**Use of a Piston Mechanism in an Educational Rig to Observe Plasma-based Carbon Dioxide Decomposition in Atmospheric Pressure Environments**

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Team 7

Experimental System Design Report for ME360

# Abstract

The primary focus of this work is to make an effective and streamlined educational process to highlight the ability of plasma to decompose CO2, while maximizing the accuracy, safety, and reproducibility of this process. Design criteria and constraints are centered around designing a low-cost rig that transports the gas with minimal leakage from the treatment chamber into the gas cell. Plasma, specifically dielectric barrier discharge (DBD), was used to decompose samples of air containing 400-10,000 ppm CO­2. A LI-850 gas analyzer was used to determine the preliminary concentration of CO2 in the gas sample, and a Fourier-Transform Infrared (FTIR) spectrometer was used to analyze relative concentrations of CO2 with higher resolution. A new piston mechanism was proposed, but leakage proved to be a significant issue. Leakage testing was instead performed on the existing rig, and results show that leakage from both rigs is too extensive to reliably conduct plasma decomposition experiments.

# Introduction

Plasma, the fourth state of matter, can be used to decompose carbon dioxide (CO2), and the effects of plasma treatment on a sample of gas with a known CO2 concentration can be observed using methods such as gas chromatography or the method selected for this project, spectroscopy. However, the necessary equipment to carry out plasma treatment and the subsequent study of the changes in concentration require specially designed experimental methods to both allow plasma to form, and to allow the gas to be analyzed. The work of Dr. Kamau Wright focuses on plasma analysis, and the goal of the rig involved is to be effective experimentally and pedagogically.

## Needs Statement

The aim of this project is to design a more streamlined procedure for an educational rig which demonstrates the ability of plasma to decompose CO2 in a sample of gas. The method must maximize accuracy, and precision, while keeping sufficient resolution of the CO­2 concentration. The procedure and the results must be easily reproduced. To achieve these goals, the main objectives include holding and moving the CO­2 mixture for plasma treatment, as well as moving the mixture from the mixing chamber to the gas cell with minimal leakage. The design constraints require the rig to be relatively inexpensive and easy to use. The gas cell must not be damaged while in transit, and when creating a new rig, the permanent alterations to the cast acrylic cylinder must be minimal to prevent leakage and to promote reusability of the cylinder.

## Previous Experimental Method

### Plasma Background

Plasma can decompose CO2 into CO and O2, as the interaction of electrons with the CO2molecules, by means of thermionic emission and field emission, has enough thermal energy to break the CO2 covalent bonds. The free electrons caused by the DBD discharge impact the CO2 molecule, “enabling the direct activation of the CO2 through either electronic excitation, ionization or dissociation mechanisms” [1].

The experimental setup uses a power supply generating an alternating current flowing to two metal electrodes, one covered by a dielectric barrier, set at a small distance from each other. The large potential differences on the surfaces of the electrodes pull electrons from surrounding air molecules which cause a DBD of plasma to flow between the two electrodes.

### Gas Cell

The main apparatus to hold the treated gas is a Specac Storm 10 Short Pathlength Gas Cell FTIR, as shown in Figure 1, which holds the gas samples in a volume of 125cm­3. The cell is sealed using potassium bromide (KBr) salt windows, as they are invisible to infrared, thus allowing the FTIR spectrometer to effectively analyze the cell contents without leakage. However, because the cell windows are composed of salt, they are sensitive to moisture and must be kept dry in a glass dome desiccator with added copper sulfate desiccate.

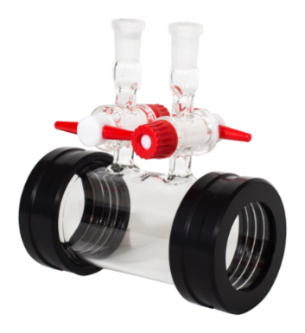


Figure 1: Specac Storm 10 Short Pathlength Gas Cell FTIR

### Existing Rig

The original setup designed by Dr. Wright, as shown in Figure 2, consists of an acrylic cylinder, with an inner diameter (ID) of 4 inches and an outer diameter (OD) of 4.75 inches, 1/8in NPT fittings, tubing, aluminum electrode nuggets 1 inch tall and 1.5 inches in diameter, and acrylic discs fitted with O-rings to seal both ends of the acrylic treatment chamber. They are attached and tightened to seal the chamber with three threaded rods. The chamber is oriented vertically, and the top electrode is suspended using metal fittings that together double as an inlet valve and an electrical connection. The bottom electrode is covered with a small disc of tempered glass, which acts as the dielectric barrier for DBD, which is inset into a machined polycarbonate dais that sits at the bottom of the chamber. The metal fittings can be screwed and unscrewed to adjust and maintain 2-3mm between the electrodes. The gas cell is connected through the exit valve on the side using the 1/8in rubber tubing.



Figures 2a & 2b: Original plasma chamber design

The initial procedure included running a Fourier-Transform Infrared spectrometer (FTIR) background scan on an empty gas cell (atmospheric air), preparing a sample of gas, initially pure CO2, by flushing the acrylic chamber with gas from a CO2 cannister, running the FTIR scan to identify CO2 peaks, performing the plasma treatment, and running the FTIR to find any difference in relative CO­2 peaks.

# Experimental Methods

## Instrumentation

### Other methods of gas analysis

There are numerous ways to analyze the presence and concentration of specific gas molecules in a captured sample of gas. One of the methods that was considered and investigated was gas chromatography: a method that consists of injecting gas, liquid, or solid samples into a gas chromatography system so that each component of the sample could be analyzed. This is done by vaporizing the mixture, separating each component, and “converting the amount of each component into an electrical signal” [2]. To separate the components, the sample is flushed with a carrier gas and each gas arrives at a column outlet at different times, differentiating them. However, this method is not as accurate as other methods could be, as the traceable accuracy is around ±0.5% of the reading [3].

Another method considered was to use an NDIR (non-dispersive infrared sensor) spectrometer to analyze the gas molecules with the FTIR spectrometer. However, many models of NDIR spectrometers are not able to accurately and precisely determine CO2 concentrations above 50,000 ppm. Those able to detect high concentrations with high accuracy and precision were still too low resolution for this project, as plasma decomposition changes the concentration slightly. Moreover, accurate NDIR sensors at high concentrations were extremely costly. When the initial concentration of the CO2 in the gas sample was later reduced to less than 20,000 ppm instead of 100% CO2, it was determined that the resolution of a cost-effective NDIR spectrometer would be sufficient to assist with creating the gas sample in the right concentration range, while the primary concentration analysis being done with the FTIR spectrometer. A full analysis of the specifications and prices of NDIR spectrometers can be found in Appendix V.

### Instrumentation used

The chosen method of gas analysis is Fourier-Transform Infrared Spectroscopy. FTIR spectroscopy measures concentrations of different gases by emitting infrared light rays of different wavelengths into a gas sample. Due to the differing geometries of different gas molecules, a machine performing spectroscopy can measure the presence of molecules at different wavelengths of light. The presence of each molecule is measured and quantified through either transmittance or absorbance (which share a logarithmic relationship). The spectrometer measures the amount of light at a certain wavelength transmitted or absorbed by whichever molecules are within the sample being analyzed. The National Institute of Standards and Technology (NIST) provides a database for the specific wavelengths of light corresponding to different gas molecules. By analyzing a graph generated by PerkinElmer spectroscopy software connected to an FTIR spectrometer, the presence and concentration of specific gas molecules in a captured sample of gas can be interpreted.

The gas before and after treatment will be analyzed with a PerkinElmer Spectrum Two FTIR spectrometer, as shown in Figure 3. To use this instrument, the proper fittings must be inserted into the FTIR spectrometer to analyze gases, as it is usually primed to analyze solids and liquids. The PerkinElmer spectroscopy software is then opened on the computer connected to the spectrometer, and the gas cell is inserted along with its holder. A background scan is run with an empty gas cell, which calibrates the instrument to the untreated gas. The gas cell can then be taken out and flushed with the intended gas. Against the same background scan, a scan is run, and data is collected in a CSV format to be later analyzed in Microsoft Excel. By comparing the scan of the untreated gas with the new scan of the treated gas, the software can detect differences in the mixture. Peaks at specific wavelengths will appear, indicating the presence of different gases, including CO2. Using the FTIR spectrometer allows for an accurate representation of the change in CO2.

A white machine with green stripe

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Figure 3: PerkinElmer Spectrum Two FTIR spectrometer

### LI-850 Gas Analyzer

To verify the readings of the FTIR spectrometer in the lab, the LI-850 gas analyzer, as shown in Figure 4, will be used to measure the concentration of gas. It measures the concentration of CO2 gas molecules through NDIR gas analysis, which uses infrared light to detect CO2 concentrations. By shining infrared light through a sample of CO2, the CO2 absorbs the band of light while ignoring other wavelengths. An optical sensor then filters out the other wavelengths, which is then read by an IR detector. To turn convert this data into a concentration, the light emitted, and the light absorbed are compared, since there is a direct correlation between the ratio and concentration. The LI-850 is also equipped with a pump to assist with the flow of air through the setup.

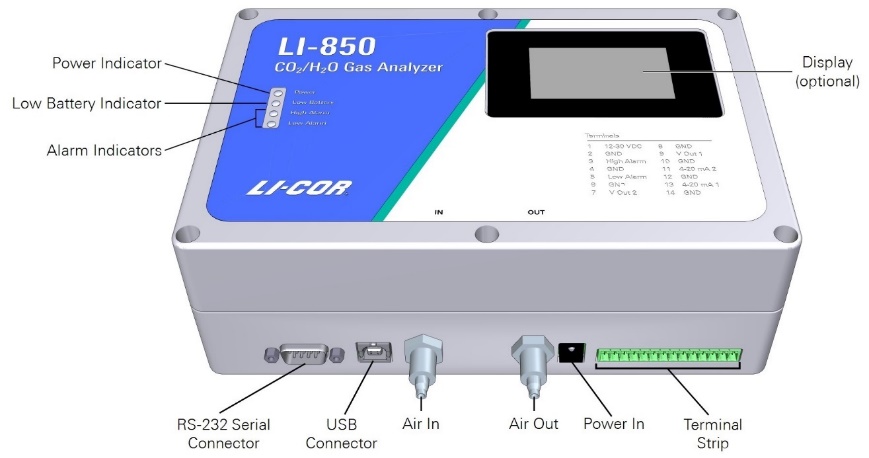


Figure 4: LI-850 gas analyzer

## Apparatus

### Piston Modification

The main modification made to this experimental set up is the addition of a piston mechanism to help push the treated gas out of the chamber and, in future uses, to depressurize the vessel, as plasma tends to form better (longer tendrils at lower voltages) in lower pressures and struggles to do so in a pressurized environment. For a sufficient volume of gas, a longer acrylic cylinder 18in in length was ordered from McMaster-Carr. Initially, the treatment chamber was designed to have three plates; one moving disc attached to the piston, and two porous, stationary discs that held the electrodes 2mm apart. However, because the acrylic cylinder is extremely expensive, the client specified that the number of permanent modifications made to the cylinder (such as drilling holes for NPT fittings) be kept to a minimum.

As a solution, the stationary discs (which were intended to be screwed into the cylinder) were redesigned to be suspended with a friction fit. The original design of suspending one electrode with a pipe nipple that doubles as both an electrical connection and an inlet valve is also present in the revised experimental setup; however, as the acrylic cylinder will be oriented horizontally, the bottom electrode will now be attached to the suspended top electrode through the 3D printed holders. Note that the client advised that this horizontal, instead of vertical, orientation tends to induce curvature in the plasma tendrils, as convection carries the ionized gas molecules upwards. Because the system would not be running for a long time where the shape of the tendril is of major interest, this phenomenon will be disregarded. The bottom electrode will be electrically connected through a wire that is in contact with an electrically insulated screw on the piston. The outlet valve will sit on the same acrylic lid as the inlet valve, as shown in Figure 5. The combination of these modifications allows the setup to require no additional holes in the acrylic treatment chamber, and the piston rig with these modifications can be seen in Figures 6 and 7.

A clear plastic disc with black holes

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Figure 5: Acrylic lid with inlet and outlet valve

Diagram of a diagram of a plasma device

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Figure 6: Diagram of piston rig with attachments

A close-up of a machine

Description automatically generatedFigure 7: Piston rig with attachments.

### Electrode holders

The electrode holders, which were 3D printed with PLA, were designed using the CAD software OnShape. The exploded view of the CAD with the electrodes can be seen in Appendix VI. They were designed to be friction fit, meaning no external adhesives were needed to suspend them, and a 2mm gap was to be held between the top electrode and the adjacent dielectric barrier. However, due to tolerancing issues, electrical tape was needed to secure both the two electrode holders to each other, and the tempered glass dielectric barrier to the bottom electrode holder. The electrode nuggets themselves fit into the holders, albeit quite tightly, and a 2mm gap was held. To ensure the gap was around 2mm in size, a washer of a measured 2mm thickness was slid in between the electrode and the dielectric barrier, as seen in Figure 6b. Plasma, in later demonstrations, was able to form successfully. The initial design covered the top electrode, the one without the dielectric barrier, completely, as the main priority was to maintain the 2mm gap, but the client insisted on the plasma being visible for educational purposes. To increase the visibility of the tendrils, the CAD for the top electrode was extruded backwards to partially expose the electrode, as seen in Figure 6a.

A blue and grey square object

Description automatically generated  A hand holding a white object

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Figure 8a & 8b: CAD and 3D print of electrode holders

### Leakage Issues

As preliminary testing for leakage with the piston, a pressure gauge was attached to the opposite end to the piston, and the piston was engaged to see if there was any significant change in pressure, as it was expected that the internal pressure would just about double (0 psig to about 15 psig) when the piston was pushed to the middle. The pressure gauge did not indicate an increase in pressure, indicating near total leakage. Moreover, when standing the cylinder upright and allowing the piston to freefall, it was discovered that the piston would slide down quite quickly, indicating a rather significant gap between the polycarbonate piston block and the cylinder, despite having been machined to fit each other.

To combat the leakage from the piston, external modifications were made to decrease the gap between the polycarbonate piston block and the cylinder. Following the idea of a rolling diaphragm, the first attempt at modification was putting a latex glove around the piston block; as a result, the polycarbonate block fell slower in freefall because of the reduced gap. This test indicated that the thickness of the gap between the polycarbonate cylinder piston and the internal diameter of the acrylic chamber is about 4-16 thousandths of an inch (the approximate thickness of 1-2 latex gloves). However, the block would stop around the midpoint of the cylinder. The cylinder itself has significant manufacturing error, which is evidenced by both the piston being unable to pass the midpoint, and the polycarbonate only fitting into one end of the cylinder.

Moreover, because the cylinder narrows significantly, the friction of the glove began affect the performance as it was pushed in, as it made it difficult to pull the cylinder without the glove being stuck. To improve the glove modification, tape was used to increase the diameter of the polycarbonate block to try and inhibit airflow around the piston. Different combinations of tape were wrapped around the block to find a minimum gap distance, but even the thickness of the thinnest tape, measured to be around 0.025mm, could not sufficiently close the gap. There was a strict tradeoff between sealing the gap towards the end of the cylinder and allowing the piston block to pass through the middle of the cylinder; the more tape applied, the better the sealing but the shorter the piston range. The best combination of adhesives that, when the piston was activated, produced at most a pressure reading of 2psig was one round of electrical tape (0.028mm) and two rounds of Scotch tape (0.025mm), for a total increase in diameter of around 0.15mm. However, the piston would not move further than 3in into the cylinder.

Moreover, the only tape that could be applied on the top layer was electrical tape, as it provided the least amount of friction when sliding through the cylinder. Other types of tape would also fray and peel at the edges, and their adhesive side would catch on the inside of the cylinder and prevent motion.

It was decided that neither the tape nor the glove outfitting the piston were viable ideas, as friction was too great. In order to combat leakage from the system as a whole, a glove was placed on the piston-side end of the cylinder to create a flexible seal that moved with the piston, as seen in Figure 9. As evidenced by the inflated and deflated glove, this method proves to be effective in keeping air within the treatment cylinder with the piston mechanism but does not adequately address the issue of air sliding over the piston itself, which would affect the ability of the piston to push out air into the gas cylinder. Pressure tests were performed with this setup, but negligible results were observed. When the piston was plunged into the cylinder, a slight pressure increase (to about 2 psig) was observed, but the reading almost immediately dropped to zero. These tests indicated that air was being moved from one section of the chamber, over the circumference of the polycarbonate piston, keeping the volume constant and resulting in effectively zero pressure increase.

A blue glove on a white surface

Description automatically generatedA clear container with a blue cap

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Figure 9a & 9b: Glove mechanism attached to cylinder, inflation showing pressure differences and air movement within the cylinder.

### Base Modifications

The linear actuator provided by the client rested on machined wooden stands drilled into a wooden plank that acted as a platform. To allow for a seamless connection of the new cylindrical apparatus to the piston, a 3D-printed stand was created that could both lift the cylinder to the appropriate height and secure it in place. The stand consists of a top section that attaches to a bottom section via clips on the side. Grooves in the arc were created where the threaded rods rested, which reduced torsion from the piston, as seen in Figure 10. However, the piston force of 400lb was often unable to overcome the friction between the polycarbonate piston block and the side of the cylinder, which would push the whole cylinder as opposed to just the piston. The length of the piston was also difficult to control, as there were significant delays in changing direction and stopping the actuator. Therefore, a wooden block had to be attached to the end of the plank to ensure the cylinder didn’t move, and an extension on the base to fit the maximum piston length. However, because the wooden plank platform was thin, the plank would also buckle if the linear actuator was not stopped in time. This posed a serious safety concern, meaning either a better piston or cylinder would have to be constructed, or the wooden platform would have to be strengthened.



Figure 10a and 10b: Demonstration of the clipping mechanism employed by the 3d printed bases.

To ensure the sheer force of the screw-based piston did not shift the stands, two holding blocks were attached at the end with multiple screws to ensure that the piston would have the effect desired.

## Procedure to analyze plasma decomposition of CO2

The following procedure was not carried out in its entirety due to the significant leakage issues encountered late into the semester. However, had the piston and cylinder been manufactured perfectly and the setup been in ideal working condition with no leakage, the procedure would be as follows:

To initiate the procedure, the gas cell filled with ambient air was brought to the FTIR spectrometer to generate a background scan, which would serve as a reference for future scans. After then returning with the gas cell, ¼’’ tubing was connected from the CO­2 gas tank to the modified apparatus, as well from the outlet to the gas cell. From the gas cell, the LI-850 Gas Analyzer was connected via tubing, using zip ties and electrical tape to secure the connection between tubes. To create the mixture of gas, the valve of the CO­2 tank was opened quickly and closed immediately after. After waiting a few minutes for the gas to reach a consistent concentration within the rig and gas cell, the approximate concentration of CO­2 is read on the LI-850. This process is repeated until the desired concentration of about 10,000 ppm CO­2 is reached.

Initially, the client requested that the plasma decomposition be conducted with pure CO­2 gas sample. A plasma demonstration test was conducted with pure CO­2 using the preexisting setup of the plasma rig. Pure CO­2 was pumped into the rig, flushing out atmospheric air into the fume hood, until it was assumed that the concentration was approximately 100% CO­2. The power supply was turned on, but a DBD discharge was not observed, and an arc discharge occurred. This broke the tempered glass acting as a dielectric barrier. This result showed that the concentration of CO­2 had to decrease, and it was decided that further tests would need to occur at around 10,000 ppm of CO­2, as advised by our client.

After the concentration in the cylinder reaches 10,000 ppm, the gas cell is closed and disconnected from the rig and LI-850 and is brought down from the seventh floor to the third floor, where the FTIR spectrometer is, for a more precise reading of CO­2 concentration. The spectrometer measures the concentration in the gas cell against the first atmospheric air background scan taken. The data is collected as a CSV file and uploaded into a flash drive. The gas cell is then taken back to the lab on the seventh floor and connected to the apparatus again, and the process for generating plasma within the rig begins.

An insulated, high voltage wire is connected to the exposed metallic region of the inlet valve at the base of the rig. The wire is secured around the inlet with electrical tape. The metallic inlet screws into the electrode. Another insulated, high voltage wire is connected to the embedded ¼” aluminum threaded screw within the polycarbonate cylinder acting as the piston. This embedded screw is in turn connected to the electrode covered with the dielectric barrier. These wires are connected to an alternating current (AC) power supply, which is then plugged into a storm surge power brick by a trained operator of the rig. When electrical connections are secured, the operator, wearing an insulating glove, powers on the power supply. A second person with an insulting glove stands by to ensure that, in case the operator accidentally makes contact with any electrified components of the setup, the operator can be removed from the electrical loop with a non-conductive tool. The frequency, current, and voltage of the power supply is adjusted by the operator until a DBD is observed between the electrodes. For this experiment, a voltage of 5-6K volts were used, which is around 20% of the max voltage. After the DBD has run for an appropriate amount of time (which was ten minutes, as decided by our client), the operator shuts off the power supply and uses an unused piece of high voltage wire to connect exposed parts of both wires connected to the electrodes. This discharges the electrical loop and renders the set up safe to interact with.

After discharging, the linear actuator is enabled to start pushing the treated gas out of the treatment chamber through the outlet valve and into the gas cell and the gas analyzer. For a less accurate reading, the LI-850 is also used for a preliminary reading of the CO­2 concentrations; it is to be noted that the LI-850 lacks the precision and accuracy to reliably detect a decrease of CO2 concentration in the gas sample after plasma treatment, which is why the FTIR analysis is necessary. To analyze the gas precisely, the procedure with the FTIR Spectrometer is repeated, with the data gathered in a CSV file to be further inspected.

## Procedure to analyze leakage

### Setup

Instead of the plasma procedure, it was decided that the focus of this project should pivot to targeting and preventing leakage. To assess leakage, the gas analyzer was used to detect any decrease in CO2 concentrations in a closed system. Leakage testing was conducted on the previous apparatus made by Dr. Wright, as decided on by the client. Instead of using the redesigned apparatus with the piston mechanism, the end plates were replaced with acrylic discs fitted with O-rings and NPT fittings, as shown in Figure 12. The electrode holders were connected via a 2in pipe nipple to an NPT fitting on one side, and stripped wires were wrapped around the fitting. This side of the treatment chamber was connected to a CO2 cannister with ¼” tubing. The setup is shown in Figure 12.

A circular object with a gold nut

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Figure 11: Acrylic disk with the rubber O-ring and NPT fitting.

Diagram of a wire connected to a device

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Figure 12: Diagram of original plasma rig.

The second electrode was inserted with a threaded rod, and a wire with an alligator clip was attached. The other end of the wire, which was also stripped, was wound around the NPT fitting on the side opposite to the CO2 inlet. A second pipe nipple was connected to the NPT, which was then attached to a pipe splitter; one of the outlets was connected to a pressure gauge and the other connected to a ¼’’ tube. This tube was connected to the inlet of the CO2 analyzer, and a tube connected the outlet to the fume hood. The system is open, as seen in Figure 13.

A diagram of a gas cell test

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Figure 13: Flow chart of CO2 before closed loop.

### First test: CO2 leakage and preliminary plasma decomposition

The creation of the 20,000 ppm CO2 gas sample was started. The LI-850 was turned on, allowed to heat up to an operating bench temperature of 51.4C, and the pump was enabled. The LI-850 embedded software was opened, and data collection started. The CO2 gas tank was opened at 10psi for around 15 seconds to let the gas transfer into the treatment chamber and the CO2 analyzer. This amount of CO2 surpassed the desired concentration of 20,000 ppm, as indicated by the erratic reading of the gas analyzer (the analyzer is only equipped to accurately measure concentrations between 0 to 20,000 ppm [4]). The cannister was then closed, and the tubing disconnected from the end of the chamber to allow CO2 to leak into the atmosphere until the desired concentration was reached. The CO2 in the lab air was monitored using an OSHA certified CO2 sensor, to ensure that the atmospheric concentration remained below the 5,000 ppm threshold [5].

Once it was noted on the CO2 analyzer that the concentration was around 20,000 ppm, the valves on both ends of the mixing chamber were closed. The tube connected to the output of the CO2 analyzer was taken out to disconnect the system from the fume hood. Because we are testing the leakage from the system as a whole, a closed system needs to be created as shown in Figure 14. To do this, a tube connected the inlet of the treatment chamber to the output of the CO2 analyzer. The time was noted, as this was the start of the leak test.

A diagram of a gas leakage test

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Figure 14: Flow chart of CO2 after closed loop.

The leak test was conducted for about 20 minutes. The plasma treatment was briefly induced to determine if the plasma decomposition could produce a noticeable change in CO2 concentration that the gas analyzer could pick up. The plasma was turned on for ten minutes, and data was continuously logged on the embedded software. The electrical loop was then discharged and the data from the embedded software was saved onto a flash drive.

### Second test: bubble leakage test and CO2 leakage test

#### Bubble test

It was observed from the initial dataset that there was significant leakage from the system. The setup was assembled as normal, but the fittings and threaded rods were made sure to be tightened as far as possible. To determine the source of the leakage, soapy water (made by mixing a tablespoon of dish soap with around 50ml of water) was applied to all orifices and connection points, such as where the cylinder met the plates, as well as the pipe nipple and splitter. It was ensured that no soap or bubbles entered the tube connected to the LI-850, even though there was a filter attached, in order to prevent any damage. The bubbles were used to identify leakage points when the chamber is pressurized. To do so, the outlet valve was closed while the gas cannister pumped air at 8psi into the cylinder for ten seconds before closing the inlet valve, and points where bubbles formed were taken note of. This test was repeated three times.

#### CO2 Leakage Test

The CO2 leakage test was conducted in a similar manner to the first test. The CO2 in the cylinder from the bubble test was allowed to leak into the atmosphere after both valves were opened, in order for the concentration to reach 10,000 to 20,000 ppm on the gas analyzer. Because the initial concentration in the cylinder was well above 20,000 ppm, the reading on the software was erratic, as expected from the previous test. When the system reached a concentration of 20,000 ppm, both valves were closed. The pipe extending out of the gas analyzer and into the fume hood was instead connected back to the inlet of the cylinder. Data was recorded for the next hour using the LI-850 and was stored on a flash drive as a CSV.

# Results

## Preliminary FTIR Analysis

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Figure 15: FTIR trial resultsA graph with orange lines

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Figure 16: NIST Infrared Spectrum for CO­2 [6]

The first test conducted with the FTIR was of pure CO­2 against a background scan of atmospheric air and was used to identify and confirm the peaks of CO­2 with existing NIST data. Graphs corresponding to the absorption and transmittance at specific wavelengths were generated which can be seen in Figure 15. Strong peaks were found at about 700 cm-1, 2400 cm-1, and 3700 cm-1, which corresponded to NIST data on FTIR presence of CO­2, as seen in Figure 16. Relative to a background scan of ambient air (approximately 0.05% CO­2), the transmittance at these peaks was approximately zero per cent, indicating an approximately 100% concentration of CO­2.

## Leakage Testing

### Adding CO2

A graph with a line going down

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Figure 17: Initial CO2 concentration for leakage test 1.

A graph with blue dots

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Figure 18: Initial CO2 concentration for leakage test 2.

Figures 17 and 18 show the readings from the gas analyzer after the CO2 was added, and while the rig was still an open system allowing leakage to the atmosphere. Above 150,000 ppm, the gas analyzer is extremely erratic and nonsensical with reading such as 100,000,000 ppm, because it is not equipped to handle this level of concentration. One data point was removed from Leakage 1 for the sake of clarity; at 13:17:35, the analyzer detected a reading of 500,000,000 ppm, which extended the y-axis in such a way that the rest of the data points were illegible. However, after 13:20, the gas analyzer managed to read reasonable measurements of CO2, as shown by the figures below (Figures 19 and 20).

Figure 18 has a lot of noise from 13:40 to 13:43 because the cylinder was tipped over various times to “spill” out the CO2, since it is denser than air. The peaks are a result of the CO2 sloshing in and out of the cylinder and into the gas analyzer. Both graphs, however, seem to show asymptotic behavior as the gas analyzer attempts to stabilize.

### Allowing CO2 to reach less than 20,000 ppm

A graph of a curve

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Figure 19: CO2 stabilization for leakage test 1.

A graph of a graph showing a curve

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Figure 20: CO2 stabilization for leakage test 2.

The initial noise in the data for Leakage Test 2 is due to the opening of the valve, as any disturbance to the system causes a sudden influx of CO2 drawn in by the pump in the LI-850. Later in Figure 20, around 13:53, there is a significant decrease of CO2 concentration to near zero. This is because the outlet value heading into the gas analyzer was briefly shut as an operator error. This does, though, indicate that the gas analyzer is sufficiently reactive to changes in concentration at low parts per million.

Both figures show an exponential decrease in concentration. If the data from Figure 20 is truncated such that the noise is disregarded, and an exponential fit is applied to both datasets, the time constant is found to be roughly the same. The free leakage from the first test can be characterized as and the second test can be characterized as , and these time constants can be used as a baseline for comparison for when the CO2 leakage test is performed.

### Leakage Testing Graphs

A graph with a line

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Figure 21: Leakage Test 1 with stabilization.

A graph with a line

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Figure 22: Leakage test 1.

A graph with a line

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Figure 23: Leakage test 2 with stabilization.

A graph with a line going up

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Figure 24: Leakage test 2.

The figures above (Fig. 22 and Fig. 24) depict the actual start of the leakage test, which started at 13:29:42 for leakage test 1 and 13:41:21 for leakage test 2(when both valves were open after reconnecting the gas analyzer outlet tube to form a closed system). Figure 22 shows a general decrease in concentration as time went on, which indicates a significant leakage in the system, even if it did not have the piston attachments. The graph follows an exponential trend, which is intuitive, as the change in concentration due to the leakage and mixing of atmospheric is directly related to the actual concentration itself and could be described as a differential equation. Figure 24 also shows a decrease, albeit much slower.

In Figures 21 and 22, there is a slight peak in value around 13:47, which indicates the time that the plasma treatment was started. However, no noticeable change in the rate of decrease in CO2 concentration could be observed. The timelines for both tests can be seen in Appendix III and IV.

### Bubble Test

A close-up of a machine

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Figure 25: Bubble test performance.

As seen in the figure above, the primary leakage in the system was through the connection between the cylinder and acrylic disk with an O-ring. This is where most of the bubbles formed. However, there were also smaller bubbles near the NPT fittings and the other acrylic disk. To prove this, the figure below shows the CO2 concentration as the cylinder was being pressurized with CO2. The times at the peaks align with the times the CO2 tank was opened, which shows that the closed NPT fitting leaked CO2 into the CO2 analyzer, even though the NPT fitting was rated for CO2 and around 8 psig of pressure. When the CO2 was pumped into the cylinder at 8 psig for ten seconds, the pressure gauge consistently read 6 psig, further indicating leakage.

A graph of a bubble test

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Figure 26: CO2 Concentration during bubble test.

During the leakage testing, there were readings on the LI850 that indicated that there was leakage through the NPT fitting, despite the fitting itself being closed, leading to predictions that either the fitting could not withstand the pressure or that there was a manufacturing error in the valve itself, leading to a tolerance error that led to leakage. As the valves were highly recommended from the client, a test using a different valve from the same manufacturer, or a more secure metallic valve might be beneficial for the leakage test.

# Discussion

The improvement in leakage performance between leaks can be attributed to the tightening of all the fittings; when setting up for the second leak test, it was ensured that all threaded rods and fittings were tightened by hand, and then further tightened with wrenches. As discovered by the bubble test, the primary source of leakage was around the O-ring; this is an issue that cannot be addressed by further tightening the threaded rods, as they began to strip the acrylic tapped threads in the end plates when screwed further. Moreover, the CO2 leaks through the NPT fittings, both into the tubing, as shown in Figure 26 where the peaks coincided with the CO2 additions, and into the atmosphere, as shown by the bubbles around the splitter in Figure 25.

In our first leakage test, after the plasma is turned on, there is no observed decrease in the slope of the graph; this indicates that the plasma decomposes at a slower rate than the rig was leaking, rendering the setup completely ineffective. However, as leakage improved significantly in the second test, it cannot be concluded if the plasma would have had a significant effect on the slope of the second test, as it was not performed. The gas analyzer, though, with an error of 1.5% (or ± 150 ppm at 10,000 ppm), will mostly likely not be able to detect the change in concentration due to plasma decomposition, unless the rig is completely airtight, and the treatment is run for a sufficiently long enough duration.

## Issues with Data Collection

As seen by the leakage tests, the results were less than ideal because of the high sensitivity of the system. For example, when turning the valves on or off, there was significant noise in the data. This was unusual because even when the valve to CO2 was closed, the data from the gas analyzer showed that there were extremely disparate concentrations of CO2 in the system, as seen at the beginning of Fig. 21. Within the span of a minute, the concentration of CO2 jumps from 22,000 ppm to 10,000 ppm.

For both leakage tests, when the concentration was allowed to drop to around 15,000 ppm, the sensor data rose slightly due to the stagnant CO2 in the tube and in the gas analyzer being agitated. CO2, being denser than atmospheric air, settled into the gas analyzer, which then caused the peaks and severe drops when tubes were changed. The pump was likely trying to draw in air from the closed valve, which depressurizes the tube; when the NPT valve does open, this causes a rush of air into the CO2 analyzer, explaining the peak. The figure eventually settles, which indicates that the reading on the gas analyzer had stabilized, and any accumulation of CO2 in the analyzer had reintegrated into the system.

# Conclusion

In conclusion, the rig test is not sufficiently airtight to effectively conduct experiments on the plasma decomposition of CO2. The rig leaks CO2 at a faster rate than plasma can decompose, and even if the plasma had the ability to decompose CO2 at a fast rate, even with the analysis of the extremely accurate FTIR, the leakage of any air into the atmosphere will drastically affect the reliability of the reading.

## Future Work

There are several improvements that could be made to both the existing rig and piston rig to reduce leakage. Future recommendations for the rig with the piston include a softer, more flexible piston material which will allow expansion to better hug the inner diameter of the cylinder to prevent leakage around the piston while being malleable enough for friction to be negligible. Some options include inch thick rubber and silicone. Issues with the material being too flexible can be dealt with by attaching it to a solid piston core; the rubber can be attached to the bottom of the polycarbonate block, or the silicone could be cast in a thin ring around the block. A groove could also be machined into the polycarbonate block for the fitting of an O-ring; this was not implemented in the piston design due to time constraints, and because a suitable O-ring could not be found. Another solution could be to ensure that the acrylic cylinder and piston block are machined correctly, such that the cylinder does not narrow in the middle and the piston block slides easily through the cylinder. However, this level of tolerancing is unrealistic, and does not completely address the issue of air escaping around the piston.

To prevent leakage outside the cylinder, a formalized version of the glove fix could be implemented, such as using latex, a balloon, or a rolling diaphragm. A flexible material that allows the depressurization of the portion of the cylinder behind the piston while also not inhibiting the movement of the piston is key. Additionally, thicker O-rings or external sealant could be implemented, but the reusability of the rig will be reduced if an effective permanent sealant is applied.

In further iterations, the number of components could be reduced; examples include machining the cylinder and top head plate to be one continuous piece. By limiting how many separate parts are involved in assembling the rig, the number of junctions where air could escape from decreases, limiting leakage. Moreover, how tightly a screw is turned is, to some degree, subjective, meaning that trials of the plasma decomposition treatment will differ in reliability.

To improve the testing procedure, a formalized procedure for adding the correct amount of CO2 to reach 20,000 ppm can be implemented; instead of timing the release of CO2 from the cannister for an arbitrary amount of time, the time can be calculated using the equation found in Appendix I. This would prevent overwhelming the sensor with concentrations above 100,000 ppm, and would decrease both the amount of time needed to drop to the desired concentration and the amount of CO2 wasted. Also, when noting down the timestamps for each section and step of the procedure, noting the second was often overlooked, but due to the high sampling rate of the gas analyzer (around two readings per second), keeping track of the experiment down to the minute is important. Further testing could be carried out with the bubble leakage testing as well. More trials of each experiment should be taken as to improve statistical reliability and to allow calculations for statistical analysis. If, even after implementing the suggested fixes for leakage, the rig is still not sufficiently airtight, the leakage can instead be characterized; by knowing how much leakage occurs, one can deduce the change in concentration due to leakage, and any further decrease in concentration can be attributed to the plasma decomposition.

Appendix I: Volume of desired co2 calculation

Where is the inner diameter of the acrylic chamber, is the length within the chamber pushed out of the cell. and approximates the volume of solids in the rig which cannot be included in the internal gas volumes.

Appendix II: Bill of Materials

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Item** | **Company** | **Website** | **Description** | **Price** | **Quantity** | **Picture** |
| Acrylic Cylinder | McMaster Carr | Link | 4.75” OD  4” ID  18” length | 600 | 1 | A close-up of a tube  Description automatically generated |
| NPT Fitting Coupler 1/4” | Glacier Tanks | [Link](https://www.glaciertanks.com/ntp-cap-tfnc-fn4.html) | 1/4 in. -SS316 | 5.30 | 1 | A close-up of a metal nut  Description automatically generated |
| Tubing ¼” | AHBLQD | [Link](https://www.amazon.com/AHBLQD-Kit-Pneumatic-Fittings-Clear/dp/B0BZ4CYZLD/ref=sr_1_17_sspa?crid=39VLEN80J10AJ&dib=eyJ2IjoiMSJ9.IE54N5k2VdjmWEQQEjLVaeKryxNTrJhVMigXZzYeAu3TS0R69jdqHOhfxewyQuLXEIiWTX6SrB5Ic727GZLEIEbI5wBj5mI17iOT_oCdmMS9bwkUhGCrraP2L7tFvpCPg60YNT_6UjEDigb_QFG4-0IEfDhR7kU7dXYkFxlZLIpt4NYPwK-0WvaHtETwG9c36hqSyyEKwGZ_KsBipZNMM8rpz5ukNh80Zi83DXxL4m8.rmwC3UP4QZwi8IkVnPNm__8yXf1qZf6WfX4Cb6gQhKo&dib_tag=se&keywords=1%2F4in+push+to+fit+tubing&qid=1711375845&sprefix=1%2F4in+push+to+fit+tubing%2Caps%2C84&sr=8-17-spons&sp_csd=d2lkZ2V0TmFtZT1zcF9tdGY&psc=1) | 5/16” OD  32.8’ length | 14.99 | 1 | A close-up of a wire  Description automatically generated |
| Gas Cell |  |  | Specac Storm 10 Short Pathlength Gas Cell FT-IR |  | 1 | A close-up of a device  Description automatically generated |
| PLA | HATCHBOX | [Link](https://www.amazon.com/HATCHBOX-3D-Filament-Dimensional-Accuracy/dp/B00J0ECR5I/ref=sr_1_2_sspa?crid=1QMJ1SAZGDIH8&dib=eyJ2IjoiMSJ9._0A0q-5aHheY2g4AQKr1--hFQbMe1rM6OhRj0FTqbSeIm9TVVP0CFl3MX2Ja6O-A09BA8etYPWkXRc2_h_YyAUrSJ5NFCguRYwUFq2Mhe09W4nnigQUzhJEP7LP6L9ZiMm0YThW9sTNQjn_nQStJIHX5GnRsTUPpy2CH6ennFANtV5on4Jwr8QLC6ZfIqyc7CVHRLRqKMKRR7C_aO646Li7LGo2_8T2AvQ6pQpfFe1g.ZQsn3lhRQ0lA9nr5KBEHP0FOqNtE5-xHnpRSQl_WbuI&dib_tag=se&keywords=PLA&qid=1711375653&sprefix=pla%2Caps%2C73&sr=8-2-spons&sp_csd=d2lkZ2V0TmFtZT1zcF9hdGY&psc=1) | 1.75mm | 25.00 |  | A spool of wire with text on it  Description automatically generated |
| Pipe Nipple | McMaster Carr | [Link](https://www.mcmaster.com/44665K122/) | 2-1/2” length  Threaded both ends  ¼” fitting | 3.56 | 1 | A close-up of a roll of foil  Description automatically generated |
| Hex Head Steel Screw | McMaster Carr | [Link](https://www.mcmaster.com/92198A502/) | 18-8  1/4"-20 Thread Size  14" Long  Partially Threaded | 8.96 | 3 | A long thin metal rod  Description automatically generated |
| NPT Fitting | McMaster Carr | [Link](https://www.mcmaster.com/products/valves/on-off-valves~/plastic-on-off-valves-with-compression-fittings/?s=npt+valves) | Plastic On/Off Valve with Compression Fittings | 8.45 | 2 |  |

Appendix III: Timeline of Test 1

|  |  |
| --- | --- |
| **Time** | **Event** |
| 1:16 | CO2 opened and closed |
| 1:27 | Closed valves connected to mixing chamber to make a closed loop |
| 1:29 | Opened valves connected to mixing chamber to start leak test |
| 1:32 | Tightened ends of reactor |
| 1:47 | Plasma turned on |
| 1:57 | Plasma turned off |

Appendix IV: Timeline of Test 2

|  |  |
| --- | --- |
| **Time** | **Event** |
| 1:30 | Logging started |
| 1:34 | CO2 opened for 10 seconds and closed for pressurized bubble testing |
| 1:35 | CO2 opened for 10 seconds and closed for pressurized bubble testing |
| 1:37 | CO2 opened for 10 seconds and closed for pressurized bubble testing |
| 1:41 | Opened all valves |
| 1:44 | Closed all valves |
| 1:45 | Tipped reactor to the side twice to decrease CO2 content |
| 1:52 | Closed valves connected to mixing chamber to make a closed loop |
| 1:54 | Opened valves connected to mixing chamber to start leak test |

Appendix V: Suggested NDIR Sensors

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Type** | **Price** | **Description** | **Link** | Accuracy | Accuracy @ 100% Range | Resolution | Measuring Range |
| SprintIR®-6S 100% CO2 Sensor | $289 | - NDIR LED - reads 100% CO2 - needs code to output - UART - inlet/outlet valves - doesnt have a monitor | [Link](https://www.co2meter.com/collections/100-percent-co2-sensors/products/sprintir6s-100-co2-smart-sensor) | 70 ppm plus  minus 5%  reading | 300 ppm plus minus  5% reading | 100 ppm | 0-100% |
| GasLab® Pro 100% Carbon Dioxide Sampling Data Logger | $2,000 | - NDIR - reads 100% CO2 - has built-in interface w/ graphing - outputs CSV file | [Link](https://www.co2meter.com/collections/100-percent-co2-sensors/products/carbon-dioxide-co2-sampling-data-logger?variant=31952656892022) | 70 ppm plus minus 5% of reading @ STP | same as left. | 1ppm (and up) | 0-100% |
| Gravity: UART Infrared Carbon Dioxide Sensor (0-50000 ppm) | $50 | - NDIR - very cheap - one tube - comes with microchip | [Link](https://www.dfrobot.com/product-1565.html) | 50 ppm plus 5% reading | N/A | unknown | 0-500000ppm |
| 100,000ppm MH-Z16 NDIR CO2 Sensor with I2C/UART 5V/3.3V Interface for Arduino/Raspeberry Pi | $68 | - NDIR - easy integration with Arduino - one tube - 400-100000ppm | [Link](https://sandboxelectronics.com/?product=100000ppm-mh-z16-ndir-co2-sensor-with-i2cuart-5v3-3v-interface-for-arduinoraspeberry-pi) | 50 ppm plus minus 5% reading | N/A | unknown | 400-100000ppm |
| RAKwireless RAK12008 CO2 Sensor Sensirion STC31 PID 100206 | $89.97 | -measurement range: 0-100 -Accuracy：0.5 vol% + 3% (0 to 25 vol%) 1 vol% + 3% (0 to 100 vol%) Size:10mmx10mm | [Link](https://store.rokland.com/products/rakwireless-rak12008-co2-sensor-sensirion-stc31-pid-100206) | 0.5 vol% + 3 (0-20% vol) | 1 vol% + 3 (0-vol%) | unknown | 0-100% |

Appendix VI: CAD of electrode and electrode holders

A blue and grey cylinder

Description automatically generated

Appendix V: Miscellaneous Graphs

A graph with a blue line

Description automatically generated

Leakage Test 1

A graph with a blue line

Description automatically generated

Leakage Test 2

A graph with a line

Description automatically generated

Leakage Test 1

A graph with a line

Description automatically generated

Leakage Test 2

# Citations

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