Coating Glass Cells with OTS

The glass cells we use to polarize xenon or to store polarized xenon need to be coated. This is because without coating wall collisions of the xenon can bring it in proximity to oxygen atoms contained in the glass, which, due to their paramagnetic nature, can depolarize the xenon.

The OTS (octadecyltrichlorosilane) we use consists of a long hydrocarbon chain with a trichlorosilane at one end. In the coating process that group binds to the silicates in the glass, relinquishing the chlorine atoms. The result is a carpet of hydrocarbon chains lining the cell walls. As xenon atoms approach the cell walls they bounce off those hydrocarbon chains, instead of the glass itself. The polarization losses due to wall collisions are thereby reduced dramatically. OTS-coated cells can produce xenon T_1's of over an hour at room temperature.

The OTS coating procedure was explained to me by Matt Rosen <rosenm@umich.edu> of Tim Chupp's group at the University of Michigan. Matt did a lot of literature research into how to reliably coat his pump cells with OTS. Originally he used the same procedure that we were using (described in Eddie Oteiza's notes) and found that Eddie's recipe needed a few modifications. In my description I will contrast the new method with the old one, because it will make clearer what Matt's recipe improves.

Before beginning, the following chemicals should be available:

- Alconox glassware cleaning detergent
- concentrated sulfuric acid
- 30% hydrogen peroxide
- methanol
- distilled water
- nitrogen tank
- octadecyltrichlorosilane [Aldrich part # 10,481-7, tel. (414) 273-3850]
- hexanes
- chloroform
- possibly carbontetrachloride

Solvent cabinet.

With the exception of the OTS, they can all be obtained from the Chemistry Department's stockroom on Oxford St., 495-4011.

Hazardous materials guidelines mandate that the solvents be stored safely. Ours are in the solvent cabinet. Because the hydrogen peroxide would decay into H_2O and O_2 at room tem-

perature too quickly, it is kept in the lab refrigerator.

Clean glassware with Alconox

To clean the glassware after it comes back from the glass blower, use a warm solution of Alconox in distilled water. If the glassware is clean enough this step can be omitted. Eddie's cleaning with a concentrated sodium hydroxide solution has the potential of etching the glass surfaces, thereby increasing the effective surface area of the glass. The negative effect could be a decrease in T_1 of ^{129}Xe or ^{3}He in the cell.

"Piranha" solution:
3 parts hydrogen peroxide
7 parts sulfuric acid

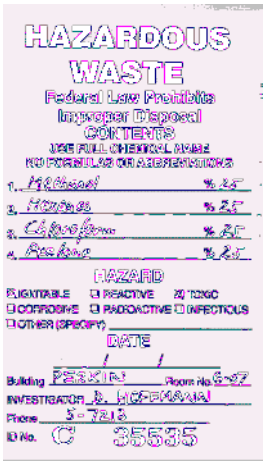
What gets the system really clean is the "piranha" solution. It consists of 3 parts hydrogen peroxide and 7 parts sulfuric acid, by volume. Make about 20% more "piranha" solution than the volume of the glass vessel you want to coat. The extra will compensate for spillage. The sulfuric acid is stored in the acid cabinet. With the piece to be coated inside the big Nalgene™ plastic tray, pour the "piranha" solution into the glassware and let it work for about an hour. Make sure that the bubbles that form can escape. Also, the piece should be reoriented a couple of times during this time in order to allow all inner surfaces to be in contact with the solution for a significant time. Needless to say, this work needs to be done under a fume hood. When handling the acid and solvents we wear acid-proof disposable gloves, along with safety glasses and lab coats. Plan on wearing ragged clothes, too. As I have painfully learned, lab coats don't provide complete protection for your clothes underneath.

Acid cabinet.

3 rinses each with distilled water and methanol

After disposing of the "piranha" solution in properly marked and stored waste bottles, rinse the piece three times with distilled water. Another three rinses with methanol will remove any leftover traces of acid. The methanol also needs to be disposed of properly.

Proper disposal can take place in empty or existing designated waste acid or solvent bottles. If you are using a container as a waste bottle for the first time, make sure that you fill out and affix a "Hazardous Waste" tag. Of course waste acids should never be disposed of in a waste solvent bottle and vice versa. If you empty a solvent bottle, mark it "MT," for "empty." and place it in the appropriate cabinet. Later we can use it as a waste container.



"Hazardous Waste" tag.

Whet cell with distilled water
Dry with N2

To prepare the cell for the coating portion of the procedure it should be whetted again with distilled water. Since OTS is hygroscopic and would rapidly oxidize when coming in contact with water, the water needs to be drained again. Any remaining drops of water should be dried with flowing nitrogen (e.g., from a nitrogen tank). This is a key point of departure from

Eddie's recipe. Drying with nitrogen ensures that the glass won't be bone-dry, as it would be without whetting the cell after the methanol rinse. The residual traces of water are important for the catalysis occurring when the OTS chains bind to the glass.

Dissolve OTS in 1 part chloroform, 4 parts hexanes 0.8 ml of OTS per 1 L of solvents

The coating solution consists of OTS solution dissolved in a base of 1 part chloroform and 4 parts hexanes, by volume. To prevent oxidation of the OTS, we keep it in the lab refrigerator, after opening it for the first time. Ideally, the solution should be two millimolar. With 1 L of the chloroform-hexanes mixture this works out to be 0.8 ml of added OTS. To dispense this small quantity you should use disposable pipettes. This solution is about twenty times weaker than Eddie's but is allowed to be in the cells for longer, instead.

Lab refrigerator.

After opening the OTS for the first time, store it in a refrigerator to slow down the oxidation rate. The OTS will be frozen into a solid after retrieving it from the fridge, but it can be quickly melted by partially submerging the OTS container in a bath of hot tap water.

Soak glass in solution for 5 min

The coating solution should be poured into the glassware, filling it completely, and be allowed to sit for five minutes. When that time has passed, the coating solution should be poured into a properly labeled and stored waste bottle. The glassware should be exposed to air for another five minutes, before the rinse cycle begins.

3 rinses with chloroform

Next, rinse the glassware with chloroform, covering all OTS-coated surfaces. Dispose of the chloroform properly, when pouring it out. Rinse three times. This gets rid of any OTS molecules that are not bound to the glass surface.

Bake cell under vacuum for 24 h at 200° C

Now put the cell on the cell filling station and use the turbo pump to eliminate the remainder of the chloroform. The more has been poured out, the easier it is on the pump. While pumping, heat the cell to 200° C for about 24 hours. This is best done by wrapping the cell with one layer of aluminum foil to spread the heat uniformly. Next wrap a heater tape around the foil-covered cell. For measuring the bake temperature, a thermocouple should be placed under the heater tape, with its tip not touching the tape. Surround the cell and heater tape with two more layers of aluminum foil, for insulation from the outside. Heating the cell helps with the polymerization of the OTS molecules, which form cross-bindings between individual molecular strands. Also, anything that could later on contaminate the alkali metal introduced into the cell should be baked off in this manner. In the absence of alkali vapor, the coating is apparently quite heat resistant.

Eddie's procedure relied on having the polymerization take place under exposure to air and at room temperature.

"Bead-test" cell

For diagnosis, the following day, pour in a drop of distilled water. This drop should roll around on surfaces well-coated with OTS much like mercury. Those spots (hopefully none of them), where the water sticks, were not reached by either the "piranha" or coating solutions.