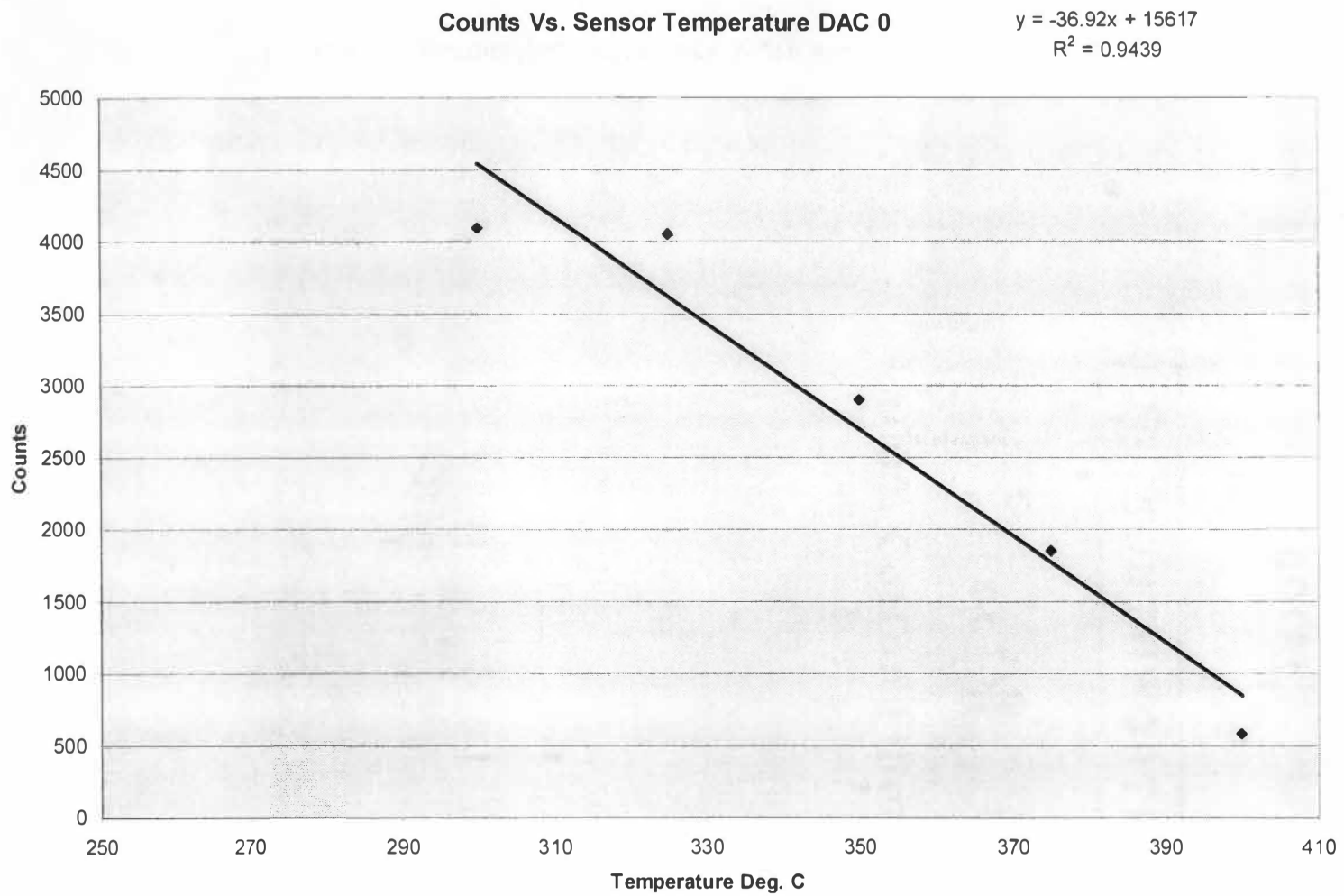


Figure 16. DAC 0 Counts Vs. Sensor Temperature Set Points

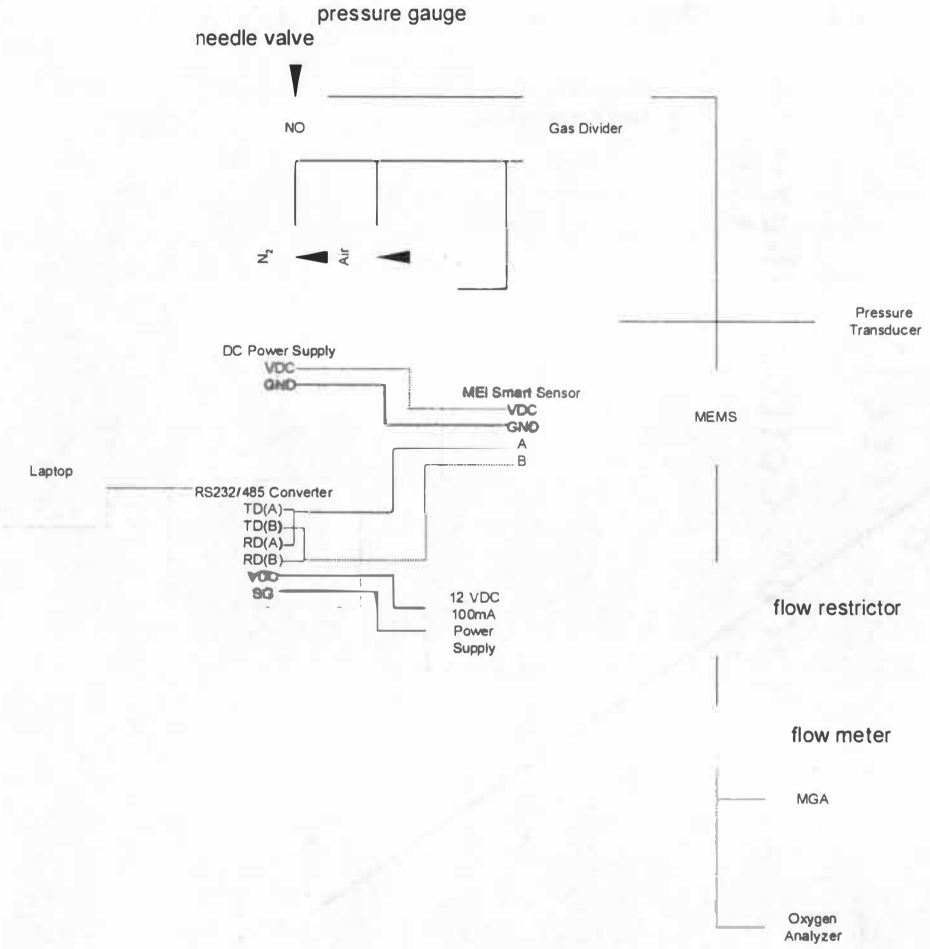


Figure 17. Apparatus Used for Sensor Oxygen Sensitivity Tests

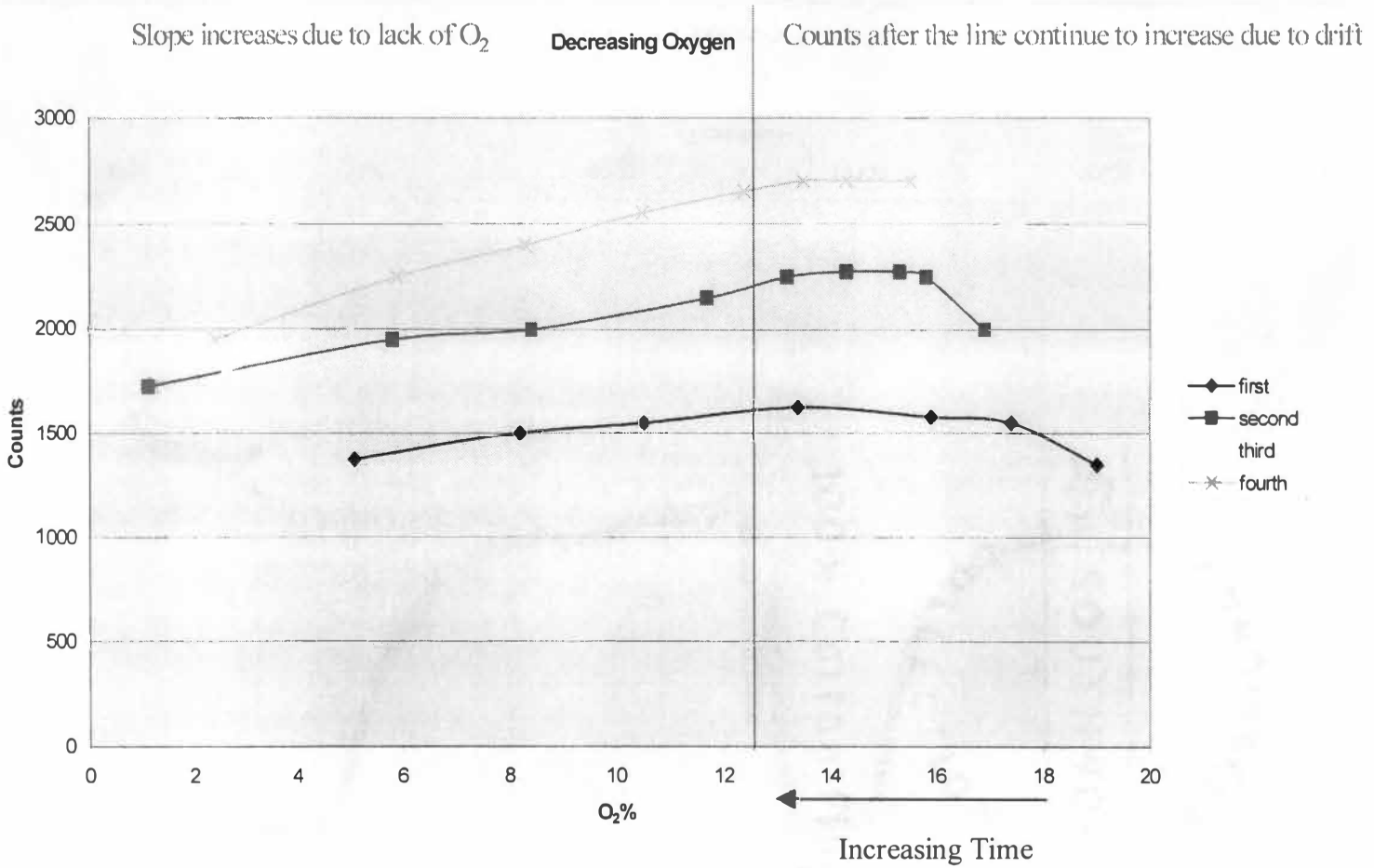


Figure 18. Required Oxygen Percentage

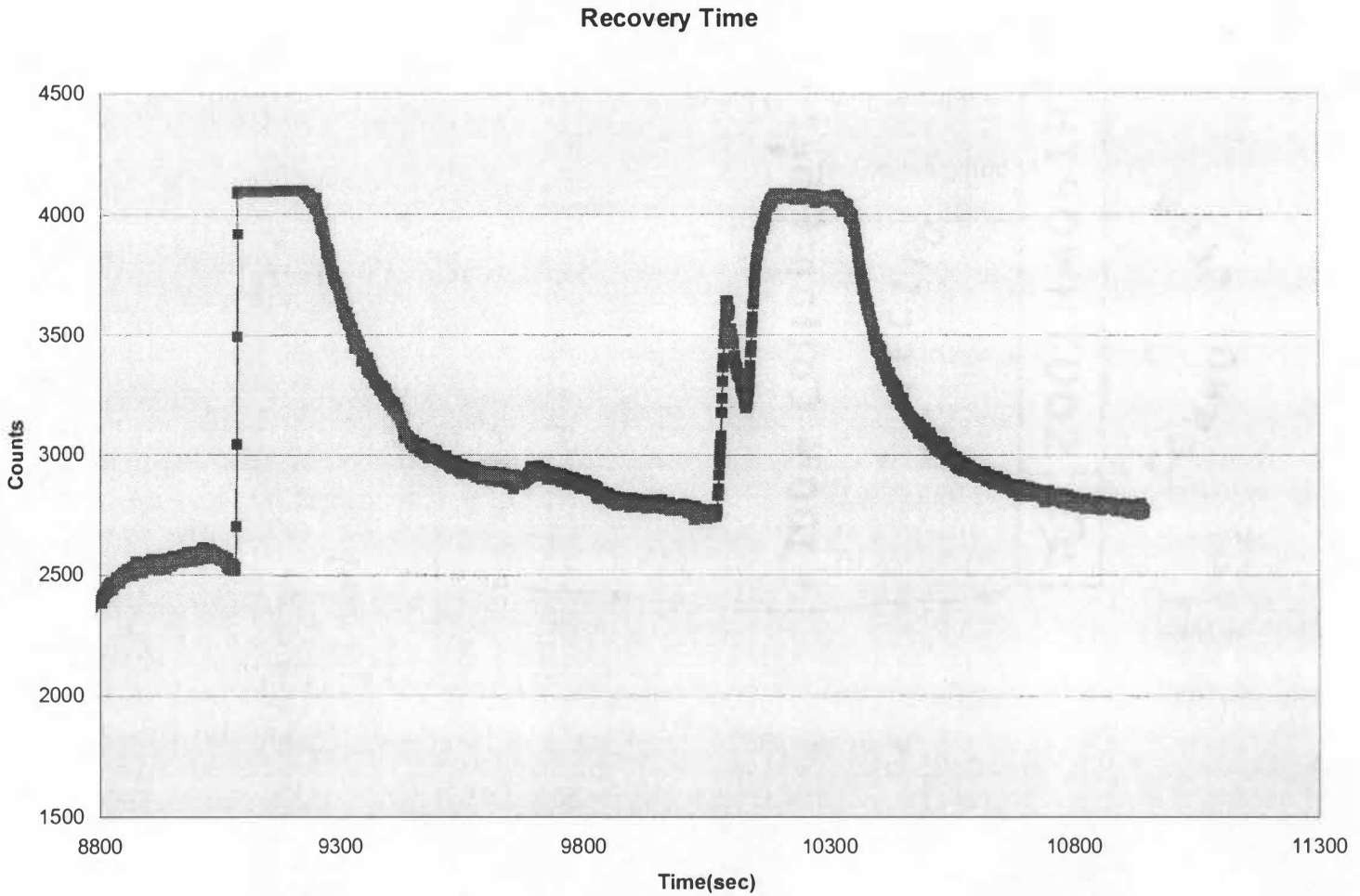


Figure 19. Recovery Time

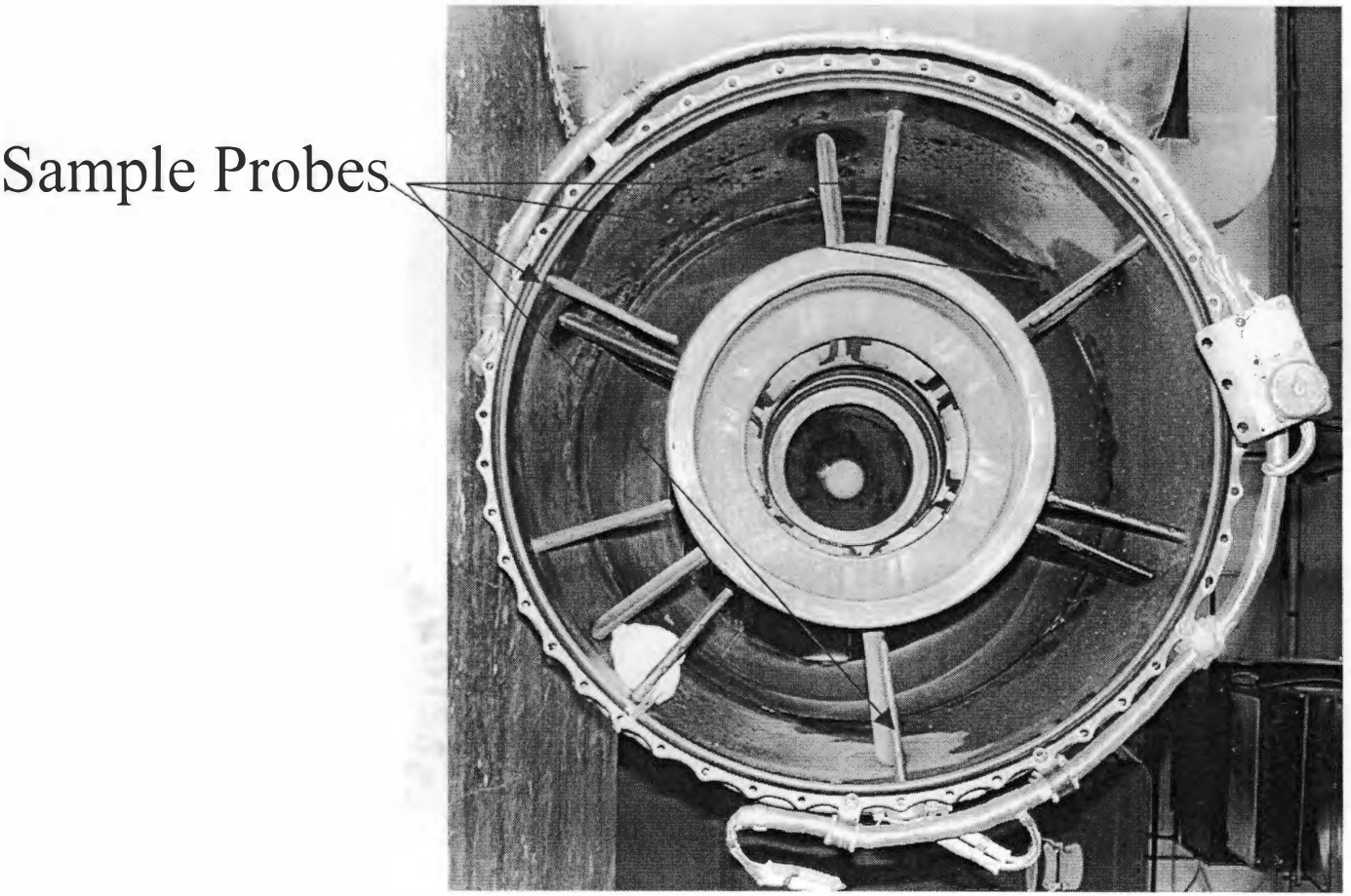


Figure 20. JT-12 Turbine Exit Probes: Viewed Forward Looking Aft (notice the 5 holes in each probe)

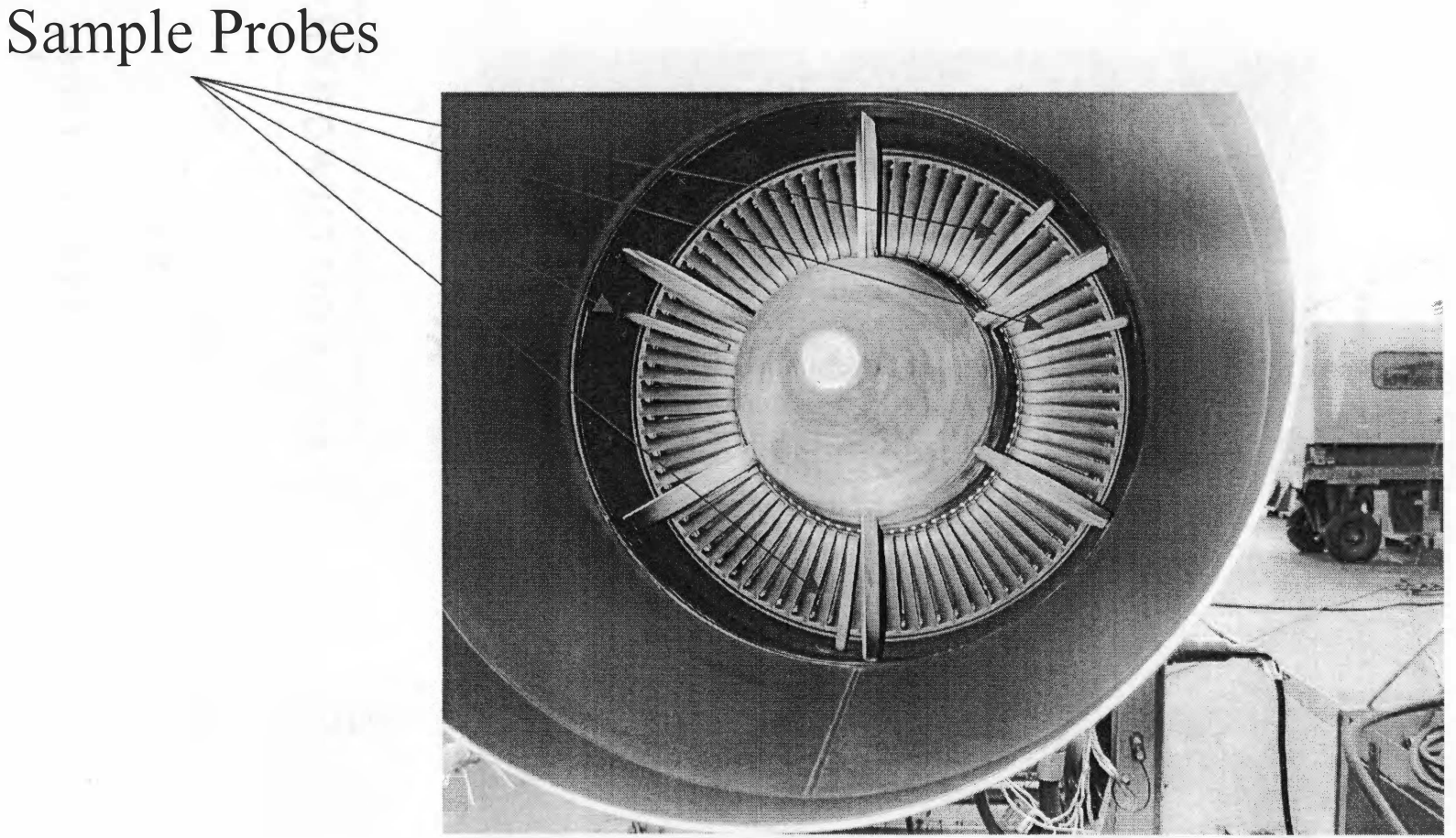


Figure 21. JT-12 Sampling Probes: Viewed Aft Looking Forward

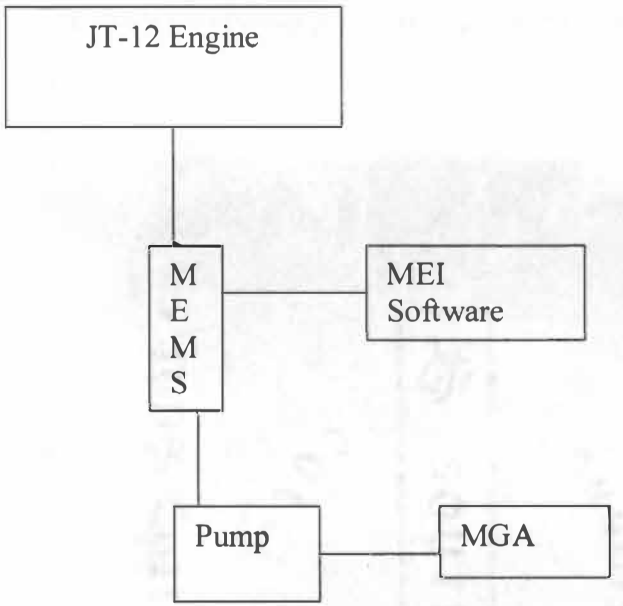


Figure 22. MTSU Schematic

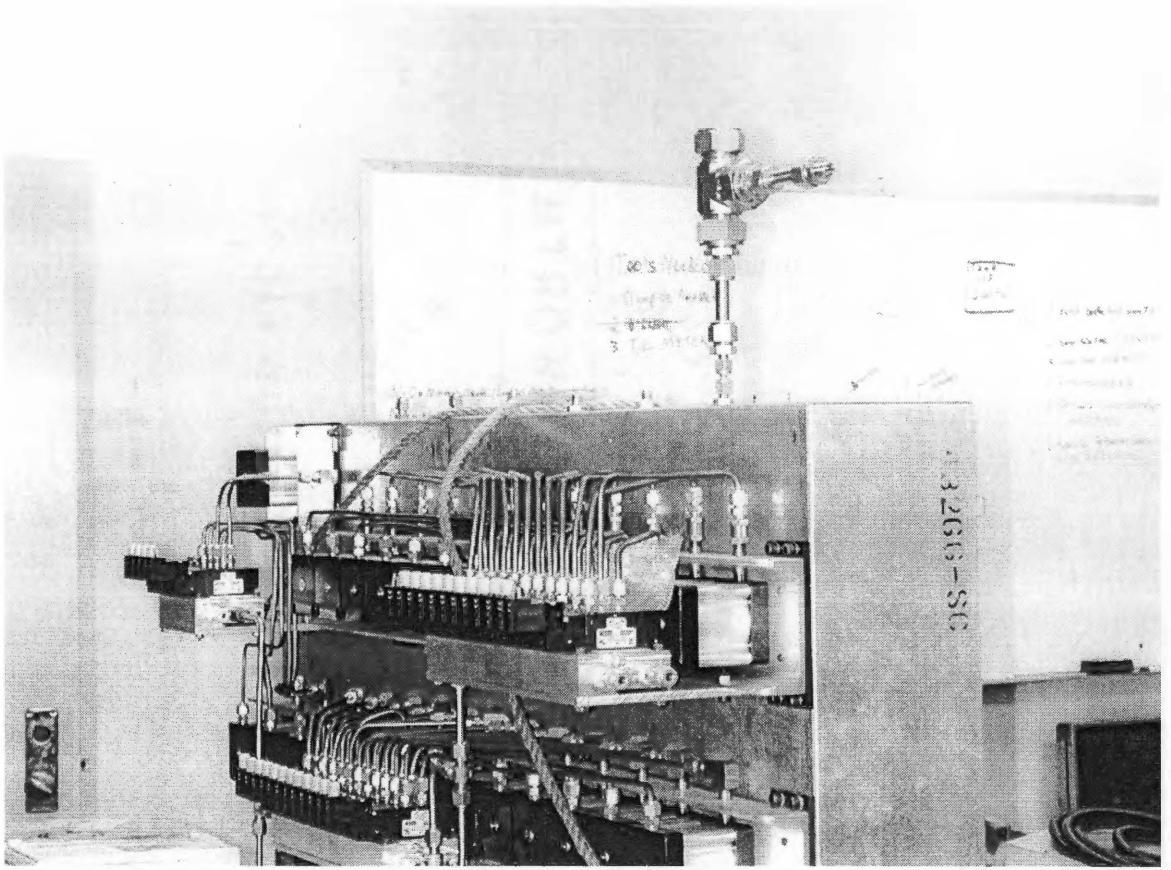


Figure 23. Heated Valve Box With MEMS Apparatus Attached

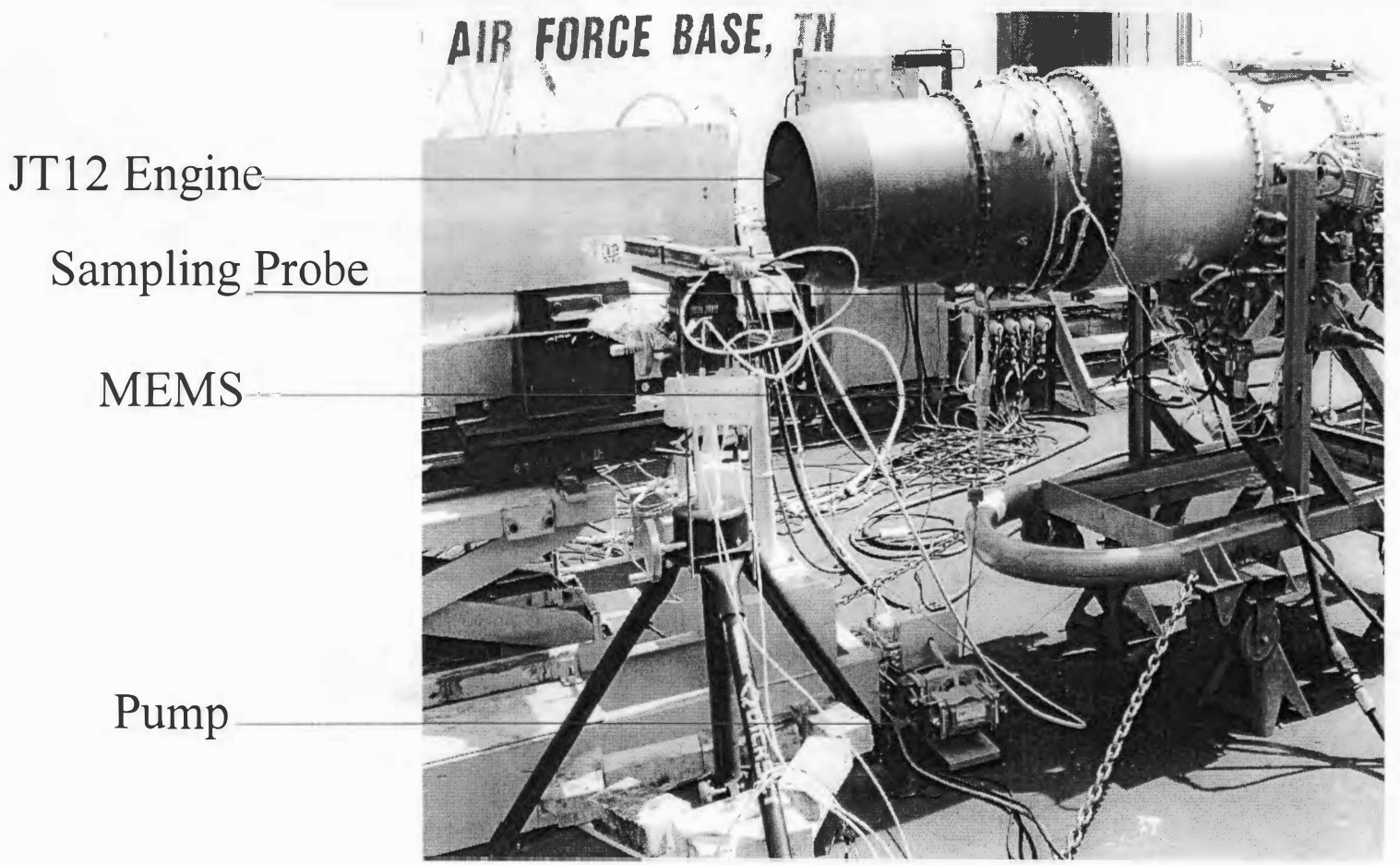


Figure 24. Field Test Apparatus

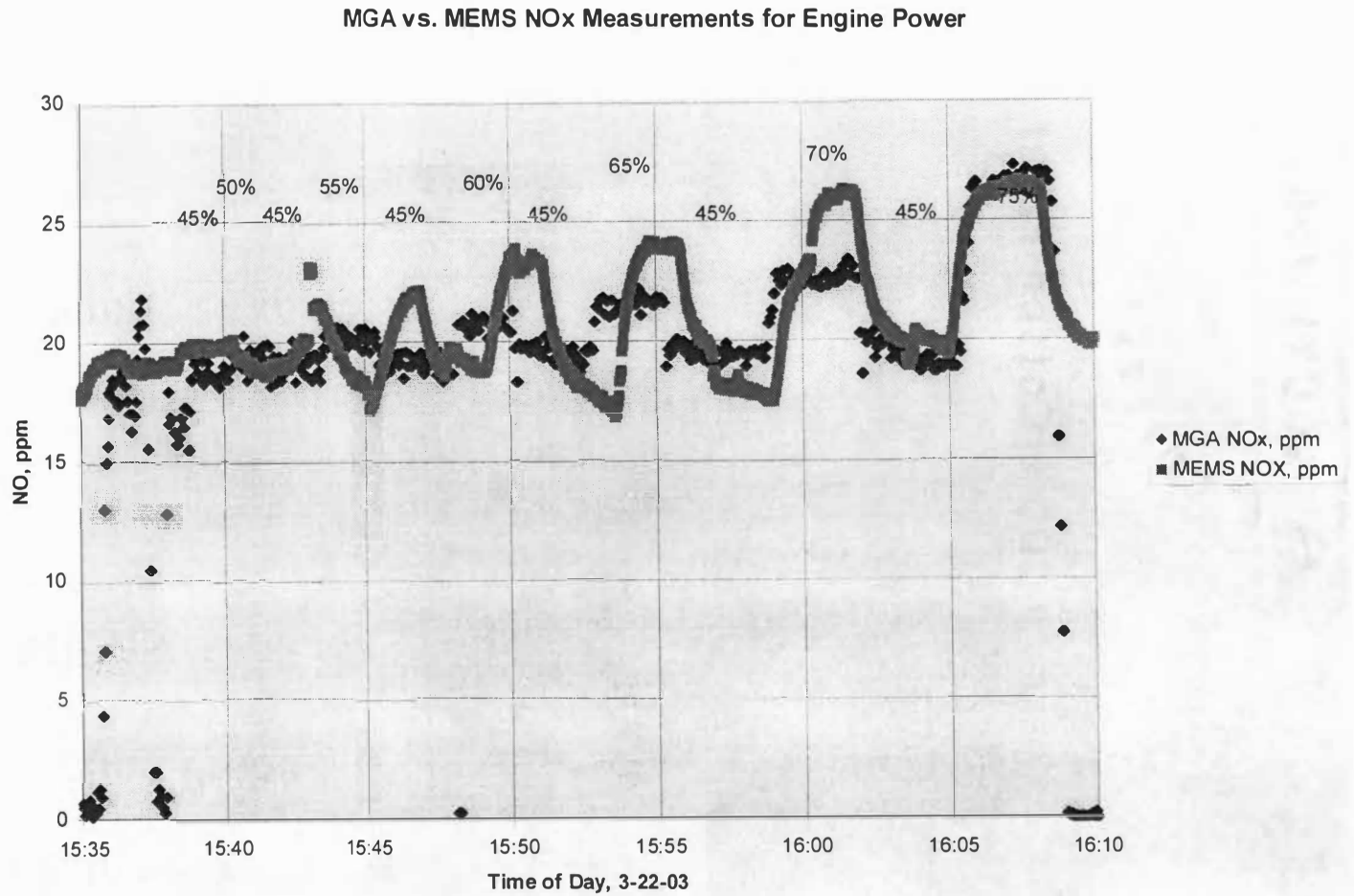


Figure 25. MGA vs. MEMS NOx Measurements

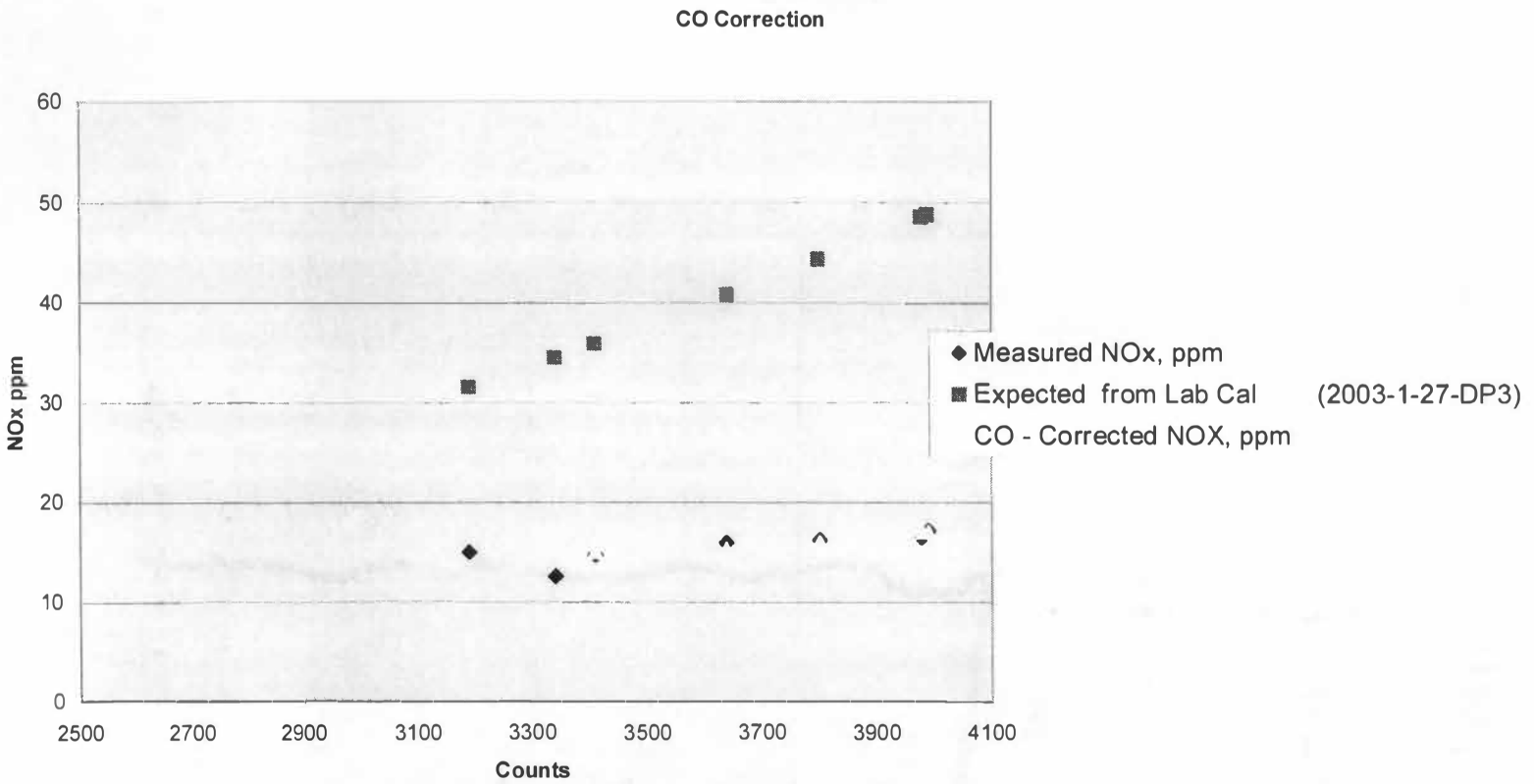


Figure 26. CO-Corrected NOx

53

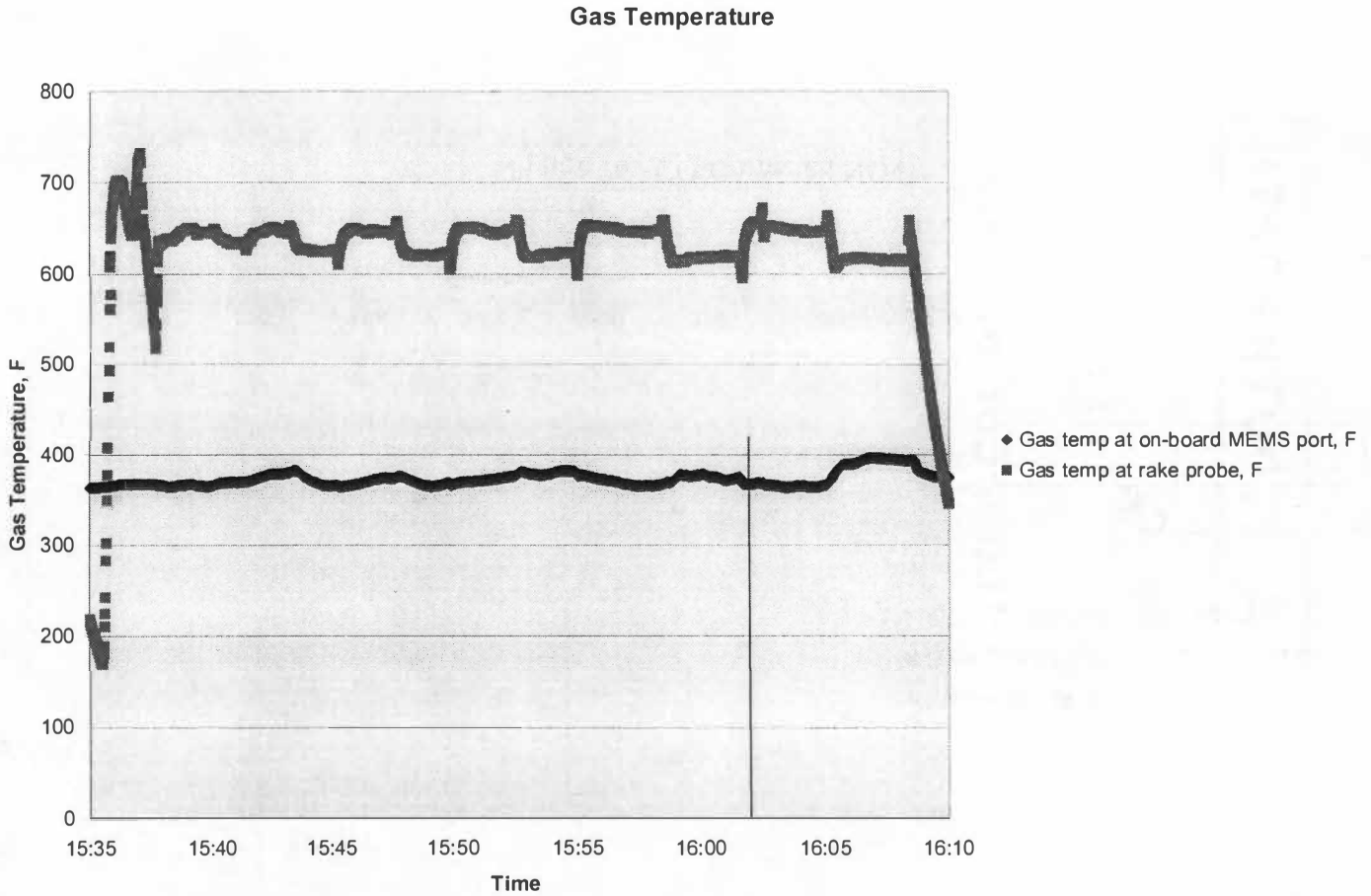


Figure 27. Gas Temperature

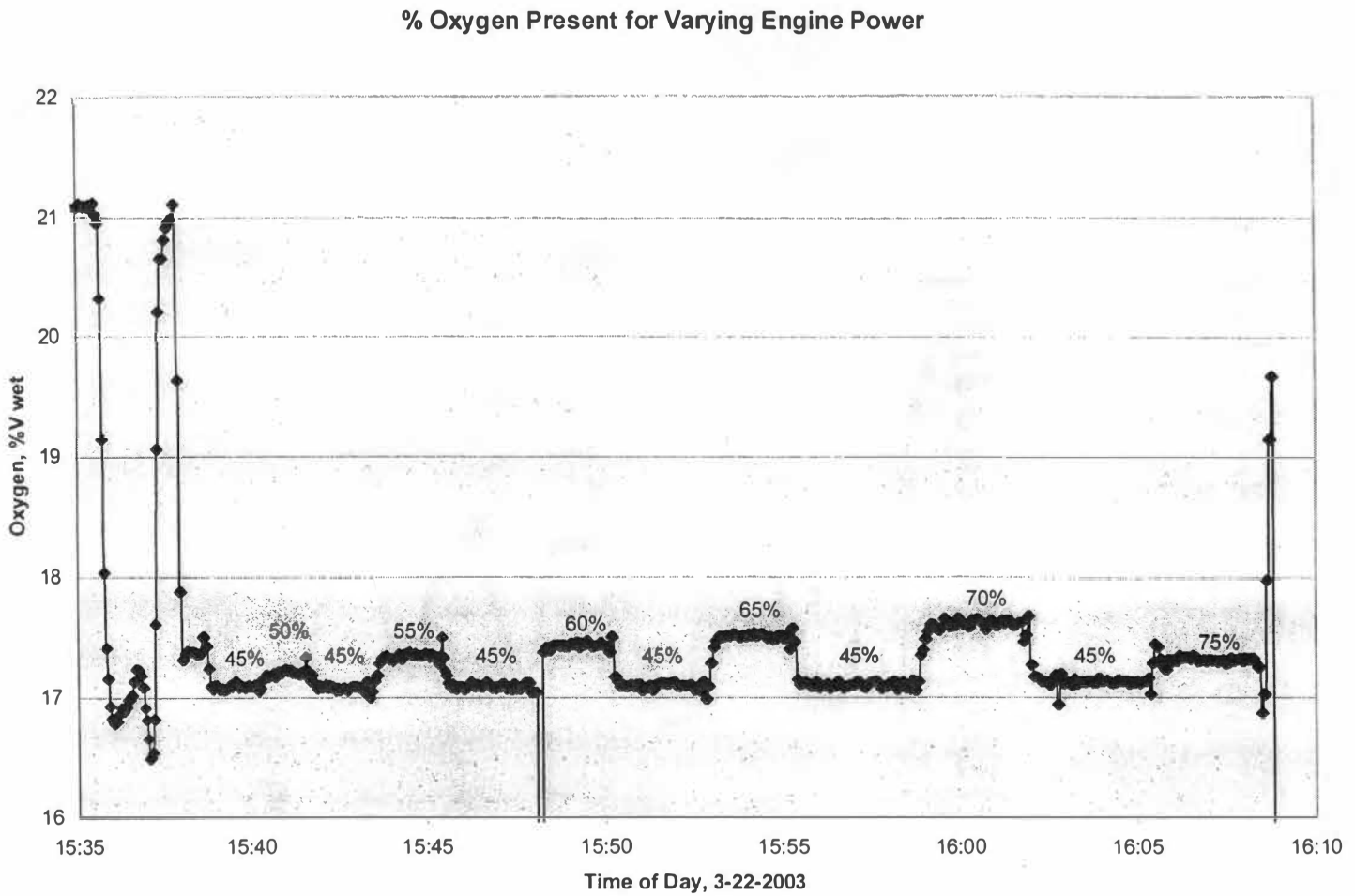


Figure 28. % Oxygen Present For Varying Engine Power

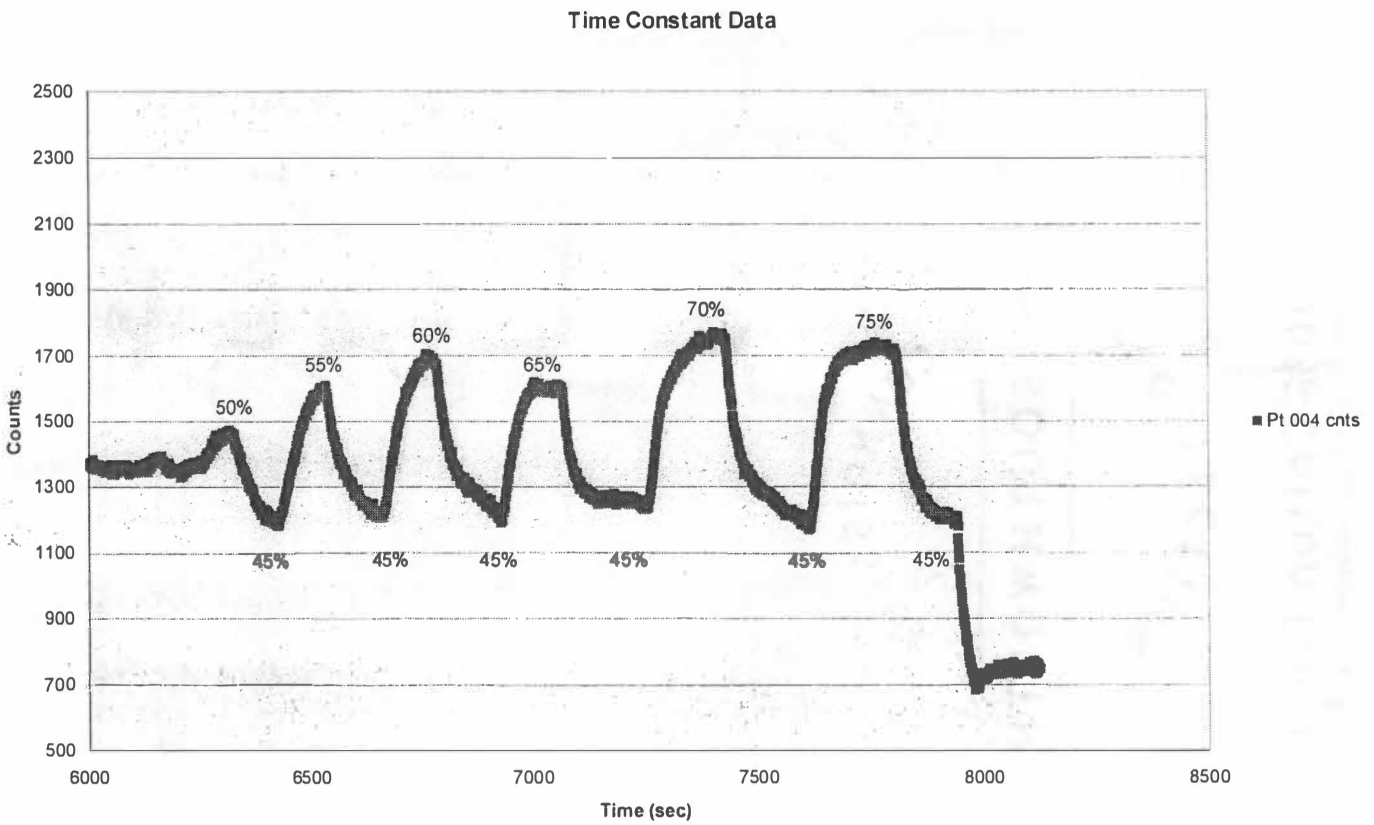


Figure 29. Time Constant Data

APPENDIX B

TABLES

Table 1. Test Matrix

Objectives of Laboratory Bench Tests	Approach	Apparatus Required	Success Criteria
Determine sensor warm-up time.	Measure sensor output vs. time to determine how long it takes for the null level to stabilize.	MEMS NOX sensor, electronic and laptop PC.	The minimum time required for the sensor null level output to stabilize, XX minutes.
Determine sensor calibration stability.	Repeated calibrations a constant conditions will indicate stability.	MutliGas Analyzer, NOX calibration gas, bottle dry air.	A plot of the results of many calibrations will show precision, bias, and linearity (best curve fit).
Determine sensor measurement range.	Utilize high concentration calibration gases to determine maximum measurable NOX concentration.	MutliGas Analyzer, NOX calibration gas, bottle dry air.	The maximum measurable NOX concentration will be determined.
Determine sensor time constant.	Apply step changes in NOX calibration gases to quantify the sensor time constant. Determine the "order" of device.	MutliGas Analyzer, NOX calibration gas, bottle dry air, ograph recorder.	The order of the sensor will be evaluated and a time constant will be determined.
Determine sensor recovery time and rate.	Determine time required for sensor to recover after measuring high NOX levels.	MutliGas Analyzer, NOX calibration gas, bottle dry air, ograph recorder.	The length of time required for the sensor to return to null after sensing a high NOX concentration will be evaluated.
Determine sensor performance at varying oxygen levels.	Measure changes in sensor output at constant NOX concentration with varying oxygen content.	MutliGas Analyzer, NOX calibration gas, bottle dry air, bottle of nitrogen.	Operational and data reduction guidelines will be established for use of the sensor in changing oxygen levels.
Determine sensor performance at varying chip temperature set points.	Measure changes in sensor output at constant NOX concentration with varying chip temperature set points.	MutliGas Analyzer, NOX calibration gas, bottle dry air.	Variation of sensor output with varying sensor set point temperature will quantify this elemental error for
Determine sensor measurement interferences from other gases (CO, HC).	Measure changes in sensor output at constant NOX concentration with varying CO and HC content. This is also an MTSU test objective.	MutliGas Analyzer, NOX calibration gas, bottle dry air, bottle of CO, bottle of propane or methane.	Operational and data reduction guidelines will be established for use of the sensor in in the presence of interfering gases.
Determine sensor sensitivity to harsh environment (vibration, temperature, pressure, moisture, soot)	Measure changes in sensor output at constant NOX concentration when exposed to a non-destructive harsh environment.	This is an MTSU test objective.	These are non-destructive tests so the conditions will be limited to the worst cases expected in planned applications.
Determine chip health monitoring parameters	Determine which parameters from the tests above seem to indicate changes in sensor health.		Track zero drift, calibration deviation, increasing interferences, sensitivity loss, time constant change, etc, for indications of failure.
Determine sensor longevity.	Keep a careful record of the time duration of tests that have been completed with each sensor.		XXX hours of operation.

Table 2. Required Oxygen Percentage

Test 2003-2-14DP2 NO = 18ppm, DAC = 500		Test 2003-2-17DP2 NO = 18ppm, DAC = 750		Test 2003-2-18DP2 NO = 9ppm, DAC = 500		
MEMS counts	**Oxygen Content, %**	**MEMS counts**	**Oxygen Content, %**	**MEMS counts**	**Oxygen Content, %**	**Average Oxygen Requirement**
14	20.5	17	20.6	14	20.8	12.3
2800	16.3	1075	16.4	1350	19	
2900	15.6	1230	15.2	1550	17.4	
3100	15.2	1350	12.9	1580	15.9	
3400	14.6	1400	11.9	1625	13.4	
3300	12.7	1350	9.7	1550	10.5	
3200	10.5	1275	8.5	1500	8.2	
3050	8.3	1200	6.7	1375	5.1	
2800	4.8	1050	5	1400	6.5	
2600	3.1	1000	4.1	1560	8.4	
2400	1.4	850	2.7	1700	10.6	
1650	0	400	0.2	1850	12.7	
2500	2.5	800	3.5	1950	14.5	
2730	3.6	1000	5	2000	16.9	
2850	4.7	1220	6.9	2250	15.8	
2900	5.5	1400	8.5	2275	15.3	
2950	6.7	1550	10.5	2275	14.3	
3050	7.5	1700	11.5	2250	13.2	
3100	8.6	1800	12.4	2150	11.7	
3200	9.6	1850	13.2	2000	8.4	
3300	10.5	1950	15	1950	5.8	
3400	11.9	2000	14	1725	1.1	
3450	12.9	2000	13.5	2050	7.9	
3520	13.9	1950	12.5	2150	10.2	
3600	14.6	1900	11.6	2300	12.2	
3600	14.1	1800	9.7	2475	14.8	
3500	12.2	1700	8.8	2500	16.1	
3350	9.8	1600	6.8	2500	17.6	
3220	8.5	1400	4.2	2550	15.3	
3050	6.9	1500	6.5	2500	13.6	
2850	4.8	1650	8.4	2400	12	
2300	0.7	1725	9.2	2330	10.3	
1670	0	1800	10	2250	8.3	
3050	7.2	1850	10.6	2080	6	
3250	8.8	1950	11.3	1800	2.6	
3370	10.5	2025	12.2	2200	8.3	
3400	11.6	2100	12.9	2300	9.9	
3500	12.8	2150	14.9	2400	11.1	
3580	14.1	2200	16.3	2600	13.3	
3620	15.1			2700	15.5	
3700	16.5			2700	14.3	
3800	15.5			2700	13.5	
3675	13.7			2650	12.4	
3600	12.7			2550	10.5	

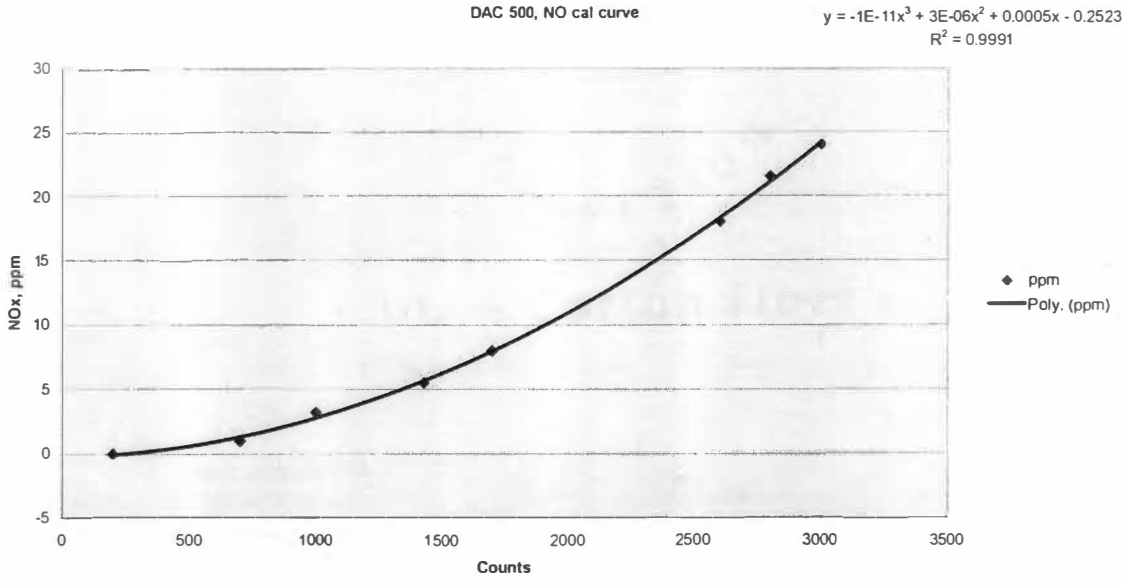
APPENDIX C

CO CORRECTION CALCULATION

MEMS NOx sensor mounted on JT-12 Engine, June 11, 2003

Power Setting	Measured NOx, ppm	CO, ppm	O2, %	NOx MEMS Counts	Expected from Lab Cal (2003-1-27-DP3)	Expected - Measured Counts	CO - Corrected NOX, ppm	Goalseek Column
45	12.6	833	17.6	3340	34.51	1401.2	-7.53	20.13
50	15	668.5	17.6	3190	31.55	987.5	11.47	3.53
50	15.95	522.5	17.7	3640	40.83	1333.1	15.30	0.65
60	16.16	422.3	17.8	3800	44.42	1470.0	16.01	0.15
65	16.47	324	17.9	3980	48.63	1615.9	17.01	-0.54
45	14.7	725.4	17.6	3409	35.92	1239.5	15.06	-0.36
65	17.15	317.1	17.9	3990	48.87	1551.2	17.04	0.11
	average =	0.0000	The average does not include rows 3 and 4.					
	Standard Deviation =	0.467151	The STD DEV does not include rows 3 and 4.					
CO Slope =	-0.0135098		0.071	-0.003221				
Intercept =	44.139667		-5.0429					

First, data from the lab on January 27, 2003 was graphed to find the relationship between counts and NOxppm.



$$Y = -1\times10^{-11}X^3 + 3\times10^{-6}X^2 + 0.0005X - 0.2523$$

$$R2 = 0.9991$$

This relationship was also used to determine the column "Expected Measured Counts." The CO corrected NOx line was created by subtracting the best-fit COppm line from the measured NOxppm line.

MEMS NOx ppm (from engine):
From the above graph, this is determined from the recorded counts using

$$Y = (NOxCounts) \times 0.0129 + 5.4276$$

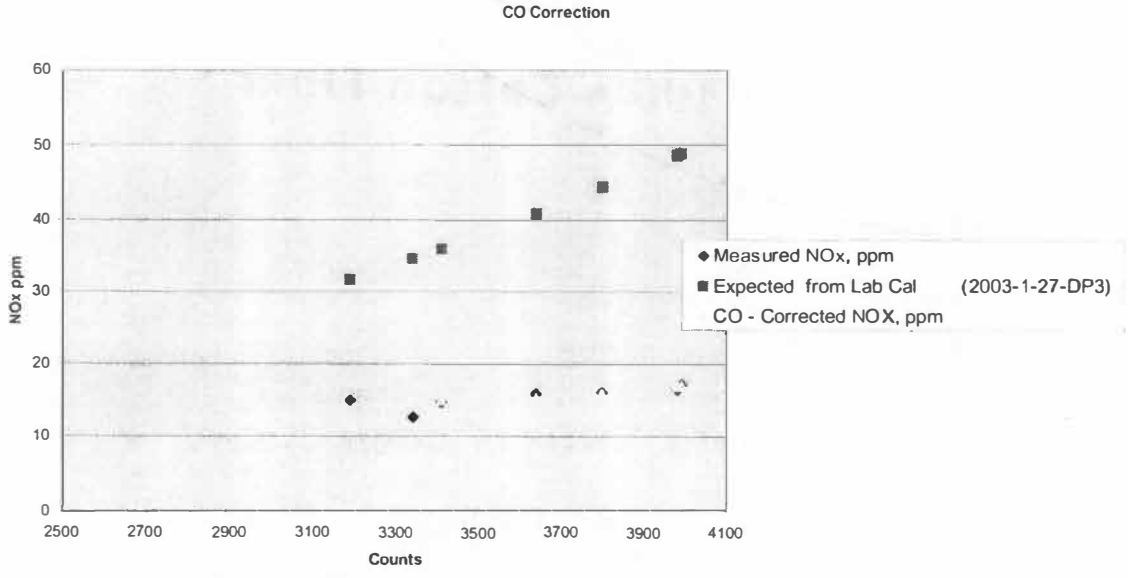
MGA CO ppm (from engine):

$$Y = (COSlope \times MeasuredCOppm) + CO\,\text{int}$$

Next take MEMS NOxppm equation and subtract the MGA CO ppm equation, while adjusting the CO slope and intercept accordingly, to find the CO-corrected NOx.

$$Y = (NOxCounts) \times 0.0129 + 5.4276 - (COSlope \times MeasuredCOppm + CO\,\text{int})$$

The Result:



APPENDIX D

INSTRUMENT DETAILS

INSTRUMENT DETAILS

MultiGas 2030 Analyzer

MultiGas™ 2030 Analyzer - Continuous Gas Analysis

The MultiGas 2030 is an FTIR based analyzer capable of ppb to ppm sensitivity for multiple gas species in a variety of applications, such as stack emissions monitoring, process monitoring, ambient air monitoring, purity monitoring, and selective catalytic reduction performance monitoring. The MultiGas 2030 can perform analysis in gas streams that contain up to 30% water, and can simultaneously analyze and display more than 30 gases. With permanently stored calibration spectra, the need for costly gas cylinders is reduced. In addition, operators will find the robust, fully automated MultiGas 2030 easy to operate and maintain.

http://www.mksinst.com/cgi-bin/product.exe?pid=online2030

VITA

Lisa Urman was born in Des Moines, IA on October 20, 1979. She was raised in New Richmond, WI and graduated from New Richmond High School in 1998. From there, she attended Hamline University in St. Paul, MN, where she received a B.A. in physics with minors in math, religion, and philosophy in 2002. She then went to the University of Tennessee Space Institute and received a M.S. in aerospace engineering in 2004.