The University of Iowa
Quarknet 2016 Student Research

Teachers and Students from Bettendorf High School, left to right:
Mike Grannen, teacher, Pete Bruecken, teacher, Katherine Braught, Class of 2016, Thomas Crowley, Class of 2017, Roger Wttmer, Class of 2018, Bridget Quesnell, Class of 2017, Sam Snow, Class of 2017, Andrew Del Vechio, Class of 2018 and Moira Truesdell, teacher.

During the summer of 2016, three teachers and six students from Bettendorf High School engaged in research for the Compact Muon Solenoid (CMS) forward calorimeter group at The University of Iowa. This team of researchers focused on creating and testing materials for use in the forward calorimeters in CMS at The European Organization for Nuclear Research (CERN). The research consisted of creating and testing materials for use in the high radiation regions of CMS.

The team began by evaporating organic-fluorescent compounds on borosilicate plates in an attempt to create plates that pumped visible light when activated by an ultraviolet (UV) LASER. The students evaporated the dyes in solvents and then annealed them in an oven in the absence of oxygen to make crystals that would not only fluoresce but conduct the light of

fluorescence to the edges of the plates. The team also honed their skills in testing the plates using an oscilloscope and Photomultiplier Tube (PMT).

After the students worked on the evaporated plates, they worked on creating epoxy plates laced with fluorescent chemicals. In an attempt to make radiation hard plates of fluorescent materials, the students worked with a radiation hard epoxy and their fluorescent dyes. An attempt was made to create radiation hard plates of epoxy that would compare with the current plastic plates used in the Electromagnetic Hadron (HE) forward calorimeter. The students dissolved the dyes in the epoxy and made tiles that were tested with the UV LASER and oscilloscope. Data was taken and analyzed in reports.

During the week of July 25-29, twenty-six teachers from all over the state of Iowa attended an institute for particle physics in high schools. They spent the week studying The Standard Model, making an audio transducer to take back to their schools, touring Fermilab, working through the Quarknet activities with 2 Quarknet presenters, attended lectures from educational and physics professors and had a live online conversation with 2 researchers at CERN.

Quarknet 2016
Scintillators and Borosilicate Plates

Katherine Braught (Bettendorf High School), Pete Bruecken (Bettendorf High School), Thomas Crowley (Bettendorf High School), Andrew Del Vecchio (Bettendorf High School), Mike Grannen (Bettendorf High School), Dr. Yasar Onel (The University of Iowa), Bridget Quesnell (Bettendorf High School), Sam Snow (Bettendorf High School), Moira Truesdell (Bettendorf High School), Dr. James Wetzel (The University of Iowa), Roger Wittmer (Bettendorf High School)

Purpose: The calorimeters in the Compact Muon Solenoid (CMS) at the Large Hadron Collider (LHC) at CERN are adversely affected by the radiation to which they are exposed. Currently, plastic scintillating plates are used in the Electromagnetic Hadronic (HE) calorimeters. These plates are found to darken when exposed to large amounts of radiation, thus decreasing their sensitivity. Several methods and materials that can withstand the radiation have been tested in an attempt to develop a replacement for the HE plates and also maintain the required amount of sensitivity. One method used in an attempt to develop the components is the dissolving of organic scintillators in solvents and subsequently depositing them onto borosilicate plates. Dissolving organic scintillators into volatile liquids, such as benzene and toluene, and pouring them onto plates is helpful to the production of coated glass plates because it coats the plates evenly and quickly with the necessary scintillating materials. This process involves super saturating a volatile liquid and allowing it to evaporate, leaving a thin coating of the scintillating materials.

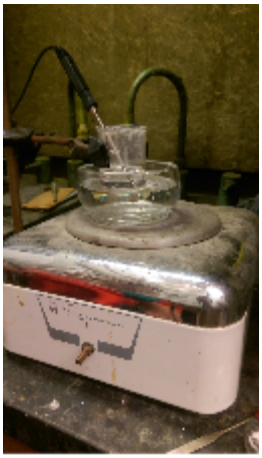


Figure 1

Procedures: Because of the volatile nature of the chemicals, the entire process was performed under a fume hood for safety. The first step for the plating was to heat one of the solvents, benzene or toluene, in a hot water bath. A 100 mL beaker filled with 20mL of the solvent was clamped onto a ring stand and placed into the hot water bath (Figure 1). Next, the amount of solute, either Anthracene or p-Terphenyl (PTP), was measured out using an electronic balance. The scintillating materials were then scooped into the heating liquid. The mixture was then stirred with a stirring rod for 10-15 seconds and allowed to sit as it was heated. Every 3-4 minutes, the solution was stirred again and the temperature was checked to ensure no solvents (i.e. Benzene, Toluene) exceeded their respective boiling points. Once the mixture reached approximately 45^{o} C, which was determined

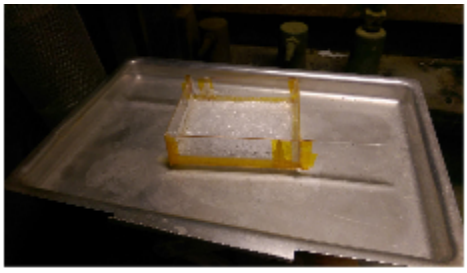


Figure 2: An example of a glass boat

to be a sufficient temperature for dissolving the amount of the substance required for plating. and all the scintillating materials were dissolved, the saturation process was complete.

Following the saturation of the solvents, the mixture needed to be distributed onto the plate. Before the heating process began, a borosilicate glass boat was built to hold the mixture onto the plate and evenly coat the plate. The sides of the boat were created by using a glass cutter to cut up other plates into smaller pieces that would fit along the side of a full plate. Kapton tape, which is resistant to extreme temperatures from –269°C to 400°C, was placed along one entire side of the full glass plate, with around half of the strip of tape stuck onto the plate and the other half hanging off. The glass plate was then flipped over so that the sticky side of the tape is facing upwards, and the corresponding glass piece was then placed onto the tape. A small gap was kept between the plate and the side in order to allow room for the side to be rotated upwards to form a wall. The process was then repeated for each side of the plate, and all four sides were rotated upwards to form the walls. More kapton tape was then used to secure each side together at the corners. A small strip was used to initially hold the sides together, and a larger piece was placed along the entire corner in order to keep it sealed. (Figure 2) After each corner was taped, a small amount of water was poured into the boat and observed to make sure that none escaped through the corners. If some did, the corner was resealed and then tested again. After the boat was completed, it was rinsed with acetone and allowed to dry in order to ensure no water or other materials were left over inside the boat when it was used.

Once the boat was built, tested, and dried, it was placed on a level surface in a fume hood. Then, the saturated solution was slowly poured into the boat. Once in the boat, the solvents were allowed to evaporate undisturbed overnight, leaving the scintillating materials coating the plate. (Figure 3)

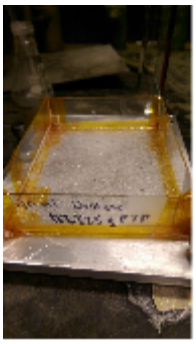


Figure 3

In further trials, more scintillating materials were needed on the plate than a solvent could dissolve. In this case, the process of double or triple evaporation was used. This involved the same exact procedure, only repeating it again the next day and pouring the new saturated solution on the plate with a scintillating material already on it. For example, triple evaporations took three days to complete. (Figure 4)

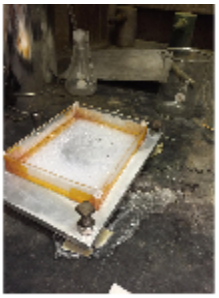


Figure 4: A triple coated boat

After all the solvent had dissolved from the plate, the sides of the boat needed to be removed and the plate needed to be sandwiched. In order to remove the sides, a razor blade was used to cut the tape and pull the sides off. (Figure 5) The remaining tape on the plate was then gently pulled off. In order to sandwich the plate, a clean plate was placed on top of it, with the coated side in the middle. Kapton tape was then

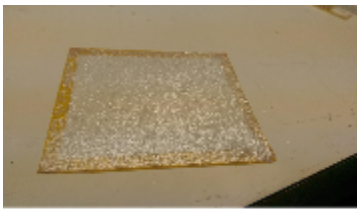


Figure 5

Figure 6

placed around the edges and folded over in order to hold the two plates together. The length of tape was longer than the side of the plate, allowing for excess at the corners. This excess was then cut into small flaps that were folded down in order to ensure the corners were airtight. After each side was taped and sealed, the sandwiched plate was completed. (Figure 6)

The final step in producing a borosilicate plate coated with a scintillating material was to anneal the plates in a nitrogen environment oven (Figure 7). Annealing is the process of slowly heating and cooling a material in an attempt to change the material's crystal structure. Before starting, the oven was inspected to make sure there was no residual material inside that needs to be cleaned. If necessary, the oven was cleaned before use with acetone to prevent any impurities for the annealing process. First, the plate was placed in the center of oven, where it was visible through the window, along with the thermometer. The oven door was shut and sealed tightly with two knobs, and the connected mechanical vacuum pump was turned on. The pump was allowed to run until a vacuum of around 30 in. of Mercury was shown on the pressure indicator. Then, the oven was filled to normal pressure using a nitrogen tank connected to it. After the oven was filled with nitrogen, it was turned on and placed on the lowest heat setting. The heat was slowly increased until it reached just below the melting point ($M.P._{ptp}$=414℉; $M.P._{anthracene}$=420℉) of the substance being annealed, and was kept at that temperature until the plate had a uniform, clear appearance. The oven was then simply turned off and allowed to cool over several hours. Once cooled, the plate was removed and ready to be tested. (Figure 8)

Figure 7

Figure 8: Annealed Plate, Ready for Testing

We tested PTP and Anthracene as solutes in Benzene and Toluene as solvents for PTP. We tried single volumes, double volumes, triple volumes, and quadruple volumes of solutions to see if more crystals made a significant difference in plate output.

Testing Process: The borosilicate plates coated with annealed, organic materials are tested for their scintillating features using a pulsing laser. Inside a dark box, a laser pulses through fiber optic cables which carry the light from the laser to shine upwards through a plate. A trigger PMT (Photomultiplier tube) is located above the testing area, and another PMT is directly against the side of the tested plate. The trigger PMT receives the laser signal first, and it communicates to the side PMT when to collect data. The side PMT

measures how much scintillating light is being transferred through the side of the tested plate. (Figure 9) Before testing, a small cut is made in the kapton tape of each side of the borosilicate plate, so a small window can be made to place against the side PMT in the laser testing box. (Figure 10) The data measured by the side PMT is collected and transferred to a Tektronix TDS 5034 Digital Phosphor Oscilloscope, which displays a graph of the scintillated light over time. After the laser has fired for about 30 seconds, the oscilloscope can display a graph with the average values over the duration of time. Amplitude, area, and burst width of the graph are all recorded in a data book, and pictures of each graph are saved within the program on the oscilloscope. (Figure 11)

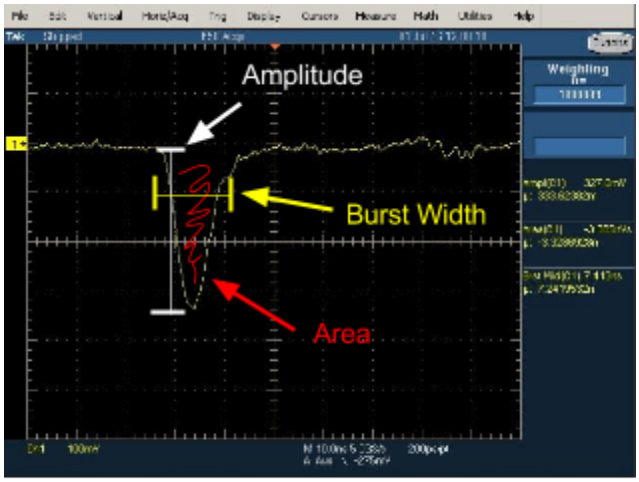


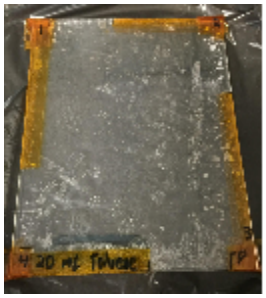
Figure 11: Example of Oscilloscope Data

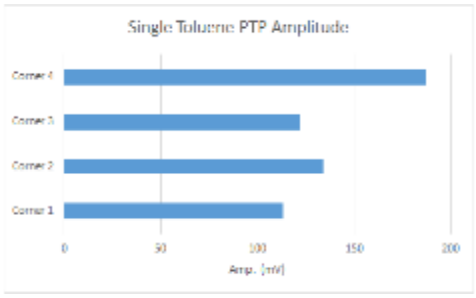
Results: After we took the data for all of the borosilicate plates, we made graphs to make comparisons. However, before making graphs, the data needed to be normalized because the control, or HE plate, changed its signal strength from test to test. The reasons for this were not completely clear. The LASER source could have varied from time to time and the oscilloscope had some failures and fixes that seemed to change the data. Normalization using the control signal from the HE plate seemed to be the best way to adjust for these variances.

To normalize the data, we divided the data from the tests by the data from the HE plate. We measured the signal from the HE plate before and after measuring the test plates. We compared the HE plate signals before and after and if they were not close, we repeated the tests. So our test sequence went from HE plate, to test plates back to HE plate to ensure the test equipment was consistent during the test plates.

First, the four corners of each plate were placed on two graphs, one comparing the amplitude, and the other comparing area. There seems to be no correlation between the visible concentration of the organic material on a specific corner of the plate and the values of the data collected on the oscilloscope. We expected that the corners with the most scintillating material would result in a higher amplitude and area, however, that was not the case. This could be due to bubbles in regions with a higher concentration resulting in unpredictable data. In order to further analyze the borosilicate plates, the corners of each plate with the highest and lowest amplitudes were eliminated, and the remaining two corners were averaged. The average amplitude and area of each plate was then used to compare the different chemicals and concentrations. The images of the plates and their graphs follow:

Plate Comparisons: Single strength Toluene PTP Plate





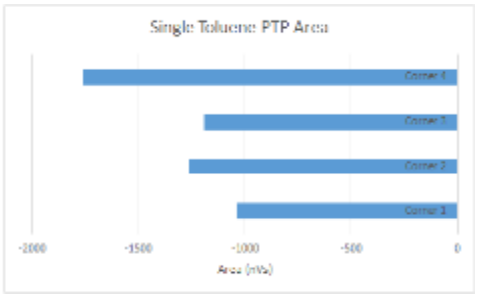
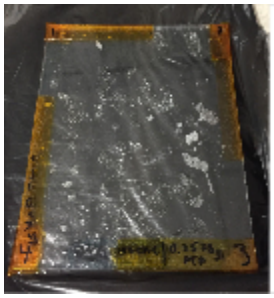
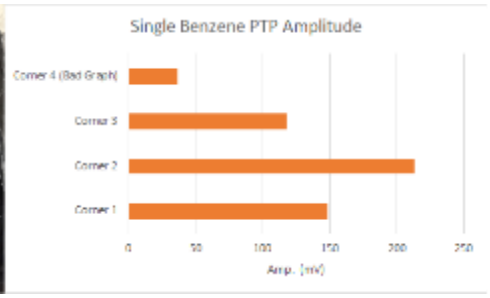


Plate Comparison: Single strength Benzene PTP Plate





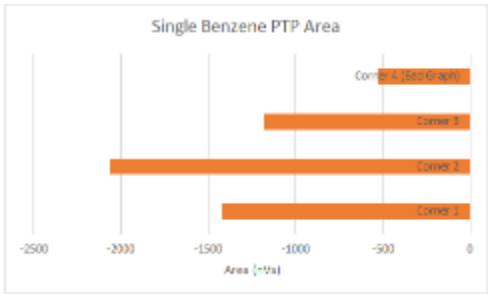
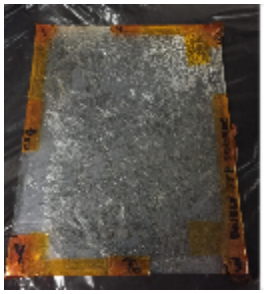
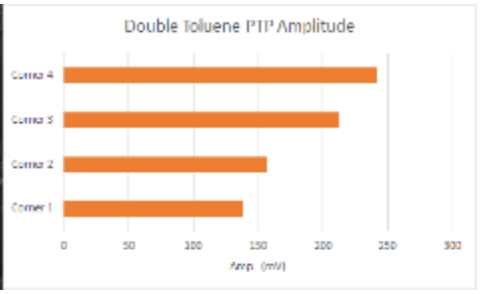


Plate Comparison: Double Strength Toluene PTP Plate





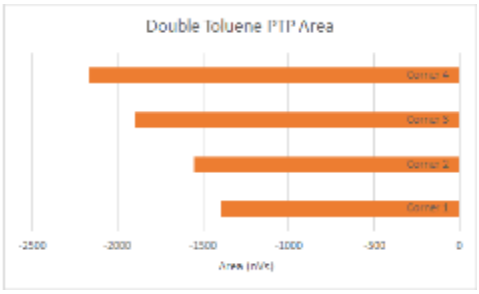
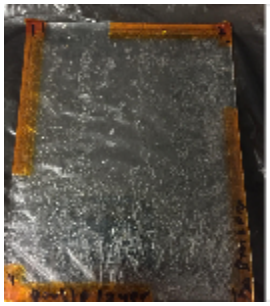
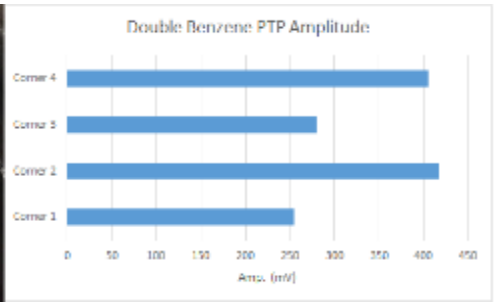


Plate Comparison: Double Strength Benzene PTP Plate





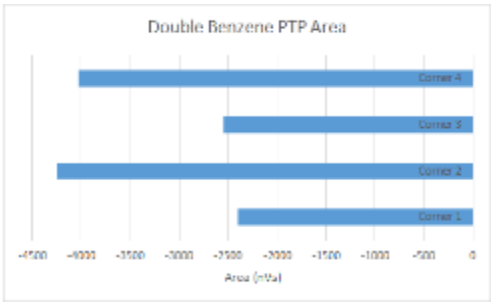
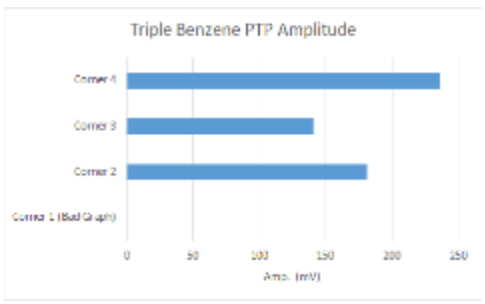


Plate Comparison: Triple Strength Benzene PTP Plate



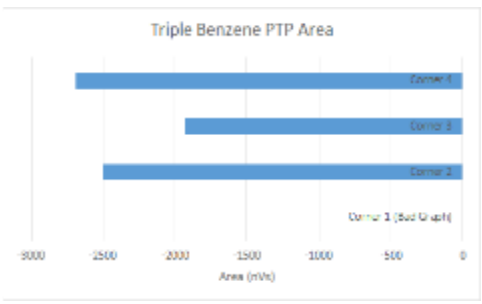
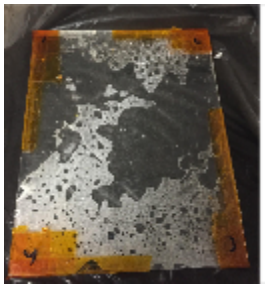
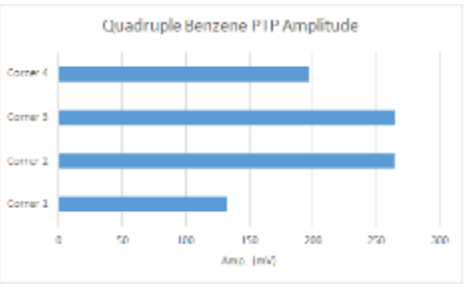
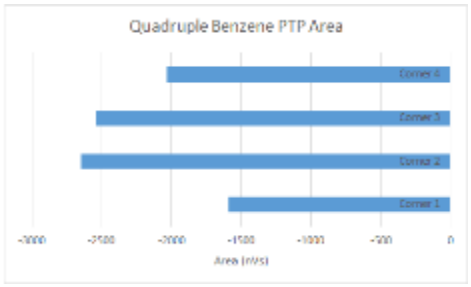


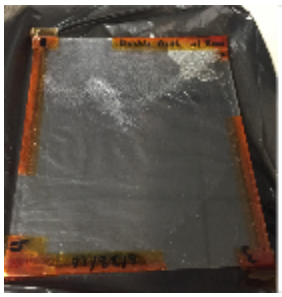
Plate Comparison: Quadruple Strength Benzene PTP Plate

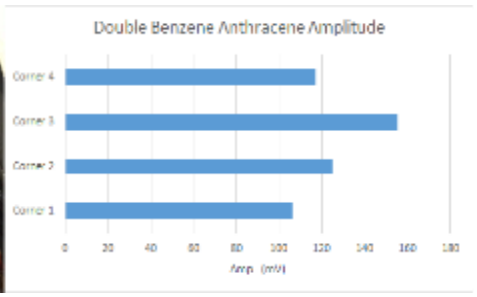






<u>Plate Comparison: Double Strength Benzene Anthracene Plate</u>





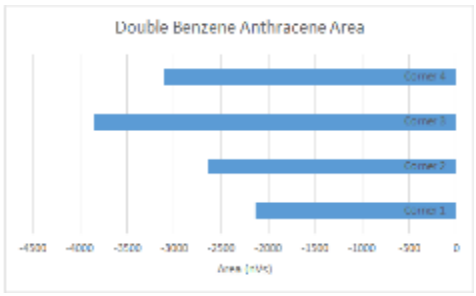
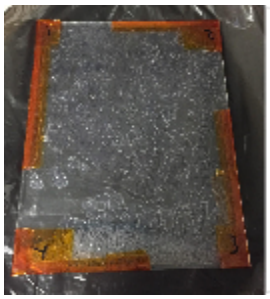
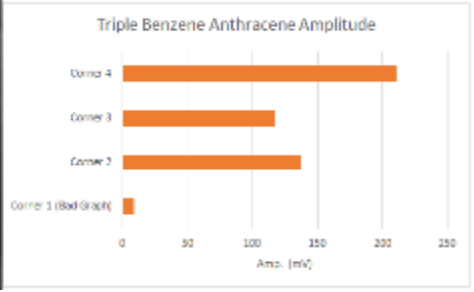


Plate Comparison: Triple Strength Benzene Anthracene Plate





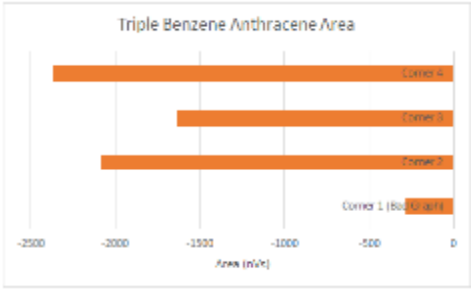
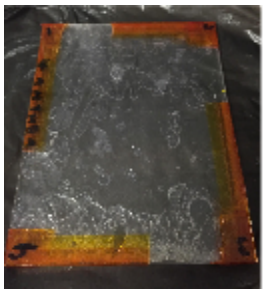
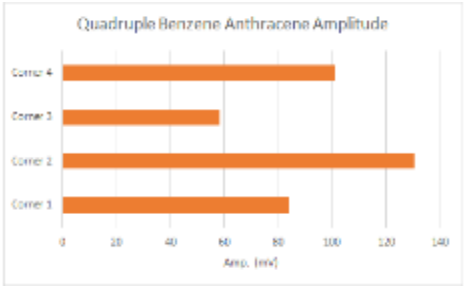
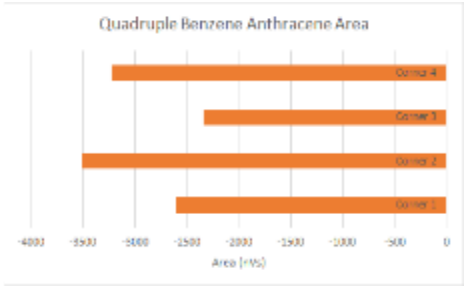


Plate Comparison: Quadruple Strength Benzene Anthracene Plate

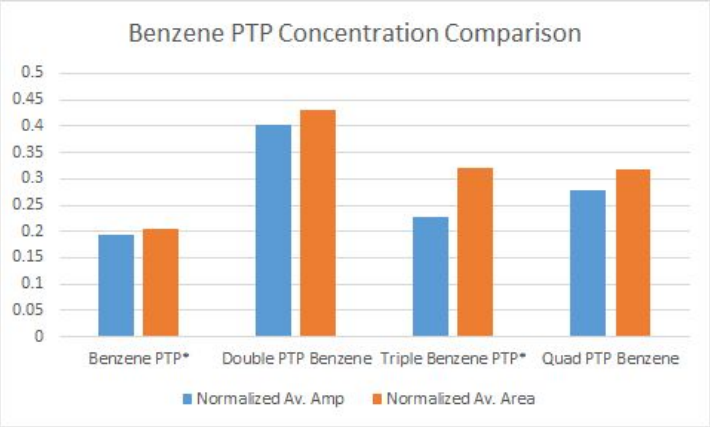






The above charts indicate that Benzene seems to give better results than Toluene for a solvent for PTP. Anthracene has almost double the pulse width as PTP so it is not as desireable as PTP for use in HE. We focused on concentrations of PTP in Benzene and graphed the amplitude and area of the PTP from Benzene evaporation vs concentration. The graph doesn't seem to show a trend for concentration of PTP.

The following graph compares concentrations of PTP in Benzene:



Conclusion:

Based on the above data, using borosilicate plates coated with organic, scintillating materials yielded highly erratic results. For instance, not only is the data for the different corners of a single plate irregular, but there seems to be no correlation between the concentration of the scintillating material on a plate and that plate's performance. While it seems logical that the thicker concentrations would result in more scintillation and higher amplitude, that was not validated by our results. For example, when PTP and benzene are mixed and evaporated on a borosilicate plate, the results show that the amplitude is best when the amount of PTP is doubled, rather than the expected quadrupled. However, doubling the amount of PTP when mixed with toluene does result in a far higher area, but hardly affects the amplitude. Our condensated material did not seem to have consistent crystalline structures and perhaps the clarity of these crystals didn't allow light to pass through them easily thus making the plates inconsistent. In conclusion, evaporating organic materials in this fashion on a borosilicate plates produces erratic results. This is process is too unpredictable to produce effective, scintillating plates for the Compact Muon Solenoid at the Large Hadron Collider at CERN.

Quarknet 2016
Scintillator X

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Purpose: The calorimeters in the Compact Muon Solenoid (CMS) at the Large Hadron Collider (LHC) are adversely affected by the radiation to which they are exposed. Currently, the Electro-magnetic Hadronic (HE) calorimeters use scintillating plastic plates to detect the energy of particles. These plates are found to lose sensitivity by darkening when exposed to large amounts of radiation. Several methods and materials have been tested in an attempt to develop a replacement for the HE plates that can withstand the radiation and also have the required amount of sensitivity. One replacement material, an epoxy with dissolved organic fluorescent materials such as bis-MSB (o-methylstyryl) and PTP (p-terphenyl), designated as scintillator X, is proposed to be a replacement for the current plastic HE plates. The purpose of this experiment was to make and test plates made from the epoxy with the fluorescent materials.

Procedure: The process of creating scintillator X took several trials. The first trial included the use of a glass mold coated in teflon tape. To create the 2cm X 10cm X 1.2cm mold, a glass plate was cut with a glass cutter into a 2cm X 10cm base. Next, two 10 cm X 1.2 cm pieces and two 2 cm X 1.2 cm were cut by the same glass plate. The four pieces and the base were all completely coated with teflon tape. (Figure 1) Next, the four pieces were attached to the base using more teflon tape.The taping process began with taping each piece to the base. Each side was folded up, meeting at the corners. Then, the corners were taped together several times. Upon completion, the effectiveness was tested by pouring water into the mold. No leaks were found, therefore, the mold was determined to be able to hold the solution.

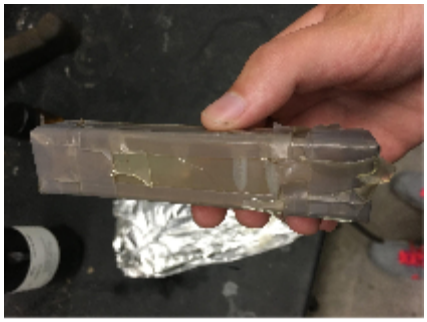


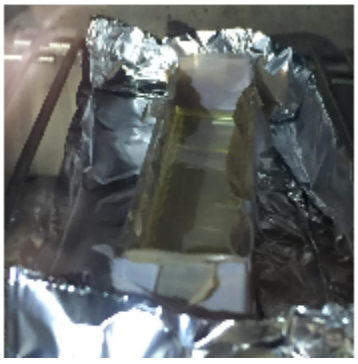
Figure 1: Teflon Tape Covering Glass Mold

The epoxy was delivered in 2 parts; part A was the resin and part B was the hardener. The manufacturer's instructions were to mix 1% part B with 99% part A and heat for 6 hours at 150℃ and then for 2 hours at 200℃. Using the density of Part A, at 1.1 grams/mL, 11 grams was used to create a 10 cm X 2cm X 0.65 cm sized plate. Using this amount, 10.89 grams of Part A and 0.11 grams of Part B would be necessary to fill the mold. Precisely 10.84 grams of Part A

was poured into a beaker and placed under a fume hood. Next, 0.11 grams of PTP (approximately 1% of the mass) was measured onto a small piece of aluminium foil and subsequently dumped into the beaker. The mixture was then stirred but did not dissolve. Also measured out on aluminum foil and placed in the beaker was .011 grams (approximately 0.1% of the mass) of Bis(MSB). The Bis(MSB) seemed to dissolve in the beaker after stirring. In an attempt to completely dissolve the PTP in Part A, the beaker was placed in a water bath, and heated to 60 ℃. At 60 ℃, the PTP did not all dissolve, so the solution was heated further. Even though the attempt to completely dissolve the particles failed, the process continued. Once the temperature of the solution reached about 85 ℃, 0.11 grams (1% of the mass) of Part B was added to the beaker. The mixture was stirred and added to the mold preheated to 150 ℃. In a precautionary measure, the mold was placed in an aluminum foil boat, and then in an oven at 150 ℃.

Figure 2: 1 % in glass mold during vacuum

Figure 3: 1% in glass mold after heating



Once the Scintillator X was in the oven, all the atmosphere was pumped out of the oven using a vacuum pump(Figure 2). This removed any dissolved air from the solution and allowed us to remove any oxygen from the oven. Then, the oven was flooded with nitrogen to return to a normal pressure. The scintillator X was then heated at 150 ℃ for 17 hours (overnight). The temperature was increased to 200 ℃ for two hours (Figure 3, 4, and 5).

Figure 4: 1% in glass mold after heating and out of oven

The same procedure was used to create two 2% scintillator X tiles in an aluminum mold. The aluminum mold itself consists of five pieces of aluminum held together by six screws and six washers. Multiple methods of coating the aluminum in Teflon tape were used, including a single piece of tape being shaped into a boat/open box and being placed inside, the aluminum base being tightly fitted with a piece of tape while the sides were wrapped with a more flexible type of tape, and each aluminum piece was tightly fitted with a sheet of tape before being screwed together as a mold. (Figure 6, 7)This time, 2% of Part A's mass was used to measure out

the PTP and .2% of Part A's mass was used to measure out the Bis(MSB). Part A and Part B were unchanged as the mass remained 11 grams while Parts A and B remained 99% and 1% respectively. For the first trial, Part A and B were heated up to 70 ℃ in the hot water bath. Then, the hardener was added and placed in the oven. The procedure of both the glass and aluminum molds, as well as both 1% and 2% solutions was largely the same. Most often, the heated (though rarely dissolved) solution and hardener were added to a preheated mold and placed on aluminum foil and an aluminum tray. In order to ensure the plates would come out even, a level was placed on a tray to determine the angle of the oven. Once the mold was filled, it was placed in the level oven and the vacuum was switched on for approximately half an hour. Bubbles in the solution had to be carefully watched during this time to prevent the evaporation of the solution or its components. When the vacuum reached its potential (the meter reached 30 ”Hg), it was turned off and the chamber was filled with nitrogen to maintain pressure levels. The oven was heated to about 150 ℃ and kept there for six hours. After six hours, the

Figure 5: 1 % final product

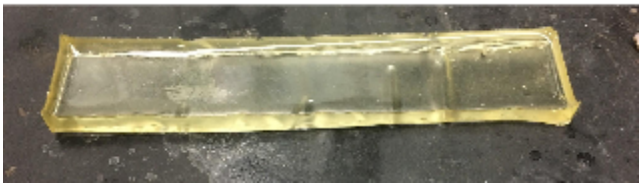


Figure 6: 2 %, First trial after heating in aluminium mold

Figure 7: 2%, second trial, product after unscrewed mold

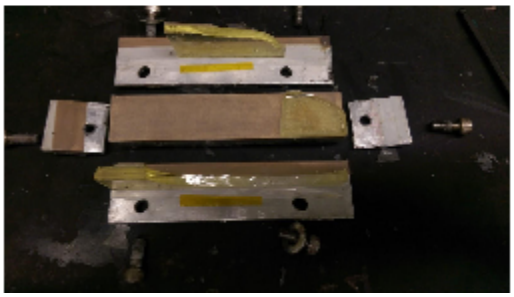


Figure 8: 2 % second trial, Removal of product from mold

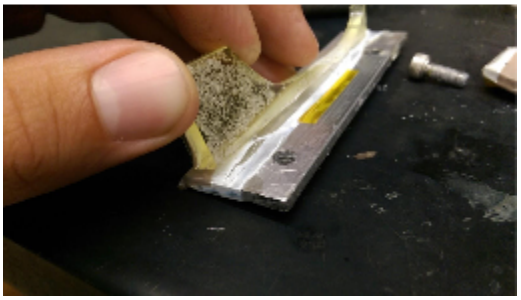


Figure 9: 2%, second trial removal of product from mold

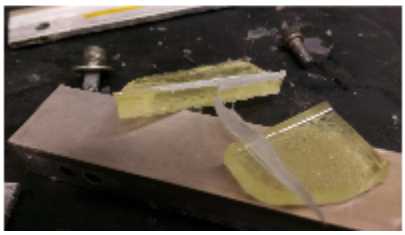
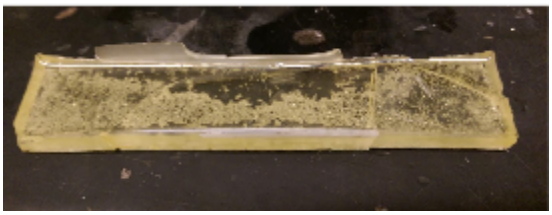


Figure 10: 2% final product



temperature was increased to 200 ℃ for two hours.(Figure 8, 9 and 10)

Testing Process: Different pieces of Scintillator X are tested for their scintillating features using a pulsing laser. Inside a dark box, a laser pulses through fiber optic cables which carry the light from the laser to shine upwards through a plate. A trigger PMT (Photomultiplier tube) is located above the testing area, and another PMT is directly against the side of the tested plate.

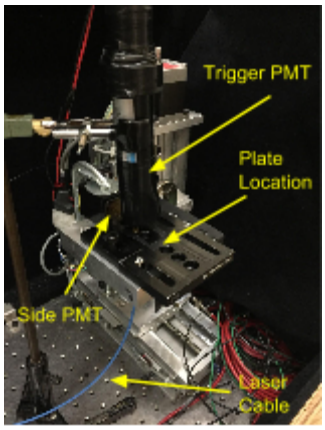


Figure 11: Laser & PMT Setup

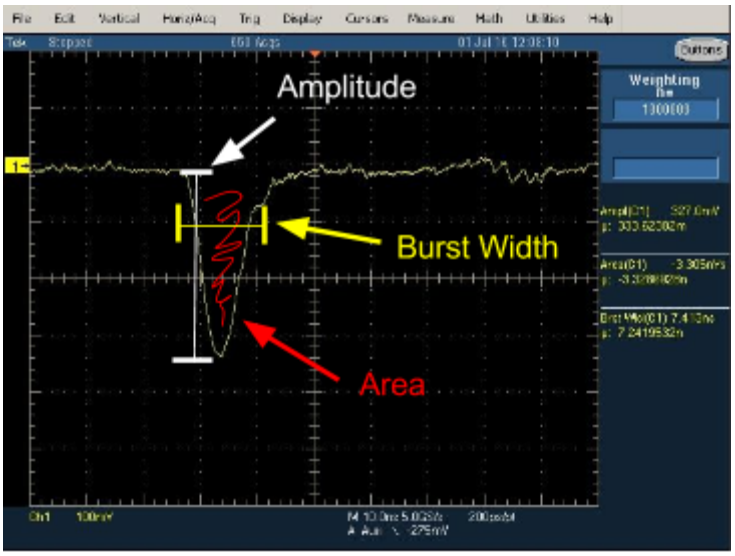


Figure 12: Example of Oscilloscope Data

The trigger PMT receives the laser signal first, and it communicates to the side PMT when to collect data. The side PMT measures how much scintillating light is being transferred through the side of the tested plate. (Figure 11)

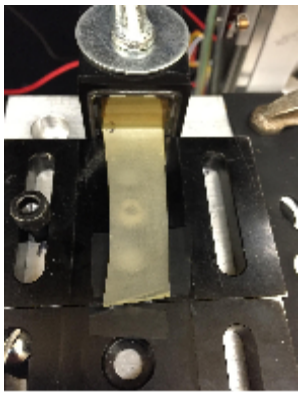


Figure 13: Tape bordering Scintillator X

The data measured by the side PMT is collected and transferred to a Tektronix TDS 5034 Digital Phosphor Oscilloscope, which displays a graph of the scintillated light over time. After the laser has fired for about 30 seconds, the oscilloscope can display a graph with the average values over the duration of time. Amplitude, area, and burst width of the graph are all recorded in a data book, and pictures of each graph are saved within the program on the oscilloscope. (Figure 12) Small strips of electrical tape were used to keep each piece in place, to maintain consistency during testing (Figure 13).

Results: The use of 2% PTP and Bis(MSB) caused no change in the plate or its performance, except to add more undissolved powders to cloud the bottom of the plates. Both the 1% and 2% solutions had little success in the aluminum mold as the plates cracked either in the oven or while being polished before being tested.The only method that resulted in no leaks was the boat/open box method. The downside of that method was that the mold had to be previously

assembled, and no method of removing wrinkles from the tape could be found. These wrinkles expanded in the oven and created ripples and deformities in the plates.

Plates in which a greater total mass was used (the masses of these ranged from 13-16 grams) had largely similar results to those of smaller masses. However, the plates removed from the molds were often smaller than the space in the mold the solution had occupied, even in cases where no leaks could be found. This implies that the plates shrink within the mold, and the successful plates with larger mass were closer to the the plates originally envisioned.

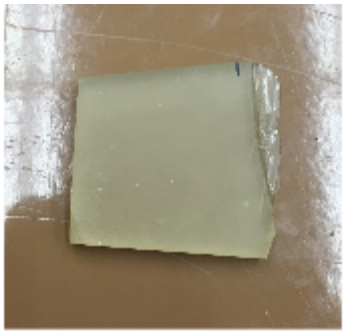
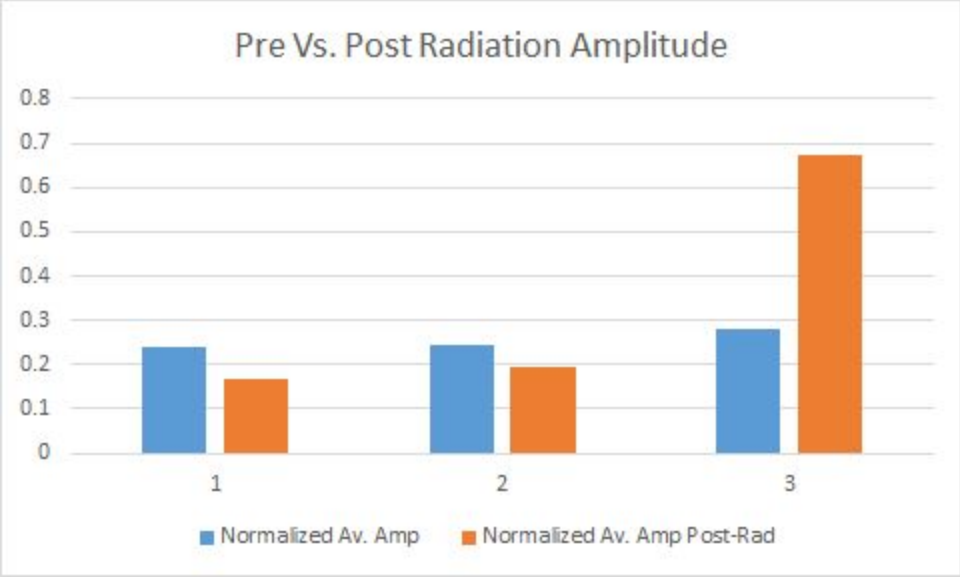


Figure 14: Coe College, Piece 1

Figure 16: Coe College, Piece 3

Figure 15: Coe College, Piece 2

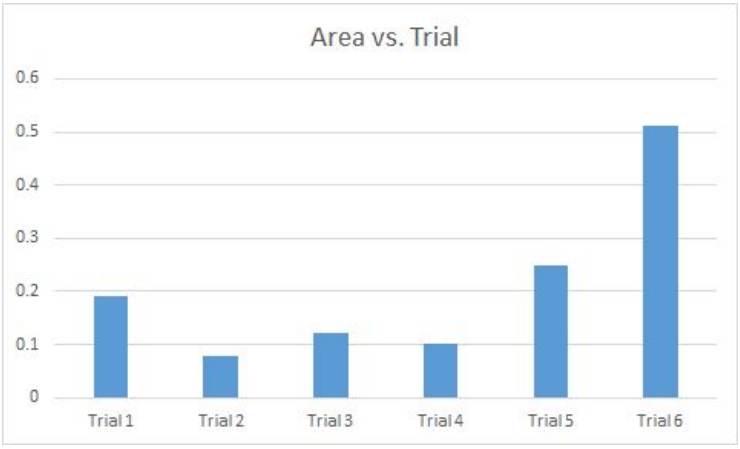
Plates created at Coe College were also tested (Figures 14, 15, 16) These plates were tested ten times and sent away to get gamma radiation. After being exposed to radiation for about three days, the plates were then retested ten times. It is unknown why, but the post-radiation results were better than the preradiation results. The amplitude is shown in graphs to compare the pre and post radiation plates. Before making graphs, the data needed to be normalized. This was done by dividing the data from the tested by the data from the HE plate. The reason for this is that the laser strength seemed to change from day to

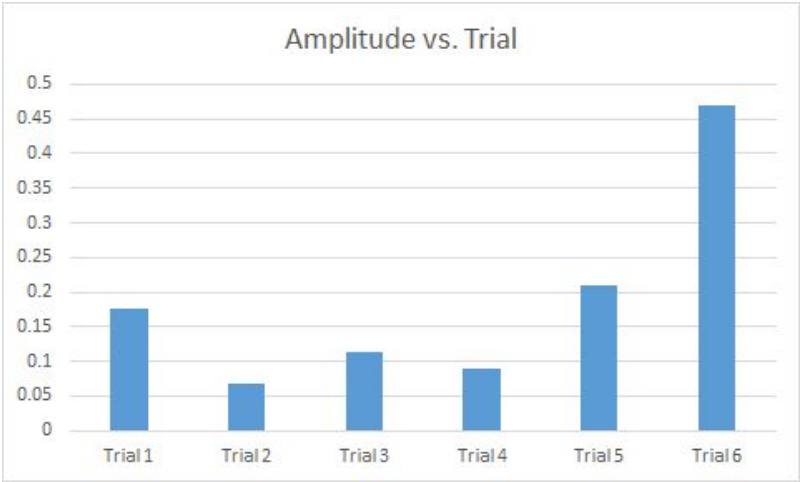


day. The HE plate was tested before and after each trial, so it would give a consistent result to compare our data.

The following graphs compare the multiple trials of creating Scintillator X. After normalizing the data, ie dividing by the control plate to take into account the varying laser strengths, the numbers were averaged and compared seeing there is little correlation between the ability of the scintillator X and their respective mold and percentage.

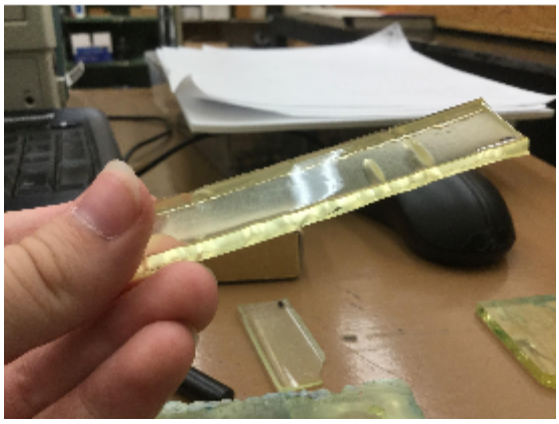
The trials were as follows:
Trial 1: Glass Mold 1% (the only non broken piece)
Trial 2: Glass Mold 2% (a broken piece)
Trial 3: Glass Mold 2% (a wrinkly bottom impeding scintillation)
Trial 4: Aluminum 2% (broken)
Trial 5: Aluminum 1% (extremely thin)
Trial 6: Aluminum 1% (broken in half)



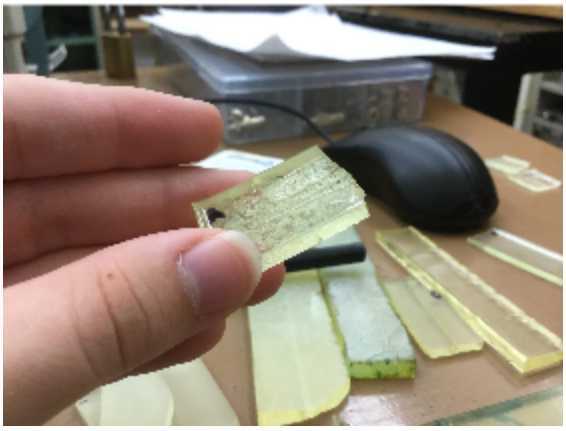


The following are pictures of each trial piece. Trial 6 is in the testing station with the LASER coming from below and the PMT reading on the side.

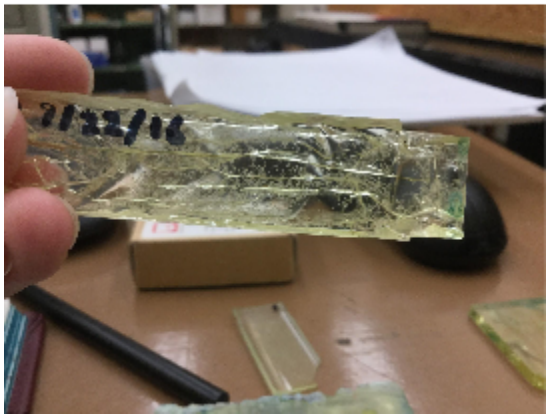
Trial 1



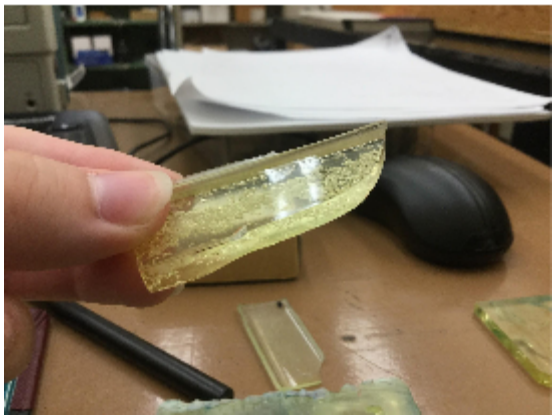
Trial 2



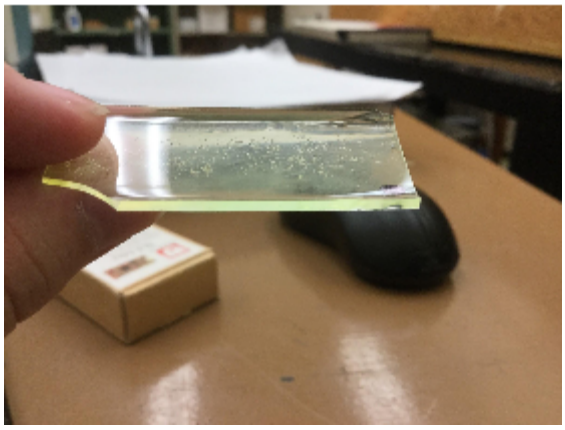
Trial 3



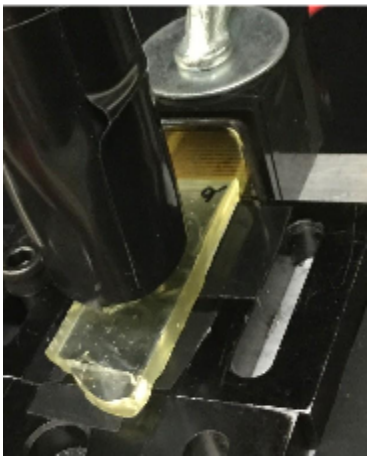
Trial 4



Trial 5



Trial 6



Conclusion: Scintillator X is a difficult compound to manufacture, owing to the rarity of the materials and the precision required to make it. However, there are several conclusions that can be drawn from the data collected. First, many different molds were tested to discover which created the best tiles. It was discovered that while all molds were prone to leaking, the glass molds retained the most material. Cracking of the tiles was another recurring issue. Only one of the five tiles did not crack during the process, the one created in a glass mold using only teflon, with no kapton tape used whatsoever. Aluminium molds performed much worse than glass molds with regard to cracking, leaking, and ease of construction/deconstruction. This could be due to the aluminium's high expansion coefficient. Conversely, the glass mold might have been held steady by the teflon and kapton tape, which is resistant to high temperatures. The tiles themselves also exhibited erratic behavior. For example, even after normalizing the data, the third tile received from Coe College seems to have functioned better after radiation, while the other two performed marginally worse. Out of the 6 created pieces, trial 6 performed significantly better than any of the others. This piece had the least amount of internal cracks, and the mold had the smallest amount of wrinkles. The only downfall for this piece was that it broke in half, reducing the amount of area that the plate had to scintillate. In conclusion, scintillator X shows promise as a reliable replacement for the HE plates in the Compact Muon Solenoid at the Large Hadron Collider at CERN. More investigation is necessary to get a consistent procedure to make the tiles. Perhaps injection molding is necessary to prevent cracks, It should also be tested with particle radiation as opposed to gamma sources.