

NEXT-100 Quartz Tube coating Proposal

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1 Purpose and Summary

This is a proposal to set up a small operation inside a fume hood for the purpose of developing a coating process for coating quartz tubes with a fluorescent paint. The paint will be formulated from scratch; the tubes will be rotated on a spindle while a translating stage moves a paint applicator along the tube.

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2 Introduction

LBNL is collaborating with a number of other institutions, including Texas A&M and Instituto de Física Corpuscular (IFIC), in Spain; the collaboration is called NEXT (Neutrino Experiment with a Xenon TPC). Next-100 is a proposed detector for the purpose of directly observing neutrinoless double beta decay (or setting new exclusion limits).

A critical component of NEXT-100 is a light collection unit which consists of a quartz tube having a scintillating bar/photomultiplier tube inside. NEXT-100 will utilize 72 of these units. On the outside of the tube a fluorescent (wavelength shifting) paint is applied. We (LBNL collaborators) need to develop this paint formulation and tube coating process.

3 Description

This is an proposal to coat a small number (10-20) of quartz tubes, each 42mm dia. x 3mm wall thk. x 14 inch long, with a fluorescent (wavelength shifting) material: p-terphenyl (TPH) mixed with an acrylic lacquer binder, polyethylmethacrylate (PEMA, AKA Paraloid B-72) and diluted in a solvent, toluene, or perhaps xylene, or dichloromethane. We may also try using a mixture of TPH with Teflon AF fluoropolymer solution as a coating on the tubes. Each tube will require mixing several ounces of coating solution, applying it and allowing to dry. We plan to try a drip method, and a spray method, using a small airbrush or spray gun. These tubes are short prototypes for the actual tubes which will be 60 inch long.

Assuming this prototyping effort is successful, we propose to proceed, at some later date, with a further effort to coat 80 full length tubes for the experiment. We may also proceed to develop similar coatings and coating processes for other components or substrates, such as PTFE (Teflon) or polystyrene panels.

4 Who

Derek Shuman will initially be the person performing the formulation and coating operations; he will take Chemical Safety (EHS0348) and Hazardous Waste Generation (EHS0604) courses before proceeding. He is also the designer of the coating equipment and will be overseeing its commissioning. Dave Nygren is the originator of the concept and will be advising along the way. We may also receive assistance from Tom Miller, Azriel Goldschmidt, or some student assistants, as yet unknown, at some point; Derek will provide on the job training as needed, in addition to insuring that any help has taken chemical Safety and Hazardous Waste Generation training. Derek may be replaced, at some point.

5 Where

All operations will take place in rm. 70-208. This room has a fume hood is 90 inches wide x 27 inch tall x 27 inch deep, as shown here 3. The fume hood has integral chemical storage cabinets below the working surface. An eyewash/shower stand will be reinstalled in this room (it was removed, but connections and mounts remain, as shown here 2).

6 When

It is expected that all materials and tooling needed to proceed with the investigation will be available for use by late February. We would like to proceed without delay. It is expected that there will be several phases. First there will be an initial development phase to formulate several coatings and develop the coating process to find the coating process which gives the best appearance. This should take several weeks, at the most. These first coatings will then need to be measured for performance in a separate operation, which may also take several weeks. Once characterized, it is expected that some further variations in coating formulation may be necessary, this would be phase 2, essentially a repeat of phase 1. Assuming positive results, there will be a second waiting period, whereby materials will be purchased in large quantities for a production run. In this production run, the coating machine will be lengthened to accommodate full length tubes of approx. 60 in. length. This machine will still fit easily within the fume hood. We will then proceed to coat approx. 80 tubes. After this is finished, the coating machine will be mothballed and this effort will be complete. We may try coating on other substrates and geometries, such as PTFE flat panels, at some point if successful in the first phases. We may need to redesign the coating equipment for this, but will nevertheless perform coating only inside the fume hood.

7 What

We have common organic solvents and organic chemicals related to common polymers and fluorecents (no acids, bases, or other reactive materials), a coating machine and associated equipment for mixing solutions and applying them. Coating solutions will be mixed and stirred till dissolved and stored in plastic coated glass bottles, using proper PPE and safe chemical practices, inside a fume hood. The coating apparatus will also be operated inside the fume hood.

7.1 Chemicals

Table 1: Chemicals and maximum amount on hand

Chemical	amt.	units
toluene	2	L
dichloromethane	2	L
xylene	2	L
polyethylmethacrylate (PEMA)	500	gm
p-terphenyl (TPH)	100	gm
Teflon AF	100	mL
mixed solutions	1	L

The MSDS's for these materials are attached to the end of this document. The total amount of flammable liquid is under 10L, so a flammable material cabinet is not needed.

7.2 Chemical Formulation Equipment

- pipettes, disposable 10 mL, with syringe-type actuators

- beakers, 200 mL
- bottles, 250 mL, plastic coated brown glass
- scale, 50 gm, ± 0.1 gm
- Kemwipes, inside fume hood
- cotton balls, for tube cleaning
- large sharps container
- container for used Kemwipes, cotton balls, stirrers, etc.
- disposable padded paper for broken glass disposal
- labels for caps and bottles

7.3 Coating Process Machinery

Fig 1 is an isometric view of the tube coater (not all parts shown yet).

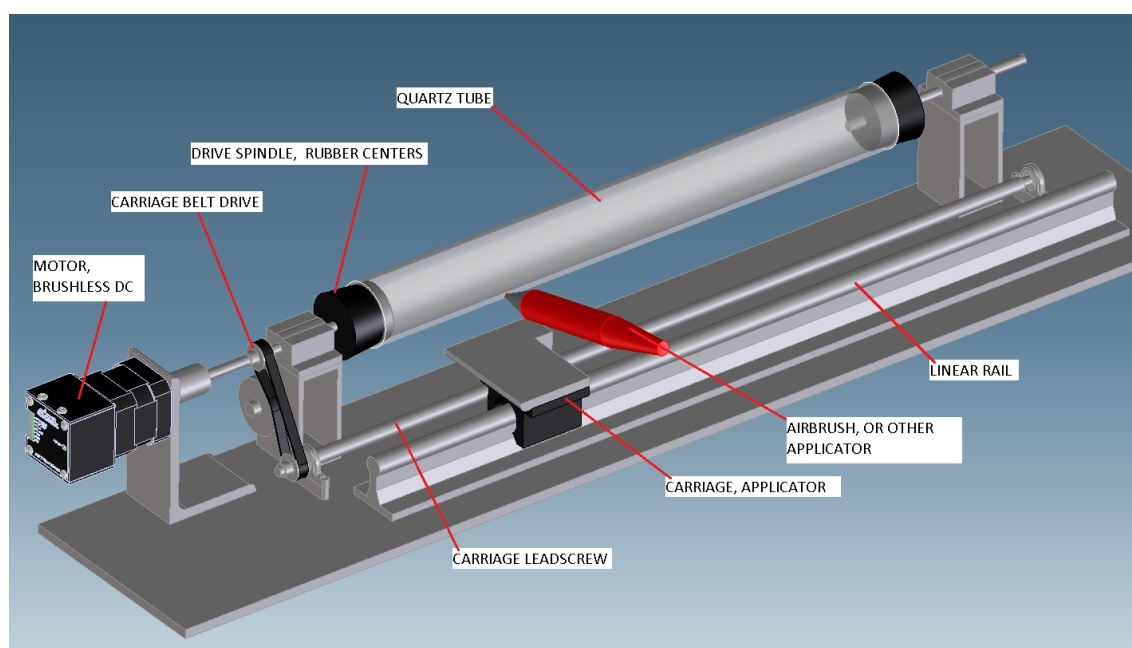


Figure 1: Tube Coater

It consists of the following parts:

- tube holding and rotation spindle, with sliding tailstock spindle
- applicator translation stage, acme screw driven; screw belt driven from spindle sprocket
- airbrush or spray gun
- spindle rotation motor (brushless DC or stepper type only)
- enclosure and conduit for motor
- motor driver
- motor controller
- carriage, for gun travel
- carriage leadscrew
- leadscrew belt drive system
- inductive switch (sealed)

The tube is held in the spindle by friction; the spindle is formed from two rubber corks mounted on stainless steel axles that rotate in bronze bushings mounted to a baseplate. The left spindle is driven by the motor, the right spindle freely spins, and is clamped to the base through slots which allow it to slide left and right for tube mounting and removal by loosening the clamp screws. The machine operates very similar to a typical bench lathe. The spindle will be spun at a low speed, likely from 1-3 revolution per second, just enough to keep the coating from sagging. The translation speed and flow rate of the spray gun are related and one or both should be adjusted to produce the smoothest coating. The spindle is reversible by the motor direction control. The travel carriage for the gun will move the gun from left to right when the tube is rotating CCW as viewed from the left end. When it reaches the end of its travel, an inductive switch be set up will sense the carriage passing by and reverse the spindle motor. The tube will then rotate CW, as viewed from the left end, and the gun will travel back toward the left end. Another inductive switch will then reverse direction once more. The motor controller will be located outside the fume hood. This way the machine cannot be actuated when the operator has their hands in the fume hood.

7.4 Personal Protective Equipment

The following equipment shall be worn during all operations where solutions are handled

1. Safety glasses, with side shields
2. Full face shield (optional, to be worn over safety glasses)
3. Two gloves shall be used. Outer glove (Ansell 92-600) used as a disposable after each use to protect the inner glove (Ansell 2-100). The inner glove provides excellent skin protection and the outer glove provides economy. If spray should hit the outer glove, then the outer glove and the inner glove should be replaced. Each user keep their own pair, stored flat, and in a clean place to protect the glove. This inner glove shall be replaced with wear.
4. Lab Coat

7.5 Safety and other General Lab Equipment to be Present

1. First aid kit
2. Fire extinguisher, located just outside fume hood (no fire extinguishers in fume hood)
3. Waste receptacle in fume hood
4. Eyewash/Shower station
5. Heavy leather gloves for handling broken glass

8 How (Tasks)

Note: The operator shall wear safety glasses with side shields at all times. A face shield may be worn over safety glasses, and is recommended, if feasible. Two gloves shall be used. Outer glove (Ansell 92-600) used as a disposable after each use to protect the inner glove (Ansell 2-100). The inner glove provides excellent skin protection and the outer glove provides economy. If spray should hit the outer glove, then the outer glove and the inner glove should be replaced. Each user keep their own pair, stored flat, and in a clean place to protect the glove. This inner glove shall be replaced with wear.

8.1 Coating Formulation

Coating solutions will be mixed in 50-100 mL batches and stored in plastic coated glass bottles (250 mL). Each bottle will be labeled with the specific mixture, and each cap will also be labeled to correspond with each bottle. All the mixtures are similar and there are no hazards if one mixture is accidentally combined with another; they are all chemically compatible. There are no chemical or exothermic reactions among the listed chemicals, to our knowledge.

8.2 Coating Operation

The goal of this operation is to find the best method of application, which is likely to be spray, although brush or drip methods may be worth trying. For spray application, the process variables are:

- spray gun type, airbrush, HVLP, or other
- air pressure, 25 psi maximum is available in fume hood, and should be sufficient.
- gun settings (fluid jet opening)
- distance from nozzle to tube
- rotation speed
- translation speed or pitch
- solids to solvent ratio

The tube will be mounted in the coating machine by first unclamping the tailstock and sliding it to the right to allow the tube to be inserted onto the rubber cork of the drive spindle. The tailstock will be pushed onto right end of the tube with just enough force to grip the tube and center it; this is very little force. Then the tailstock is clamped by tightening its mounting screws to the baseplate.

The tube will need to be cleaned just prior to coating; this will be done using cotton balls wetted with toluene and held with plastic tongs or tweezers; they may not be held with gloves, as the tube can grab rubber gloves and pull them into the spinning tube. During this operation, the tube must be rotated so that the surface facing the operator is moving upward (CCW when viewing rightward, from the left side), so that if the cotton grabs on the tube, it is pulled over the top of the tube instead of under it, which could possibly pull the tongs into the translation carriage. Used cotton balls must be placed in an open waste pail, inside the fume hood, and not removed until dry of solvent.

In all cases we desire to have a given areal density of TPH deposited on the tube, regardless of the ratio of TPH to PEMA. Thus, a measured volume of coating fluid, as determined to give the correct areal density of TPH, will be pipetted into spray gun cup for each tube, and will be sprayed until the cup is emptied. So therefore, the fluid flow will need to be adjusted so that the cup empties in a whole number of gun passes, if possible, to avoid running out of solution in the middle of a pass. Afterward, a solvent overcoat may be sprayed to level the surface, if desired.

Tubes will be rotated until dry; some testing will be done to find the time it takes. Once dry, the tubes will be removed by unclamping the tailstock and sliding to allow the tube to be removed.

When it is found that a poor coating is being formed, the coating process can be aborted by turning off the spray gun, and then waiting until the tube is rotating CCW (as viewed from left) and cleaning using tongs, cotton balls, and solvent.

In case of a broken tube, the coating process shall be stopped immediately. Once stable, the applicator reservoir should be emptied and the applicator cleaned, only if possible to do safely.

Then the tube should be allowed to fully dry. The operators outer glove should be replaced then the heavy leather gloves put on over the new clean glove before proceeding to clean up the broken tube.

9 Safety Analysis

9.1 Chemical Hazards

The chemicals are all common organic solvents, common polymers and fluorescents. Mixing solutions produces no noticeable exothermic or endothermic reaction. The substances are flammable but not reactive in any other way, to our knowledge.

9.2 Toxic Exposure

Inhalation, skin or eye contact with any of the chemicals involved is to be fully avoided. All coating formulation shall take place inside the (running) fume hood, including the weighing of solids, which are typically powders. All coating operations will be performed in the fume hood. The operator shall wear safety glasses at all times. A face shield may be worn over safety glasses, and is recommended, if feasible. The operator shall wear two gloves for all operations where there is any potential for skin contact with any of the chemicals used. An outer glove (Ansell 92-600) is to be used as a disposable after each use to protect the inner glove (Ansell 2-100). The inner glove provides excellent skin protection and the outer glove provides economy. If spray should hit the outer glove, then the outer glove and the inner glove should be replaced. Each user keep their own pair, stored flat, and in a clean place to protect the glove. This inner glove shall be replaced with wear.

In case of a broken tube, the coating process shall be stopped immediately. Once stable, the applicator reservoir should be emptied and the applicator cleaned, only if possible to do safely. Then the tube should be allowed to fully dry. The operators outer glove should be replaced then the heavy leather gloves put on over the new clean glove before proceeding to clean up the broken tube.

9.3 Flammability hazards

The total amount of flammable liquid is under 10L, so a flammable material cabinet is not needed. There is a chemical storage cabinet under the fume hood which should be used for storing all chemicals and solutions. These should not be left in the fume hood when not being used.

A fire extinguisher shall be present just outside the fume hood during all operations. There should not be any additional fire extinguisher inside the fume hood.

The coating machine will be fully tested prior to using in order to assure that all parts are functioning properly, and no metal to metal contact that could create a spark is occurring.

The motor shall only be of a brushless DC or stepper motor design, as these motors have no brushes where electrical sparking can occur. The motor cables must be contained in a metal conduit; If the conduit cannot be properly connected to the motor housing, the motor itself will be mounted in an enclosure such that a proper conduit connection can be made. Unless the motor has an integral driver/controller, only the motor will be located in the fume hood, all drive and controlling electronics will be outside the fume hood. There will need to be sensors for detecting the carriage end of travel, so as provide a signal for reversing its direction; these will be inductive

proximity sensors which are fully enclosed and do not present an ignition source. Their cabling must also be enclosed in metal conduit.

At installation, or whenever there is a change in equipment or configuration, a safety check will be made to ensure the spray is captured by the hood and directed away from the motor. The use volume of solvent is low enough and the hood flow sufficient for preventing a concentration that would exceed 20% of the LEL. A check will be performed at set-up to confirm. If the process changes to production-mode, or automated, or off-hours, then additional approval will be sought.

9.4 Other Hazards

The tube will be rotating during the coating process, as will the leadscrew and belt drive mechanism. It is not possible to shield the rotating tube, as this will disturb the spray flow. A guard will be made to shield the belt drive mechanism from fingers, however motor power and speed is extremely low, and cannot do serious damage to fingers if caught in the mechanism. There may be frequent gear changes to the belt drive mechanism, so this mechanism must be made accessible

The tube must not be touched with gloves when rotating; therefore all cleaning, which needs to be done while rotating, must be done using only plastic tongs or tweezers and clean cotton balls. No Kemwipes, paper, cloth may be used as it may drag around the rotating tube and pull the operators hand into the tube

Special care and preparation must be taken to deal with a broken quartz tube, a large sharps container, a hammer and some disposable padded wrapping paper or a blanket of some sorts must be immediately available, to be able to safely dispose of a broken tube. Proper tube holders and packaging must be in place to safely store tubes when not being handled. Tubes will be spun on the coating machine until dried, so no provision for handling wet tubes is needed. Should a tube break while coating, the proper procedure is to stop the coating machine and let the fractured tube dry, then put on heavy leather gloves over new clean rubber gloves, wrap it up in padded paper, break it up with the hammer, and dispose of it in the sharps container, paper and all.

10 Location Photos

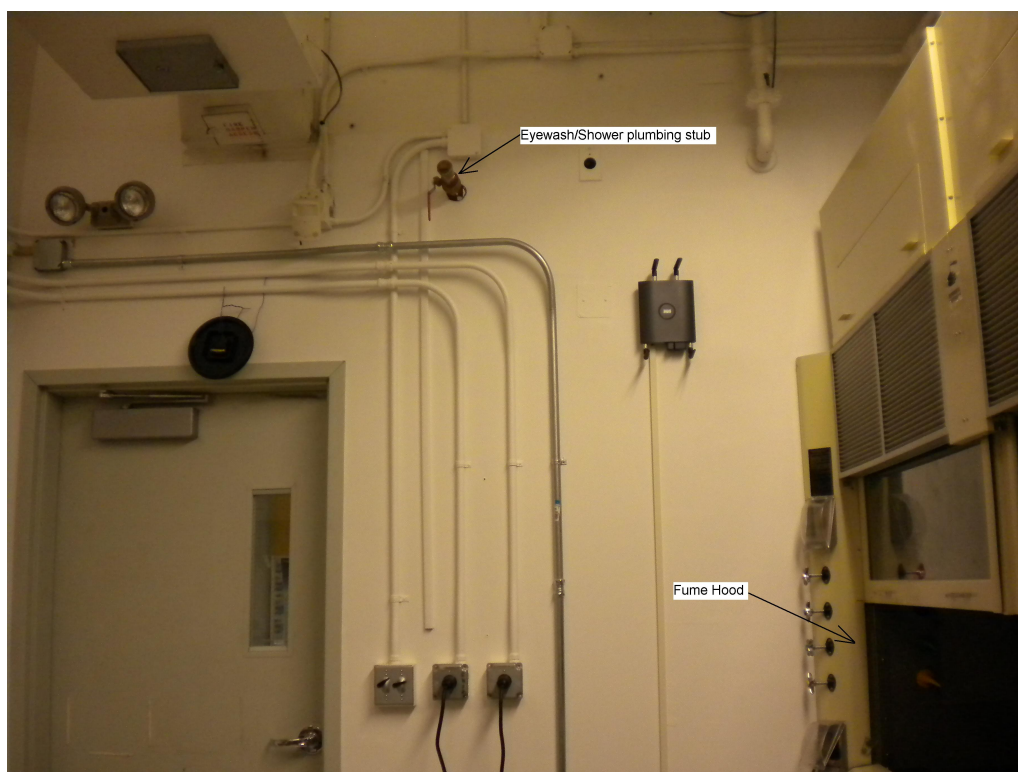


Figure 2: 70-208 fume hood and eyewash/shower plumbing stub

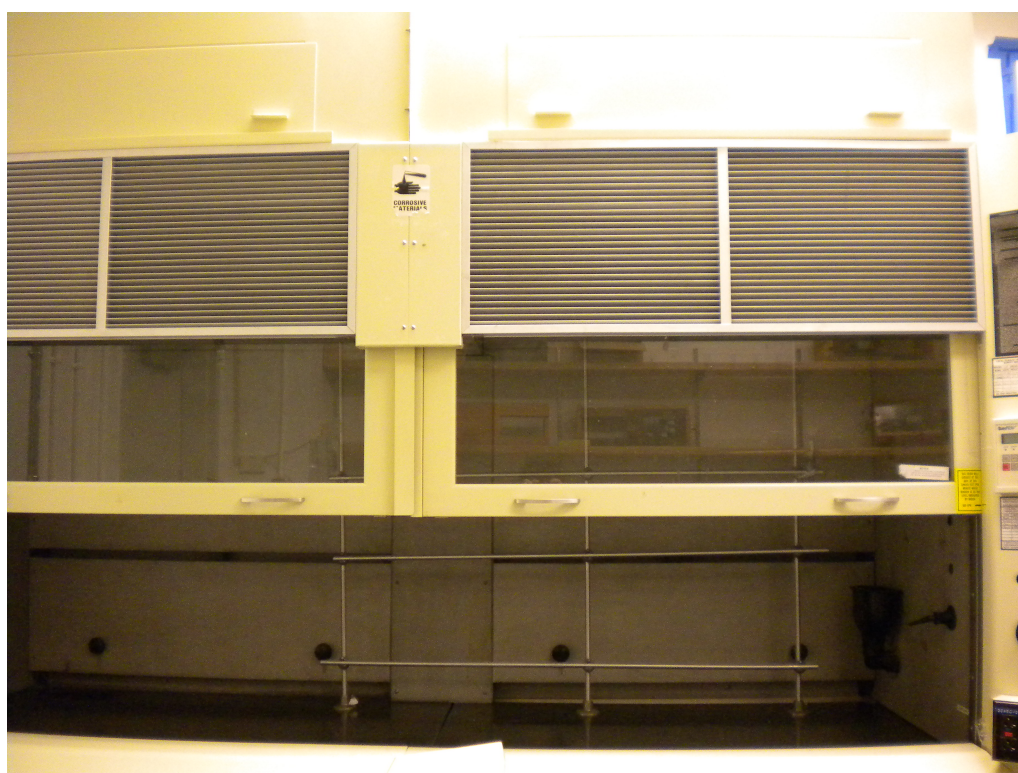


Figure 3: 70-208 fume hood