

Quarknet 2015

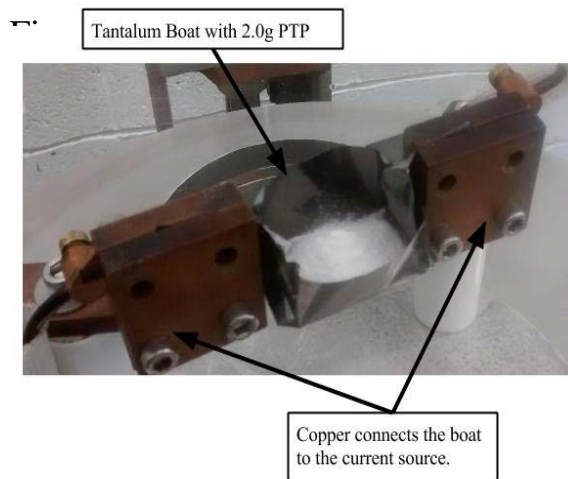
Quartz Plate Preparation

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Purpose: The Compact Muon Solenoid (CMS) calorimeters at the Large Hadron Collider (LHC) at CERN have been exposed to excessive radiation, causing them to quickly suffer enough damage to negatively affect their function. The components need to maintain a high sensitivity for particle detection when they are replaced by materials that can withstand the large amounts of radiation exposure. Materials have been tested to replace the current materials in the detector; however, none have been truly successful, and the search continues. In an attempt to achieve a radiation hard component that is able to sense particles as they pass, organic materials were deposited, and often annealed on Quartz plates.

Procedures: The vapor deposition process is critical in the production of the quartz plates, because it coats the plates with the necessary scintillating material. Vapor deposition involves heating and vaporizing the chosen substance in a vacuum, which allows the particles to freely float around in the vacuum and evenly coat the plates. The first steps taken in the vapor deposition process is to prepare the vacuum pump. Both the mechanical and diffusion pump are turned on. After the diffusion pump is turned on, the valve for the cooling water is opened. Additionally, the power for the ion gauge is turned on to give it ample time to warm up. It is best to complete these steps at least 30 minutes prior to beginning vapor deposition. To prepare for the vapor deposition process itself, the vacuum chamber was cleaned using acetone to remove any contaminants or materials that may outgas. Items cleaned include the bottom of the chamber, the metal stands, the platform in the middle of the chamber, the stand on which the plates were placed, and the glass cylinder which the stand sits on. Additionally, the glass bell for the vacuum chamber was cleaned to make sure nothing would obstruct vision of the inside.

Figure 1



After cleaning, the amount of material to be used for vapor deposition, 1,4-diphenylbenzene (PTP), or Anthracene, was measured out on an electronic balance. The material was taken out of the electronic balance and then placed into the boat¹, (see Figure 1) inside the vacuum chamber where it was held for vapor deposition. Once the chamber was cleaned and the material to be used placed into the boat, the quartz plates were then cleaned with acetone to ensure they had no fingerprints, smudges, or other materials which might upset the coating process. Following this, they were balanced on the sides of the aluminum stand, which is located on the glass cylinder (Figure 2). One edge of each plate must lean against the stand to ensure they would not fall during the process. However, the edge that was touching the stand would not be covered by the material. The blank quartz plate was simply placed on one side of the stand. More importantly, the plate with the groove for the optic fiber had to be placed with the groove facing away from the boat containing the materials to prevent the material from entering the groove.

Figure 2

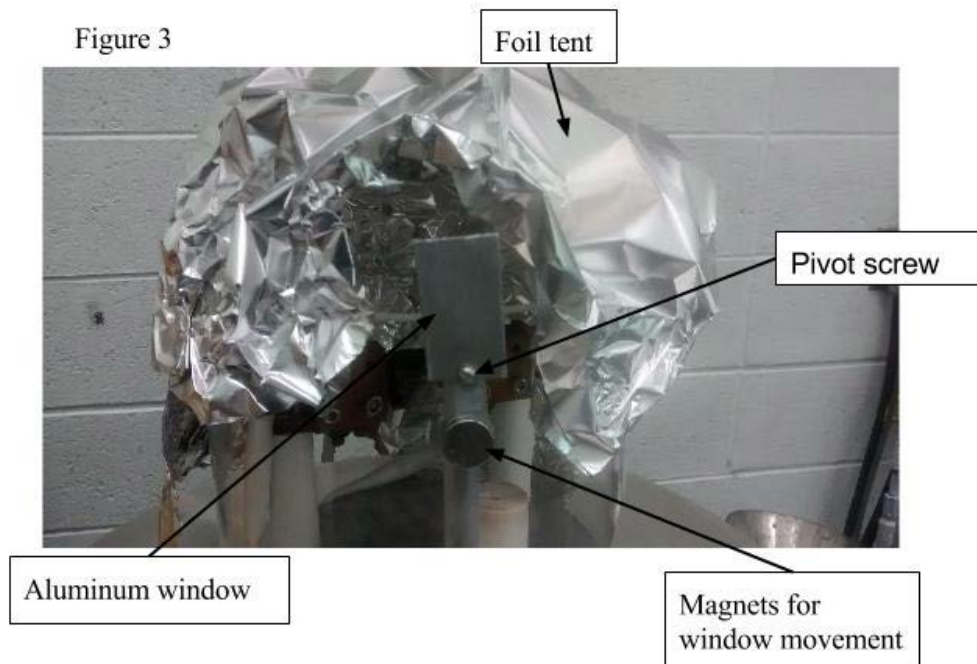


Once the plates were in place, an aluminum foil tent was placed over the stand with the plates, and extended down to the glass cylinder. Each plating material had its own foil tent to prevent cross contamination. The tent serves to prevent the entire vacuum chamber from becoming coated with the vaporized PTP or anthracene. This makes the preparation process for the next run quicker. The aluminum foil tent has a small opening at the front which can be used to see inside. In front of the opening is a stand holding a small piece of metal with a magnet on it. This piece of metal prevents material from escaping the front opening and coating the section of the vacuum chamber bell that is used to check the progress (Figure 3). To move the metal window, a

¹ Boat: an open tantalum container connected to two copper wires on opposite ends. The wires are also connected to a current source. The purpose of the boat is to hold the chemical to be vaporized.

magnet was placed near it on the outside of the chamber, which repelled the magnet on the piece of metal inside, and effectively moved it out of the way to allow vision inside the aluminum foil tent.

When it was ensured that all materials to be used were cleaned, inside, and in place, the bell of the vacuum chamber was closed. The chamber was initially pumped out with a



mechanical pump, which can be used until pressures below 10 millitorr are achieved. Once the mechanical pump achieved a low enough pressure, the roughing valve connecting it to the chamber was closed. Then, the valve connecting the diffusion pump to the chamber was opened and the diffusion pump removed more gas. Once the pressure inside the chamber reached 5×10^{-5} millitorr, liquid nitrogen was poured through an outer pipe and into a chamber located within the vacuum. Cold from the liquid nitrogen caused the vapor inside the vacuum chamber to condense on the plates of the cryo trap. That condensation made the particles heavy enough to fall into the diffusion pump, so they could be removed. The liquid nitrogen was refilled periodically as the diffusion pump ran in order to keep removing particles and create the best environment for coating by keeping the material pure. Although the diffusion pump was effective, it generally took between 30-60 minutes for it to achieve the desired pressure. Occasionally, the 'degas' switch was used to help force any particles out of the ion gauge, used to measure pressure, and into the diffusion pump, where they would be pumped out of the system. This allowed a more

accurate pressure reading, in addition to the removal of the gases. The optimal pressure to begin the vapor deposition process is around 5×10^{-7} torr. However, the diffusion pump was often unable to achieve this pressure and the process began around 2×10^{-6} instead. It can be noted that the difference in pressure did not have any measurable effect on the quality of the plates produced.

Once the desired pressure was met, the vapor deposition process was started. Since PTP and anthracene do not readily vaporize in the vacuum at room temperature, the sample had to be heated to achieve vaporization. The sample was heated by producing a current through the boat which causes the sample to heat up. This was done with the use of a rheostat plugged into the wall and connected to the copper wires attached to the boat inside the vacuum. To measure the current, a clamp ammeter was placed on one of the wires between the rheostat and the boat. The ammeter dial was rotated until the '200A' marker was reached. The rheostat adjusts current through the boat by rotating the dial on the front. The dial was rotated until 20 amps are measured on the ammeter. Using a lower current over a longer period of time caused the sample to bind to the plate loosely, resulting in low quality plates.

After the rheostat was adjusted so that 20 amps of current flow, the time, pressure, amps, and any other notes were recorded in a logbook. While running, the current tends to change slightly, and requires monitoring and adjusting back to 20 amps when needed. During the time that the current was flowing, visual observations of the condition of the plates as well as the amount of sample remaining in the boat were made. Once it looks as though the material was completely gone, the rheostat could be left on for another 5-10 minutes to ensure everything has been vaporized. After the rheostat was shut down and unplugged, valves to the diffusion pump were closed which isolated it from the rest of the chamber. The bell segment of the chamber was ensured to be completely isolated from both pumps, then the air was vented back into the chamber through a valve until the pressure equalized. Once the chamber repressurized, the bell on the chamber was opened. The aluminum foil tent was removed from the chamber, and the plates -provided they were sufficiently coated- were placed in the desiccator².

Unfortunately, the material used to coat the quartz plates also coats everything around it as well. This means that after the plates are removed from the vacuum chamber, it must be thoroughly cleaned to remove the large amounts of material coated onto parts of the chamber. Generally, a razor blade is used to scrape off large amounts of material which are not removed by acetone. The three primary surfaces coated were always the metal stand, the glass cylinder, and the bottom of the chamber. These three surfaces were always cleaned to ensure no material was left in the chamber. Additionally, the rest of the chamber was cleaned, including the inside of the bell, where material often coats the window near the opening in the aluminum foil tent. Then, unless another set of plates is to be coated, everything is placed back in the chamber and

² A desiccator is a container that uses a small substance called a desiccant to remove moisture from the air inside of said container. The desiccants used resemble small rocks and kept the coated plates from being exposed to humidity. It was necessary that the plates not be exposed to humidity, because the chemicals would oxidize.

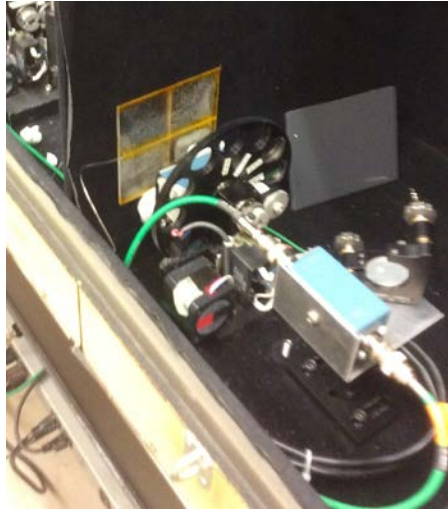
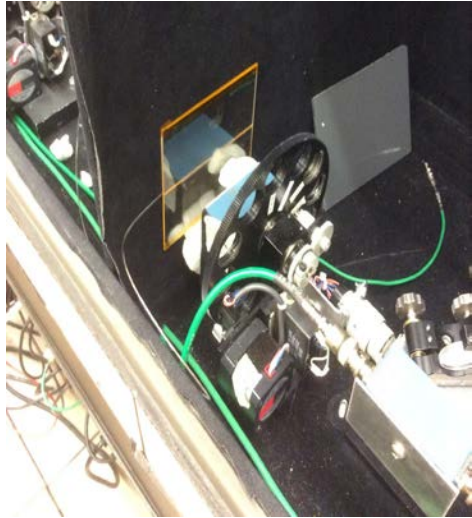
the bell is sealed. The mechanical pump is then allowed to pump out the chamber, to ensure no excess moisture is allowed into the system, which makes it more difficult for the pumps create the vacuum.

The coated plates were then annealed in a vacuum oven filled with N₂ gas. The purpose of this step is to heat the coating to liquefy it, then to cool it, in order to allow the coating to crystallize in a transparent film. The plate was placed into a vacuum oven, with a mechanical pump attached. The pump was turned on, and the pressure was pumped down as low as possible on the pressure gauge. A tank of nitrogen gas was also attached to the oven. Nitrogen gas was added to the chamber to resume atmospheric pressure readings. Then the heating element was turned on, and the heat was increased slowly to the melting point (M.P._{ptp}=414 °F ; M.P._{anthracene}=420 °C) The oven was then turned off, and the plate was allowed to cool slowly before it was removed for testing.

To prepare the plates for testing, fibers were attached. A blue wave-shifting fiber was used for the PTP plates, and a green wave-shifting fiber was used for the anthracene plates. The plates were then tested for a signal using a light tight dark box and a UV LASER for activation. The signal was read out using an oscilloscope. Both the green and blue fibers were tested on the PET and ZnO plates, although in both cases the green fiber produced a larger signal.

Results: The results of the vapor deposition trials were often similar, although changes did occur. Notably, neither the variation of the time nor of the amount of sample (above 2 grams) provided a clear pattern regarding the effectiveness of the process. The time required to plate with both PTP and Anthracene varied from under thirty minutes to over two hours, while the process typically ran with 2.0 to 5.0 grams of the material. Those differentiations seemed to have no effect on each other, or on how well the finished plates were coated. Rather, a change in the current produced the most obvious differences; if the current began low or ever dropped, the plates would not be coated well, if at all.

To test the LASER procedure for plates, two 4" x 4" plates were put in front of the 337 nm LASER. All plates had the "sigma" shaped groove except the 2014 PTP-deposited plate. It had 4 half-size quartz plates that formed a "cross" slot in the plates. This made quadrants separated by grooves. One of the slotted grooves had a blue wave-shifting fiber attached. All the other plates had a "sigma" shaped groove on the outside of the plate with optical coupling paste to link it to the plates to the fibers. All plates were put in the path of the LASER as shown below:

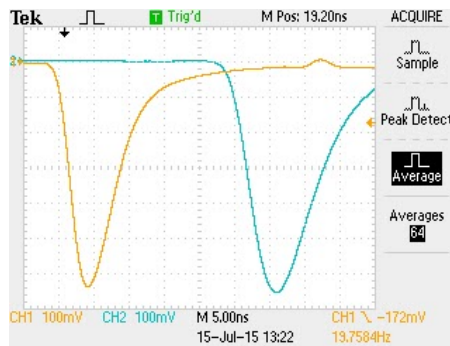
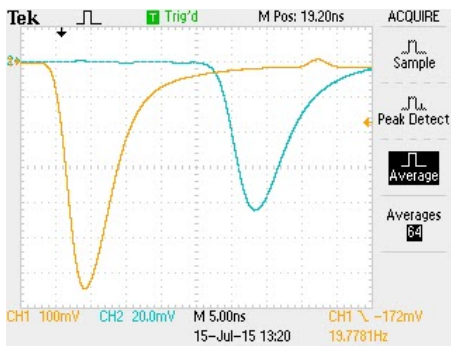


The photo on the left is the plain plate and the one on the right is the PTP annealed plate. The LASER is to the right. The fiber was lead behind the screen and fed into a light guide with a PMT at the end.

The LASER activated a photodiode (the turquoise box) as a

trigger and standard of comparison. Signals were saved on an oscilloscope. The signals were as

follows:



The yellow pulse is the photodiode pulse and the blue pulse is the PMT pulse from the fiber. The photo on the left is the plain plate and the one on the right is the PTP coated plate.

Notice the scales on the scopes.

The area of the pulse is proportional to its light intensity since the area is a function of the charge on the PMT. The plain plate pulse is about 82 mv deep and about 10 ns wide.

Approximating it as a triangle, that gives an area of about 410 (ns-mv). The other pulse is about 650 mv deep and about 20 ns wide to give an area of about 4875 (ns-mv). It seems the PTP enhances the signal by about a factor of almost 12. Upon comparison of the prepared plates, per the graph below, the two strongest signals by far were from the PET plastic and the PTP quartz plates.

July 2015 beam tests.

Equipment

PMT: R7525HA-2 CA 2643 '02.10 Gain @ 1KV=2.62 x 10⁴; @1.2kV = 6.99 x 10⁴

LASER: SRS NL100 337 nm w/Filter

SCOPE: Tektronix TDS 2002B

Full pulse:

HV 1000V

Pin: 768 mv 8.00 ns

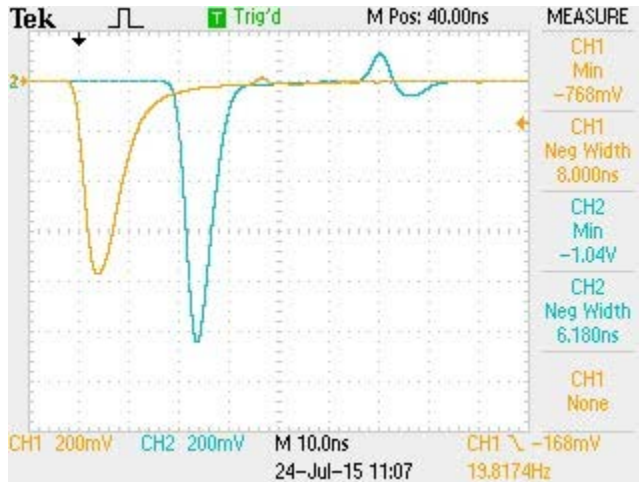
PMT: 1040 mv 6.18 ns

Filters:

Wheel B = 5.0

Wheel A = 0.9

Light Attenuation 1.26×10^{-6}



$$\text{Charge of PMT pulse: } \frac{1}{2} 1040 \cdot 6.18 \div 50 = 64.27$$

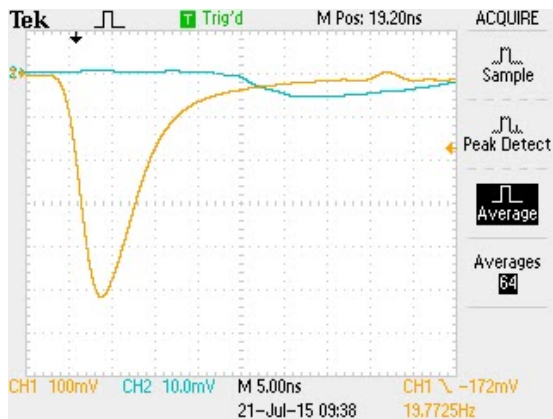
$$\text{Charge of Trigger: } \frac{1}{2} 768 \cdot 8.00 \div 50 = 61.44$$

Blank "sigma" plate: green fiber

HV 1200 V

Pin: 608mv 7.240ns

PMT: 16.0mv 7.380ns



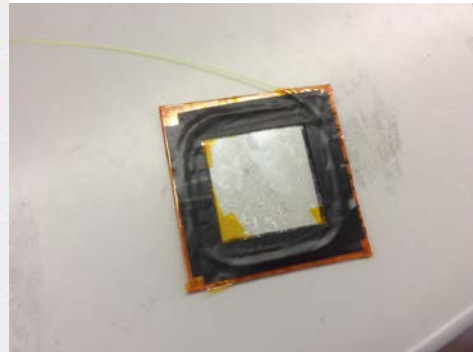
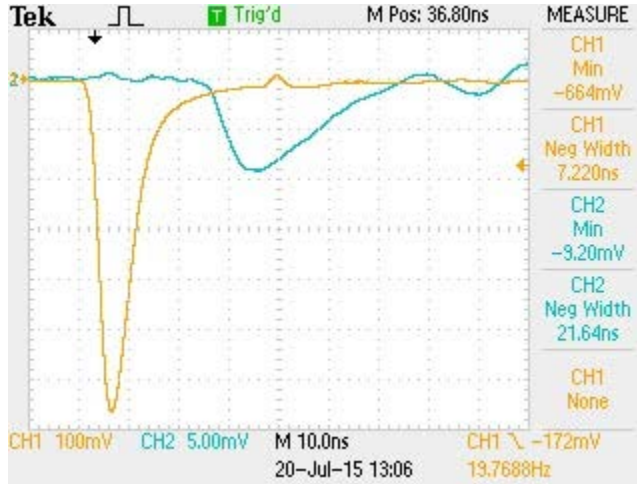
Very minor drop on PMT

Anthracene "Sigma" Plate: green fiber

HV 1200 V

Pin: 664mv 7.22ns

PMT: 9.2mv 21.6ns



$$\text{Charge of PMT pulse: } \frac{1}{2} \cdot 9.20 \cdot 21.64 \div 50 = 1.99$$

$$\text{Charge of Trigger: } \frac{1}{2} \cdot 664 \cdot 7.22 \div 50 = 47.94$$

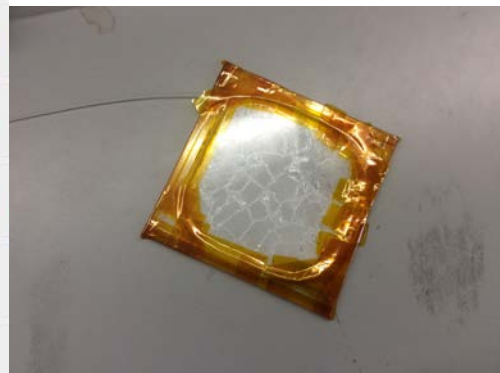
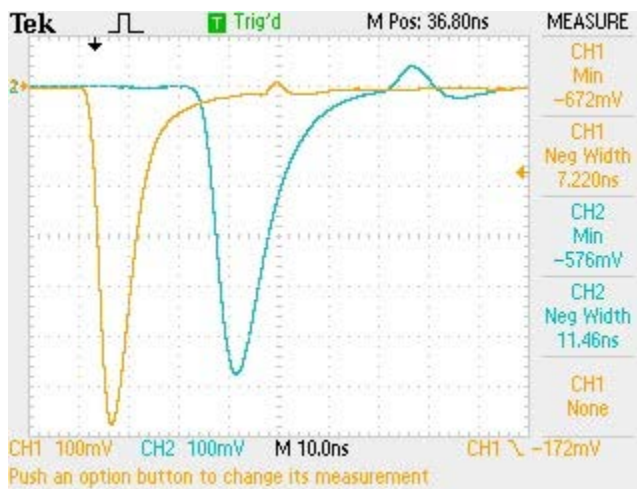
PTP "sigma" plate blue fiber

HV 1200 V

Pin: 608mv 7.280ns

PMT: 300mv 10.44ns

If plate is oriented with wire at top left, the left side (clear) produces larger curve than clouded side



$$\text{Charge of PMT pulse: } \frac{1}{2} \cdot 576 \cdot 11.46 \div 50 = 66.0$$

$$\text{Charge of Trigger: } \frac{1}{2} \cdot 672 \cdot 7.22 \div 50 = 48.5$$

PET "Sigma" plate: green fiber

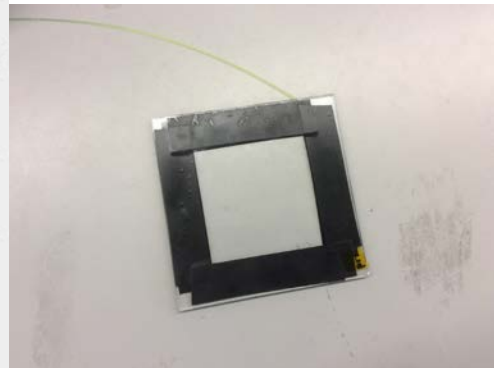
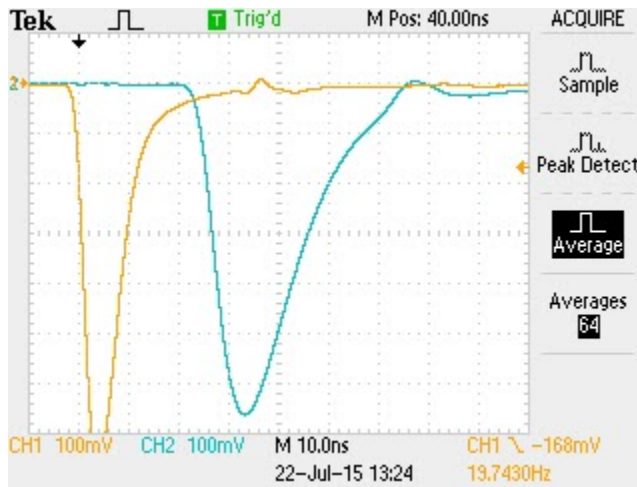
HV 1200V

Pin: 700mv 0.6ns

PMT 550mv 16ns

Filters

Wheel A: 0.6



$$\text{Charge of PMT pulse: } \frac{1}{2} \cdot 660 \cdot 18.0 \div 50 = 118.8$$

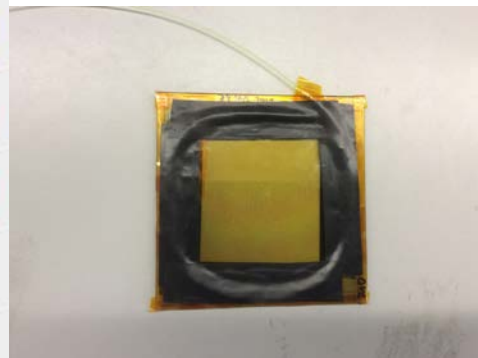
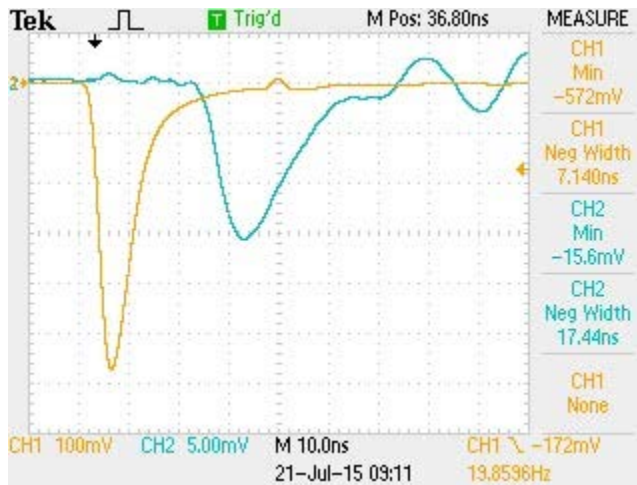
$$\text{Charge of Trigger: } \frac{1}{2} \cdot 750 \cdot 9.9 \div 50 = 74.25$$

ZnO "sigma" plate: green fiber

HV 1200V

Pin: 572mv 7.14ns

PMT: 15.6mv 17.44ns

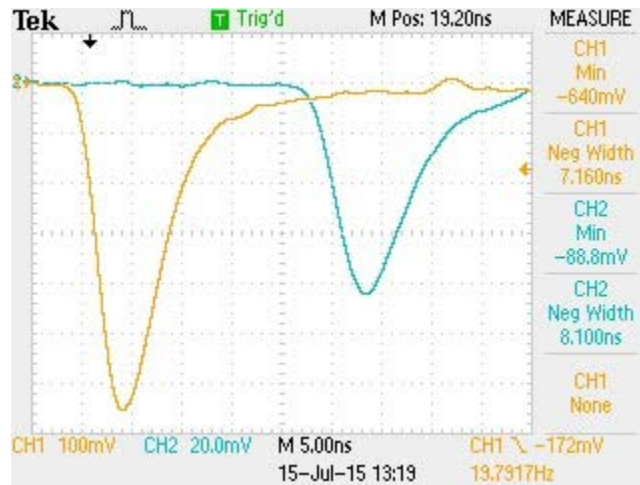


$$\text{Charge of PMT pulse: } \frac{1}{2} \cdot 15.2 \cdot 17.44 \div 50 = 2.6$$

$$\text{Charge of Trigger: } \frac{1}{2} \cdot 572 \cdot 7.14 \div 50 = 40.8$$

PTP Line Plate

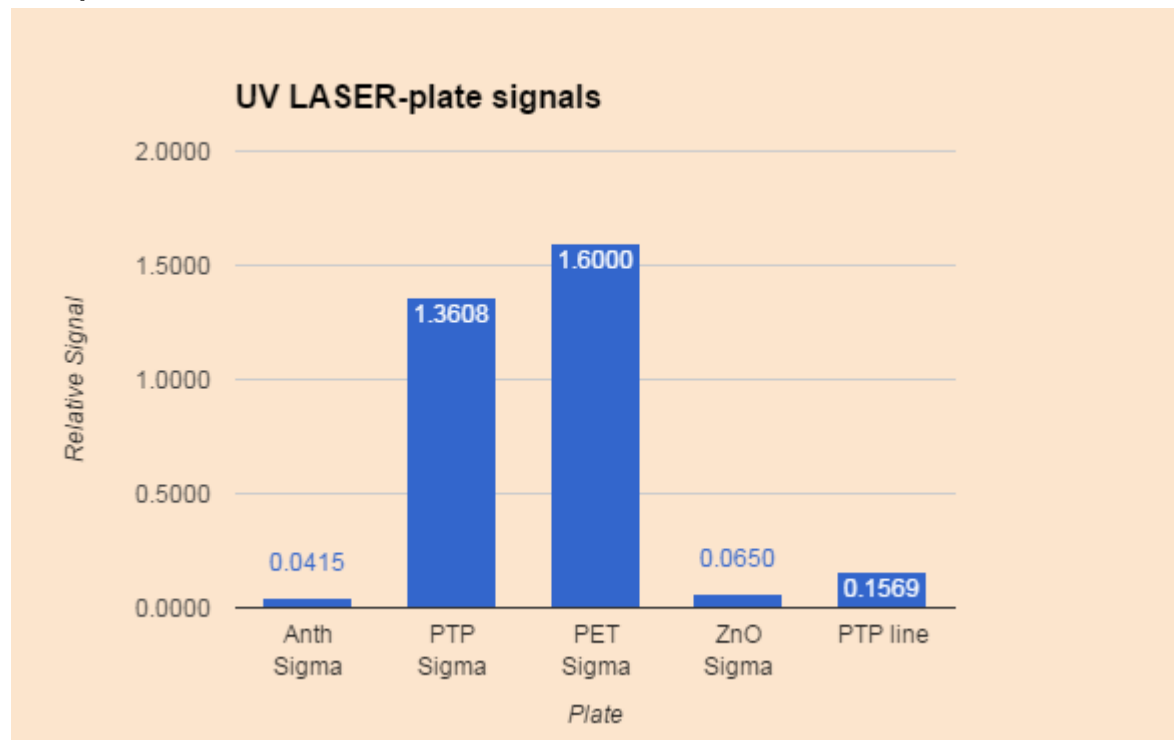
HV 1200



$$\text{Charge of PMT Pulse: } \frac{1}{2} \cdot 88.8 \cdot 8.10 \div 50 = 7.19$$

$$\text{Charge of Trigger: } \frac{1}{2} \cdot 640 \cdot 7.16 \div 50 = 45.82$$

Plate Comparison Chart



Further Study:

The procedure developed in this study for coating and annealing could use some further work and improvement. One idea would be to vary the amount of organic substance used to coat the plate. Another idea would be to study the process of annealing in depth. Variations in the procedures of heating and cooling the plate regarding the timing could be studied to check for any effect on the coating. One specific idea might work with the vacuum coated plates as well: cooling annealed plates even faster by removing them from the oven and placing them in something cold. It could be also be beneficial to research alternative methods, such as crystallization, for coating the plates. Testing these methods might be worthwhile, as a successful crystallization and test would save hours of time spent coating plates with the vacuum.