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Development of a Micro-Catbed for Miniature Thrusters

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DEVELOPMENT OF A MICRO-CATBED FOR MINIATURE THRUSTERS

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2010

Dedication

This thesis is dedicated to my family and all their support, and my close friends that helped me through the hardest times.

DEVELOPMENT OF A MICRO-CATBED FOR MINIATURE THRUSTERS

by

CHANCE PAUL GARCIA, B.S. Mechanical Engineering

THESIS

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Abstract

The goal of this work is to develop a micro catalytic bed for miniature thrusters. A test bed was constructed for experimental testing to characterize several parts of the micro catalytic bed. The reactant fluid studied is 90% Hydrogen Peroxide (H_2O_2) by weight. Three different cross sectional area designs were tested to determine decomposition efficiency, temperature reaction propensity, optimum flow rate, and temperature distribution across each bed. The products of decomposition were condensed to measure the oxygen released from decomposition. The results will be used to develop the basis for a micro catalytic bed design to implement into future miniature thruster design.

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Chapter 1: Introduction

The focus of this research is towards the development of a cat-bed for miniature thrusters. The thrust class proposed for the cat-bed to be developed is in the micro Newton class. The cat-bed will decompose 90% hydrogen peroxide (H_2O_2) to hot gas for use in the thruster as a monopropellant. This research is aimed at determining which size cat-bed of the three tested yields better decomposition. Recent interest in H_2O_2 propulsion solutions for small scale satellites has surfaced to offer a solution in place of using traditional cold gas or toxic hydrazine propulsion systems (Whitehead, 1998).

1.1 Catalytic Bed

A catalytic bed (cat-bed) is a device used to lower the energy of a chemical reaction, usually allowing it to occur at an accelerated rate (Chang, 2005). The use of the cat-bed in propulsion has shown extensive use on what is called monoprop or monopropellant thruster. Various space systems rely on their design simplicity and adaptability. These propulsion systems usually generate thrust ranging from 0.5 N thrust to 4500 N thrust (Brown, 2002). Thruster pulse widths as low as 7 ms have been demonstrated (Brown, 2002). Short pulses of this nature depend on catalyst design to achieve such fast reaction rates. It should be noted that propellant instability can also produce a fast reaction rate, but for general use in space systems unstable propellants are usually not used for obvious reasons. These include but are not limited to: ground handling safety, shock sensitivity, propellant degrading, etc. In short, the catalytic bed of a monopropellant thruster is desired to have long reliability, induce fast reaction on the propellant, and take up as little space as possible.

1.2 Hydrogen Peroxide

There are a variety of propellants that have been studied for use with liquid propulsion systems. Liquid propulsion can be divided into three categories, bi-propellant, monopropellant, and hybrid propellant (liquid and solid propellant) systems. Bi-propellant propulsion systems combust an oxidizer and fuel generate hot gas for expansion through a nozzle. Hybrid systems use solid propellants and liquid propellants for the same result. Speaking on the basis of monopropellants, several compounds have been characterized. Currently hydrazine is the most widely used of these compounds. Hydrogen

peroxide has also been used with some success. Verner Von-Braun used this propellant with some success when the German V-2 rocket was used during World War II (Griffin and French, 2004), the main engine pumps on the Russian Soyuz launch vehicle (Whitehead, 1998), and the AR airplane super-performance rockets (Huzel and Huang, 1992).

1.3 Purpose and Objectives

The cat-bed being developed investigates the optimal flow rate and channel size for a decomposition chamber. The channel sizes tested were based on 3 different aspect ratios on the millimeter scale. The cat-bed designed is made of 99.9% pure silver and was tested at 5 different flow rates. Testing was initially done on the first cat-bed to characterize a test matrix and decomposition temperatures in the reactor.

To characterize the best cat-bed aspect ratio and flow rate, measured and calculated parameters are made. Temperature measurement on the cat-bed were made along the length of the cat-bed, flow rate of H_2O_2 to the cat-bed, oxygen flow rate of the exhaust products, and oxygen meter to validate oxygen exhaust. With this data cat-bed decomposition efficiency and oxygen flow rate can be determined.

1.4 Thesis Organization

This work is divided into five chapters. Chapter 1 will provide a brief overview of cat-bed uses in propulsion, hydrogen peroxide uses, and statement of purpose for the research done. Chapter 2 will detail the experimental set up, materials used, and equipment capabilities. This chapter will have three subchapters: fabrication and design, additional materials and devices, and the data acquisition. Chapter 3 focuses on the procedure used to conduct safety and experimentation. Data obtained from testing is then analyzed, presented, and discussed in the results and discussion section in Chapter 4. A final section in Chapter 5 is dedicated to a summary of findings and future work to be done, improvements made, and recommendations for further testing.

Chapter 2: Experimental Setup

The experimental set up was composed of a syringe pump, onsite manufactured silver cat-bed, on site manufactured cat-bed stand, condensing tube, separation flask, rotameter, and oxygen sensor. The design and manufacture of the cat-bed will be explained in detail. The parameters and capabilities of equipment used will also be described in detail. Testing and fabrication was conducted in the Center for Space Exploration Technology Research (cSETR) in Room E105 and E203 of the engineering building at the University of Texas at El Paso. This research was done in conjunction with the University of Maryland Combustion Research Center. The lab focuses on energy production, transfer, and propulsion. The capabilities of the center allow for characterization of experiments in these areas with DAQ systems, optical hardware, laser systems, altitude simulation, and miniature component manufacturing.

2.1 Fabrication of the Cat-Bed

The cat-bed was machined on a 3-axis CNC milling machine. Silver 1 inch x 3/16 inch (in) stock for the cat-bed was purchased from D. F. Goldsmith Chemical. It was decided that the cat-bed would be simpler if designed in two pieces, a top and lower piece. The dimensions for the three aspect ratios tested are 1 x 5 millimeters (mm), 1 x 3 mm, and 1 x 1 mm. The 1 x 5 mm cat-bed was manufactured and tested first. Characterization of the experimental set up and cat-bed capabilities were also done on this aspect ratio.

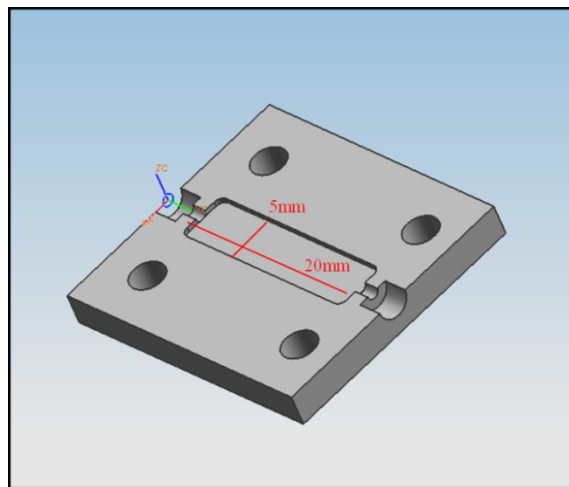


Figure 2.1: NX CAD Model of 1 x 5 mm Cat-Bed.

All cat-bed designs were done using Unigraphics NX 6.0 computer aided design (CAD) software. An example of the models can be seen above in Figure 2.1. Once the NX CAD model was completed a G-code was derived for the CNC to properly manufacture the cat-beds.

After the G-code was made the silver stock was then end-milled to proper dimensions. The pieces were polished and checked for obstructions in the channel and shavings left over from machining. Bolt holes were then tapped for 10-32 screw threads and once bolted, the cat-bed was again drilled on the ends to accept two 1/8 in NPT to 1/8 in Swagelok stainless steel(SS) pipe fittings. The cat-bed manufactured can be seen below in Figure 2.2. Each cat-bed was manufactured in a similar fashion.



Figure 2.2: 1 x 3 mm Cat-Bed Tested With H_2O_2

S-type thermocouples (TC) were attached to each reactor through holes drilled at measured locations into the top and bottom of each reactor. The arrangement of TCs on the cat-bed can be seen below in Figure 2.3. The TCs on the bottom of the reactor were held in using Loctite epoxy. Omega Kapton wrapped heaters were then attached to the cat-bed being tested. The cat-bed was then wrapped in fiberglass insulation and heat tape in an effort to reduce heat loss to the surroundings.

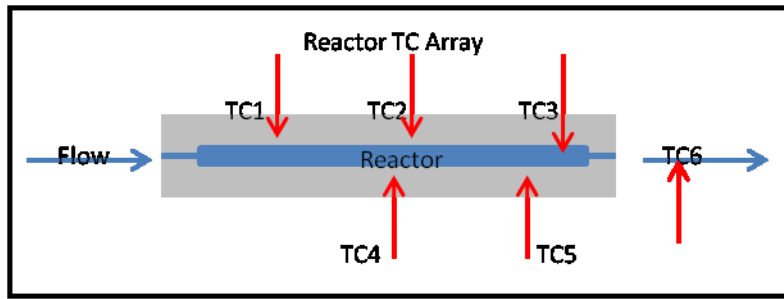


Figure 2.3: Cat-Bed Thermocouple (TC) Array

During testing the TCs began to absorb heat into the thermocouple connectors through the ceramic housing holding the S-type wires. This began to generate some off temperature TC readings. To render a solution, only two S-type TCs were used on the top side of the cat-bed. It was believed that the cluster of TCs on top and bottom of the cat-bed gathered too much heat, thus affecting temperature measurement. The final TC array composed of TC 1 and TC 3.

The cat-bed stand is made of aluminum bar stock and round bar stock from scrap metal left over in the university machine shop. Aluminum is fully compatible with H_2O_2 as long as it is correctly passivated. The base of the stand remains in a 1 – 2 centimeter (cm) high pool of de-ionized (DI) water. The stand was previously used by the center for previous research on combustor and injector parts for meso-scale propulsion rockets.



Figure 2.4 Cat-Bed Test Stand with Cat-Bed

Once the machining, TCs, and insulation were completed, the cat-bed was bolted into the cat-bed stand where it could mount firmly to the 1/8 inch (in) SS lines to integrate it into the system. The connections to the SS lines were then checked for leaks with Swagelok Snoop liquid.

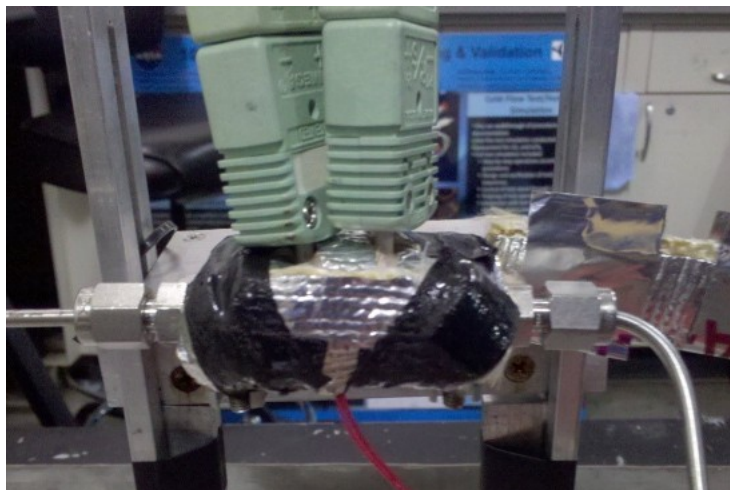


Figure 2.5 Assembled 1 x 1 mm Cat-Bed

2.2 Equipment

The complete experimental set up contains a syringe pump, 19 mm Teflon syringe, 1/8 in SS tubing, cat-bed, cat-bed test stand, neoprene stoppers, condenser, separation flask, rotameter, oxygen sensor, a 306 SS catch tray, 90% H_2O_2 by weight, 40% Nitric Acid, 60% Sulfuric Acid, and de-ionized (DI) water. A simple schematic of the layout is seen below in Figure 2.6.

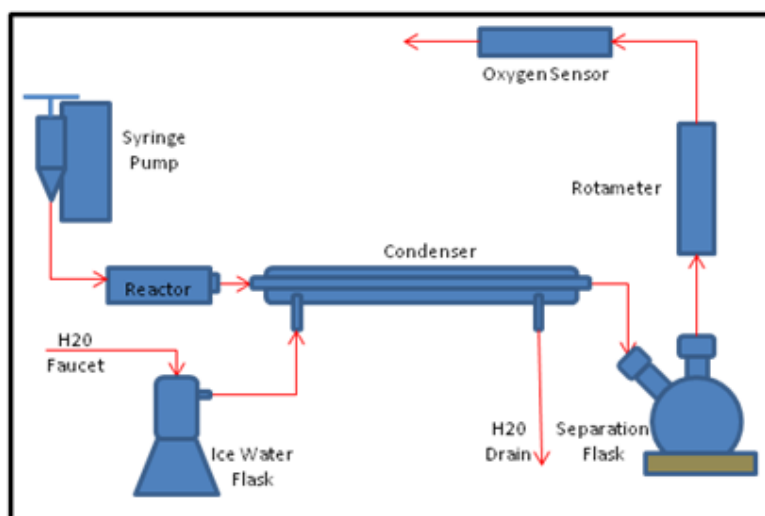


Figure 2.6 Experimental Setup Schematic

2.2.1 Syringe Pump

The syringe pump is a Genie Plus made from Kent Scientific. It can be manually or remotely operated by computer, infuse or withdraw fluid, and accepts 15 micro liters (μL) to 60 cubic centimeters (cc) syringes. The syringe pump is widely used in medical and scientific testing laboratories. In this experiment the pump was remotely operated due to safety precautions.

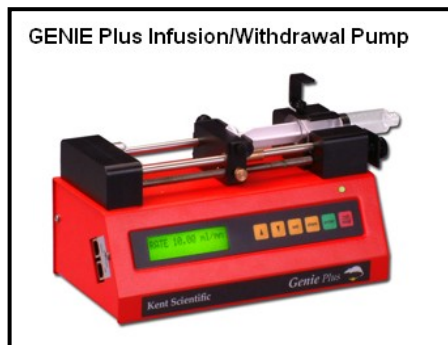


Figure 2.7 Kent Scientific Infusion/Withdrawal Syringe Pump

The syringe used to supply 90% H_2O_2 to the system is made of Teflon. The flow diameter is 19 mm and holds about 10 milliliters (mL). At any given point in time approximately 5mL was contained for safety precautionary measures. Concluding testing, the syringe was rinsed with de-ionized water and dried before subsequent use. The purpose behind this was to ensure no residual peroxide remained inside the syringe.

2.2.2 Heaters

Thin resistive heaters were used to heat each cat-bed tested to the respective initial testing temperatures. The heaters are Kapton wrapped and purchased from Omega Engineering KHLV series. The power supplied to the heaters was done via DC power supply. The voltage run to the heaters was 28 VDC at 0.9 Amps.



Figure 2.8 Omega Kapton Heaters

2.2.3 Glassware

The primary glassware pieces for this experiment are comprised of a filter Erlenmeyer flask, west condenser, and distillation (separation) flask. Neoprene stoppers were used to seal glassware with holes cut into them to tightly accept 1/8 in SS lines.

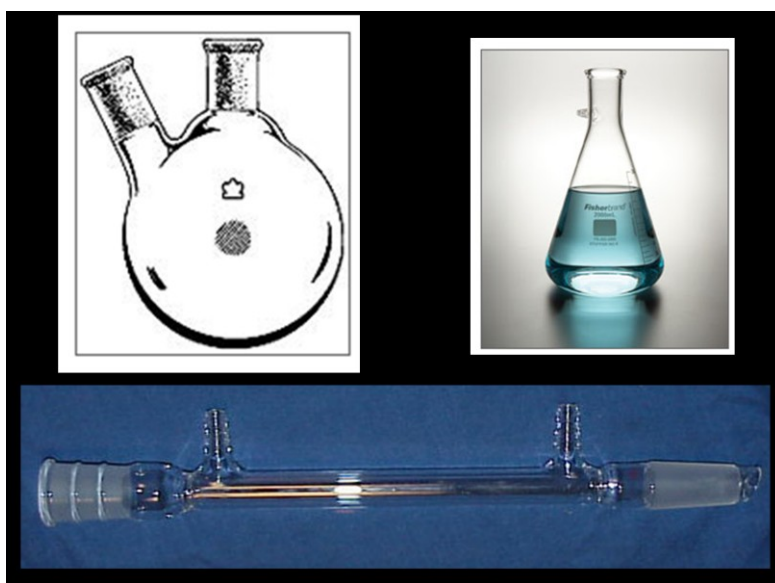


Figure 2.9 Glassware Used for Experimentation (Top left: Distillation Flask, Top right: Filter Erlenmeyer Flask, Bottom: West Condenser)

Once the stoppers and SS lines were fitted, the stoppers were sealed onto the glassware using RTV. The parts were left overnight to cure. They were then tested for leaks with water and Snoop.

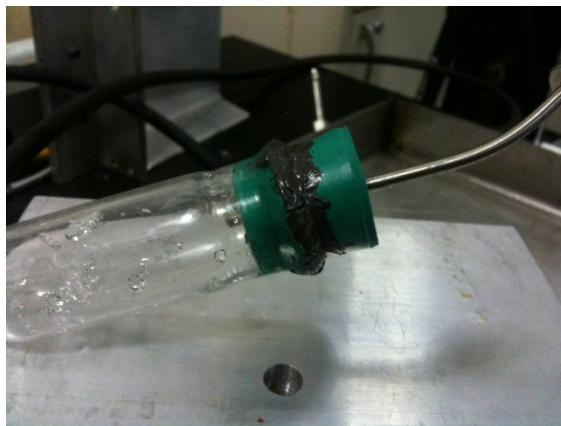


Figure 2.10 RTV Sealed Neoprene Stopper with SS Line

Secondary glassware was used for experimental preparation of material to come in contact with the H_2O_2 . These mainly consisted of 800 and 600 mL beakers. All parts were placed inside these beakers to complete the passivation process. This process and its reasoning will later be explained in the preparation procedure in Chapter 3.

2.2.4 Rotameter

The rotameter used to determine the flow rate of the oxygen product from decomposition is an Omega FL-1444-G unit. The max reading capacity of the meter is 820 cubic centimeters per minute (cc/min) air with the minimum flow rate to accuracy reading of 10% of the maximum reading (82 cc/min).



Figure 2.11 Omega FL-1444-G Rotameter

The reading face has a 150 mm gauge to read as the glass ball inside floats with flow rate. The meter itself comes with a calibration sheet, but the meter was calibrated in-house with pure nitrogen to avoid any conversion issues and errors from the factory. The device used to calibrate the meter is a DryCal DC-Lite with an accuracy reading of 1.25% from BIOS International Corp.

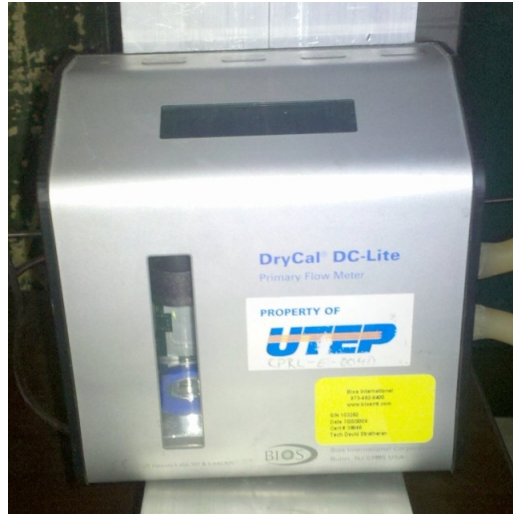


Figure 2.12 DryCal DC-Lite

The calibration on the flow meter was done at 10 mm on the flow meter to 135 mm at just before fully open conditions. The needle valve inside the rotameter was not opened fully because the max flow rate of the meter was not predicted to be used as the cat-bed product velocities at the lower flow rates were very small. Once completed the flow meter calibration data was tabulated in an excel spreadsheet for further conversion. The data was first converted using a square root density equation.

$$\text{Corrected Flow Rate} = (\text{Calibration Flow Rate}) \sqrt{\frac{\text{Density of Calibration Gas}}{\text{Density of Gas Used}}} \quad (2.1)$$

Concluding the density ratio calibration, a temperature adjustment factor was also incorporated on the flow rates. The exhaust flow temperature was measured to be 180 °C. The equation incorporates pressure and temperature into the calculation. The temperature input was 815.7 Rankine (°R) and the pressure input was 762 millimeters of mercury (mm Hg).

$$\text{Corrected Temperature and Pressure Flow Rate} = (\text{Flow Rate}) \sqrt{\frac{\text{Pressure}}{760} \times \frac{530}{\text{Temperature}}} \quad (2.2)$$

Once the calibration data was tabulated and corrected a plot of the calibration done with the DryCal DC-Lite is shown below in Figure 2.13. A trend line was added to be used as a conversion function for rotameter readings.

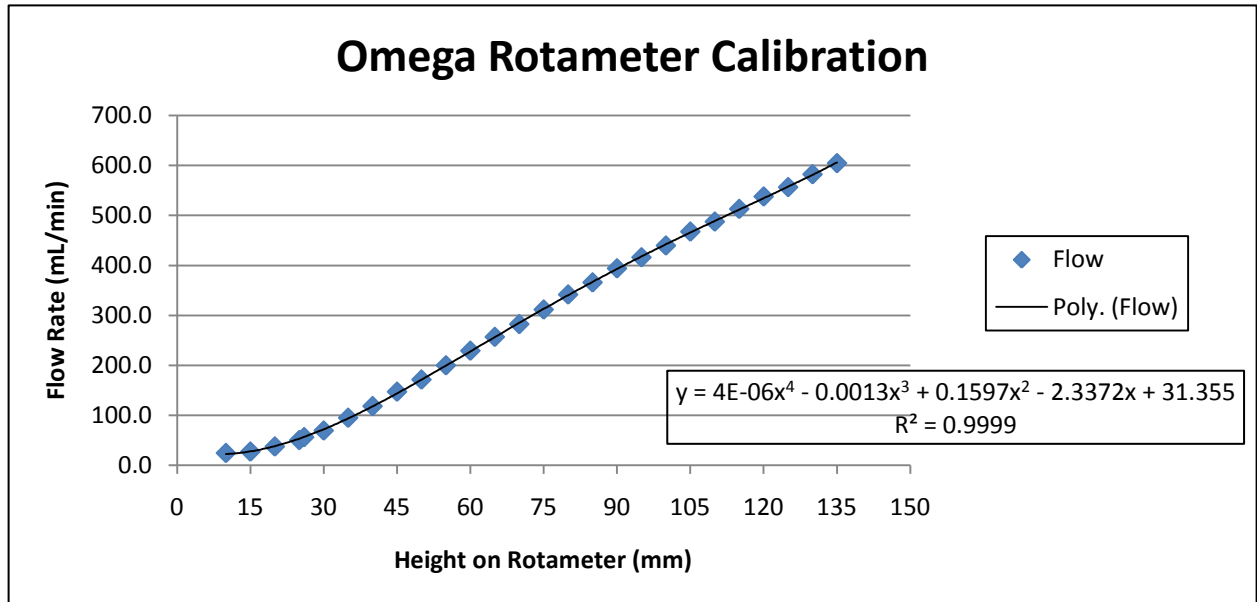


Figure 2.13 Calibration Plot of Rotameter

2.2.5 Oxygen Sensor

The oxygen sensor used is a MSA MiniOx III Oxygen Monitor system. It is mainly used in the medical field for patient monitoring. The sensor can give a 0 to 100 percent reading of oxygen in a given flow field. The sensor works in a humid environment but only operates between 0 to 40 degrees centigrade (°C). This was the driving factor for condensing and cooling the exhaust products of the cat-bed.



Figure 2.14 MSA MiniOx Oxygen Sensor

The sensor calibrates in ambient air or in a 100 percent oxygen environment. The sensor was calibrated in ambient air to 21% where the sensor will operate and read out flowing oxygen from the experimental set up. The recommendation from MSA is to calibrate the sensor in the environment closest to the sampling conditions the sensor will see.

2.2.6 Thermocouple and Data Acquisition

The temperature data on the cat-bed was recorded via Labview 8.1 software. The interface between the thermocouples used is a National Instruments (NI) data acquisition (DAQ) USB 9213 DAQ card. The sample rate fully loaded can support a sample rate of up to 100 hertz (hz). The DAQ is compatible with virtually any differential voltage measurement device +/- 5 volts (V). Platinum – 90% Platinum/Rhodium (S-Type) thermocouples were used in experimentation.

2.2.7 Hydrogen Peroxide

The hydrogen peroxide used for this experimentation was in two concentrations 35% and 90% H_2O_2 by weight. As will be discussed in chapter 3 the 35% H_2O_2 was used to passivate the test equipment before use with 90% H_2O_2 . The origins of both peroxide concentrations are different. The 35% H_2O_2 was ordered from Fischer Scientific while the 90% H_2O_2 procured through FMC Global. Obtaining peroxide from FMC Global in high concentrations requires a company process that includes full lab inspection, personnel training, security requirements for the lab, fire department notification, health and safety department notification, and proper display of personal protective equipment (PPE). Concluding this process FMC will send reference material on H_2O_2 . This material can be obtained from FMC without purchase of peroxide or services.

2.2.8 Nitric Acid and Sulfuric Acid

70% Nitric acid (by weight) was used to prepare equipment for use with hydrogen peroxide. This acid is part of the passivation process, in particular for the aluminum and stainless steel (SS) test equipment. 35% Sulfuric acid (by weight) was used to passivate all glassware used in test preparation and sample preparation. The main use was to ensure all glassware contained no residual organic material. Both are general acids and were purchased from Fischer Scientific.

2.2.9 De-Ionized Water

De-ionized (DI) water was used in all parts of experiment preparation, testing, and post testing. All equipment prepared or cleaned was done using DI water. This is recommended from the FMC H₂O₂ peroxide manual. The experiment stainless steel tray was also filled with DI water prior to any experimentation. DI water was procured from a DI filtration system located in the biology department at the university.

2.3 Safety and Equipment

Handling high concentrations of H₂O₂ can be a nominal task if the safety precautions are followed. To prepare for use of H₂O₂ wrist length Nitrile gloves were used at all times to handle any equipment in contact with H₂O₂. DI water bottles were always on hand to wash anything down that may have H₂O₂ residue. All testing and H₂O₂ filling of the syringes was done with elbow length Nitrile gloves, protective rubber rain coat, rubber steel toed boots, splash guard goggles and face shield. All H₂O₂ was first poured into a beaker then transferred to Teflon syringe inside a hood with over 250 cubic feet per minute (cfm) air flow. Also a 10 gallon reservoir of DI water was kept near to douse and clean larger items and gloves with H₂O₂ residue. A collection of documents were generated to standardize the use of H₂O₂, facilities, testing, and clean up. These can be found in the appendix.

Chapter 3: Experimental Procedure

A series of characterization tests were performed on the experimental set up using the 1 x 5 mm cat-bed. Water tests were performed to ensure pump and leaks were not evident in the system. Also dribble volume was calculated in the line entering the cat-bed. This value was used to initially fill H_2O_2 in the line before testing was initiated. The goal of the procedure was to obtain data for H_2O_2 decomposition at an initial test temperature of 30 °C and an initial temperature of 150 °C.

3.1 Passivation

Each piece of equipment that will come into contact with H_2O_2 must be properly passivated before it can be used. This process prevents contamination of the H_2O_2 before use in the system, giving erroneous results, and preventing the system from getting damaged from the flowing H_2O_2 . The exact method with time values and quantities is described in detail in the passivation procedure found in the appendix.

The general procedure for passivation required first for all items that will be passivated to be cleaned with a general cleaning agent such as Alcanox. After cleaned, the pieces were rinsed with DI water and air dried. A beaker was prepared with 70% Nitric Acid solution to passivate all stainless steel and aluminum parts with the exception of the cat-beds. The parts were left in solution for the predetermined hours stated in the procedure attached to the appendix. The time varied between 1 and 3 hours. Most of the parts were passivated along with the inside of the stainless steel catch tray due to the large volume it held. Glassware to be used for H_2O_2 transfer was passivated with a 35% sulfuric acid solution. The time value is again stated in the procedure in the appendix. All passivation processes took place inside a 250 cfm hood.

To finalize the passivation process all parts were again passivated with 35% H_2O_2 to ensure no areas on all parts were left to react with the high concentration H_2O_2 . This process did not require as much time but it was repeated three times. The first emersion of the parts in the 35% H_2O_2 encompassed a full immersion of each piece into the beaker containing the H_2O_2 mixture. The part was then removed and washed with DI water. The next step required the entire test rig to be assembled and 2 mL of 35% H_2O_2 pumped through the system using the Teflon syringe and syringe pump. The assembly was then

left to sit for an hour. The third and final process with the 35% H_2O_2 was to run another 2 mL through the system. Concluding this process the system is either immediately ready for testing or for rinsing with DI water for storage.

Once all materials were cleaned and passivated they wrapped in plastic bags and rinsed with DI water after every use to avoid contamination. Again it can not be emphasized enough that before any experimentation or handling of high concentration H_2O_2 all parts must be passivated before use.

3.2 Test Preparation

To prepare for a test, a test matrix was formulated to follow and characterize data to be found. Test number and volume to be used should be clearly defined to avoid any excess H_2O_2 in any area as this could create a hazard.

Once the test matrix is defined the test area, hood, and PPE were prepared for testing. This included cleaning and assembling the test set up. Testing signs were then posted at the appropriate labs and facilities being used for testing. All measurement devices were first set up and initialized to record. The TCs were set to record at a sample rate of 20 hertz (hz). The pump control parameters were set for the first test. DI water bottles and the 10 gallon DI tank were filled in case of any emergency.

All PPE was put on before any further preparation. The first task preparing to test with H_2O_2 is to extract it from the container in a hood. Nitrile matting was placed on the bottom of the hood for further safety in the event of a spill. The water source inside the hood was turned on and allowed to run at a slow flow rate. The container was then removed from its secure storage locker and poured into a passivated beaker. Only 5 to 6 mL was poured. Teflon syringes were then filled from the beaker. Any excess H_2O_2 left over was immediately diluted with DI water and further diluted into a larger beaker.

Once the syringe was full of H_2O_2 it was taken to the test set up and installed for testing. The safety shield was installed in front of the test set up as a precautionary measure. The water for the condenser was turned on to properly function. The heaters were turned on to ensure the cat-bed was at the proper starting temperature. The oxygen sensor was also turned on and calibrated at 21% (ambient air).

3.3 Testing

To begin the decomposition process the syringe pump was set to the target flow rate, pump diameter, and target volume. Each of these parameters was taken from the test matrix generated seen below.

Table 3.1 Cat-Bed Test Matrix

Test Matrix					
1 x 5 mm	Cat-Bed	1 x 1 mm	Cat-Bed	3 x 1 mm	Cat-Bed
Flow Rate	Init. Temp	Flow Rate	Init. Temp	Flow Rate	Init. Temp
.1 mL/min	25 C	.1 mL/min	25 C	.1 mL/min	25 C
.1 mL/min	150 C	.1 mL/min	150 C	.1 mL/min	150 C
.5mL/min	25 C	.5mL/min	25 C	.5mL/min	25 C
.5mL/min	150 C	.5mL/min	150 C	.5mL/min	150 C
1mL/min	25 C	1mL/min	25 C	1mL/min	25 C
1mL/min	150 C	1mL/min	150 C	1mL/min	150 C
2mL/min	25 C	2mL/min	25 C	2mL/min	25 C
2mL/min	150 C	2mL/min	150 C	2mL/min	150 C

Each test was done at the same initial conditions for each cat-bed. A 30 °C test series and 150 °C test series was completed on each of the cat-beds. The choice of these two temperatures was to represent a slightly warmer than ambient environment test condition and the higher temperature was chosen because of the boiling temperature of H₂O₂. The goal and thought was that at higher temperatures a stronger decomposition reaction would result.

During each test the rotameter and oxygen meter were monitored. The values recorded on the rotameter were the highest steady reading and peak value. The reading was taken from the middle of the ball inside. The value taken from the oxygen sensor was the highest value shown as it was open to the atmosphere. Concluding each test the cat-bed being test was allowed to cool or heat up to the correct testing temperature. Upon reaching the test temperature the pump was started and the heaters were turned off. Temperature measurements were recorded as soon as temperature conditions were met. This process was repeated until completion of the test matrix or no H₂O₂ remained in the syringe. The previously mentioned process was repeated to refill the empty syringe.

3.4 Post Testing

Concluding completion of the test day or test matrix all parts of the assembly were first allowed to cool down. This was monitored with the TCs on the cat-bed. Once cool, the set up was flooded with DI water with a laboratory style water bottle. It was then disassembled and watered down further with DI water. All components were then sealed for storage. The hood and Nitrile matting were picked up and washed with water.

Chapter 4: Experimental Results and Discussion

Data obtained from experimentation was compiled from its raw form into plotted and tabulated formats. The data compiled was temperature °C, flow rate in milliliters per minute (mL/min), and oxygen percentage (%). The data was grouped mainly by initial testing temperature, cat-bed size, and flow rate. As discussed earlier each flow rate and initial test temperature was run 3 consecutive times. For comparison purposes the rotameter readings were averaged. The three runs of H₂O₂ flow rates between cat-beds were then averaged and compared between different cat-bed sizes. For example at a H₂O₂ flow rate of 0.1 mL/min all three cat-bed results were plotted together with the three runs for each cat-bed size averaged.

4.1 Results and Discussion

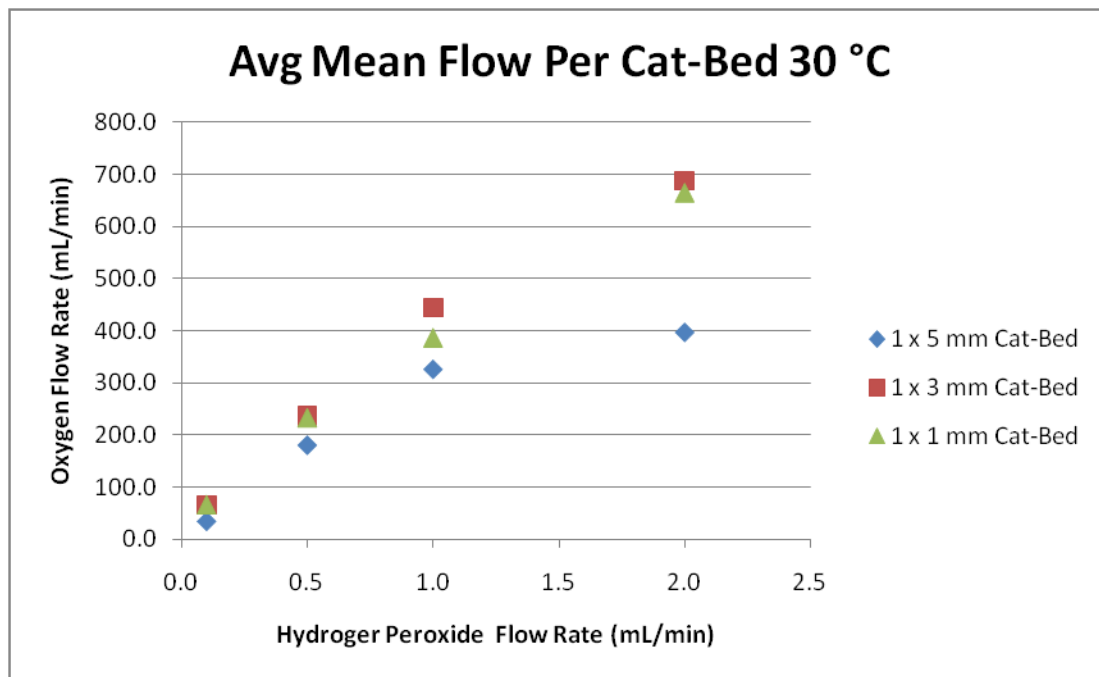


Figure 4.1 Cat-Bed Average Mean Flow Rate at 30 °C

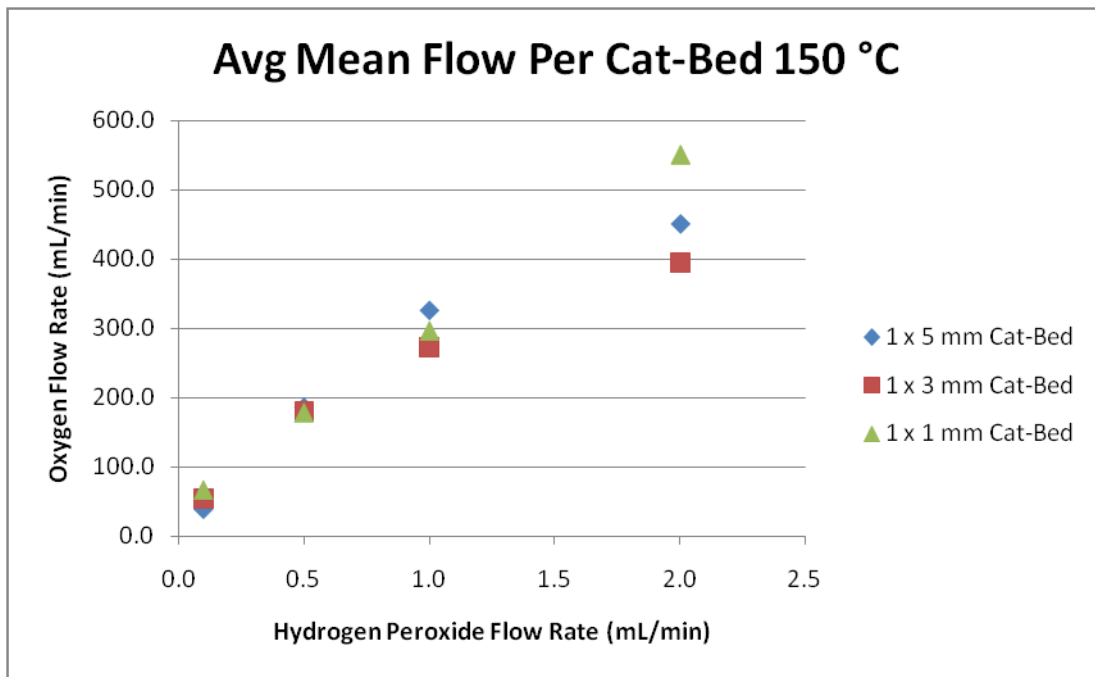


Figure 4.2 Cat-Bed Average Mean Flow Rate at 150 °C

The figures above display output oxygen flow rate after decomposition of H₂O₂ pumped into each cat-bed. Both charts differ in initial cat-bed decomposition temperature only. Each cat-bed displayed different results and differences between cat-beds were both small and large depending on the flow rate of H₂O₂.

The results in figure 4.1 show that both the 1 x 3 mm and 1 x 1 mm cat-beds had greater flow rate of oxygen at 1.0 and 2.0 mL/min flow rates. The 1 x 5 mm cat-bed showed lower flow rates for all 30 °C tests. This results change slightly between cat-bed flow rates when tested at 150 °C. The data only moves apart at the higher flow rates of 1.0 and 2.0 mL/min, 2.0 being more apparent. If both figures are compared however, the higher flow rates show a difference greater than 100 mL/min oxygen flow rate between the lower and higher temperatures tested at. The H₂O₂ pumped into the system was at 30 °C. When testing at the same initial temperature the change in temperature from the fluid to the cat-bed was minimal if not zero, but when entering the cat-bed at the higher initial temperature of 150 °C the change in temperature was 120 °C. It was perceived for this reason that the change in temperature to the fluid in the cat-bed would aid decomposition and generate higher flow rates of oxygen. This evidently was not the case as previously shown. The temperature profile of these tests located in the appendix for each cat-

bed and test run show results of the reactor cooling during testing. The decomposition clearly increases in the beginning of the experiment, but the flow rate seems to be too high, affecting heat generation inside the cat-bed. The cooler 30 °C H_2O_2 flowing in the cat-bed has more of convective cooling affect than heat release of decomposition. This was the general trend for all cat-beds tested.

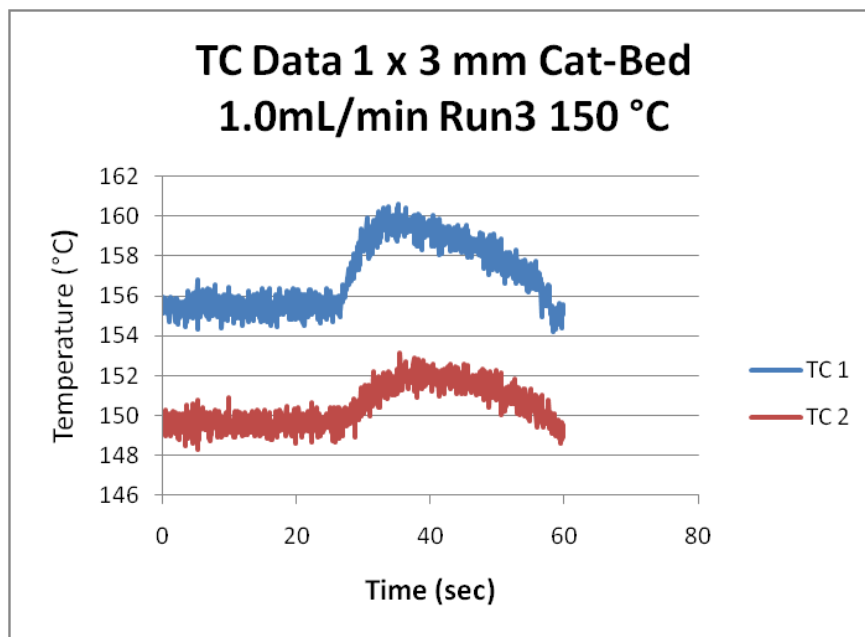


Figure 4.3 Temperature Profile of 1 x 3 mm Cat-Bed at 1.0 mL/min Flow Rate

Further testing methods into trying to generate higher decomposition temperatures should be done. Literature suggests in one test matrix that heating the H_2O_2 to roughly 120 °C initially and then flowing into the catalyst generates promising results (Ventura and Wernimont, 2001). A possible implementation to the current test set up to heat the H_2O_2 is by using thin copper plates, Kapton heaters, thermocouple, power source, and proper insulation on the Teflon syringe.

Another parameter analyzed between cat-bed performances is the decomposition efficiency attained. This process requires a theoretical max oxygen mass flow rate output to be known. This value was found for each of the four different flow rates of H_2O_2 .

To characterize the cat-bed decomposition efficiency, the maximum theoretical oxygen released during decomposition must be calculated. The maximum oxygen released can be found by investigating

the homogenous chemical decomposition of H_2O_2 . To begin, 90% H_2O_2 (by weight) is considered in a 100 gram (g) sample:

$$100 \text{ g Solution} = 90 \text{ g H}_2\text{O}_2 + 10 \text{ g H}_2\text{O}$$

The mass is then converted using the density of H_2O_2 and H_2O at 30 °C

$$\rho (\text{H}_2\text{O}_2) = 1.463 \text{ mL (g/mL)}$$

$$\rho (\text{H}_2\text{O}) = 0.997 \text{ (g/mL)}$$

$$90 \text{ g H}_2\text{O}_2 \rightarrow 61.52 \text{ mL}$$

$$10 \text{ g H}_2\text{O} \rightarrow 9.97 \text{ mL}$$

The total volume (Vol) and volume fraction was then calculated

$$\text{Total Vol} = 71.49 \text{ mL}; 86.05\% \text{ H}_2\text{O}_2 \text{ (by Vol)}$$

For every 0.5 mL sample 86.05% is H_2O_2 or 0.43 mL H_2O_2 and 0.070 mL H_2O . Again using the density of each compound we have:

$$0.629 \text{ g H}_2\text{O}_2 \text{ and } 0.0695 \text{ g H}_2\text{O}$$

$$\text{Total mass} = 0.699 \text{ g}$$

The Mole (mol) fraction of the mixture is then calculated using the molecular weight (MW) of both compounds:

$$\text{MW (H}_2\text{O}_2) \rightarrow 34.0147 \text{ (g/mol)}$$

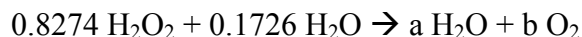
$$\text{MW (H}_2\text{O)} \rightarrow 18.0153 \text{ (g/mol)}$$

The mol fraction (η) for each compound was:

$$\eta (\text{H}_2\text{O}_2) = 0.8274$$

$$\eta (\text{H}_2\text{O}) = 0.1726$$

The chemical equation for theoretical decomposition of the solution was then balanced:



Where a and b are:

$$a = 1 \text{ and } b = 0.414$$

The weight of each compound was found and the mass fraction (mf) was calculated:

$$1 \text{ mol H}_2\text{O} = 18.0153 \text{ g H}_2\text{O}$$

$$0.414 \text{ mol O}_2 = 13.238 \text{ g O}_2$$

The mf for compound are:

$$\text{mf H}_2\text{O} = 0.576$$

$$\text{mf O}_2 = 0.424$$

The mass flow was then solved for using a volumetric flow rate of the H₂O₂ and sample size of 0.5 mL for all flow rates.

Table 4.1 Volumetric Flow Rate H₂O₂ Values Converted to Mass Flow Rate

Vol Flow Rate (mL/min)	Mass Flow Rate (g/s)
0.1	0.00233
0.5	0.01165
1.0	0.02330
2.0	0.04660

Using the mass fraction (mf O₂) solved for earlier and the density of oxygen taken at exhaust conditions (180 °C) the max theoretical oxygen was calculated.

Table 4.2 Maximum Theoretical Oxygen Flow Rate by Mass and Volumetric Flow Rate

Max Theoretical Oxygen Mass Flow (g/s)	Max Theoretical Oxygen Volumetric Flow (mL/min)
0.000987	68.6
0.004935	343.1
0.009869	686.2
0.019739	1372.3

The theoretical oxygen flow rate was chosen to be displayed in mL/min for data comparison purposes. Having the theoretical values in the same units as those measured in the test set up allows for quick calculation and dissemination of cat-bed efficiency. Efficiency was calculated as follows:

$$\frac{\text{Vol Flow O}_{\text{experimental}}}{\text{Vol Flow O}_{\text{Theoretical}}} \times 100 = \text{Cat_Bed Efficiency} \quad (4.1)$$

Using these values the follow plots were made to display cat-bed efficiency for the 30 °C and 150 °C initial test temperatures.

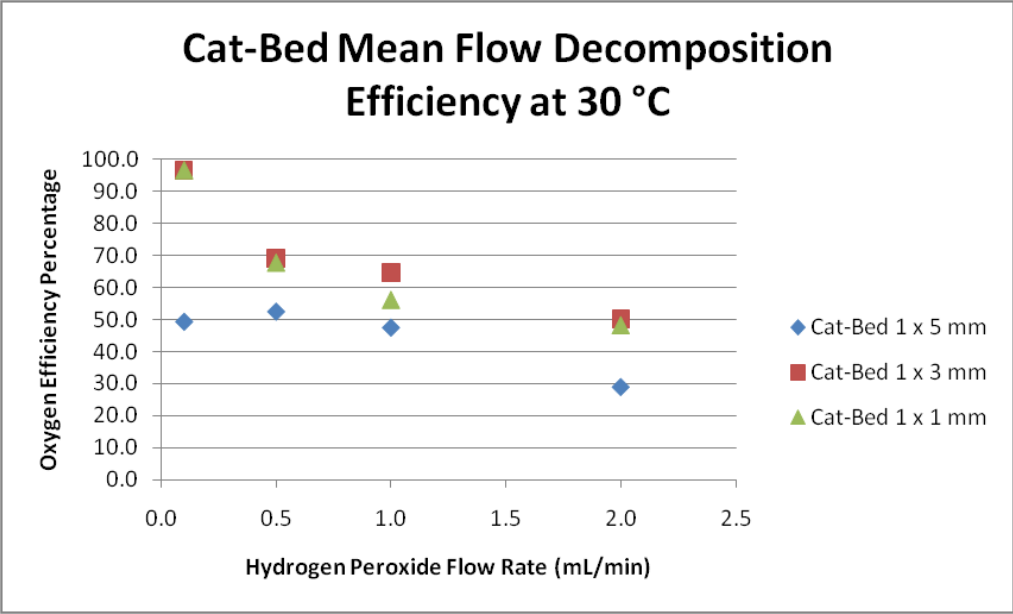


Figure 4.4 Cat-Bed Efficiency at 30 °C

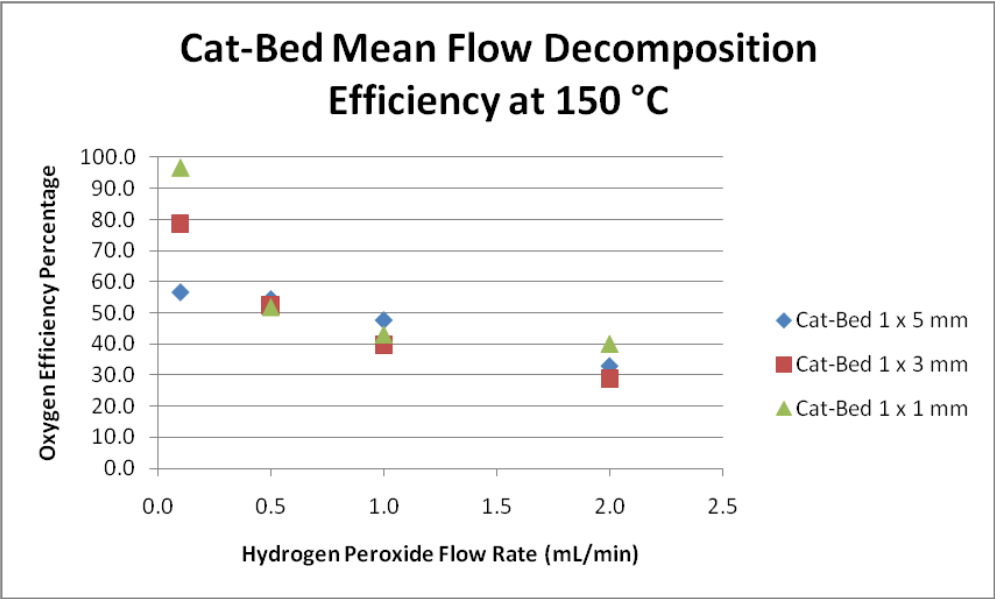


Figure 4.5 Cat-Bed Efficiency at 150 °C

In figure 4.4 the smaller cat-beds (1 x 3 and 1 x 1 mm) show significantly better decomposition at 0.1 mL/min of H₂O₂. Overall these two cat-beds also showed similar results. This flow rate also

displays as one of the highest efficiency rates for all cat-beds. In both figure 4.4 and 4.5 the general trend of efficiency for all cat-beds shows a downward trend as the H_2O_2 increased. In figure 4.4 and 4.5 the decomposition efficiency for the 1 x 5 mm cat-bed showed the least variability in comparison to the other two cat-beds.

The general consensus of all cat-bed performance shows that each decomposes H_2O_2 better at a specific flow rate and initial cat-bed temperature than the others. The overall efficiencies at a flow rate above 0.1 mL/min showed values between 30 and 55 percent. The values found for all cat-beds can in turn be used to investigate the potential of the products released by the cat-bed for propulsion purposes. Essentially high efficiency is desired for better specific impulse (Isp) on a rocket. The flow rate values will also aid in the design of a multichannel cat-bed chamber, considering the plenum delivers a similar H_2O_2 flow rate to each channel.

One aspect that directly relates to a propulsion application is how close the decomposition temperature of the products is to the adiabatic decomposition temperature. This in turn relates how well the reaction is generating heat. For this comparison, the oxygen percentage was multiplied by the enthalpy generation of the experimental results and then divided by the adiabatic enthalpy to give heat efficiency. This was calculated using the specific heat of oxygen at the exhaust temperature of 180 °C.

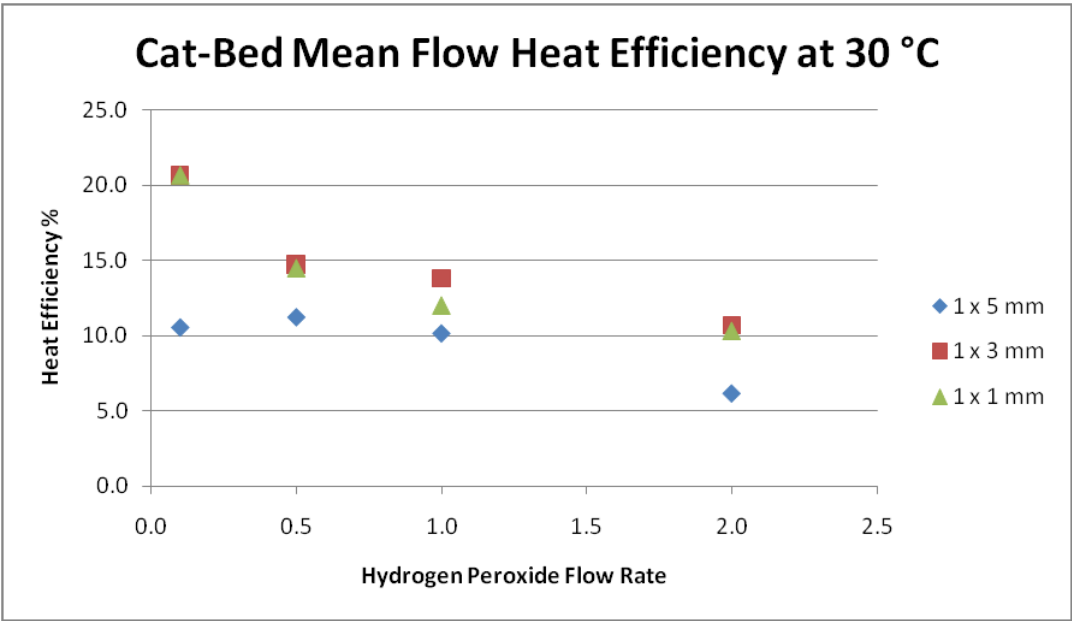


Figure 4.6 Cat-Bed Mean Flow Heat Efficiency at 30°C

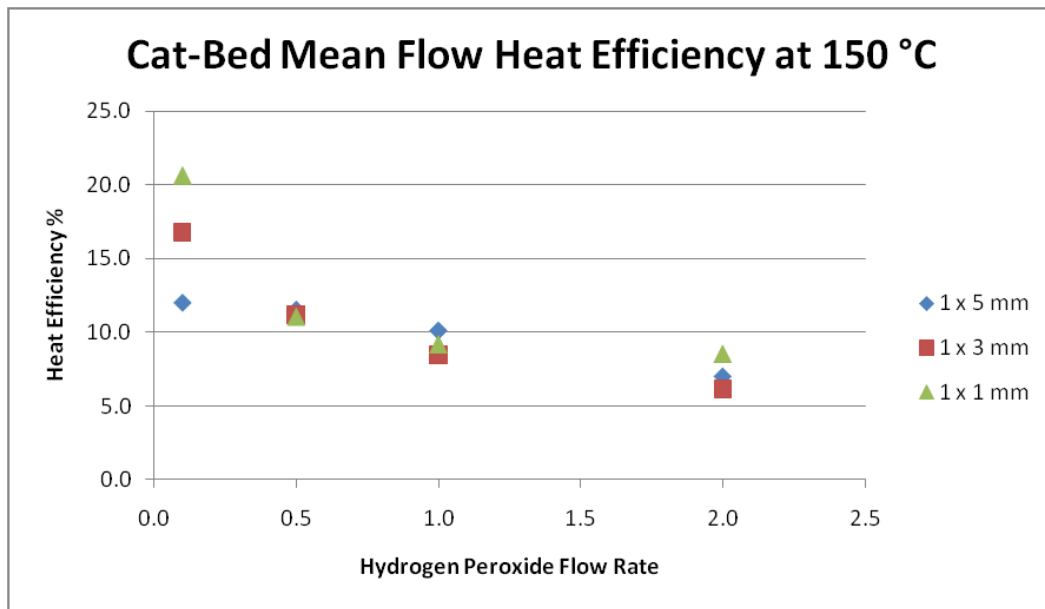


Figure 4.7 Cat-Bed Mean Flow Heat Efficiency at 150°C

The results show that although oxygen efficiency was high at 0.1 mL/min H_2O_2 flow rate but the heat generated was not very applicable to use for combustion or as a monopropellant. However, further testing including chamber pressure measurement would need to be done to verify this applicability for the micro Newton class of thrusters.

The Reynolds number for inlet and outlet conditions was determined. Assumptions stated that the inlet density remained constant at 30 C for the 90% H_2O_2 mixture. Another assumption was based on mass flow calculated using this density. This was then applied to an assumed product calculation based on U.S. Peroxide data posted on mol fraction results for products and Colorado State University data regarding fluid properties of multiple gases. This data was used to calculate a Reynolds number considering an all liquid flow of reactants and a gas flow of products. The Reynolds numbers can be seen below in table 4.3 and table 4.4.

Table 4.3 Reynolds Number of all Liquid Reactants

Flow Rate (mL/min)	RE 1 x 5 mm Cat-Bed	RE 1 x 3 mm Cat-Bed	RE 1 x 1 mm Cat-Bed
0.1	0.73	1.22	3.65
0.5	3.65	6.09	18.27
1.0	7.31	12.18	36.53
2.0	14.61	24.36	73.07

Table 4.4 Reynolds Number of all Gas Products

Flow Rate (mL/min)	RE 1 x 5 mm Cat-Bed	RE 1 x 3 mm Cat-Bed	RE 1 x 1 mm Cat-Bed
0.1	51.42	77.13	154.27
0.5	257.11	385.66	771.33
1.0	514.22	771.33	1542.65
2.0	1028.44	1542.65	3085.31

These Reynolds values show that the inlet stream is very subtle with almost no mixing. The low Reynolds number suggests that a single boundary layer may be found inside the catalyst bed before decomposition. However, the almost detonation like reactivity H_2O_2 has with the catalyst can easily disrupt a smooth decomposition process of incoming H_2O_2 (Hitt and Zhou, 2003). Another insight brought to light by the Reynolds number calculation is the order in magnitude difference between both the liquid phase and gas phase. The exact point inside the cat-bed as to where the phase change occurs is unknown. What is known is that the low Reynolds number promotes a laminar flow and low mixing. This could be a reason in fluctuation of oxygen efficiency and cooling of the cat-beds. The diffusion of H_2O_2 in the solution may not be enough to promote constant decomposition throughout the entire solution as it flows through each cat-bed.

4.2 Summary

The oxygen flow rate was shown and discussed. A decomposition of efficiency of 95% was recorded but at a heat efficiency of 21%. Both the 30 °C and 150 °C tests all showed an increase in oxygen flow rate as the H_2O_2 flow rate was increased. The temperature and efficiency for these tests decreased as the H_2O_2 flow was increased. The heat efficiency of all cat-beds was below 25%, suggesting the reaction temperature is low. The 1 x 5 mm cat-bed varied the least and did not have the highest efficiencies. The other two cat-beds (1 x 3 and 1 x 1 mm) did not show a large difference in test results. The heat efficiency for all three cat-bed designs was calculated and showed values between 7 and 21 percent. This suggests the reaction temperature inside is very low. Reynolds numbers calculated for each cat-bed at all liquid and all gas assumptions were made. The values were all low indicating laminar flow while H_2O_2 was infused into each cat-bed and as the decomposition products expanded.

Chapter 5: Conclusion and Future Work

A test set up was designed and prepared for H_2O_2 use. All safety considerations and guidelines were followed to comply with the procurement of H_2O_2 from FMC Global. Once procured, the test procedure, test matrix, and safety action plan was created to ensure a safe testing environment. Three cat-bed designs were manufactured and tested at two initial conditions, 30 °C and 150 °C. The results from testing show high oxygen efficiency at certain flow rates, but at a low heat efficiency. Oxygen flow rate and cat-bed efficiency decreased for both initial test conditions as the H_2O_2 was increased. The 1 x 5 mm cat-bed did not have the highest efficiency or flow rate, but it varied the least. Both the 1 x 3 and 1 x 1 mm cat-beds showed high flow rates, higher efficiencies, and heat efficiencies. The difference between these two cat-beds is very small.

5.1 Future work

To improve cat-bed performance several parameters can be varied. The flow rate of course directly affects residence time of the H_2O_2 inside the cat-bed. As the flow rate is increased, heat generation in turn should increase as long as H_2O_2 is decomposing. The catalyst can also be changed to induce greater wet ability and reaction rate. The designed surface area can be increased or manipulated in a fashion to provide more area for H_2O_2 to decompose on and increase the cat-bed load. Heating the H_2O_2 before injection into the cat-bed can raise reaction rate temperatures and support greater decomposition at these higher temperatures (Ventura and Wernimont, 2001).

Investigation into trying to characterize the Damkohler number should be done in conjunction with a numerical analysis on the reactor geometries to identify fluid characteristics. The Reynolds number calculated can then be compared to investigate a refined geometry size or how a multi channel design should be approached. The Damkohler number is a ratio of resident time over chemical kinetics time. Relating the length of the cat-bed to decomposition.

Further investigation of the transport characteristics and fluid characteristics can also be done. This can be accomplished by designing a cat-bed with quartz side panels instead of an all metal construction. The cat-bed channel size can be varied as well.

Recommendations for further testing with the current set up are to use possible one more heater on the cat-bed instead of 2. Also, each cat-bed should have its own set of heaters as they corrode and become less efficient with each removal. Future tests should also incorporate obtaining exhaust samples in a sealed cylinder for analysis with the gas chromatograph in the lab. The results obtained would show the exact particulates in the exhaust.

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Appendix

1.0 Passivation: Supplies and Procedure

The amount of each supply needed will be dependent upon the size of the Passivation Operations undertaken for this project. Provided are the mixing ratios for the purpose of estimating the volumes/amounts of materials needed for the parts and storage tanks to be passivated.

1.1 Supplies:

I. Cleaning and Passivation Solutions:

A. For Preparation of Cleaning and Passivation Solutions

i. Detergent: For cleaning equipment and/or hardware

1. 1% solution of
2. Powdered commercial detergents such as:
 - a) Dreft
 - b) Nacconal
 - c) Tide
 - d) Swerl
3. Liquid Detergent such as:
 - a) Kyro-EO
4. DO NOT USE strongly alkaline cleaning solutions
5. Amount/Mixture Ratio:
 - a) 1 part by weight of detergent
 - b) 99 parts by weight of potable water

ii. Trichloroethylene or Perchloroethylene (Commercial Grade)

iii. Sodium Hydroxide

1. For a NaOH Solutions
2. Cleaning Aluminum Equipment: ($\approx 1/15$ N) or 0.25% by weight
 - a) Amount/Mixture Ratio:
 - i) 10 grams, flaked or pelleted NaOH (99+%)

- ii) 1 gallon of clean potable water
 - 3. 10% by weight
 - a) Amount/Mixture Ratio:
 - i) 10 parts NaOH (99%) flaked or pelleted
 - ii) 90 parts by weight of distilled or deionized water
 - b) Large Pyrex Beaker for preparation of solution
 - c) Polyethylene container for storage
- iv. Sulfuric Acid
 - 1. For a solution
 - 2. 35% by weight
 - 3. Amount/Mixture Ratio:
 - a) 1 Volume (approx 95%) Sulfuric Acid (H_2SO_4)
 - b) 3 Volumes of Clean Potable Water
- v. Nitric Acid
 - 1. For a solution
 - 2. Passivation of Stainless Steel Equipment: 42° Baume ($\approx 70\%$ by weight)
 - 3. Passivation of Aluminum Equipment: 35% by weight
 - 4. Amount/Mixture Ratio:
 - a) 1 Volume of Nitric Acid (approx 70%)
 - b) 1 Volume of Clean Potable Water
- vi. Hydrogen Peroxide
 - 1. For a stabilized solution
 - 2. 35% (stabilized)
 - 3. H_2O_2 of concentration to come into contact with the passivated surface

4. If diluted 90% H₂O₂ will be used, a stabilizer is necessary to dilute the H₂O₂ and adjust the pH of the solution
- vii. Clean Potable Water
- viii. Fresh Distilled or Deionized Water
- ix. Hydrofluoric Acid (for Hydrofluoric Acid-Nitric Acid mixture)
 1. 3%-10% Mixtures (respectively)
 2. For pickling and cleaning of Stainless Steel
 3. Amount/Mixture Ratio:
 - a) 1 Volume Hydrofluoric Acid (approx 52%)
 - b) 2 Volumes of Nitric Acid (70%)
 - c) 16 Volumes of clean potable water
- x. Lint free cloths
- B. For Storage of Cleaning and Passivation Solutions
 - i. Detergent
 1. Non Rusting container
 - a) Preferably can be heated to 120°F (49°C)
 - b) Have a lid preventing contamination with dirt
 - ii. Trichloroethylene and Perchloroethylene
 1. Galvanized iron, black iron, or steel container
 - a) Must prevent contamination from water, dirt, and escape of solvent fumes
 2. DON'T USE Perchloroethylene WITH ALUMINUM!
 - iii. Sodium Hydroxide
 1. Stainless Steel Drum or Polyethylene Container
 - iv. Sulfuric Acid
 1. Polyethylene or Polyethylene-lined Container

or

- 2. Glass carboys in which it is received
- v. Nitric Acid
 - 1. Polyethylene-lined or A.I.S.I. 300 Series stainless steel container
- vi. Hydrogen Peroxide (35% and of respective solutions that will actually come into contact with the passivated surfaces)
 - 1. Pure Aluminum 1060, Aluminum Alloy 5652 or 5254 container
 - a) Must have a vent line of a porous stone filter or plastic vent
 - b) Must still keep out dust or dirt
 - 2. H₂O₂ Shipping Drum
- vii. Potable water, distilled water, or deionized water
 - 1. Non-rusting container or piping system
 - 2. Avoid Copper lines and equipment
- viii. Lint free cloths

II. Cleaning and Passivation Facilities and Personal Protection Equipment:

- A. Dust free area for Cleaning, Passivation, Conditioning areas
- B. Must be at least 25 foot distance between the cleaning, passivation, and storage areas and anything that could spark, combust, etc.
- C. Adequate water supply and drain for flushing spilled acid and hydrogen peroxide
- D. Safety showers and eye wash deluge stations
- E. Good ventilation (i.e. Fume hoods)
- F. Safety Warning Signs appropriate for each section
- G. Safety Attire
 - i. Face Shield or goggles
 - ii. Acid Resistant Rubber gloves
 - iii. Acid Resistant Rubbers
 - iv. Apron
- H. Polyethylene beakers of various sizes appropriate to the parts to be treated

- i. Are NOT to be utilized for conditioning or surveillance tests with H₂O₂, unless immersed in water
- I. Glass beakers (can be a substitute for the polyethylene beakers except when utilizing the hydrofluoric-nitric solution)

1.2 Procedure:

- I. Preparation
 - A. Cleaning/Requirements of Passivation Prep Area
 - i. Clean and free of combustibles and ignition sources (i.e. sparks and open flames) for at least a 25 foot radius from the passivation, cleaning, and storage areas
 - ii. °Adequate water supply and drain for disposal of spilled or used acid and hydrogen peroxide
 - iii. Presence of adequate ventilation (i.e. fume hoods)
 - iv. Presence and readily accessible safety showers and eye wash stations
 - v. Presence and readily accessible protective clothing for personnel
 - vi. Storage for and presence of polyethylene and glass beakers for the treatment of parts and surfaces to be in contact with H₂O₂.
 - vii. Presence of storage area for cleaning and passivation solutions
 - B. Cleaning of Surfaces to come in contact with H₂O₂
 - i. Use of commercial grade detergent solutions (preparation details found in the following section) to clean the equipment to be passivated, preparation area, and glassware
 - C. Preparation of Cleaning and Passivation Solutions
 - i. Cleaning Solutions
 - 1. Detergent, 1% Solution

- a) Indications: cleaning equipment and glassware
 - b) Components:
 - i) Powdered commercial detergents such as Dreft, Nacconal, Tide, or Swerl
 - ii) Liquids such as Kyro-EO
 - c) DO NOT USE: strong alkaline cleaning solutions
 - d) Mixing Ratio/Instructions:
 - i) Dissolve: 1 part by weight of detergent in 99 parts by weight of potable water
 - ii) Mix at room temperature
2. Trichloroethylene or Perchloroethylene (Commercial Grade):
- a) Indications: degreasing heavily soiled or greasy metallic equipment
3. Sodium Hydroxide (NaOH) Solution (approx 1/15 N) 0.25% by weight
- a) Indications: cleaning Aluminum equipment
 - b) Components: NaOH and potable water
 - c) Mixing Ratio/Instructions:
 - i) Prepare in a large Pyrex beaker, store in a properly cleaned polyethylene container
 - ii) Dissolve: 10 grams, flaked or pelleted NaOH (99+%) in 1 gallon of clean potable water
 - 1) Dissolve NaOH in ½ gallon of water by constantly stirring at a slow rate
 - 2) Dilute solution with remaining ½ gallon of water; mix thoroughly

- iii) Note: heat is given off when dissolving the NaOH in the water

4. Sodium Hydroxide (NaOH) Solution, 10% by weight

- a) Indications: cleaning heavily soiled glassware, or glassware previously containing unknown solutions
- b) Components: NaOH 99% flaked or pelleted, Distilled or deionized water
- c) Preparation Container: Large Pyrex beaker, Storage Container: Polyethylene container
- d) Mixing Ratio/Instructions:
 - i) 10 parts by weight of NaOH 99% flaked or pelleted
 - ii) 90 parts by weight of distilled or deionized water
- e) Heat will be emitted during the preparation of this solution

ii. Passivation Solutions

1. Sulfuric Acid, 35% by weight

- a) Indications: Passivating glassware
- b) Components: Sulfuric Acid (approx 95%) and clean potable water
- c) Mixing Ratio/Instructions:
 - i) Constant stirring, adding slowly
 - ii) Always add acid to water
 - iii) Dissolve: 1 volume of sulfuric acid (approx 95%) in 3 volumes of clean potable water
- d) Overheating of the container could occur when making large volumes—alleviate by:
 - i) Add acid to the solution slowly

- ii) Use a water bath around the beaker to dissipate the heat
 - iii) Try to avoid cracking the mixing container, especially if it is glass
 - 2. Nitric Acid, 42° Baume (approx 70% by weight)
 - a) Indications: Passivating Stainless Steel equipment
 - b) Store in the containers in which it is received
 - 3. Nitric Acid, 35% by weight
 - a) Indications: Passivating Aluminum equipment
 - b) Components: Nitric Acid (approx 70%) and clean potable water
 - i) Standard or technical grade acid may be used
 - c) Mixing Ratio/Instructions:
 - i) Constant stirring, add slowly
 - ii) Always add acid to water
 - iii) Dissolve: 1 volume of Nitric Acid (approx 70%) to 1 volume of clean potable water
 - d) DO NOT INHALE FUMES
- iii. Conditioning Solutions
 - 1. Hydrogen Peroxide (H₂O₂), 35%, Stabilized
 - a) Indications: Conditioning of parts, drums, and tanks after passivation
 - b) Components: H₂O₂, distilled or deionized water, stabilizer
 - c) Mixing Ratio/Instructions:
 - i) If already in 35% stabilized solution from the manufacturer, use as is
 - ii) If diluted from 90% H₂O₂

- 1) Add a stabilizer to the diluted H₂O₂
 - 2) Adjust the pH of the solution
 - iii) Dilution water must be sufficiently distilled or deionized
 - iv. Pickling and Cleaning Solution
 1. Hydrofluoric Acid-Nitric Acid Mixture, 3%-10% respectively
 - a) Indications: Stainless Steel with rust or surface contamination that cannot be removed by the passivation acids
 - b) Components: Hydrofluoric Acid (approx 52%), Nitric Acid (70%), clean potable water
 - c) Mixing Ratio/Instructions
 - i) Add slowly with constant stirring
 - ii) Always add acid to water
 - iii) Dissolve: 1 volume of hydrofluoric acid and 2 volumes of nitric acid in 16 volumes of clean potable water
 - iv) Example: for a 10 gallon mixture:
 - 1) 2 quarts of Hydrofluoric acid (52%)
 - 2) 1 gallon Nitric acid (70%)
 - 3) 10 gallons of water
 - v. Rinsing Materials
 1. Clean potable water
 - a) Indications: Rinsing parts during passivation
 - b) Components: drinking water
 2. Fresh distilled and deionized water
 - a) Indications: Rinsing parts after passivation

- b) Cannot be stored in aluminum for longer than one week before or during usage
- D. Storage of Cleaning and Passivation Solutions
 - i. Detergent
 - 1. Non Rusting container
 - a) Preferably can be heated to 120°F (49°C)
 - b) Have a lid preventing contamination with dirt
 - ii. Trichloroethylene and Perchloroethylene
 - 1. Galvanized iron, black iron, or steel container
 - a) Must prevent contamination from water, dirt, and escape of solvent fumes
 - 2. DON'T USE Perchloroethylene WITH ALUMINUM!
 - iii. Sodium Hydroxide
 - 1. Stainless Steel Drum or Polyethylene Container
 - iv. Sulfuric Acid
 - 1. Polyethylene or Polyethylene-lined Container
 - or
 - 2. Glass carboys in which it is received
 - v. Nitric Acid
 - 1. Polyethylene-lined or A.I.S.I. 300 Series stainless steel container
 - vi. Hydrogen Peroxide (35% and of respective solutions that will actually come into contact with the passivated surfaces)
 - 1. Pure Aluminum 1060, Aluminum Alloy 5652 or 5254 container
 - a) Must have a vent line of a porous stone filter or plastic vent
 - b) Must still keep out dust or dirt
 - 2. H₂O₂ Shipping Drum
 - vii. Potable water, distilled water, or deionized water

1. Non-rusting container or piping system
2. Avoid Copper lines and equipment
- viii. Lint free cloths

II. Passivation

A. Passivation

- i. Aluminum and Aluminum Alloy Parts and valves
 1. Degrease
 - a) Agitate parts in a warmed (to approx 120°F / 49°C) 1% detergent solution
 - b) Tubing and Piping: pump solution through or agitate with the solution inside of them
 2. Rinse with Potable Water
 - a) **Handle parts wearing gloves or with tongs until the completion of the drying process**
 3. Immersion in 0.25% NaOH
 - a) Duration: 15-20 minutes
 - b) Room Temperature (approx 25° C)
 - c) Caution: hydrogen gas release; no spark or open flame within 25 feet of operation
 4. Rinse with potable water
 5. Immersion/Fill with 35% Nitric Acid
 - a) Duration: 1 hour
 - b) Room Temperature (approx 25° C)
 6. Rinse thoroughly with Distilled or Deionized water
 7. Continue to Conditioning of Aluminum and Aluminum Alloy Parts and Valves (detailed under like titled section)
- ii. Aluminum Tanks

1. Degreasing
 - a) Degrease if tank is really greasy
 - b) Wash with a 1% detergent solution
 - i) AVOID
 - 1) chlorinated solvents due to their hazard potential
 - 2) Flammable solvents
 - 3) Residue forming solvents
 - c) Rinse with clean potable water
2. Pump clean potable water into the tank being treated until $\frac{1}{2}$ full
3. Pump or pour the required Nitric Acid (Solutions approx 5% Nitric Acid)
4. Fill remainder of the tank with clean potable water
5. Acid solution to stay in the tank for 1 to 2 days
 - a) Leave nipples and manholes open
 - b) Protect from dirt and contamination by covering with polyethylene or a vinyl sheet
6. Utilize Residual Acid solution for the treatment of up to 3 tanks/make decision based on the amount of dirt and grease in the acid
 - a) Discard after use
 - b) DO NOT STORE nitric acid in aluminum tanks
7. Rinse treated tank with clean potable water
8. Dry with a clean, lint-free cloth
 - a) Use rubber gloves
 - b) Avoid scratching the bottom of the tank
9. Examine the inside of the tank for iron spots

- a) Dig out with a knife point
 - b) Scrub unpassivated spots with detergent to remove grease preventing contact with of the acid with the metal surface.
10. Repeat steps 2-9 for a second time
11. If the interior of the tank is coated uniformly with a dull, velvety finish, the tank is passivated satisfactorily. If not, repeat the passivation process.
12. Continue to the conditioning process (see following section entitled “Conditioning of Aluminum Tanks”)
- iii. Special Aluminum Tank Treatment: Modified Passivation Procedure
 1. Treat with approx 0.25% Sodium Hydroxide
 2. Add 35% Nitric Acid to the tank by hand pump or by a small motor driven pump to fill the tank $\frac{1}{2}$ way
 3. Seal the tank and roll intermittently
 - a) Duration: 1-2 hours
 - b) Room temperature
 4. May fill tank completely with 35% Nitric Acid and allowed to stand for 1 to 2 hours at room temperature in lieu of step 3
 5. Rinse thoroughly with distilled water
 6. Condition tank with stabilized 35% H₂O₂ in the manner described in the “Conditioning of Aluminum Tanks” Section
- iv. Stainless Steel Parts
 1. Disassemble part assemblies into components
 2. Degrease
 - a) Degrease with trichloroethylene or Perchloroethylene and allow to drip dry before washing in detergent solution if exceptionally greasy

- b) Wash by agitating in a 1% detergent solution warmed to 120°F/49°C
 - c) Scrub with a stiff brush
 - d) Pump solution through or agitate with solution in the tank, tubing and piping
 - 3. Flush with clean potable water immediately following degreasing/washing.
 - a) **Handle parts with gloved hands or clean stainless steel tongs from this point forward**
 - 4. Immerse or fill with 70% Nitric Acid
 - a) Duration: 4-5 Hours
 - b) Room Temperature
 - 5. Rinse thoroughly with distilled water
 - 6. Alternate Passivation Procedure
 - a) Indications: successful on hardened stainless steel such as Armco 17-7 PH
 - b) Immersion in 2% Na₂Cr₂O₇-2H₂O – 20% HNO₃ Solution
 - i) Duration: ½ hour
 - ii) Temperature: 120-130°F (49-54°C)
 - c) Flush with clean potable water and then distilled water
 - d) Dry
 - 7. Continue to “Conditioning of Stainless Steel Parts” Section
- v. Stainless Steel Tanks
 - 1. Degrease
 - a) First with trichloroethylene or perchloroethylene
 - b) Then subject to a 1% detergent wash
 - 2. Rinse thoroughly with clean potable water

3. Examine tank for welding scale, rust, or inorganic contaminants; if observed:
 - a) Brush welds with A.I.S.I 300 series stainless steel brush
 - b) Fill tank with 3% Hydrofluoric acid and 10% Nitric Acid Pickling solution
 - i) Duration: 2-3 hours
 - ii) Room Temperature 65° -70° F (18°-21°C)
 - c) Remove and rinse with clean tap water
 - d) Brush welds again with stainless steel brush
 - e) Clean out debris and make sure bristles are not stuck in the welds
 4. Fill tank with approx 70% Nitric Acid
 - a) Duration: 4-5 Hours
 - b) Room Temperature
 5. Previous step can be substituted with 35% Nitric Acid
 - a) Duration: 16-24 hours
 6. Rinse with distilled water
 7. Continue to “Conditioning of Stainless Steel Tanks” Section
- vi. Glassware
1. Cleaning:
 - a) Heavily Soiled:
 - i) Immerse in 10% NaOH
 - ii) Duration: 1 hour
 - b) Relatively Clean:
 - i) Wash with 1% detergent
 - ii) Omit NaOH immersion to avoid decrease in inertness of surface to H₂O₂

2. Rinse in clean potable water
3. Immerse in 35% Sulfuric Acid
 - a) Duration: 1 hour
 - b) Room temperature
4. Rinse in distilled water
5. Air dry or dry in an oven at 110° C (230°F)
6. Store ready for immediate use by covering with aluminum foil

B. Conditioning

i. Conditioning of Aluminum and Aluminum Alloy Parts and Valves with H₂O₂

1. Duration: 3-4 Hours
2. Note: Rate of H₂O₂ Decomposition (Further detailed in the section titled Assessment of Passivation Coating)

3. Process:

- a) Immersion in H₂O₂ of concentration mimicking that of the conditions under which the part, etc. must function
- b) Block outlets of large parts (i.e. valve bodies, tubing, piping, etc.) and fill with H₂O₂ of concentration mimicking that of the conditions under which it must function
- c) Watch for:
 - i) Heating of the part
 - ii) Decomposition of H₂O₂

4. Move on to Assessment of Passivation Coating

ii. Conditioning of Aluminum Tanks

1. Fill with stabilized 35% H₂O₂ for a 1-3 day duration
2. Fill with concentrated H₂O₂

3. Move on to “Assessment of Passivation, Section ii. Aluminum Tank”
- iii. Conditioning of Stainless Steel Parts
 1. Duration: 3-4 hours
 2. Note: Rate H₂O₂ Decomposition (Further detailed in section titled Assessment of Passivation Coating)
 3. Process:
 - a) Immerse small parts directly into the H₂O₂ solution of concentration mimicking the conditions under which the part must function
 - b) Block outlets of large parts (i.e. valve bodies, tubing, piping, etc.) and completely fill with H₂O₂ of concentration mimicking that of the conditions under which it must function
 - c) Watch for:
 - i) Heating of the part
 - ii) Decomposition of H₂O₂
 4. Move on to the Assessment of the Passivation Coating section specifically for Stainless Steel Parts
- iv. Conditioning of Stainless Steel Tanks
 1. Fill with 35% H₂O₂ (FMC Becco Commercial grade)
 - a) Duration: 1-3 Days
 - b) Prior to filling with high concentration H₂O₂
 2. Assess the passivity of the tank surfaces (see Assessment of Passivation Coating, Section iv. “Stainless Steel Tanks”)
- C. Assessment of Passivation Coating
 - i. Aluminum and Aluminum Alloy Parts and Valves with H₂O₂

1. Signs of Excessive Decomposition
 - a) Great deal of bubbling or gas streamers
 - b) Heating of the part or of the H₂O₂
2. Procedure for Occurrence of Excessive Decomposition
 - a) Remove the part from the H₂O₂
 - b) Wash with water
 - c) Inspection of the part
 - i) If no defect is found:
 - 1) Repeat steps 5-7 of the Passivation Process for Aluminum and Aluminum Alloy Parts
 - 2) If excessive decomposition continues to occur after the 2nd treatment, REJECT the part
 - 3) Be primarily concerned with the areas of the part that will come into contact with the H₂O₂ in the final assembly.
 - 4) Activity on surfaces that won't be in contact with the H₂O₂ in the final assembly should not be cause for rejection
3. Rejection of Parts-reject parts if:
 - a) Possesses a defect that causes excessive decomposition (esp. on surfaces that will contact the H₂O₂ in final assembly)
 - b) Fails during the repeat of the passivation and conditioning processes after the initial failure
 - c) the part discolors the H₂O₂
4. Remove part from H₂O₂, flush with distilled water and Inspect

- a) Return parts if they exhibit blackening, rust streaks, or signs of corrosion
- 5. Disposal of H₂O₂ utilized for Conditioning upon completion of the process
 - a) Dilute H₂O₂ with 10 volumes of water
 - b) Discard down the drain
 - c) Avoid contamination by not handling waste H₂O₂ in the systems specific for the fresh H₂O₂
- 6. Air dry or oven dry conditioned parts
 - a) Only use the oven drying if the part does not contain plastic inserts
 - b) Keep the dust and dirt from the parts during drying
- ii. Aluminum Tanks
 - 1. Check Compatibility by observing; Watch for:
 - a) Indicators of Incompatibility
 - i) Steady bubbling in tank
 - ii) Gas streamers in tank
 - b) Compatibility
 - i) H₂O₂ solution is quiet and cool
 - 2. Pump solution out of the tank
 - 3. Rinse tank with distilled or deionized water
 - 4. Pump in the concentrated H₂O₂
 - 5. Dry with a clean, lint-free cloth after washing if the tank is to be stored for more than one week.
- iii. Stainless Steel Parts
 - 1. Procedure for occurrence of excessive decomposition
 - a) Indicators of excessive decomposition:

- i) Great deal of bubbling or gas streamers
 - ii) Heating of the part or the H₂O₂
- b) Remove from H₂O₂, wash with fresh water and inspect
- c) Repeat nitric acid immersion and/or alternative passivation procedure if no defects are found
- d) Pickle the part with 3% HF – 10% HNO₃ Solution if excessive H₂O₂ decomposition continues after secondary treatment (see Section II, Part d “Pickling of Stainless Steel”)
- e) Take into account the necessity of the aforementioned process, if the unpassivated area will not come in contact with H₂O₂ after final assembly, this is unnecessary
- f) Conditions indicating the need for pickling:
 - i) Discoloration of the H₂O₂
 - ii) Causes of Discoloration
 - 1) Light Pink to Purple: Chromium contamination
 - 2) Blue: Copper Contamination
 - 3) Yellow: Iron or titanium Contamination
- 2. Remove the parts from the H₂O₂ and rinse with distilled or deionized water
- 3. Inspect for rust and corrosion
 - a) If rust is found repeat Nitric Acid immersion and/or alternate passivation procedure
 - b) Pickle again if rust persists
- 4. Dilute and discard H₂O₂ after completion of the conditioning
- 5. Parts considered conditioned if there are no unfavorable results

6. Air dry or oven dry the parts below the melting point of any plastic inserts within the part
 - a) Prevent contact with dust and dirt during drying
 - b) Dry tanks unless immediately using
7. Reassemble the parts
- iv. Stainless Steel Tanks
 1. Passivity determined by observation
 2. Tank is compatible if:
 - a) No steady bubbling or gas streamers
 - b) H_2O_2 solution is quiet and cool
 3. Pump out 35% H_2O_2 and either reuse or dispose of it
 4. Rinse tank with distilled or deionized water
 5. Fill with the concentrated H_2O_2
 6. **For the first filling with concentrated H_2O_2 , observe for 16 to 24 hours for temperature rises of the tank or excessive gas formation**
- D. Pickling of Stainless Steel
 - i. Indications: cast stainless steel surfaces, stainless steel welds, and rusted surfaces
 - ii. Process:
 1. Coat gaskets and surfaces subject to damage by HF- HNO_3 solution with petrolatum
 2. Immerse in 3% HF acid and 10% Nitric Acid
 - a) Duration: 30 minutes
 - b) Temperature: 100°F (38°C)
 - c) Could also immerse for 3 hours at 65-70°F (18-21°C)
 - d) Keep HF- HNO_3 away from sealing surfaces

- e) Immersion can be substituted with use of a paste made of 3% HF – 10% Nitric Acid in graphite on surfaces to be treated
- 3. Flush with potable water
- 4. Scrub with stiff brush to remove welding scale and petrolatum coating
- 5. Repeat the passivation process specific to the Stainless Steel (for either the part or tank)
- 6. If passivation is still not achieved, repeat passivation and conditioning steps
- 7. Reject the part if it still isn't passivated after the second treatment.

E. Cleaning Plastics

- i. Indications: for plastic parts, “O” Rings, and gaskets
- ii. Process:
 - 1. Wipe with a lint free rag to clean as much as possible
 - 2. Degrease
 - a) Agitate part in 1% detergent solution, warmed to 49°C (120°C)
 - b) Do not heat to boiling as it may damage part
 - 3. Rinse thoroughly with distilled or deionized water
 - 4. Immerse into or fill with 20% by weight nitric acid
 - a) Duration: 1 hour
 - b) Temperature: 20-22°C (68-72°F)
 - 5. Submerge in H₂O₂ of concentration it will encounter during service
 - a) Duration: 16-24 hours
 - b) Note rate of H₂O₂ decomposition

6. Flush with distilled water and inspect
7. Reassemble or package if part is satisfactory

III. Packaging

- A. Either use or seal immediately after completion of the process
- B. Seal should block against dust, dirt, and other contaminants
- C. Label packaging with the name and number of the part, passivation date, and name of the inspector
- D. Storage Process
 - i. Small parts sealed in polyethylene or vinyl bags
 - ii. Large parts should have polyethylene plugs inserted in the external threads and openings
 - iii. Wash all air out before sealing bags or parts with plugs using a dry nitrogen gas (to remove atmospheric moisture).

IV. Passivity Checks

- A. Small amounts of H₂O₂ decomposition is expected during initial filling of equipment
- B. Surveillance of the system should be conducted for the duration of a specific amount of time after the initial filling of equipment with H₂O₂ to make sure that significant decomposition is not occurring.
- C. Conditions that affect the decomposition rate:
 - i. Temperature
 1. Rate of decomposition will increase with increase in temperature
 2. Rate:
 - a) $+10^{\circ}\text{F} = 1.6 \times \text{rate of decomposition}$
 - b) $+10^{\circ}\text{C} = 2.4 \times \text{rate of decomposition}$
 - ii. Amount of surface

1. H₂O₂ Decomposition almost exclusively occurs on the surface of the container/contact surface, not within the body of the solution
 2. Rate of Decomposition= (Area of surface wetted)/(Volume of solution)
- iii. Type of Surface
1. Stainless steel decomposes H₂O₂ at a rate that is 3 to 10 faster than pure aluminum
 2. The smoother the surface the lower the rate of H₂O₂ decomposition
- D. Passivity Checks of Individual Components of Flow Systems
- i. Indication: for the quantitative determination of passivity
 - ii. Testing should be done with H₂O₂ of solution concentration that system will encounter during service
 - iii. Satisfactory equipment for use, H₂O₂ must meet these limits:
 1. Max loss of concentration: 0.7%
 2. Max temp rise (above ambient): 2.7°C (5°F)
 3. Min stability after the test: 90%
- E. Passivity Checks of Hydrogen Peroxide Flow Systems
- i. Surveillance
 1. Duration: 6 hours
 2. First time complete system is filled with H₂O₂
 - ii. Measure:
 1. Temperature
 - a) Check temperature of all parts of the outside of the tank
 - b) Can be measured qualitatively by feeling with one's hand

- c) Remotely measured using a thermocouple wrapped around the outside of the container; allows for the determination of hot spots as well

2. Rate of Decomposition

- a) Criteria to determine if system is passive:
 - i) Detection of Hot spots (2.7°C warmer than surrounding)
 - 1) Component replaced and repassivated
 - 2) Passivity test will be repeated
 - ii) Rate of decomp is excessive without being attributed to specific component(s)
 - 1) Repassivation of entire system
 - 2) Excessive rate of decomposition
 - a) Reduce the H_2O_2 concentration to point where it can no longer be utilized in the specific system, for the determined use within the desired amount of time
 - b) Cause H_2O_2 to heat 2.7°C above ambient
 - 3) Be sure to provide a margin of safety, don't deem acceptable if it is within a "safety margin" of the maximum and minimum of the criteria

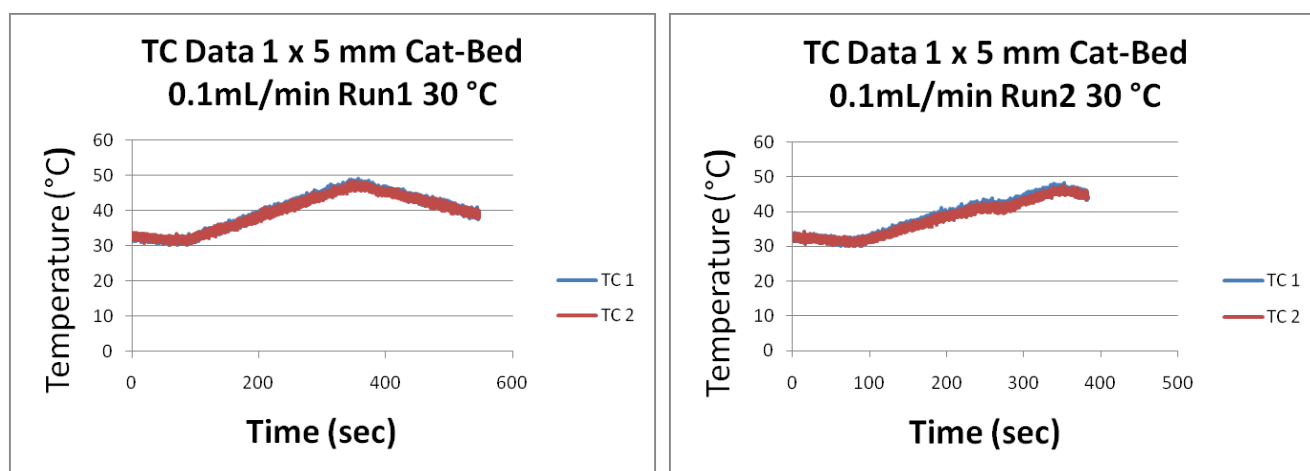
F. Passivity Checks of High Strength H_2O_2 Aluminum Storage Tanks

i. Surveillance

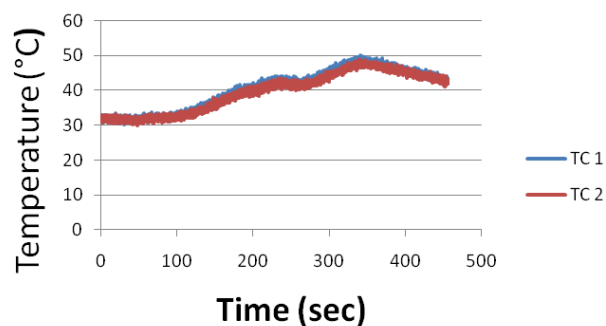
- 1. Duration: Minimum of 24 hours, Preferred to last 72 hours

2. Observe hourly
 3. Temperature of the solution and the surrounding air monitored with thermocouples and thermometers
 4. Temperature of all parts outside of tank quantitatively measured by touch
 5. Mark all hot spots found
- ii. Rate of Decomposition determined by sampling at the start and end of surveillance period
 - iii. Tank is passive if it meets the following criteria during surveillance:
 1. No steady bubbling or gas streamers from the H₂O₂
 2. No local hot spots (approx 2.7°C warmer than surrounding areas)
 3. No increase in temperature of solution (not related to increase in air temp)
 4. Rate of H₂O₂ decomposition is within acceptable range
 - iv. Tank must be repassivated if it fails to meet any of these criteria
 - v. Check H₂O₂ removed from improperly passivated tank for stability before being transferred to another tank

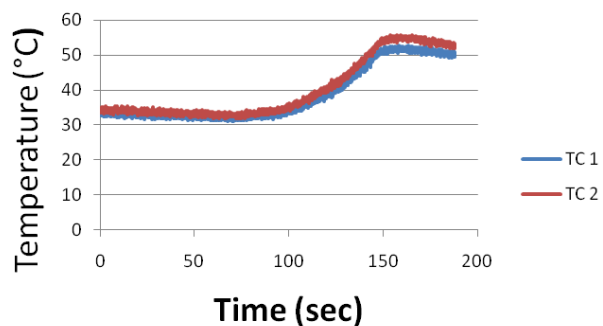
2.0 Cat-Bed Temperature Data



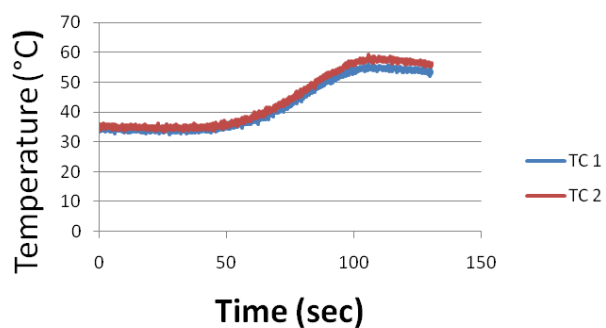
**TC Data 1 x 5 mm Cat-Bed
0.1mL/min Run3 30 °C**



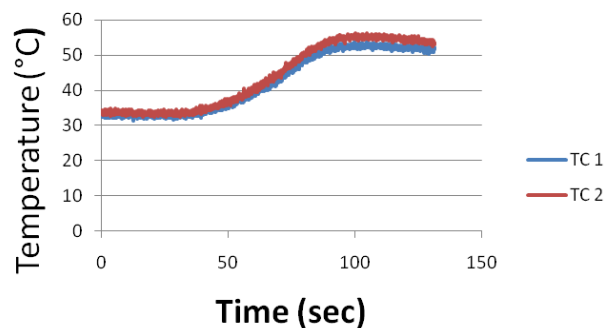
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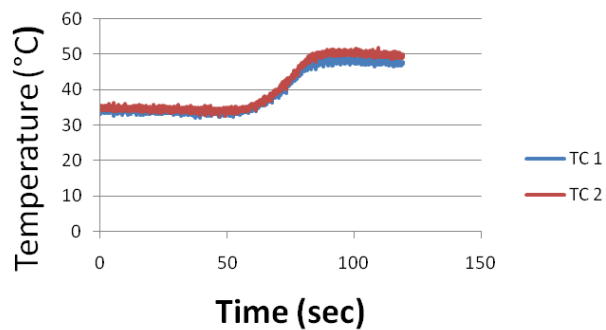
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0.5mL/min Run2 30 °C**



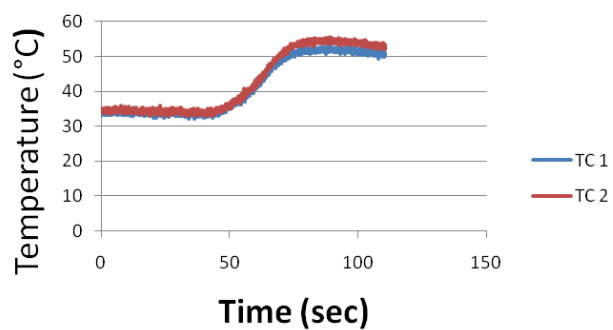
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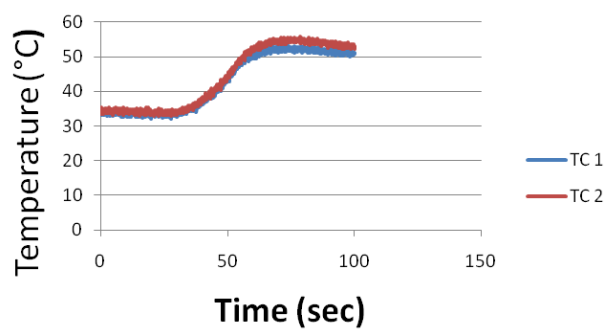
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1.0mL/min Run1 30 °C**



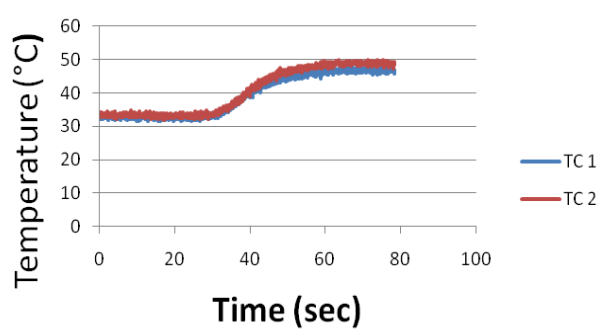
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1.0mL/min Run2 30 °C**



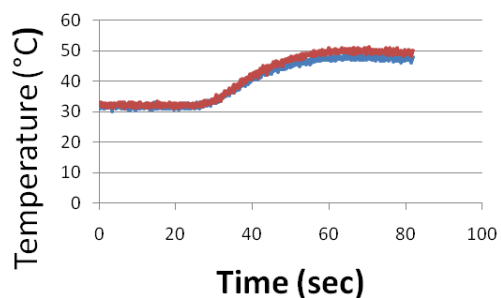
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1.0mL/min Run3 30 °C**



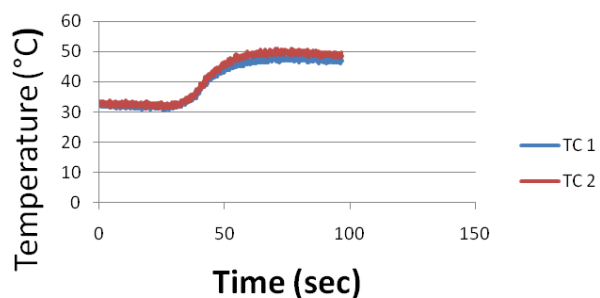
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2.0mL/min Run1 30 °C**



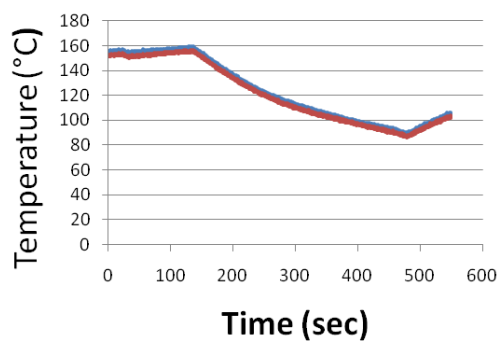
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2.0mL/min Run2 30 °C**



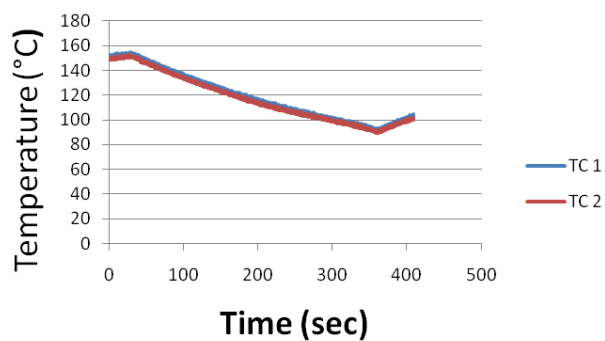
**TC Data 1 x 5 mm Cat-Bed
2.0mL/min Run3 30 °C**



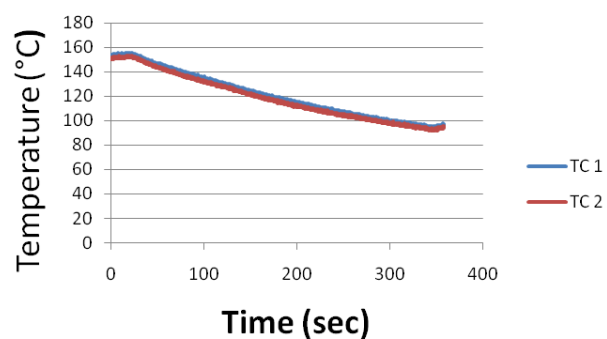
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0.1mL/min Run1**



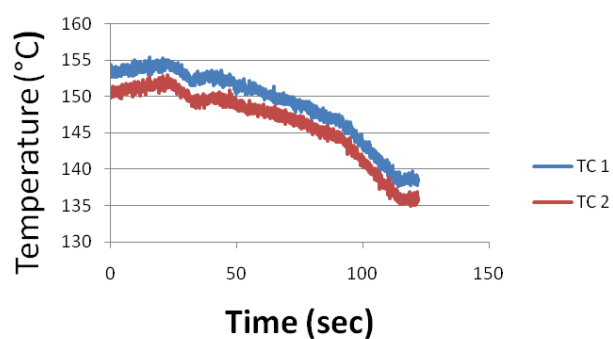
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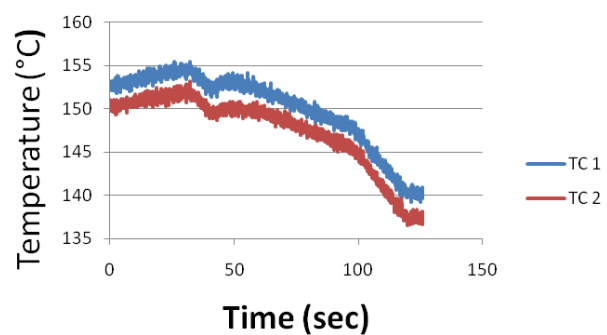
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0.1mL/min Run3**



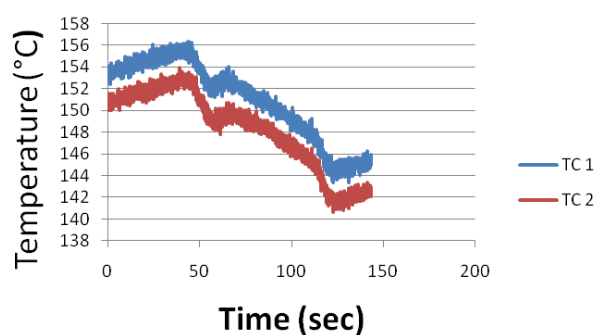
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0.5mL/min Run1**



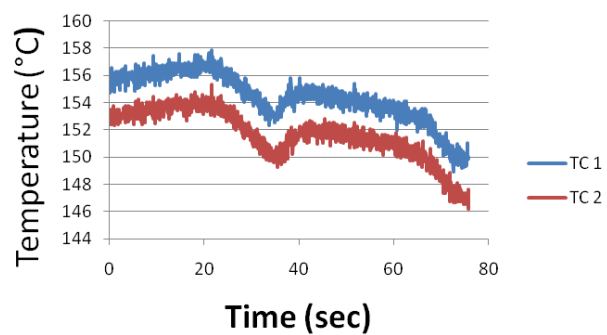
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0.5mL/min Run2**



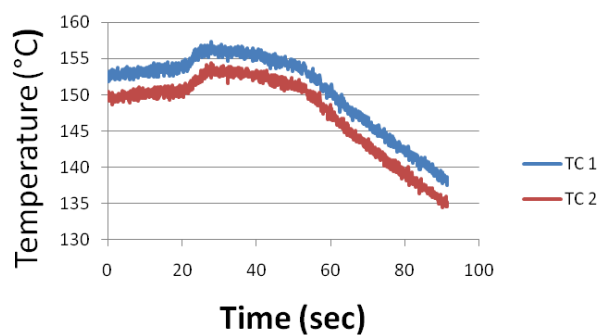
**TC Data 1 x 5 mm Cat-Bed
0.5mL/min Run3**

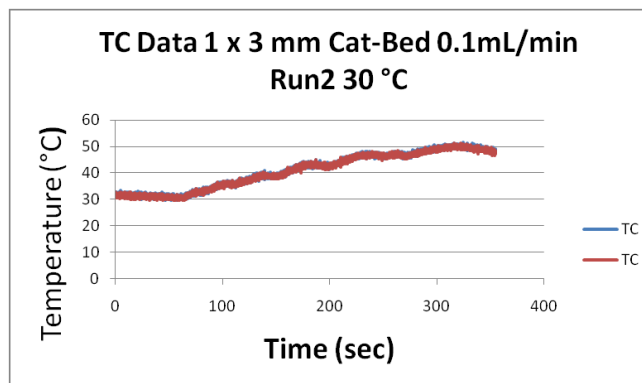
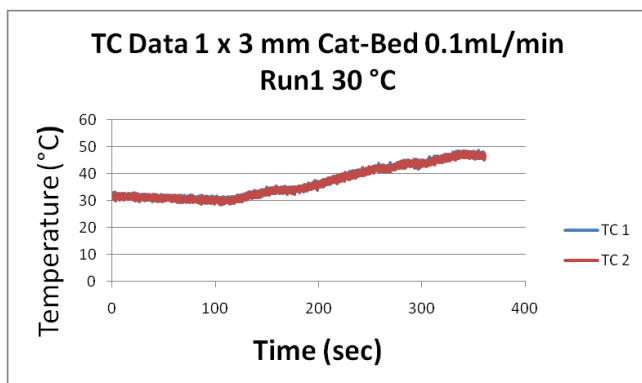
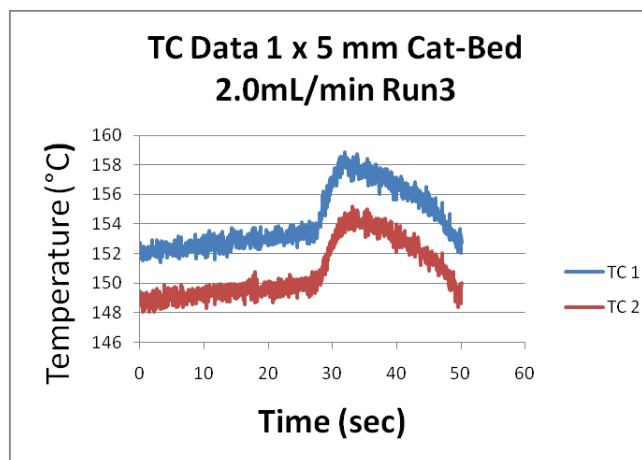
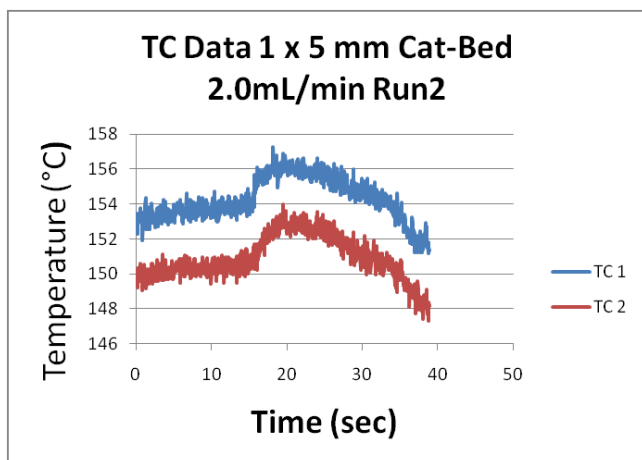
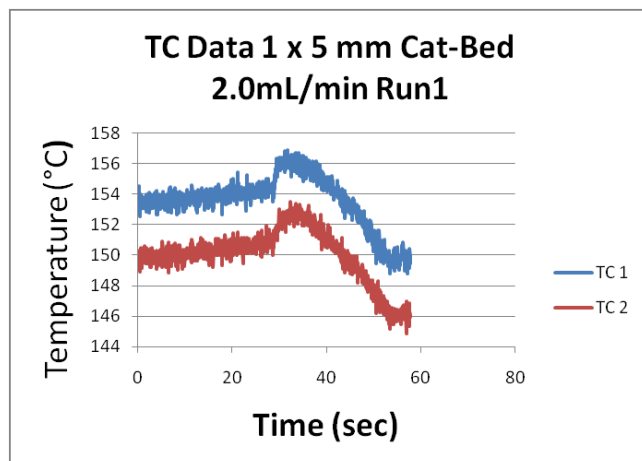
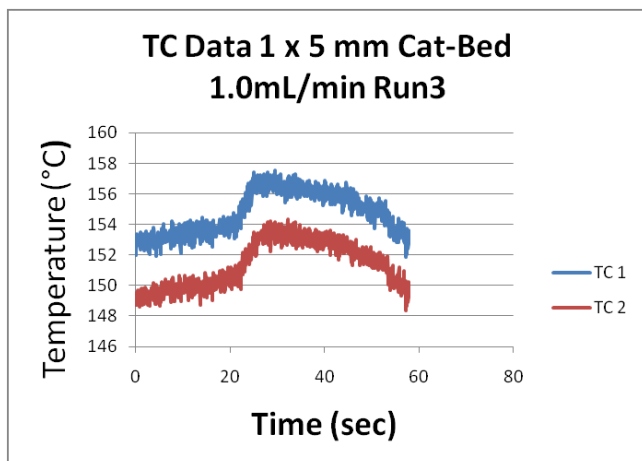


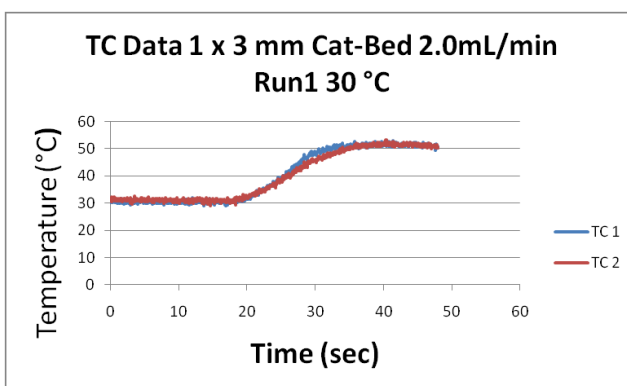
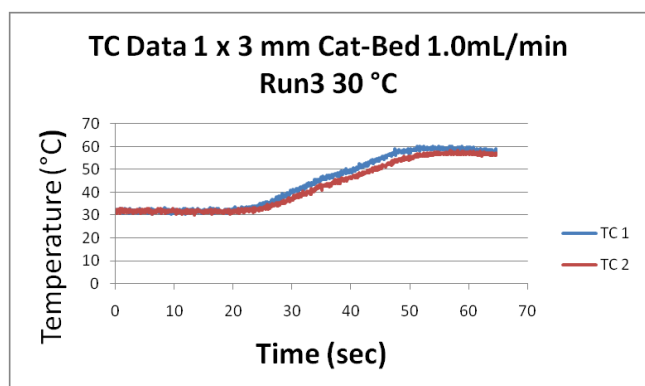
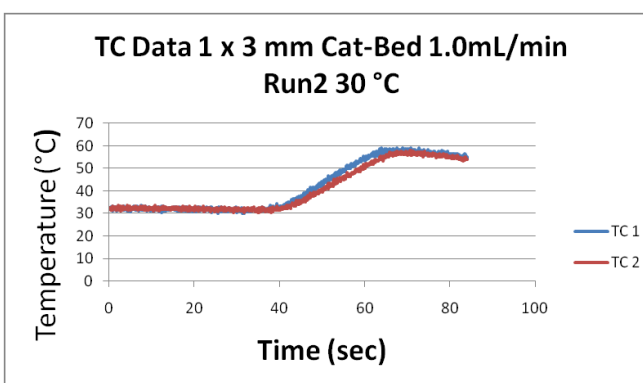
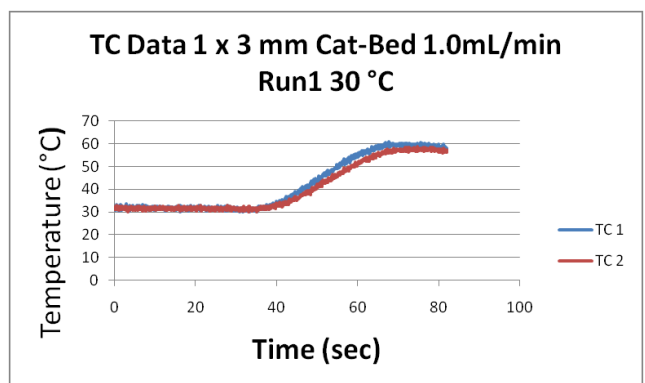
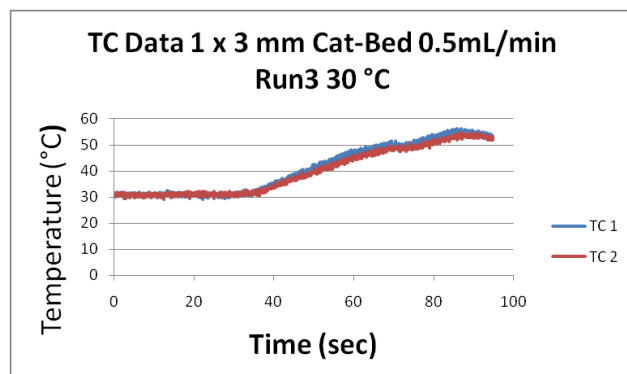
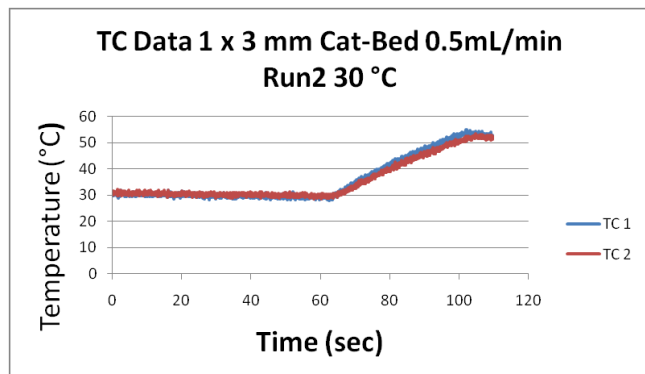
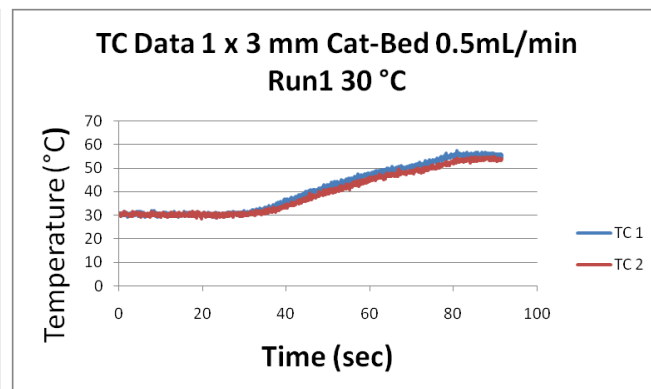
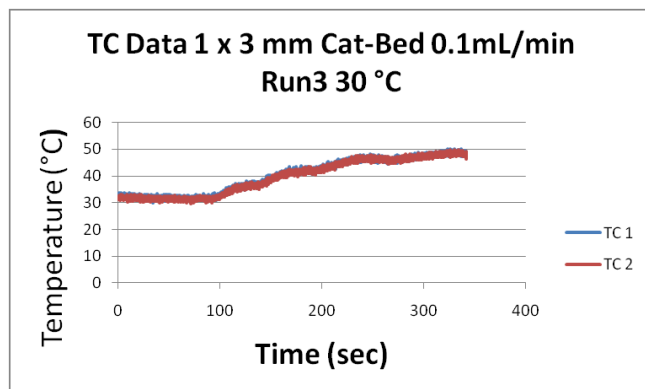
**TC Data 1 x 5 mm Cat-Bed
1.0mL/min Run1**

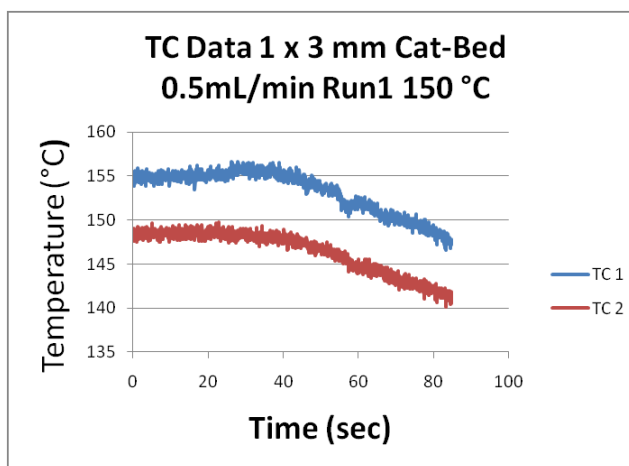
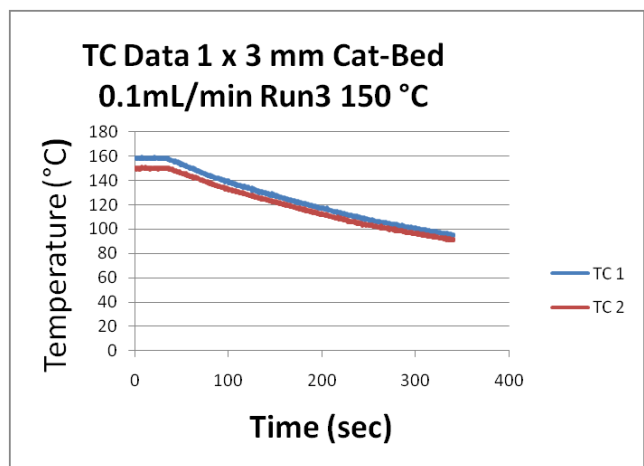
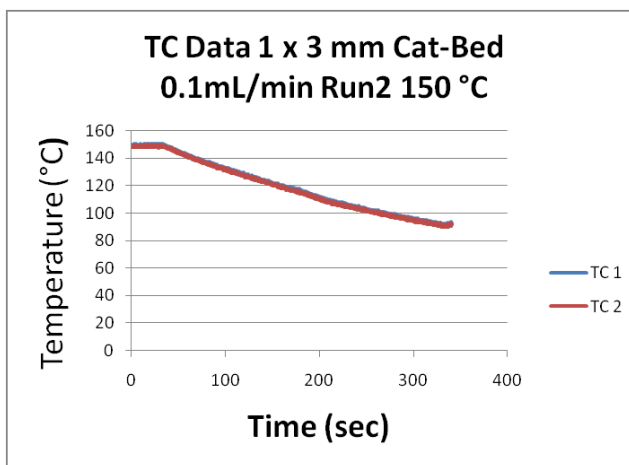
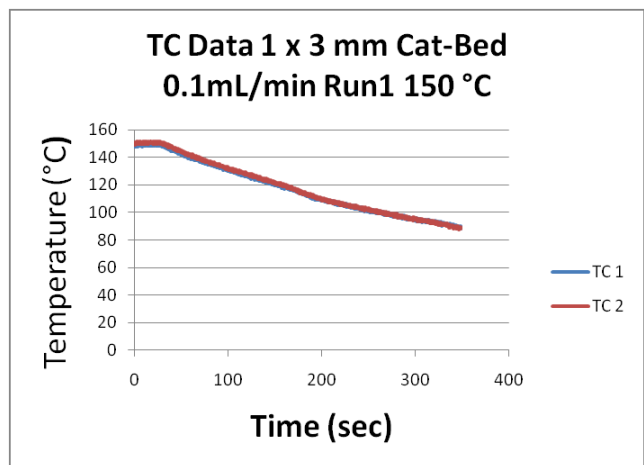
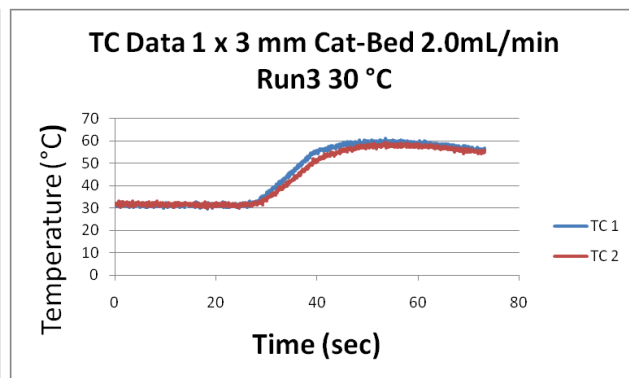
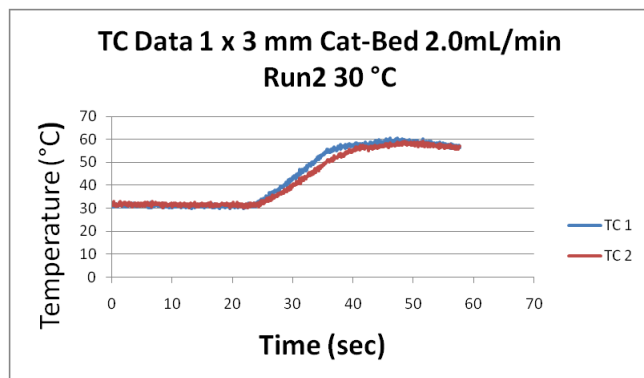


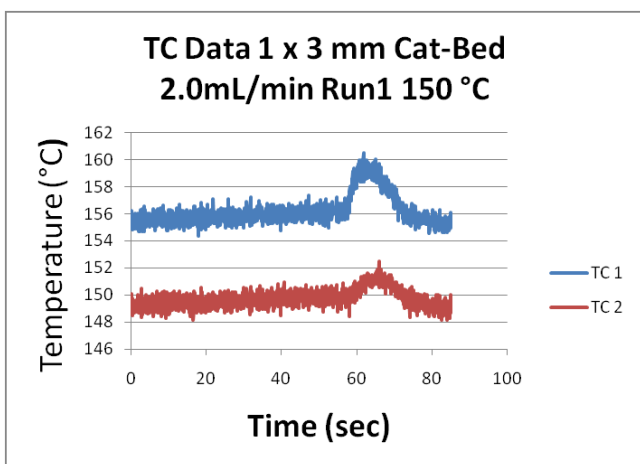
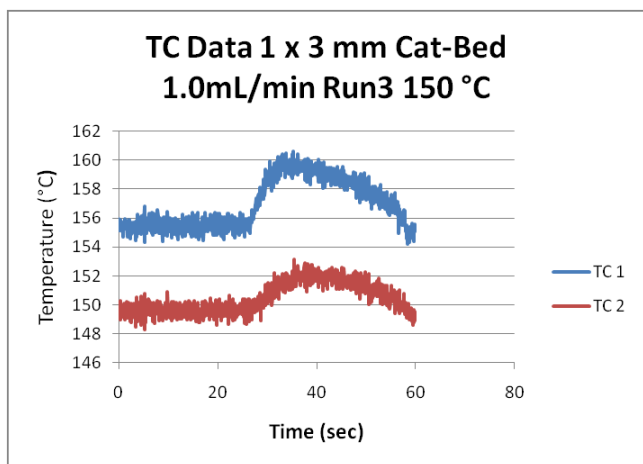
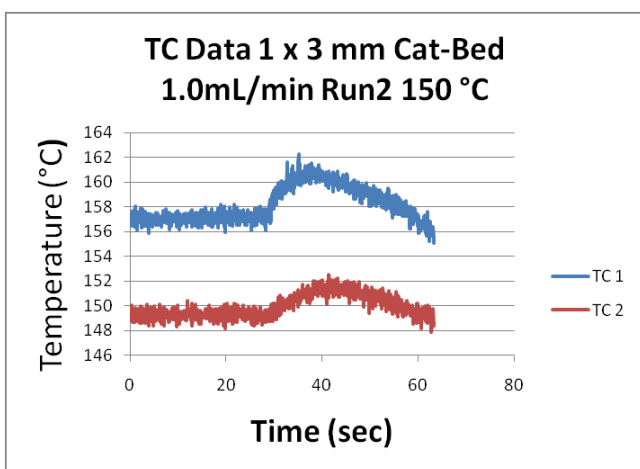
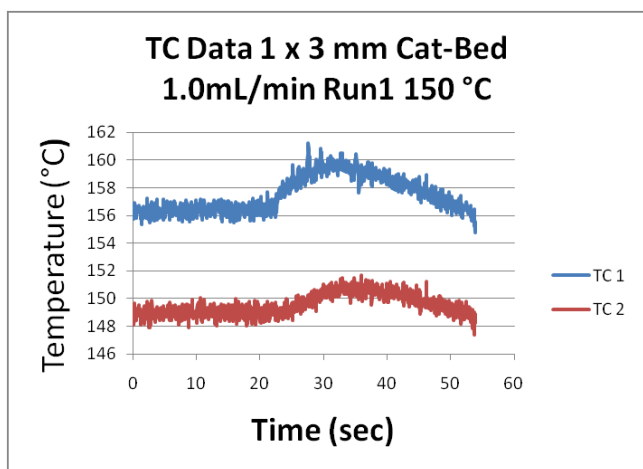
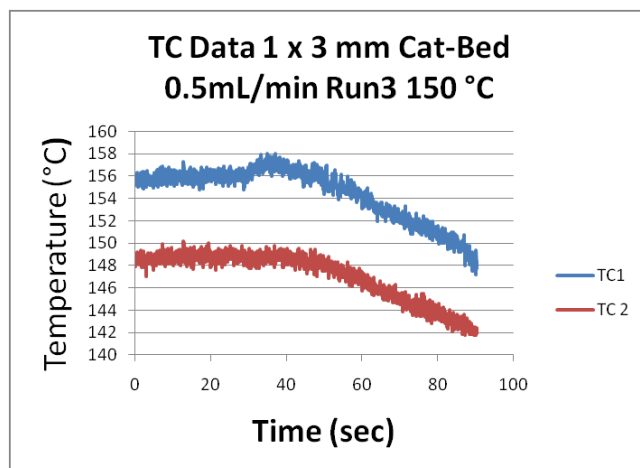
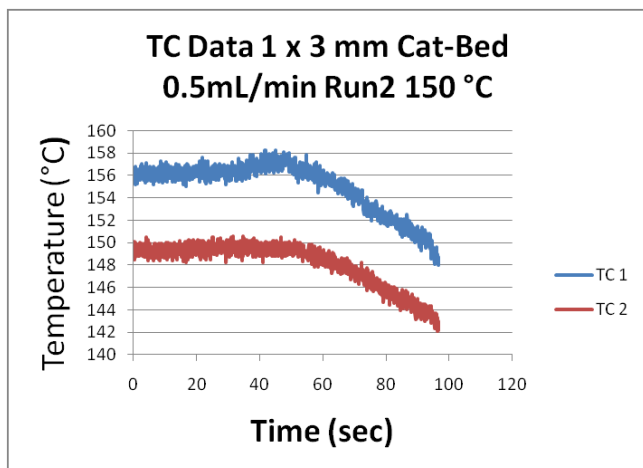
**TC Data 1 x 5 mm Cat-Bed
1.0mL/min Run2**

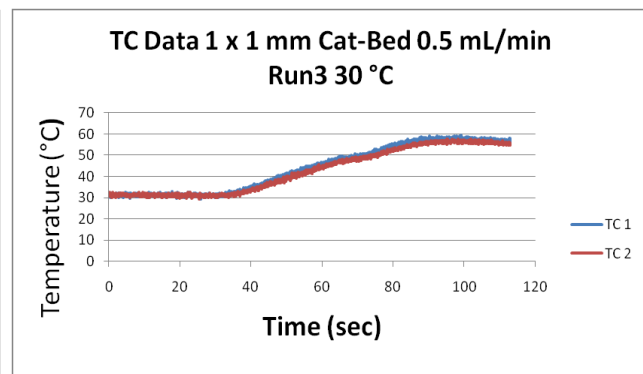
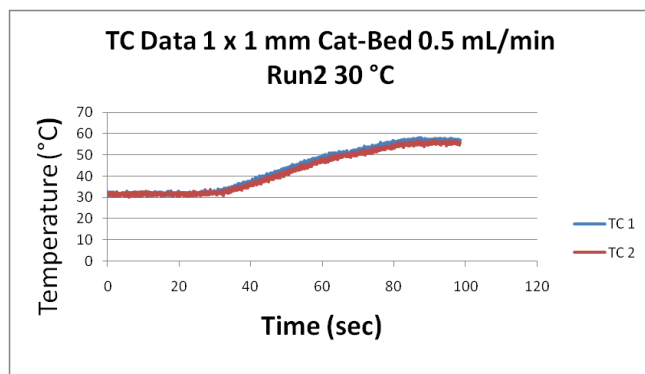
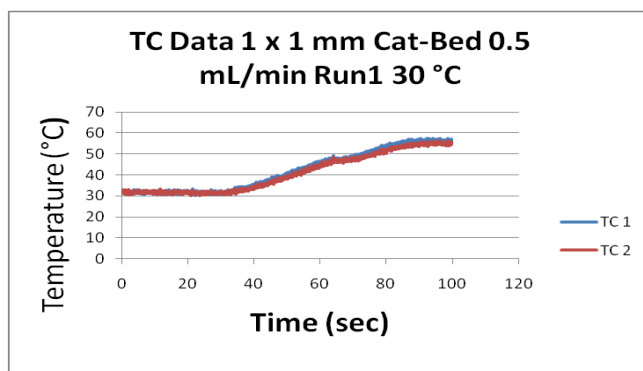
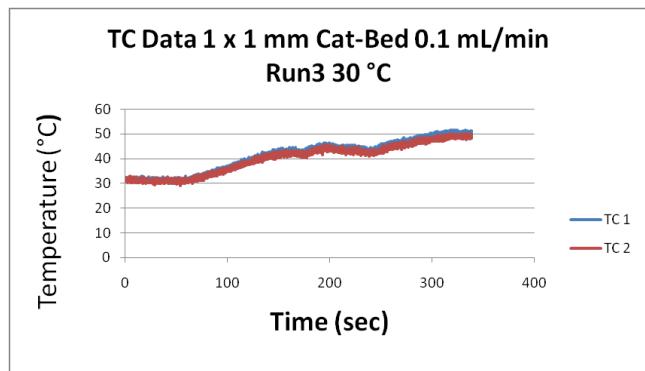
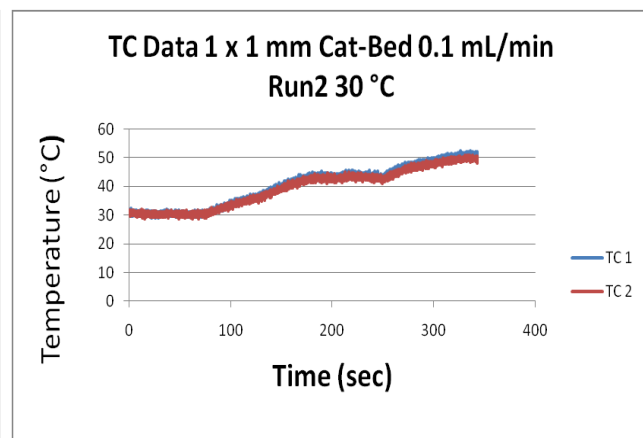
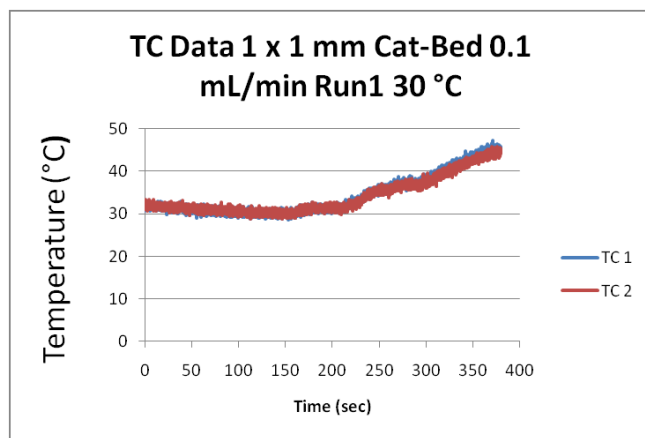
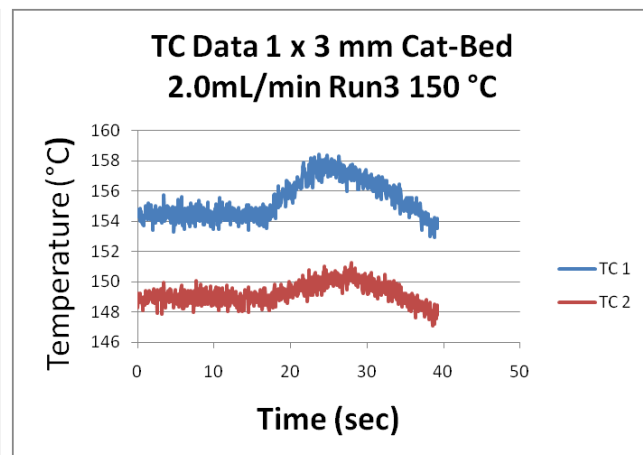
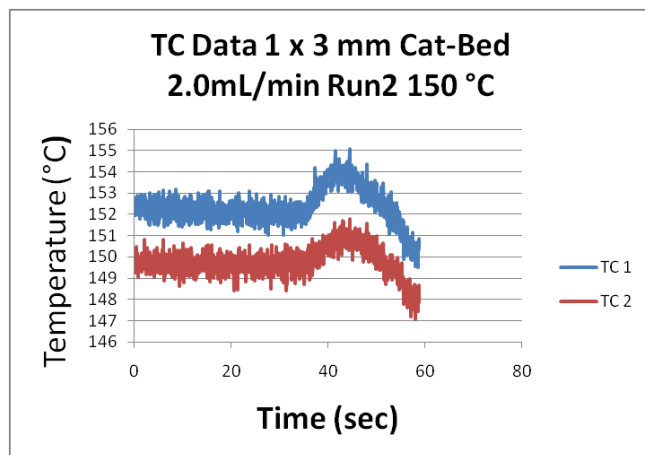


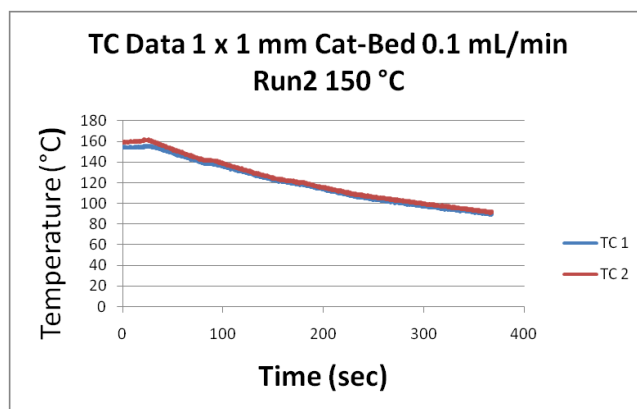
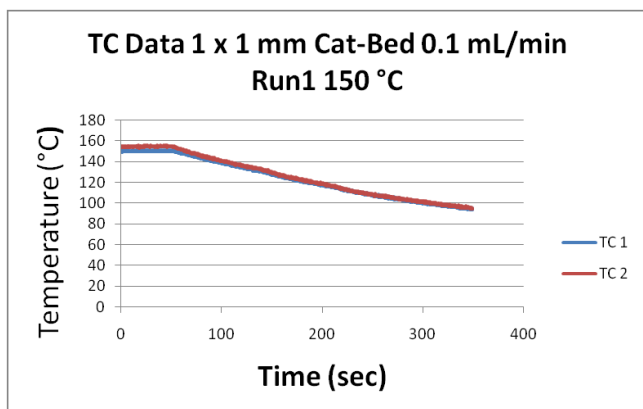
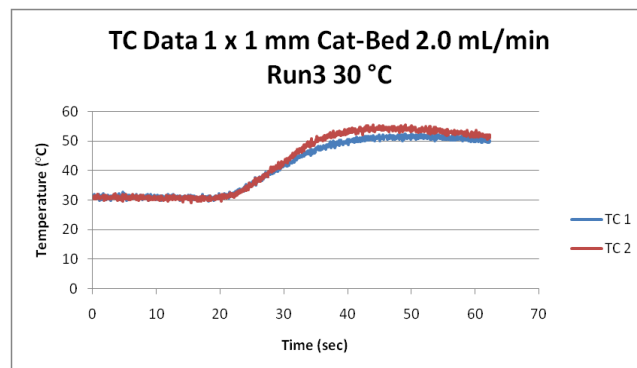
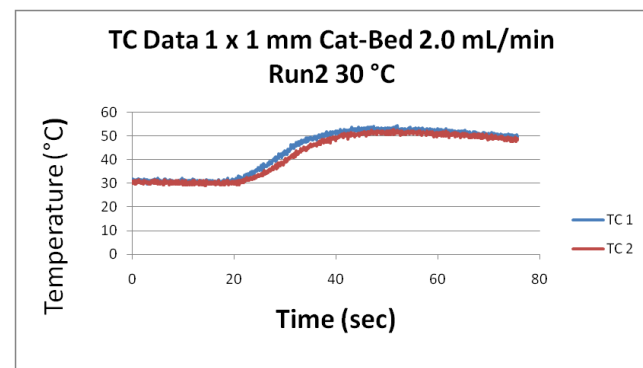
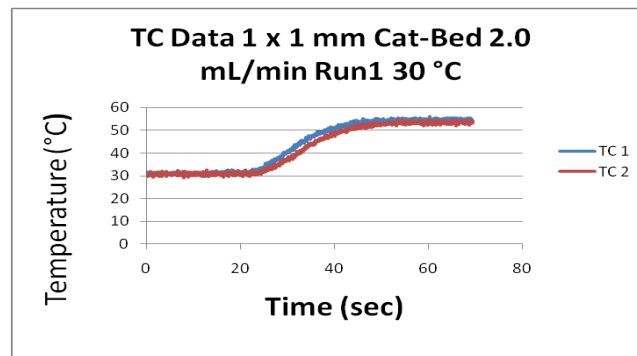
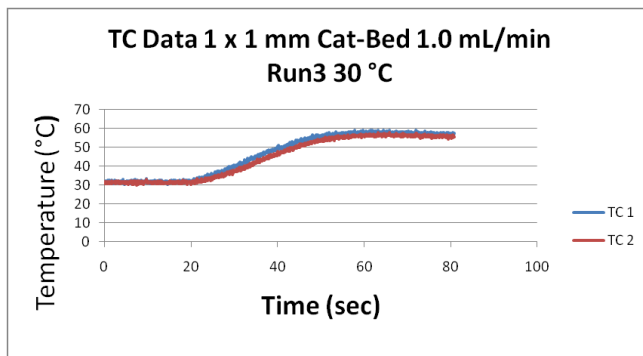
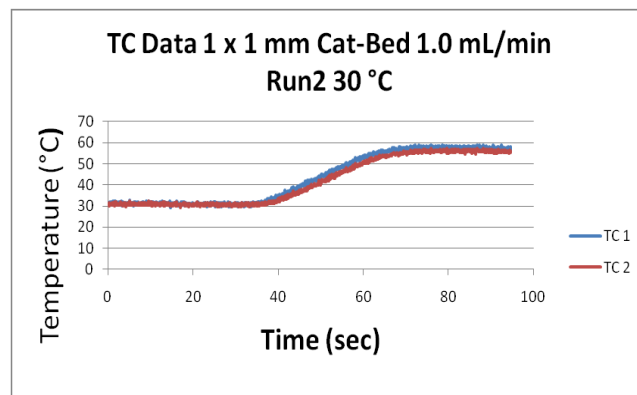
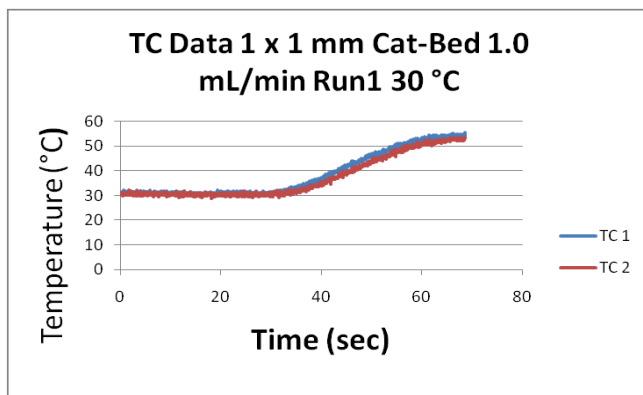


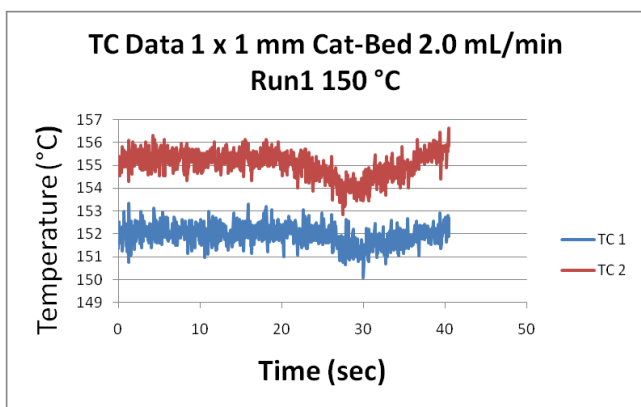
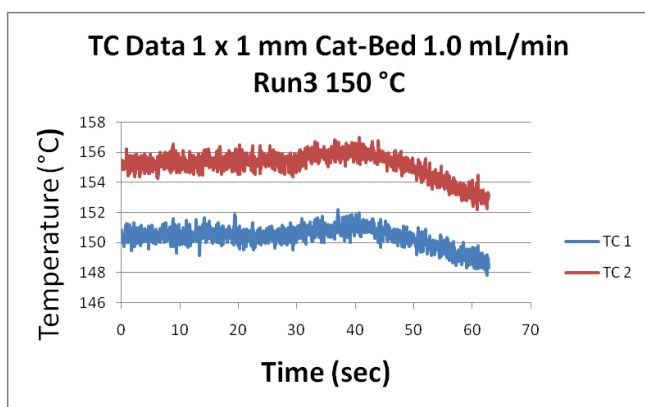
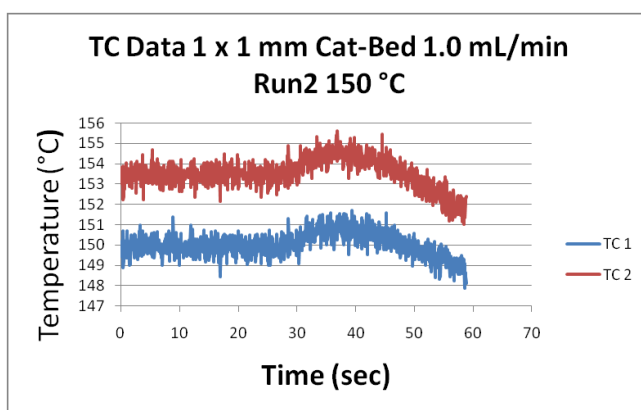
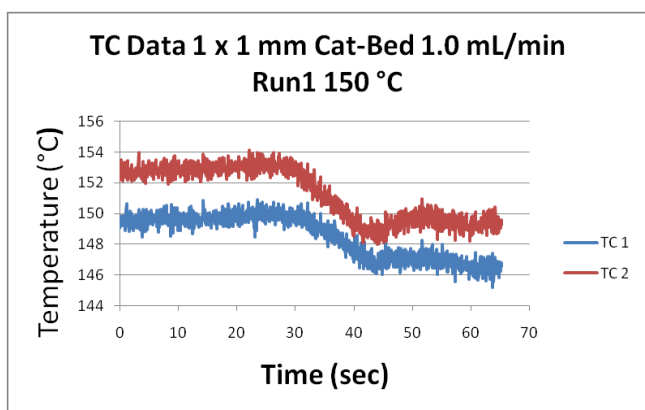
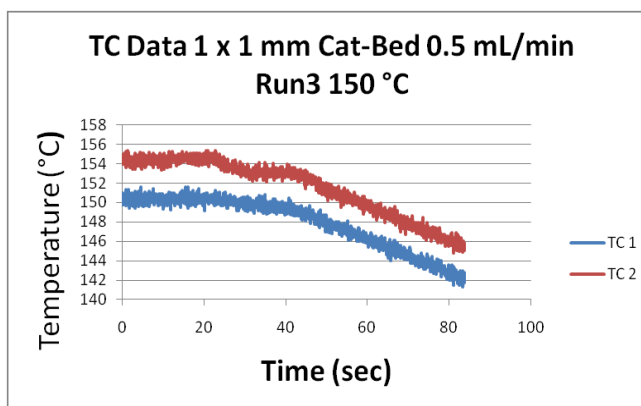
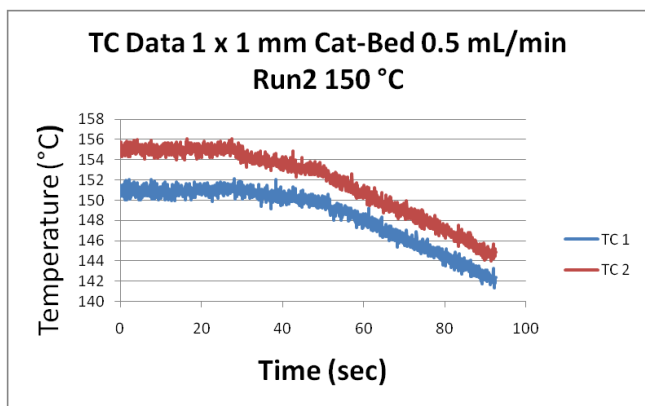
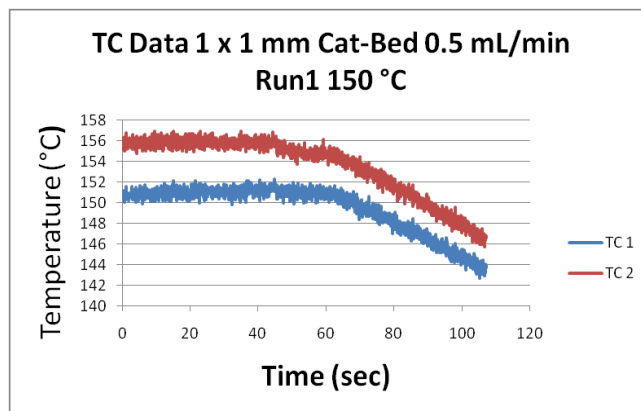
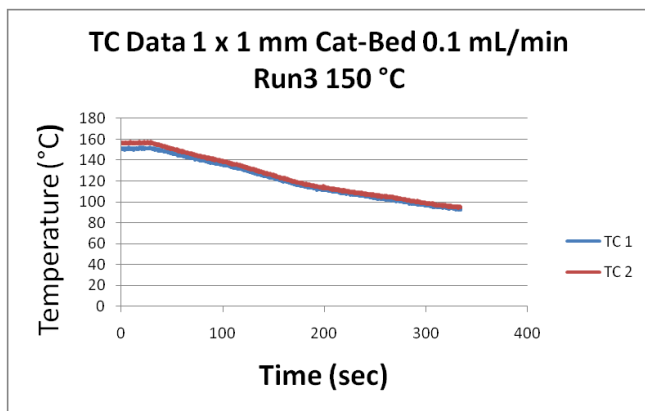


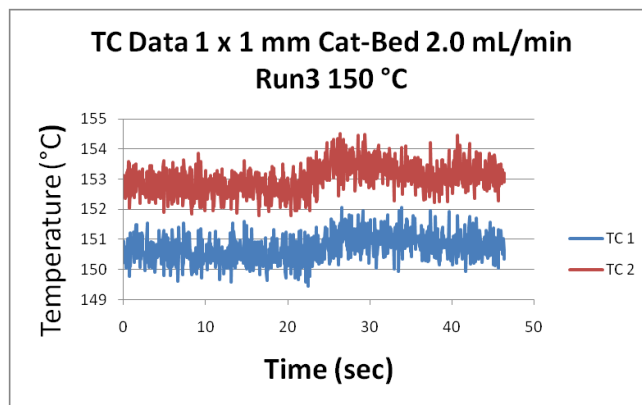
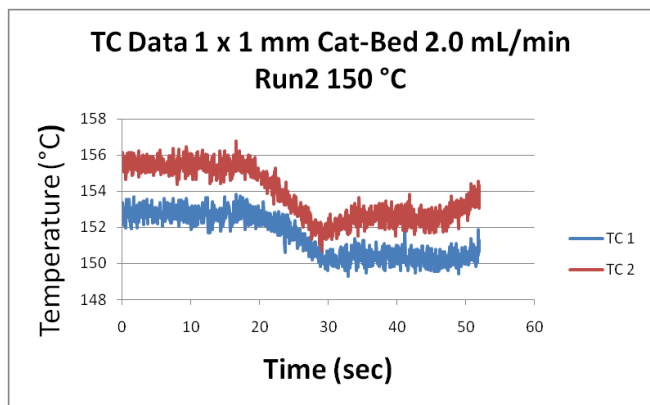












Vita

Chance Paul Garcia was born in Cannon Air Force Base, Clovis, NM on June 7, 1984 to Pablo Garcia and Patricia V Vasquez. He was born and raised in El Paso TX the majority of his life. He studied at local schools and graduated from Montwood High School Top 10 in the summer of 2002. He then continued his studies at the University of Texas at El Paso where he attained his Bachelor of Science in Mechanical Engineering. He will graduate with his Master's of Science in Mechanical Engineering in 2009 and will further pursue a Doctoral Degree in the same field at the Univeristy of Texas at El Paso as well.

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This thesis/dissertation was typed by Chance P Garcia.