Straw Assembly for CO_2 Leak Testing

David Rivera

Marjorie Corcoran

Aseet Mukherjee

Rice University

October 17, 2013

dor1@rice.edu

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1 Introduction

The tracker for Mu2e will be made of Mylar® straws which are aluminum plated on the outer surface and gold plated on the inner surface. Inside the straws a thin gold-plated tungsten sense wire will be surrounded by $ArCO_2$ (80%/20%) serving as the drift gas. The straw tubes will operate in vacuum, so it is crucial that the leak rate of $ArCO_2$ gas into the vacuum be at a rate low enough for the vacuum pumps to handle. The gas will permeate the straw material at a certain rate, and in addition there might be flaws in the straw material creating much larger leaks. It is therefore important to leak-test the straws prior to installing them in the gas manifolds and assembling the tracker. The collective limit for gas leaks has been provisionally set at 7 cubic cm/min (ccm)[1]. The current design has: 48 straws per layer, 2 layers per panel, 6 panels per plane, 2 planes per station, and 18 stations for a total of 20,736 straws, hence the maximum allowed limit for each straw is around $33x10^{-5}$ ccm. It has been proposed that *each* straw be leak tested to ensure that this limit is not overshot. Furthermore efforts are going into minimizing the leak rate. While there is a greater concern for reducing the leak from the straws, the straws are not the only possible source of leaks. The manifolds will feed the $ArCO_2$ mixture into the straws and are hence are likely to contribute a fraction of the total leak rate. The lower the leak rates on the straws the more margin of error we have for other possible sources of leaks.

1.1 Assembly of Straws for Leak Testing

The straws are packaged by the manufacturer (PPG) with an inner paper wrapper which first has to be removed. This wrapper is glued to the inner surface of the straw and can be removed easily with tweezers and a free hand to hold the straw. The best way to remove the wrapper is to insert the tweezers between the paper and the straw on both ends ensuring that the paper becomes unstuck all the way around from both sides and then pulling the paper out from one end while holding the straw with the other hand for support as the paper unravels from within. **NOTE:** If one pulls on the paper without properly holding the straw, the paper may wrinkle the straw and may ultimately introduce a leak.

Before leak testing, the straws will have to be plugged. An end piece will be glued in place on both ends using epoxy. The straws have a diameter of 5mm while the end pieces will have a diameter slightly smaller than this in order to leave room for the epoxy to cure between the plastic end pieces and the straw. (It was found that making the pieces exactly 5mm for a perfect fit resulted in the scraping off of the epoxy which in turn led to poorly glued ends). For prototype testing a 3D printer was utilized to make the ends out of ABS (Acrylonitrile butadiene styrene). The orignal design for the end piece had an overall cylindrical shape; however, the pieces were meant to have a small chamfer to make them easier to insert into the straw. The 3D printer used to make these pieces has a resolution of 100 μm but this was too low to actually make this chamfer. The newer design took into account the fact that the printer did not have enough resolution to include a 45° chamfer a quarter of a mm high and instead the height of the chamfer

[1] This limit may change as other gas containing parts of the tracker are leak tested.

was increased to 1mm. The second piece as seen on figure 1, is tapered off in the opposite end as the original piece to make them easier to pull through the manifolds later on in the assembly process. The first prototype had a slightly smaller diameter than the second prototype which had a diameter of 4.95mm compared to 4.89mm. The end pieces worked well with the mixture of epoxy for a 24 hour cure which was viscous enough to stick well to the pieces without being completely scraped off by the straw during the insertion process.

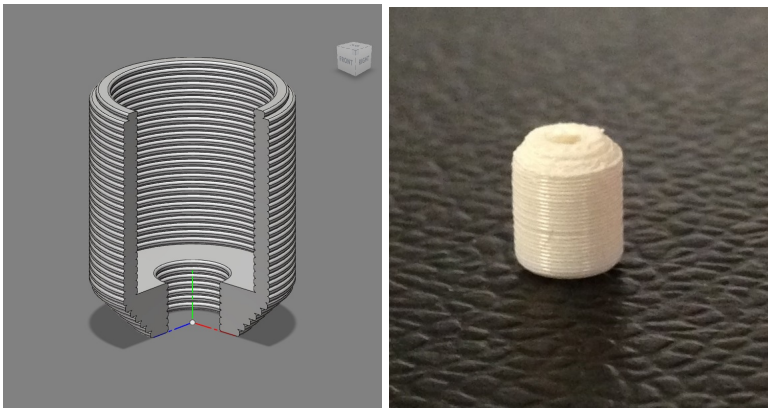

Figure 1: The prototype end pieces used for leak testing were made on a 3D printer. On the left is the model of what we expected the pieces to look like and on the right is what the printer actually produced.

Our prototype end pieces had slight imperfections which were filed down using a small filing tool, this added an extra step to the process but it was proposed that the pieces be made by injection molding for mass production. In either case the pieces should be kept clean and should be handled with gloves once clean in order to avoid contamination with oils from the skin which could affect the curing process of the epoxy. It is recommended that the pieces be cleaned with alcohol (either isopropyl or ethyl) in an ultrasonic cleaner. The pieces can simply be placed inside a sealed plastic bag along with the alcohol which would then be submerged in the water bath within the cleaner. 10 minutes at 22 °C worked fine for our purposes. The pieces were then removed from the bag and were allowed to dry.

The combination of resin and curing agent to make the 24 hour epoxy used to attach the end pieces consisted of 7 parts by weight of Epikure 3155 Curing Agent for every 10 parts Epon Resin 828, both manufactured by the Miller-Stephenson Company. The two were typically mixed in small plastic cups using a plastic or wooden cue tip (using the plastic or wooden ends, not the cotton part). The typical working time for this epoxy is an hour after which the epoxy has cured enough to become notably more viscous and may be harder to apply to the end pieces. Using the same cue tip or mixer one can apply the epoxy onto the end piece. This is best done by inserting tweezers into the intake hole of the each piece (through the tapered end) and holding it this way while covering the lateral surface area of the end piece with epoxy. Because the end piece will be inserted into the straw on the end opposite to the intake hole it is a good idea to have any excess epoxy collect near this end so that when the end piece is inserted the excess will be smeared over the enire length of the end piece. This may also help cover any part of the end piece that may have not been coated well with epoxy and it can also occupy any

space beween the straw and the end piece for better contact. There will most likely be excess epoxy that gets pushed out as the end pieces are inserted. Any residual epoxy should be wiped away from the body of the straw as carefully as possible. Epoxy on the surface of the straw will tend to cure fast and stick to the heating pads during the heating stage and can tear the straw when trying to pry the straw loose. A solution to this is to rotate the straws every so often to keep the epoxy from curing between them and the heating pads.

We allowed the epoxy to cure for an hour before applying heat to allow for faster and more reliable curing. The heating stage consists of placing the straw on the aforementioned heating pads for 4 hours at 60°C. The heating pads utilized were rated at 50 W, 115 V and were calibrated to 60°C using a platinum RTD. The target resistance for a temperature of 60°C was calculated using a manufacturer formula provided on their website which can also be found online by searching for a generic RTD conversion chart. The temperature was found to work well and be safe enough to be handled by hand. The straws were often rotated to avoid sticking to the heating pads. After the elapsed time of 4 hours the power supply for the heating pads was shut off and the straws were allowed to cool before continuing.

1.2 Leak Testing Pre-Procedures

Leak testing the Mylar® straws involved flushing them with $ArCO_2$, sealing them with Devcon 5 Minute® epoxy and then placing them in a chamber. To flush the straws with $ArCO_2$ the gas line was fed into a small syringe with a needle tip the same diameter as the intake hole on the straws. **NOTE:** The better the fit of the feeding line into the straw the lower the impedance due to the difference in the diameters between the gas feeding tube, the diameter of the intake hole on the straw and the 5mm diameter of the straw. A 1L/min flow meter was used to control the gas flow through the tubing and was typically set to flush the straws at a flow rate of 0.6 L/min. The standard time the straws were flushed was about 5 minutes during which the last 2 minutes were spent mixing 5 minute epoxy to seal the straws. The 5 minute epoxy used to seal the straws came in a two-chamber syringe which kept the curing agent and resin separate until the time of usage. The working time on this type of epoxy was roughly 5 minutes after which it became too difficult to work with. After 5 minutes, however, the epoxy was not fully cured but rather it was remained tacky for a few minutes. *Typically* a total time of **10 minutes** elapsed between the time the 5 minute epoxy was mixed and the time the straw was actually inserted into the chamber. As with the 24 hour epoxy this one was also mixed using the wooden end of a cuetip and to apply the epoxy onto the ends. The amount of epoxy used to seal the ends was about the size of a drop of water which tended to form a dome over the aperture on the end piece. After the first end was sealed with epoxy the syringe was removed from the intake side and the epoxy was applied to this end in the same manner. The straws were then to be inserted into a copper tube section of the CO_2 chamber.

Permeability of Mylar® to Gases and Vapors

Vapors	g/100 in²/24 hr/mil*
Acetone	2.22
Benzene	0.36
Carbon Tetrachloride	0.08
Ethyl Acetate	0.08
Hexane	0.12

Gas	Temp., °C (°F)	cc/100 in²/ 24 hr/atm/mil	Test Method
Carbon Dioxide	25 (77)	16	ASTM D1434-58
Freon® 12	55 (131)	0.01	
Methane	25 (77)	1	

* Permeabilities of vapors are determined at the vapor pressure of the liquid at the temperature of the test, 40°C (104°F), using 1 mil film.

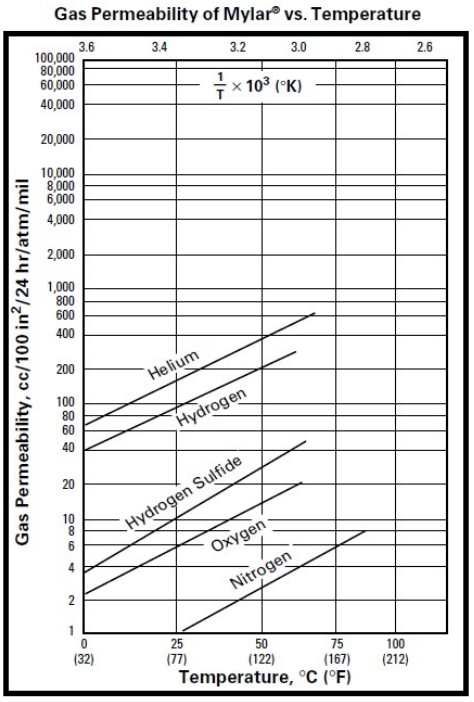

Figure 2: The chart depicts the gas permeability of Mylar® as a function of temperature. The straws will be filled with $ArCO_2$ while the chamber will be purged with N_2. Mylar® is most permeable to carbon dioxide which is not included in the plot but whose value appears in the chart.

1.2.1 Effects of Sealing Straws

Over a period of days, straws that had been left sealed had collapsed, see figure 3. We understood this effect was due to the larger permeation rate of CO_2 through the straws than oxygen or nitrogen, as can be seen in the chart in Fig. 2. The straws were flushed with $ArCO_2$ (80/20) hence the concentration of CO_2 within the straws should have been in the order of 200,000 ppm (parts per million). This concentration of CO_2 within the straw is high compared to the concentration of CO_2 in room air which was measured to be around 500 ppm i.e. 400 times less. Furthermore because Mylar®is permeable to CO_2, impermeable to N_2 (at room temperature) and less permeable to Argon and Oxygen (relative to CO_2) there is a net outflux of gas from inside the straw, resulting in a partial deflation. Upon opening a hole in the 5-Minute epoxy seal using a hand drill, the straws did not regain their shape and were permanently deformed. This permanent deformation of course is not acceptable and should be avoided. Therefore: **straws should be kept open to air after they are leak tested or at least they should not be kept sealed for long periods of time (days)**.

1.2.2 Post-Leak Test Procedures

After the straws are leak tested they would need to be tension tested and assembled into panels; thus it was proposed that having an easy way to tension the straws would be a good idea. Hooks made out of paperclip metal were found to work well; however, they had to be fashioned for this purpose which took some time and they had to have a diameter less than 5mm to get them through the manifolds. It was deemed more convenient to find eye screws which were pre-made with a proper diamter. Brass hooks meeting this requirement were found and purchased online[2] from Artfire.com and were shipped from China. They are just under 5mm with a convenient screw that effectively increases the surface area coated with epoxy (compared to a smooth paperclip surface) and which hold up well [3]. Attatching these hooks results in sealing the ends of the straws hence it was decided that the hooks would be glued in after leak testing, because they are not necessary at that point and instead they should be attached just before they are tension tested[4]. Removing the 5 minute epoxy after leak testing the straws proved to be impractical hence it was suggested that the straws be sealed using clay. This idea was tested using small chunks of APIEZON Q Sealing Compound. The straws were assembled in the same manner and were purged with $ArCO_2$ after which they were plugged with the sealing compound and were then placed into the chamber to test for leaks. The leak rates for a straw tested using this compound were $(6.43 \pm 0.02) \times 10^{-5}$ ccm for Sensor 1 and $(6.47 \pm 0.04) \times 10^{-5}$ ccm for Sensor 2 thus proving that this straw, sealed with APIEZON clay, worked just as well compared to straws sealed with epoxy, but was easier to reopen. **NOTE:** pressing the clay too hard onto the end pieces can force some of the clay into the straw which was not easy, if not impossible, to remove as well as counterproductive since the whole point was to keep the end pieces clean and to be able to insert and epoxy hooks for tensioning.

1.3 The Chamber

The CO_2 Chamber consisted of three main parts: two long copper tubes soldered together each with its own valve and a plastic box housing the carbon dioxide sensors. The copper tubes are of diameter 1.4 cm with slightly different lengths. The top tube on the diagrams below had an approximate length of 151 cm while the bottom tube was approximately 148 cm long. I will refer to the top tube as the intake tube and to the bottom tube as the outflow tube. The valve on the intake tube is a three way valve which opens two different paths while the outflow tube can only be either open or closed. The two valves were mainly used in two configurations, i.e. the internal and external modes. Figure 4 demonstrates the air flow in these two configurations depicted by the red arrows. The external mode was used to purge and load a straw into the outflow tube for testing, while the internal mode was used mainly to isolate the straws or the air within the chamber to measure leak rates reliably. Within the box there is a fan which ensures

[2] http://www.artfire.com/ext/shop/studio/Apollo-beads

[3] A hook was glued with 5 minute epoxy on each side of a straw segment and 700 grams of were hung from the straw to test their strength.

[4] For tension testing the straws will not be filled with $ArCO_2$ hence no need to worry about them collapsing.

Figure 3: A straw that was left sealed over the period of a weekend.

that the air circulates properly and promptly within the chamber. The lack thereof would mean that the only form of circulation of any leaked CO_2 would be limited by its diffusion speed which is quite low. Once the chamber was purged the chamber was put in internal mode which allowed for the circulation of air within the chamber which was *essential* for good measurements of the CO_2 levels.

It is worth mentioning that the chamber was not pressurized. Leak tests were perfomed at 1 atmosphere. Both the straws and the chamber were under the same pressure although the fan would theoretically reduce the pressure on the straw, this would be only a slight and negligible deviation from 1 atmosphere due to the relatively low rotation speed of the fan (12000 RPM), an ORION FANS brushless 5V DC fan (Model OD2510-05HB).

2 Experimental

2.1 Sensors

CO_2 readings were all taken by E+E Elektronik CO_2 sensors. Our particular chamber prototype contained both the **EE892** and **EE891** series of carbon dioxide sensors with

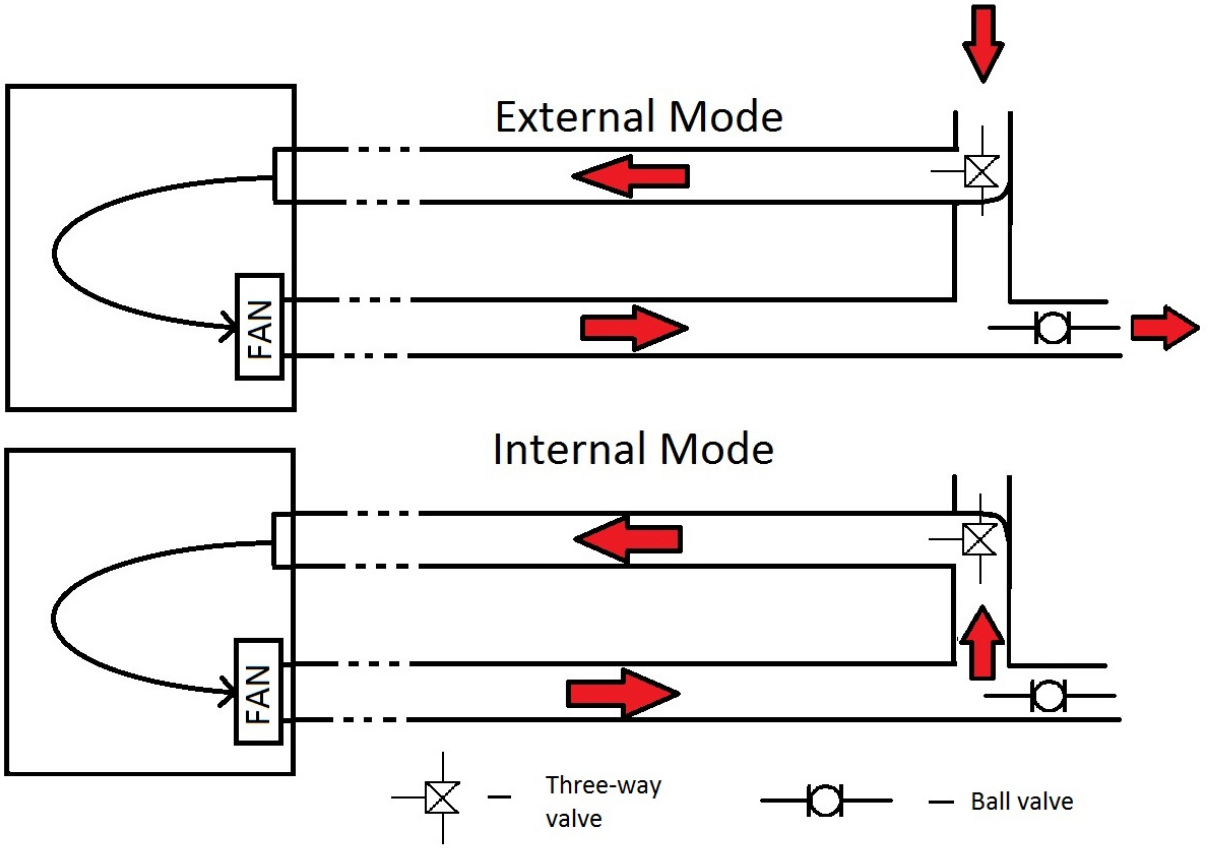

Figure 4: External and Internal modes of the CO_2 detecting chamber.

the EE891 being a newer version of the EE892, both of which utilize nondispersive infrared technology (NDIR) to yield values of CO_2 concentrations in ppm (parts per million). There are a few differences between these two models which are worth noting. In general model 892 yielded higher reading values than the 891 but for the most part these differences remained constant; this is evident in the plots where the 892 readings run parallel to the reading of the 891. The offset seemed to change over time and with consecutive jumps and drops in CO_2 levels. Perhaps the most important difference is the accuracy and precision of such sensors. Through multiple backgrounds the two sensors varied in reading values and spread. A difference between the readings persisted throughout all tests with model 892 yielding higher reading values almost every time, the gap between the two plots, however, varied in an unclear way. This difference appeared to worsen as the reading values increased. The manufacturer website for these devices quotes an accuracy which is directly proportional to the reading value i.e. a *higher* reading value will result in a *higher* uncertainty in the sensor readings. For the most part, over long periods of time, the slopes for both plots agreed within a reasonable amount.

Another difference between the models is that the 891 sensor has an adjustable concentration range of 0–2000/ 5000/ 10000 ppm whereas the 892 model has a fixed 0–2000 reading range. For our purposes both sensors were set to record values in this range in

the 0–2000 range.

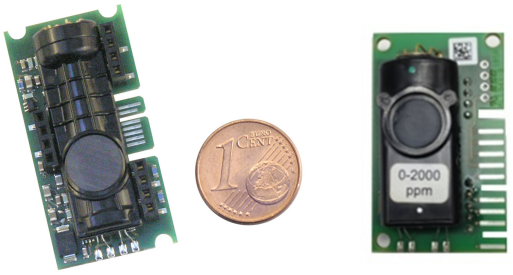

Figure 5: EE891 and EE892 CO_2 Sensors

2.2 Interfacing with the CO_2 Sensors

In order to communicate with the sensors we utilized a LPC2103 32-bit microcontroller. Both sensors were read out through the E2 interface and were on the same data bus. The code was later ported to run on an Arduino microcontroller for convenience. It was proposed that 10 chambers resembling the prototype be assembled so it would be convenient to have all sensors be read out using the same microcontroller. The sensors can be set to take readings as often as every 15 seconds hence there is a potential for having multiple chambers running in parallel while those 15 seconds transpire.

The code was written to output the reading values and the time to a text file which could be easily imported into a data analyzer such as Excel or Root. Excel worked well after installing the Data Analysis Tool add-on available in the Excel Options to perform least squares regressions. The Excel files were set to update from the text files every minute which was specially useful for monitoring the leak rates visually.

2.3 Recording Values

The original plan was to purge the chamber with N_2 and bring down the CO_2 levels to zero and ideally attribute the CO_2 readings to a leakege in the straws. It was found that purging our prototype down to zero was not practical because this took a large amount of time at high flow rates of N_2 and could not be reliably achieved. One of the main reasons behind purging is that the manufacturer quoted a dependency of the uncertainty of the readings on the reading value. The manufacturer of the sensors (E+E ELEKTRONIK) quoted the error in the sensor readings as being $\pm(50ppm + 2\% \, of \, the \, reading \, value)$. This direct dependency on the reading value suggests that the lowest errors are achieved for reading values equaling or those near zero. This was also visually apparent when comparing plots for which the reading values were high to those which were low. The spread in the plots increased as the reading value increased. The error quoted by the manufacturer was tested with our prototype chamber by plotting standard deviation vs. average reading value for segments of data (usually 1 hour long) which were flat. Figure 6 depicts the error as being less than what the manufacturer quoted but still dependent on the reading value.

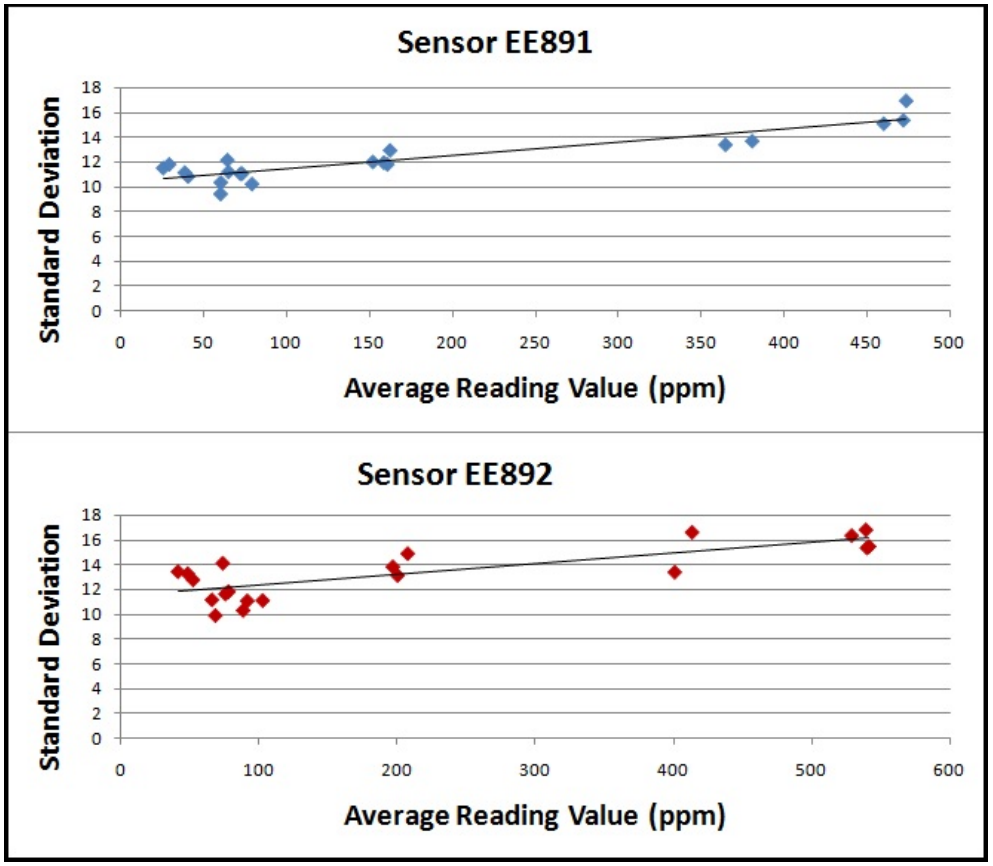

Figure 6: The linear models performed on these points estimate the standard deviation for the EE891 sensor (σ_1) to be $\pm[(10.44 \pm 0.35)ppm + (1.02 \pm 0.13)\%\,of\,the\,Reading\,Value]$ and $\pm[(11.45 \pm 0.45)ppm + (0.86 \pm 0.16)\%\,of\,the\,Reading\,Value]$ (i.e. σ_2) for sensor EE892.

2.4 Backgrounds

We expected that the leak rates would be dependent on time and, furthermore, that the relationship between CO_2 concentrations and time would be exponential in nature due to the difference in concentrations inside the straw and outside the straw (i.e. in the chamber). Initially the leak rates would be steeper and as time progressed this rate would level off as the difference in the concentration of CO_2 inside and outside approached zero. The volume of the chamber was initially approximated at around 733 cc[5] (cubic centimeters) while the volume of the straws was far less than this at around 0.785 cc per cm length with most of these being little over 100 cm long hence the time for the leak rate to equilibrate both inside an outside of the straws would be very long. The slope of the ppm vs. time plots were approximately linear with the exception of the first 7 minutes. It was suggested that we record values without bringing the CO_2 levels down to near zero to avoid the initial climb; however, there was a problem with this idea. For our purposes and for an accurate reading it would be optimal to monitor the leak rates starting from the lowest concentrations of CO_2 possible, because as suggested by the models in figure 6 starting at a higher reading value entails a higher standard error.

[5]Through a series of calibrations the volume calculation was revisited, see subsubsection 3.1.1.

To ensure that this was consistent with the experiment, three different backgrounds were cosidered: N_2, dry air, and ambient air. The latter was discarded as a possibility due to it's instability. To test this option the chamber was simply left open and was set to record values over a few days after which we obtained the plot in figure 7. Ideally we want to perform the exact same leak tests with all the straws so this was unacceptable. A bottle of dry air was available to us so we were able to perform a test with it. Dry air has a higher CO_2 content and because it is bottled it can be controlled just as easily as N_2. The results for the tests with dry air were consistent with a reading value dependency on the spread of our data. N_2 is the best candidate among the three for a background in our leak tests. It can be obtained easily and it is cheaper, it can be used to purge leak testing chambers down to near zero easily, it is cheaper than dry air, and according to the graph in figure 2 it does not diffuse significantly through Mylar® at room temperature.

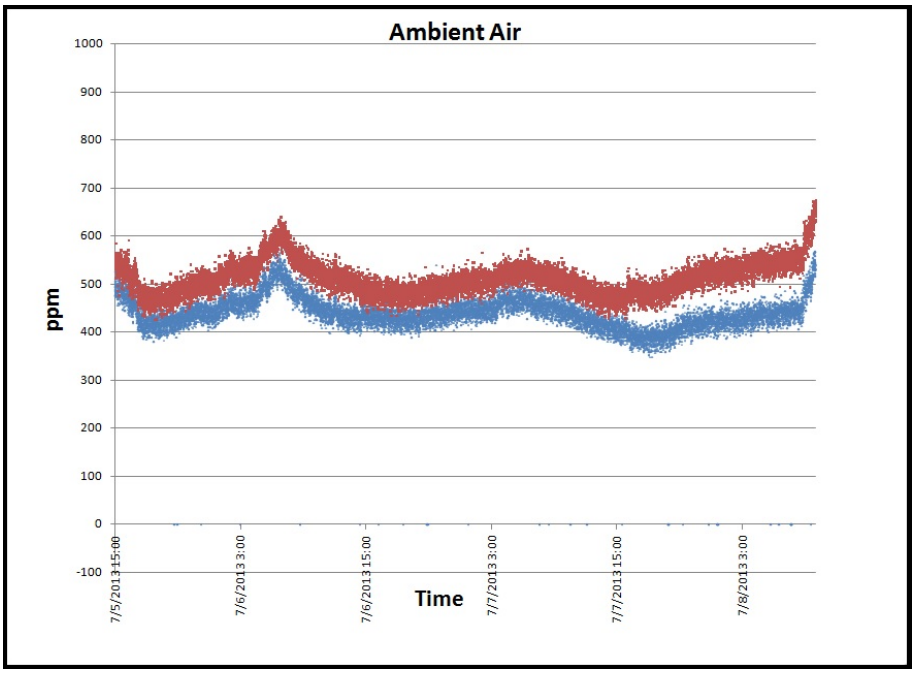

Figure 7: Readings logged for CO_2 chamber open to air over a few days.

2.4.1 Purging with $ArCO_2$

We decided that testing the response to 20% CO_2 would be a good idea hence we ran $ArCO_2$ into the chamber for a few minutes. The sensors were saturated within seconds and began spamming 2000 ppm. With the 20% CO_2 we were pumping in a 200,000 ppm CO_2 gas mixture which proved to be troublesome. Even after purging for longer than usual (30 vs. 10 minutes at more than 1L per minute) the background readings taken afterwards were steep and the concentrations of CO_2 were rising in an exponential manner. The next few backgrounds were also higher than usual. A possible explanation for this is that the CO_2 was adhering to the walls of the chamber and that even after purging there were pockets of CO_2 within the chamber. After each consecutive purge with N_2 the backgrounds slowly went back to normal. It is not a good idea to flush such high mixtures of CO_2 gas into a chamber which is designed to test the leak rate of CO_2

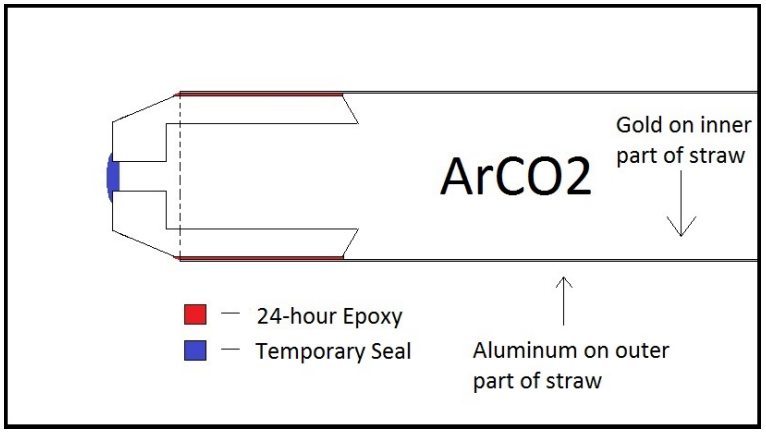

Figure 8: Concept for sealed straw.

because it takes a long time to truly purge the interior of the chamber after doing so, confounding can occur in the source of the leak rate, and because the sensors became saturated after such a short time that we did not gain any new information while they were saturated since the cut off value for the sensors was set to 2000 ppm, and any values above that were lost so there was really no point in bringing the levels up that high.

3 Results

3.1 Calibrations

To test the accuracy of our values we performed multiple calibrations tests which involved injecting a known amount of CO_2 with a fine needle and noting the jump in the readings that accompanied the increase in the concentration of CO_2 inside the chamber, see figure 9. A small hole 0.343 mm in diameter was drilled by hand into the top of the box, housing the sensors and the fan. The box was leak tested to ensure that the leak resulting from the drilling of the hole was not too large. Multiple backgrounds were taken to test the effectiveness of taping over the hole with tape and it was determined that the lowest change in the background was achieved using blue 3M Scotch tape. The two linear regression models relating the change in ppm vs. the amount of CO_2 injected had strong correlation coefficients and Δppm intercepts consistent with zero as expected.

3.1.1 Volume

The equations for the two lines in figure 9 are in the form of

$$\Delta ppm = m \times cc\,of\,CO_2\,injected$$

Where m is the slopes of the least squares regression lines, and where Δppm is related to the volume of the chamber by the equation:

$$\Delta ppm = \frac{cc\,of\,CO_2}{Volume\,of\,Chamber} \times 10^6$$
$$\text{Hence} \Rightarrow Volume\,of\,Chamber = \frac{cc\,of\,CO_2}{\Delta ppm} \times 10^6$$

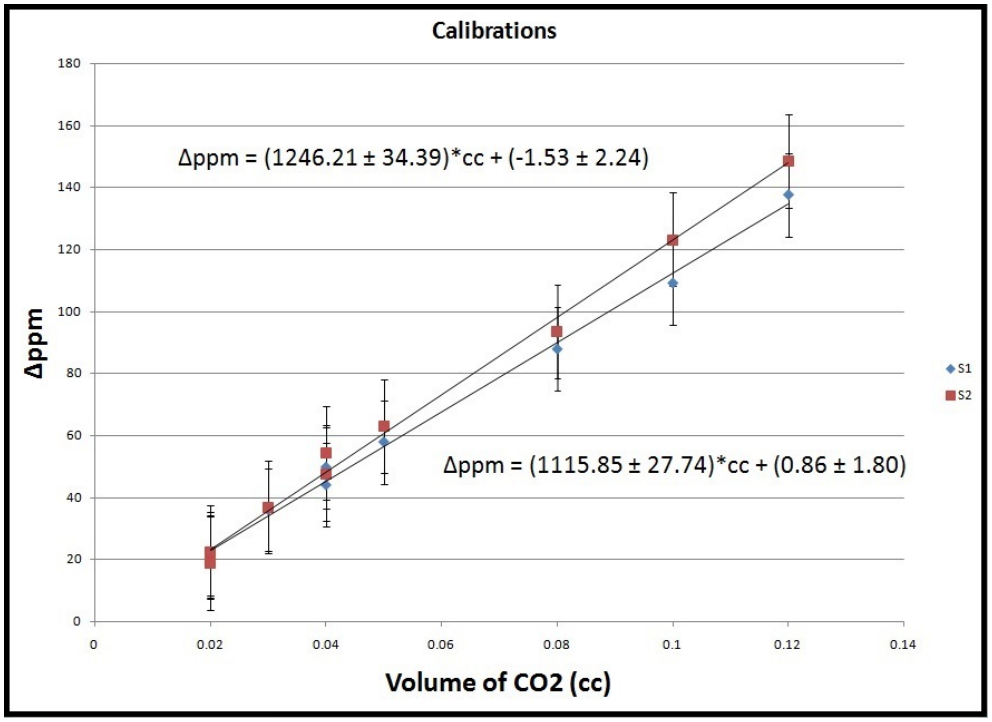

Figure 9:

Because the chamber was setup before measuring all of the necessary dimensions to calculate the volume accurately, we would like to use these formulas to calculate the volume with a higher degree of confidence. Upon calculating all of the different volumes predicted by these models and taking the average of these, the volume was calculated to be (884.7 $\pm$ 15.6) cc from the values of Sensor 1 and (843.5 $\pm$ 29.9)cc from the values of Sensor 2 both of which vary significantly from 733 cc which was the earlier estimate. Based on the 9 calibrations performed we can say that we are 95% confident that the interval (848.6, 920.8) contains the true value for the volume of the chamber. Taking more measurements would help narrow down this interval but we can *confidently* say that the chamber is actually slightly bigger than originally thought and that therefore the leak rates calculated utilizing the old estimate for the volume are a bit lower than the actual leak rates of the straws. It is *highly* recommended that a similar series of calibrations be performed on any future chambers assembled for the purpose of leak testing straws.

3.2 PPG Straws

The first leak tests performed on PPG straws were done on the seven straws, five of them as seen in figure 10. These straws had been previously used for other tests and had brass end pieces epoxied on both ends which were cut off before using. Some of the straws showed signs of creasing although most were only small wrinkles in the body of the straws. The straws were prepared by plugging with the old end pieces, flushing with $ArCO_2$, and sealing with 5 minute epoxy. The first batch of seven was prepared all at once and within an hour hence the same batch of 24 hour epoxy was used to attach the

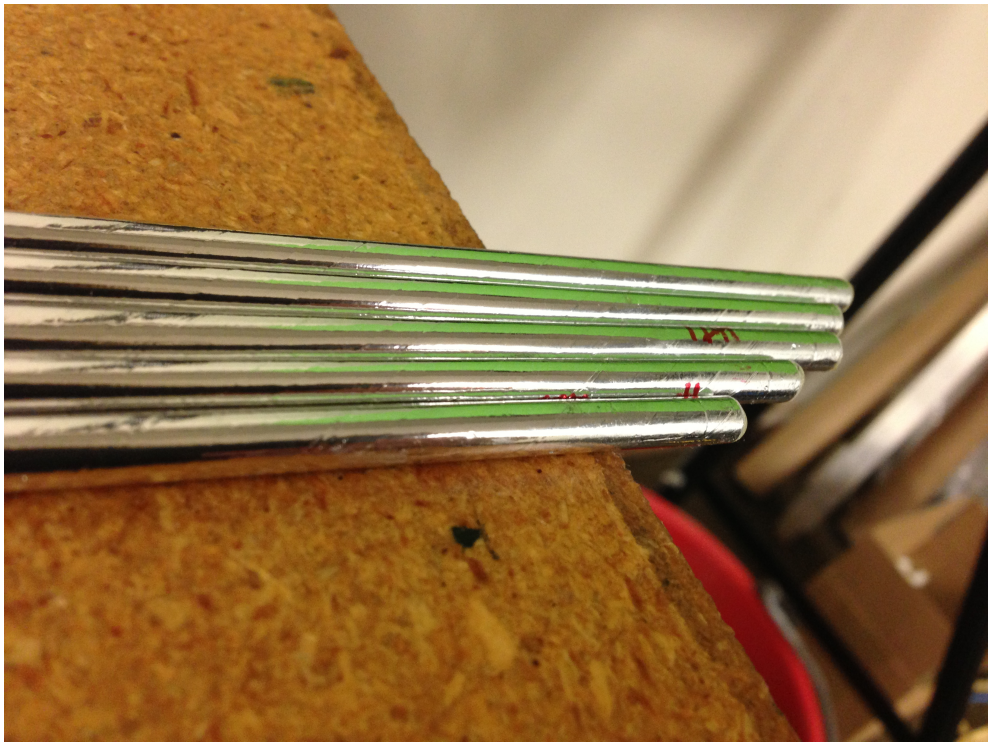

Figure 10: PPG Straws

end pieces. The straws varied in length, but only by a few inches with the shortest straw being 53 inches and the longest being 56 inches long. They were labeled 1-7 in order to keep track of each straw individually. They were flushed and sealed with 5 minute epoxy just before being placed in the chamber to avoid deflation.

3.2.1 Lamina Straws

Before testing the PPG straws the procedure for preparing the straws was practiced on the old version of the straw from a manufacturer in the UK, Lamina. Only a few tests were performed but out of those few tests one straw was found to leak badly. The straw was checked for obvious signs of leaks; however, the straw was in a better shape relative to the other straws tested previously hence it was assumed that the ends were leaking, so they were cut off and redone. Once flushed with $ArCO_2$ and after being sealed generously with 5 minute epoxy, the straw was inserted into the outflow tube of the chamber and was again monitored for leaks. Soon after inserting, the ppm readings started increasing rapidly and after an hour the two plots appeared to be running parallel suggesting that the leak was not due to the ends. Because there was no obvious cause for the leak we concluded that the straw was faulty. The straws to be used in the tracker are made by a different company and hence it was decided that tests on those particular straws would be more relevant.

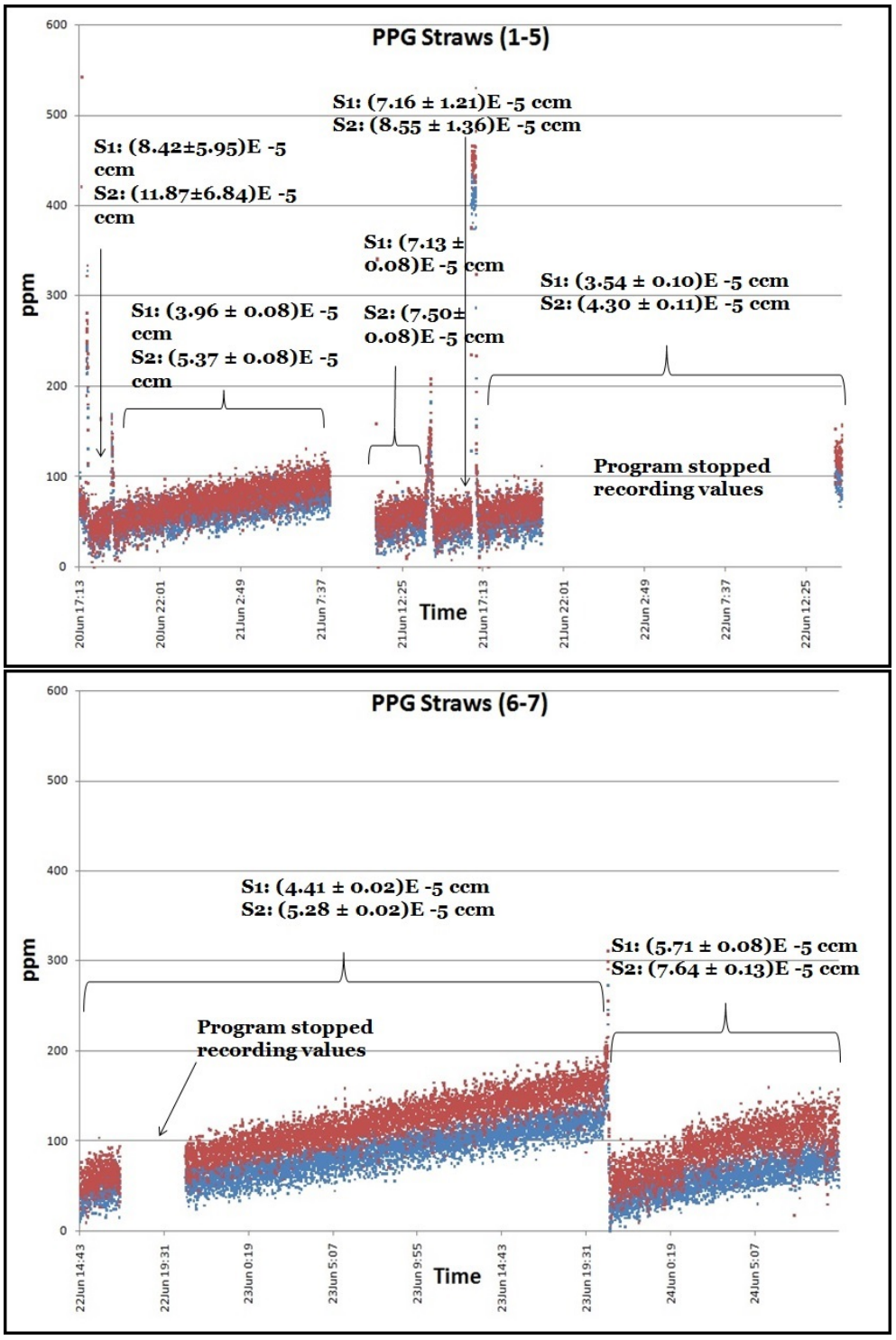

Figure 11: Plots of PPM vs. Time for the first set of PPG straws tested.

3.3 PPG Straws 1-7

Linear Least Squares Regression lines were approximated using the data corresponding to each individual straw and then the leak rates were calculated based on the slope of the regression line that Excel calculated. The slope for the line that Excel put out was in units of ppm per day hence the leak rate was derived by relating this concentration over time to the estimated volume of the chamber. The slopes have been revised to account for the discrepancy in the volume of the chamber. The volume was assumed to be the average volume from the calibration calculation based on Sensor 1, i.e. 884 ccm, which had the lower spread. Even after the leak rates increased by about 20% due to the error in the volume none of the leak rates were over the limit and, furthermore, even in the worst case scenario the maximum slope was 18.71x10^{-5} ccm[6], corresponding to the first straw tested and appearing in figure 11 which is still well below the limit. To obtain the leak rate we then subtract the background for this set of straws which was calculated a couple of days prior to be $(2.41 \pm 0.06) \times 10^{-5}$ ccm and which can be seen at the top of figure 11. Hence the maximum leak rate in the worst case scenario would be 16.30x10^{-5} ccm, which is less than half of the maximum leak rate allowed for each straw. Additionally all of the straws will be shorter (33 in - 46 in) than the length of the straws we tested so we can expect lower leak rates in the shorter straws due to less Mylar® for gas to diffuse out.

$$
\begin{aligned}
& Slope(\tfrac{ppm}{day}) \times \tfrac{1\,day}{24\,hours} \times \tfrac{1\,hour}{60\,min} \\
& = Slope \times \tfrac{1}{1440}(\tfrac{ppm}{min}) \\
& \Rightarrow Slope \times \tfrac{1}{1440} \times (\tfrac{1\,part\,CO_2}{10^6\,Parts\,Chamber\,Volume}) \cdot min^{-1} \\
& \Rightarrow Slope \times \tfrac{1}{1.44\times10^9}(\tfrac{Part\,Volume\,CO_2}{Parts\,Chamber\,Volume \cdot min}) \times Chamber\,Volume \\
& = Slope \times \tfrac{1}{1.44\times10^9}(\tfrac{Part\,Volume\,CO_2}{Chamber\,Volume \cdot min}) \times (Total\,Chamber\,Volume - Straw\,Volume) \\
& = Slope \times \tfrac{1}{1.44\times10^9}(\tfrac{volume\,CO_2}{Chamber\,Volume \cdot min}) \times (884cc - \pi(0.5cm)^2 \cdot Length\,of\,Straw) \\
& = \text{Rate of Increase} \\
& \Rightarrow \text{Rate of Increase - Background = Leak Rate of } CO_2
\end{aligned}
$$

3.4 PPG Straws 8-15

PPG straws 8 through 15 are new straws from the second batch of PPG straws made for *Mu2e* and were prepared in a similar manner as straws 1–7 with the exception of the end pieces. This set of straws was plugged using the new model for the end pieces which were 3D printed shortly before putting the straws together, see figure 1. As with the old pieces the imperfections[7] were filed down and then cleaned by placing them in a plastic bag in ethyl alcohol and then placed in an ultrasonic cleaner. 24 hour epoxy was mixed to secure the pieces onto the ends of all of the straws; the pieces were inserted up to the point where the chamfer starts.

[6] This number is the overall slope of the least squares regression line i.e. it is the leak rate plus the background

[7] The imperfections mentioned are mainly a vertical ridge along the outer surface of the pieces, both the old and the new ones.

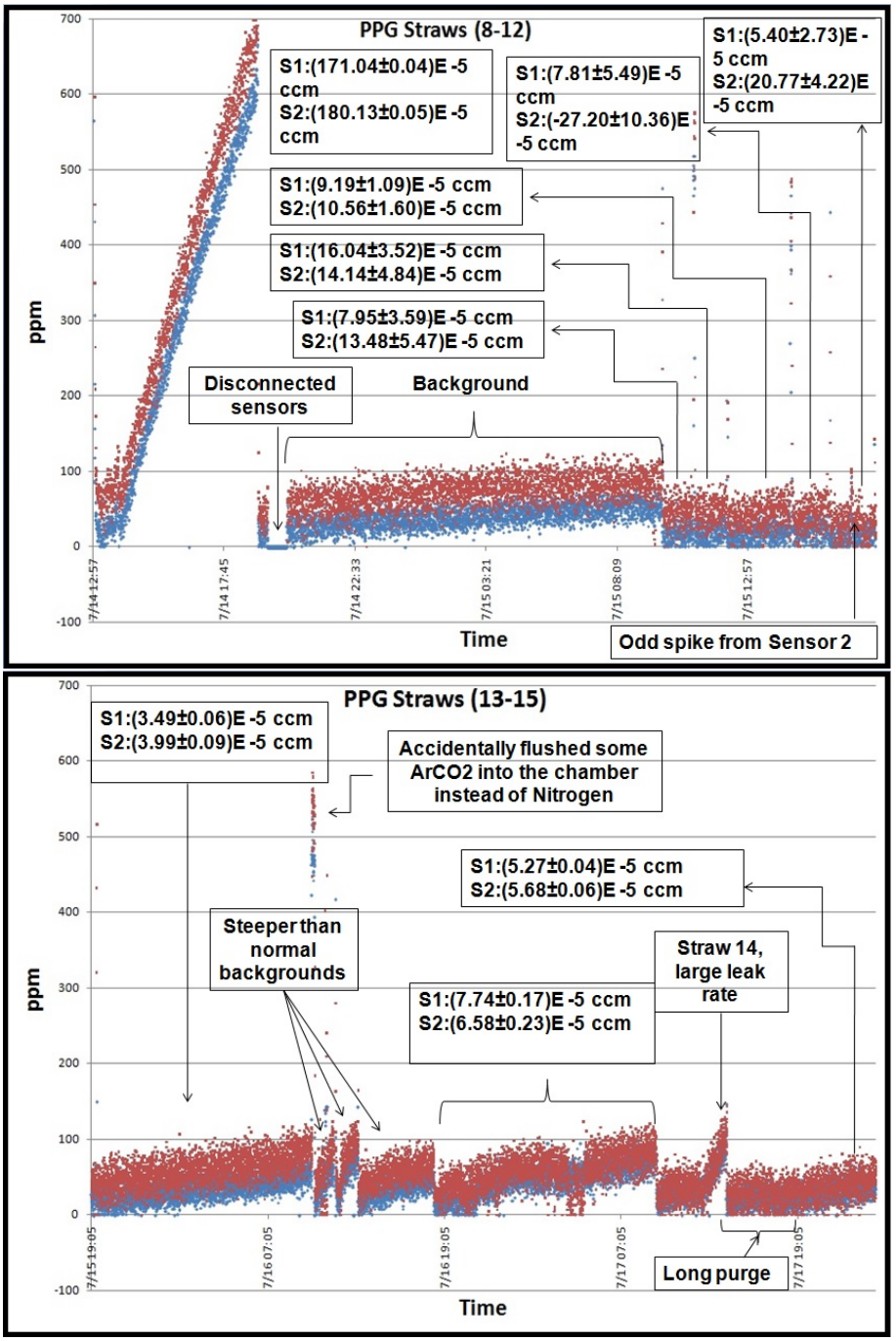

Figure 12: Second Set of PPG Straw Leak Rates

The epoxy cured at room temperature for an hour while the pieces were being attatched on all 8 straws, once the working time for the epoxy had expired the ends were heated for four hours using heating pads after which the straws sat at room temperature over a day until they were used. Straw number 8 was flushed with $ArCO_2$ for about 5 minutes and was sealed using 5 minute epoxy. It was inserted into the chamber and left to record over night. The straw leaked from the start at a steep rate of about 170.1×10^{-5} ccm (Sensor 1) and 180.0×10^{-5} ccm according to Sensor 2 (see first . Upon inspection, there was a small lip on the straw which had not glued well to the end piece while insert-

ing it. This possibility that this was the source of the leak was tested and confirmed by generously adding another coat of epoxy to the ends and specially around the edges of the straw and then monitoring the leak rate a second time. Something similar occurred with straw 14.

Straw 14 had a somewhat steep slope thus it was removed shortly after inserting into the chamber. Straw 15 was inserted into the chamber and the chamber was accidentally flushed with $ArCO_2$ and the sensors became saturated. Straw 15 was removed and the chamber was purged; however, even after purging, the CO_2 levels were rising abnormally fast. Straws 8 through 14 were reopened using a hot knife and a small hand drill to allow air to diffuse in and out of the straw without causing them to collapse. After purging two more times straw 15 was reinserted and leak tested over night. Because straw 14 had shown a higher than normal leak it was flushed again with $ArCO_2$ and then it was sealed again. Straw 14 was re-inserted into the chamber and a steep slope was again observed. The straw was removed and another coat of 5 minute epoxy was applied generously to the ends of straw 14 to ensure that they were properly sealed. The chamber was re-purged and straw 14 was inserted into the outflow tube. The leak rate was found to be low confirming that the ends were not properly sealed. On a separate note, in the process of opening the straw by drilling through the 5 minute epoxy was difficult to do without wrinkling the straws near the ends. Straw 14 was also a test in the resiliency of the straws, because it became very wrinkled while reopening it as seen in figure 13. Surprisingly the leak rates for this straw were some of the lowest in the batch hence we concluded that the the leak in straw 7 was mainly due to the poor seal between the straw and the end piece.

For this set of straws the background was measured to be $(3.42 \pm 0.10) \times 10^{-5}$ ccm. In the worst case scenario for this set of straws the maximum leak rate would be that of straw 10 which had a slope of $19.56\text{x}10^{-5}$ ccm. Taking into account the background, the worst leak rate would be $19.66\text{x}10^{5}$ ccm which is below the limit. We can expect that the true value for this leak rate is lower than this because the interval of time recording values for this straw was relatively short (an hour and 4 minutes). The same is true for most of these straws which only spent approximately an hour in the chamber and hence had higher uncertainties. We are, however, confident that the leak rate for these straws is bounded from above by the slopes calculated here with the exception of straw 12 who's approximate slope was negative according to sensor 2. There sometimes occurs a glitch in which sensor 2's readings which causes a shift in the reading either up or down. For this reason and because the error is perpetually larger for sensor 2, it would be best to use only the EE891 model of the CO_2 sensors (the newer model).

4 Pressurized Tests

4.1 Procedures

After obtaining results from leak tests perfomed in a vacuum, it was decided that the straws should be pressurized when leak tested. These pressure tests performed at the City University of New York by James Popp and Kevin Lynch involved monitoring the pressure change over time of a vacuum chamber containing four of the previously

Figure 13: Cutting and drilling through the epoxy can wrinkle the ends of the straws if they are not held firmly where the end piece is. The two pictures are also before and after pictures showing how the leaky straws tested were coated generously to rule out the ends as the source of the leak.

tested PPG straws. The straws were open to air and then sealed before being placed in a vacuum chamber, hence the air inside them was at one atmosphere, and after inserting them in a vacuum chamber, the chamber was brought down to 10^{-4} torr creating a ΔP of about 1 atmosphere. After two days when the straws were removed from the chamber, one of the straws was completely deflated; thus, it was deemed appropriate to do CO_2 leak tests with pressurized straws in order to explore this type of behavior. It was expected that the straws would leak more when under pressure since the tendency for gases to permeate through permeable material increases as the ΔP accross the material, i.e. the straw, increases. Our leak tests utilizing the CO_2 chamber did not allow us to bring the pressure in the chamber below atmospheric so instead the straws were to be pressurized to two atmospheres resulting in the same ΔP.

Initially a straw segment was tested prior to using pristine PPG straws by sealing them and attaching 1/8 inch tubing to the end piece to make it easier to pressurize. The 1/8 inch tubing was found to be appropriate because the end pieces have two holes one of which is larger than the other hole and which could be easily drilled to the proper size of the tubing using a drill bit. Regularly the end pieces are epoxied in place with the narrow hole facing out of the straw; however, since the tubing had to be on the exterior of the straw the end piece with the tubing had to be inserted backwards, i.e. with the narrow hole facing the interior of the straw. The second end piece was epoxied normally and the straw with the tubing attachment was placed on the heating pads which were set to the max voltage on the power supply (40 volts vs. the usual 17 V). Once the epoxy had cured, the straw was sealed on the end opposite to the tubing, and the tubing was connected to the gas line. For a better connection between the tubing and the gas line a 1/8 inch ferrul was placed on the tubing, and it was fastened with stainless steel connectors. The straw was then flushed with N_2 until the pressure gauge on the gas line reached the maximum level (14.7 psi or about one atmosphere above atmospheric pressure). The straws were notably stiff when at this pressure; however, the connection between the gas line and the straw was not quite as good, hence pressure dropped fairly

quickly upon shutting off the gas line.

In an attempt to detect a change in the ability of the straws to hold pressure, the time it took for the pressure in the straws to go from 14.7 psi to 10 psi was timed over a day. The straw was kept pressurized and every hour the the air flow was turned off and the time it took for the straws to lose pressure was recorded three times. We expected that if the straws were becoming less resistant to pressure then the time it took to go from 14.7 psi to 10 psi would decrease indicating that the straws were losing pressure faster. However, the opposite was noted after timing this change over a day. The time it took to go down to 10 psi increased from about 30 seconds to about 43 seconds. The straw was obviously not becoming more resistant but rather this suggested that something was probably expanding. The likeliest of all components to expand was the tubing which is able to flex easily. This test could be improved by using metallic tubing instead of plastic, but because the straw did not burst and because the pressure did not seem to have any adverse effects on the epoxy seal, it was decided that the straws were probably resistant enough to widthstand the pressure.

The test straw was found to widthstand 2 atmospheres of pressure, so we proceeded to find a way to seal them in order to keep them pressurized and be able to insert them into the chamber. It was resolved that the tubing could be melted off and clamped to form a seal which was easy to open up. For a comparison between pressurized and unpressurized straws the straws were tested twice. First they were tested unpressurized by flushing with $ArCO_2$ (80/20), sealing the open end with five minute epoxy, waiting for the epoxy to cure (about 10 minutes), clamping the tubing with pliers, and melting off the rest of the tubing attached to the gas line. The tubing was sealed by clamping the melted portion of the tubing while still hot with a separate pair of pliers. The tubing was cut according to the length of the straw, i.e. so that they straw plus the tubing could fit into the CO_2 chamber. It was noted that the straws were easier to handle once pressurized since they become notably stiff. The straws were placed into the chamber and the chamber was purged down as usual. the straws are easier to handle once pressurized since they become notably stiff.Three straws were assembled and tested in this manner and additionally the last two straws to be tested were kept sealed while the first one was opened to air by simply cutting into the tubing with a sharp razor blade. The second straw lost some pressure during the sealing process thus the pressure inside the straw was below the target. The straw that collapsed completely during the vacuum test collapsed after about a day and a half; however, the set of straws prepared for these pressurized tests qualitatively showed no signs of deflation or pressure loss after the same amount of time.

4.2 Results

The background for the set of pressurized straws was measured to be about $(6.36 \pm 0.16) \times 10^{-5}$ ccm by the readings from the first sensor. The second sensor appeared to have random fluctuations as seen in figure 15 thus the background from sensor 1 was utilized to calculate all leak rates in this set. The unpressurized test served as a comparison to the previous PPG straw tests. The leak rates for this set were between 4.52×10^{-5} and 8.53×10^{-5} ccm while the pressurized leak rates ranged between 6.32×10^{-5} and 13.30×10^{-5} ccm. We expected the leak rates for the pressurized straws to increase by

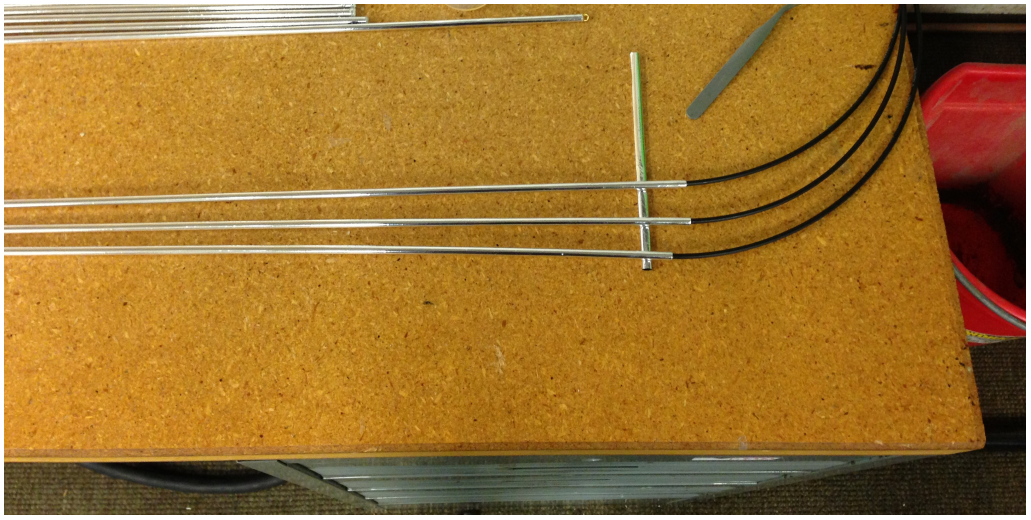

Figure 14: PPG straws with attached tubing for pressurized leak tests.

a factor equivalent to the ratio of the pressure in these straws to atmospheric pressure (ideally this would be a ratio of 2:1). What was observed, however, was a factor of about 1.53 for the first straw, 1.17 for the second, and 1.45 for the third. As expected the leak rate for the second pressurized straw was less than with the other two which were sealed without losing much pressure. As in previous tests the leak rates were safely below the proposed limit; although, they were higher than the leak rates measured for the same straws for the unpressurized tests, as expected.

5 Conclusion

5.1 Process

It is recommended that for performing leak tests on straws in higher numbers there be more students assembling straws than leak testing them since the rate limiting step in the process is the heating stage. The working time for the 24 hour epoxy utilized and described here is one hour so it would be best to mix just enough epoxy according to the number of students that will be putting these together. Although the working time for the 24 hour epoxy is one hour the epoxy can sometimes be usable for longer than this. There is a notable difference from when the epoxy is good for gluing the ends in and when it has cured too much to apply succesfully to the end pieces so 1 hour is not exactly the cut off time. Assembling the second batch of 8 PPG straws took the entire working time of the epoxy thus for 5 hour of purely putting these together at least 40 straws could be made by one person. Extrapolating to 5 students working 5 days a week for 5 hours each day would yield 20,000 straws in 20 weeks. However, these numbers may be a bit optimistic since the students will first have to learn the procedure and get accustomed to handling the straws without wrinkling them. Once they are handled properly and can be assembled at a decent rate it is a good idea to have a surplus of straws with the end pieces glued in ready to seal and test in the CO_2 chambers since these straws can sit around without risk.

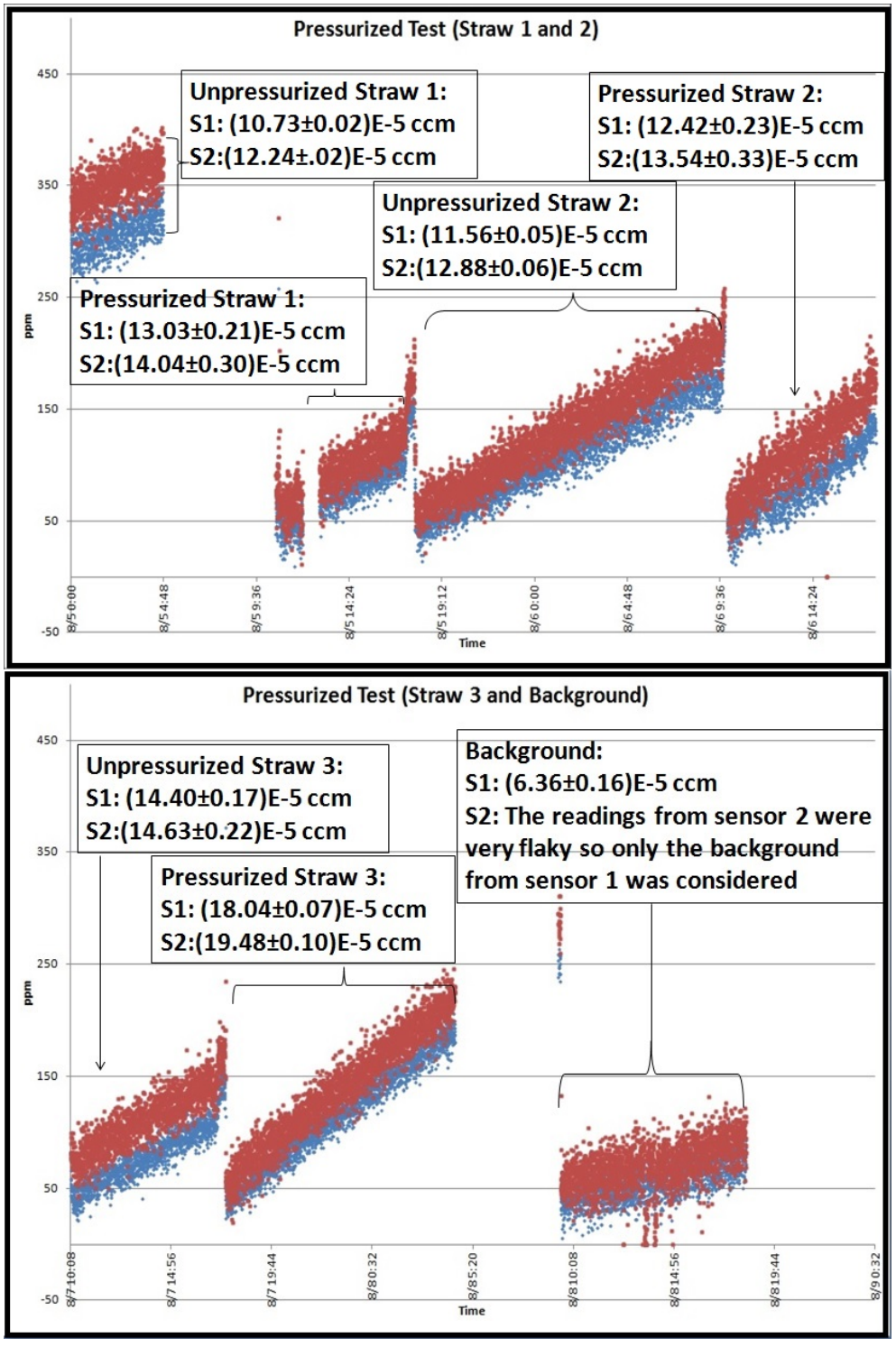

Figure 15: Pressurized leak rate tests performed on PPG straws.

The possible use of clay to seal the straws should be tested further to determine whether this is a better alternative to 5 minute epoxy. The leak rates were reasonable and low but the clay has the potential to be messier if it were to get inside the straws. A possible test to determine which provides for a better seal would be to leak test a couple of straws using APIEZON Q, then removing the clay and sealing with 5 minute epoxy and leak testing again. If the clay were to be used to seal the straws before CO_2 leak testing in a chamber afterwards a hook would be glued onto each end using 5 minute epoxy anyway. This would test whether the clay might leave residue which would not be compatible with the 5 minute epoxy that will be used to seal the straw and attach a hook.

5.2 Leak Testing

In the future it would be helpful to try flushing the straws with gases of higher concentrations of CO_2. Using pure CO_2 would reduce the time each straw has to be inside the chamber by a factor of 5. This could be used to visually detect a leak faster, instead of waiting an hour one could wait 20 minutes or one could still wait the full hour and have a higher confidence that the straws will not leak. Because the CO_2 sensors cannot take readings more often than every 15 seconds it is not recommended that one wait only 20 minutes to obtain a leak rate because the uncertainty in the linear models will be much larger that way and one could potentially accept straws which are near the limit. It is *strongly* recommended that a background reading be done prior to doing a series of leak tests and that moreover there should be multiple backgrounds taken when purging down from *high* levels of CO_2 (anything above 600 ppm). Visually we are able to tell whether the background is increasing too drastically thus it would be a good idea to purge down a couple of times before starting to record a series of leak rates for a batch of straws.

In an attempt to determine the lowest amount of time it would take to determine the leak rate within the limit of 33×10^{-5} ccm, it was found that the first 7 to 10 minutes of the readings from sensor 1 consistently yielded higher values for the leak rate that did not accurately represent the long term behavior of the CO_2 concentration in the chamber. For future reference if leak tests are performed in 1 hour intervals, omitting the first 7 to 10 minutes of readings from the **EE891** model can give a decent upper boundary on the leak rate. This follows from the observation that leak rates tend to go down as the uncertainty in the slope of the linear model decreases which decreases as more values are logged. Overall the EE891 sensor had less issues, therefore as previoulsy mentioned it would be best to use only the EE891 model of the sensors by E+E Elektronik. On a similar note having two sensors reading values in the chamber has proven to be helpful in noting discrepancies such as those found in sensor 2, and as so it is a good idea to have two of the same sensors working simultaneously in each chamber.

Out of the 18 PPG straws tested none of them were found to leak badly other than when not sealed properly. Because poorly glued end pieces can be mistaken for a leaky straw, it is important to glue in the end pieces well without having to excessively coat the ends as in figure 13. The straws will have to be assembled into panels and there's very little clearence through the manifolds; thus, there can be no excessive amounts of epoxy on the ends of the straws. More straws should be tested in order to gain a good idea about the failure rate of these straws. So far, an excessively leaky PPG straw has

not been observed. If after 100 or so straws none are found to be leaking above the limit it would save time to test more than one straw at a time in each chamber and simply make sure that the leak rate is conveniently still less than 33×10^{-5} ccm, so that in the worst case scenario if the entire leak is due to any single straw then the leak rate is safely below the maximum.

It would be advisable that these preliminary straw leak tests be perfomed with pressurized straws. The straws in the pressurized tests more closely resemble the state of the straws once the tracker is running, i.e. they will be filled with $ArCO_2$ at one atmosphere and will be in vacuum. The difference in pressure in these pressurized tests resembles the ΔP of 1 atmosphere; however, since the straws in the tracker will constantly be flushed with $ArCO_2$, and ΔP will remain constant; whereas, the straws in the leak tests will naturally lose pressure as CO_2 diffuses out hence as time increases the test will lose relevance. The most pertinent time interval over which the leak rates will predict the behavior of the straws in the tracker is; therefore, the span of time over which $\Delta P \approx 1$ atmosphere. This interval is convenient because it justifies taking readings over short intervals of time such as one hour during which the straws lose a negligible amount of pressure. The test, however, needs improvement since some of the pressure was most likely lost while sealing, and the actual pressure inside could not be quantatively measured after they were sealed.