Equipment and Materials List

- Safety Glasses (20+ pairs for students and instructors)
- Nitrile Gloves (Supreno, 15 pairs of small or medium for students, 5+ pairs of medium or large for instructors and others)
- □ Ten 1" glass squares with conductive coatings (indium tin oxide, ITO) on one side
- Degussa P25 titanium dioxide (TiO₂) (at least 12 g)
- Clear, colorless dishwashing liquid
- Distilled white household vinegar (5% acetic acid, ~1 M)
- □ Tweezers (At least 6 pairs)
- Small metal spatulas (At least 6)
- Scotch "Magic" tape (at least 3 small dispensers)
- Glass stirring rods (at least 6)
- Boxes of small Kim-Wipes (at least 3)
- Paper towels (lots)
- Cotton swabs (one pack of 50-100)
- □ Evedroppers (at least 6)
- □ 10 mL graduated cylinder
- 20 mL graduated cylinder
- Ethanol wash bottles (at least 3)
- Blender
- Deionized water wash bottles (at least 3)
- Oven capable of 450 °C
- □ Tongs for oven
- Three alumina sample boats for oven
- Fire brick where all three sample boats can cool
- Small glass petri dishes (at least 10)
- Graphite pencils (at least 5)
- Blackberries (at least 2 packs)
- □ Thin coffee filters (at least 5)
- Two filter setups (each consisting of a large glass funnel, a small glass funnel, a rubber stopper for the latter, and a lab stand)
- 20 mL glass vials (at least 3)
- Smaller glass vials (if available)
- Binder clips (at least 10)
- Iodide electrolyte solution in squeeze bottles (at least 5)
- Five digital multimeters with clean, new alligator clips for leads
- Large porcelain mortar and pestle
- Scale and weighing paper or weighing cup

WEAR GLOVES AND SAFETY GLASSES AT ALL TIMES, UNLESS SPECIFICALLY TOLD OTHERWISE BY AN INSTRUCTOR OR INSTRUCTED ON THESE PAGES

Specifically, you are making what is called a Graetzel Cell, named after Dr. M. Graetzel, its inventor. Information, solar cell kits, and many good links explaining the technology further can be found at http://www.solideas.com/solrcell/cellkit.html Support for Cornell Center for Materials Research is provided through NSF Grant DMR-0079992

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PUT GLOVES AND GLASSES BACK ON BEFORE DOING ANYTHING

Preparing the TiO₂ Suspension – All Groups Cooperate

Materials Used:

- Degussa P25 titanium dioxide (TiO₂)
- Scale
- Weigh paper or weighing dish
- Metal spatula
- Large porcelain mortar and pestle
- □ 10 mL graduated cylinder
- □ 20 mL graduated cylinder
- □ Small glass vial (20 mL, smaller if available)
- □ Vinegar
- Deionized water wash bottle
- □ Paper towels (not specifically mentioned, but chances are you'll need them)
- **1.** Measure out 12 g of Degussa P25 titanium dioxide on the scale using weigh paper or a weighing dish, and put into a mortar and pestle.
- 2. Measure out 18 mL of vinegar in the 20 mL graduated cylinder.
- 3. Measure out 2 mL of deionized water in the 10 mL graduated cylinder.
- **4.** Transfer the water to a small glass vial, and add two drops of clear, colorless dishwashing detergent to the vial. Stir together carefully, so as to avoid producing foam or bubbles.
- 5. Add about 30 drops of vinegar to the titanium dioxide in the mortar and pestle.
- 6. Mix thoroughly until the mixture is uniform and free of lumps.
- 7. Repeat steps 5 and 6 until all 18 mL of the vinegar is used.
- **8.** Add the previously prepared mixture of detergent and water to the paste, and very gently stir it, being careful not to produce bubbles or foam.
- **9.** Let the suspension sit for 15 minutes.

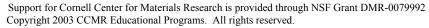




Preparing the Blackberry Juice – All Groups Cooperate

Materials Used:

- One container of fresh blackberries (containing at least 10-12 berries)
- Blender
- □ 10 mL graduated cylinder
- □ 20 mL graduated cylinder
- Two filter setups (each consisting of a large glass funnel, a small glass funnel, a rubber stopper for the latter, and a lab stand)
- Two coffee filters
- □ Two small glass vials (20 mL)
- Deionized water wash bottle
- □ Paper towels (not specifically mentioned, but chances are you'll need them)
- 1. Put one container of fresh blackberries into the blender, counting the number of blackberries.
- 2. For every 5 blackberries used, add 2 mL of deionized water to the blender, using the 10 mL or 20 mL graduated cylinder (make sure it is clean first).
- **3.** Cover the blender and run it on the lowest setting (1) until the mixture is mostly liquid; then increase the speed to a setting of 3-4, and run for another minute
- **4.** Ensure that both filter setups are ready (Cris can help with this) and that a fresh coffee filter (in the large funnel) and a clean 20 mL vial (below the small funnel) are in place in each.
- 5. Pour the liquid from the blender into the large funnel at the top of each filter setup, dividing it equally between the two; if some liquid is left over, wait and add it later, once the liquid level in the filters drops
- 6. We will now wait, with the instructors occasionally agitating the filters by bunching up the edges to form what looks like a little back and gently shaking them up and down against the large funnel, in order to speed filtration. You should be preparing your titanium dioxide coated slides during this time; you will use the filtered blackberry juice later in the day.



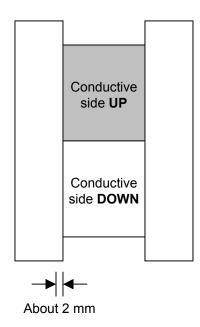




Preparing the TiO₂ Films – Groups Act Individually

Materials Used:

- Two 1" square glass slides with special conductive coatings on one side
- Kim-Wipes (these are very small white paper towels; they look like tissues, and can be found in small green and white boxes around the lab)
- Ethanol wash bottle
- One multimeter set up to measure electrical resistance
- Scotch tape
- One glass stirring rod
- Paper towels
- Metal spatula
- Eyedropper
- Titanium dioxide suspension (made earlier)
- Glass stir rod
- Petri dish
- Oven
- Tongs for oven
- Fire brick for oven
- 1. Clean both 1" square glass slides gently with ethanol and Kim Wipes.
- 2. Identify which side is conductive by using a multimeter (ask an instructor for help here); the resistance of the conductive side will be 10-30 ohms, while the non-conductive side will be too high to measure.
- 3. Clean off an area of the lab bench, and place the square glass slides on the lab bench, orienting one glass slide with its conductive side up, and another immediately below it, with its conductive side down. Carefully keeping the slides touching, tape them to the lab bench using Scotch tape, as shown, leaving most of the surface of the slides exposed and only covering a thin strip (as shown here, about 2 mm wide) on either side:

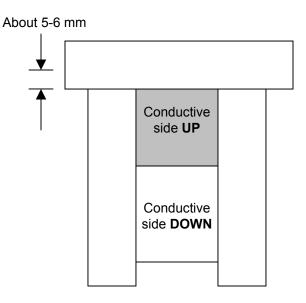


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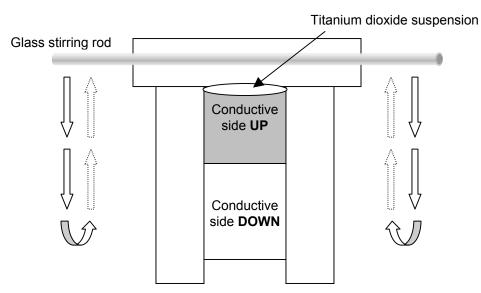
4

Place a third piece of tape over the top of the conductive slide, as shown, covering a larger area (about 5-6 mm wide):



4. IMPORTANT – THIS IS THE HARDEST STEP, SO PLEASE READ EVERYTHING HERE BEFORE STARTING.

Keep in mind that our goal in this step is to get a nice, smooth titanium dioxide film on the conducting slide; any film on the non-conducting slide does not matter. Using a metal spatula or an eyedropper (depending on how liquid the suspension is), take a small amount of the titanium dioxide suspension you made earlier and spread it in a thin line just below the last piece of tape, on the conductive slide. **Immediately** take a glass stir rod, held horizontal as shown, and in contact with the tape, and slide it (*don't* roll it) in order to spread the suspension smoothly, first moving downwards, then reversing direction. Do this 2-3 times or until the film on the conducting slide is smooth. Add more of the suspension if necessary, but do it quickly, as it will dry rapidly. If something goes wrong, you can carefully wipe the slides off and try again.

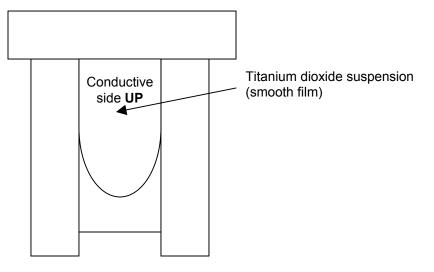


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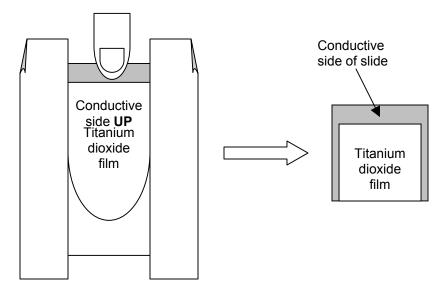




5. When you are successful, you should have something like what is shown below.



Carefully remove the top piece of tape first, and then place your finger (you should be wearing gloves, make sure they're clean ones) on the uncoated area where the tape just was (see below), **being sure not to touch the film**. If you are worried that you may accidentally touch the film, ask an instructor for help. While the conductive slide is held down in this way, have someone else very carefully pull the other two pieces of tape off, peeling from the top down. The non-conducting slide will come loose at some point. Take the slide whose conductive side has been coated with the titanium dioxide film and cover it with a glass petri dish. You may leave it on the lab bench for this. Finish removing the tape from the other slide, and gently wash the titanium dioxide suspension off of it and into the sink. Use ethanol and Kim-Wipes to finish cleaning it. Clean the glass rod in the same way.



6. Once you are done cleaning things, the coated glass slide (above, at right) you placed under the petri dish on the lab bench should be dry (make sure it has had at least one minute to dry; longer is fine). Take the dried glass slide and find an instructor to help you put it into an oven at 450 °C. The slides will be taken out of the oven by the instructors after 30 minutes of heating, and allowed to cool.





STOP HERE: DISPOSE OF GLOVES, TAKE OFF GLASSES, WASH HANDS (LUNCH!)





PUT GLOVES AND GLASSES BACK ON BEFORE DOING ANYTHING

<u>Staining the TiO₂ Films with Dye, Carbon Coating the Counter-Electrode, and</u> <u>Constructing the Cell – Groups Act Individually</u>

Materials Used:

- □ Small petri dish
- □ Eyedropper
- □ Filtered blackberry juice
- Titanium dioxide coated 1" square glass slide
- Uncoated 1" square glass slide with special conductive coating on one side
- Multimeter
- Special graphite pencil
- Deionized water wash bottle
- Ethanol wash bottle
- Kim-Wipes
- Paper towels
- Two binder clips
- □ Squeeze bottle of iodide electrolyte solution
- Cotton swabs
- 1. Take a small petri dish (ensure that it is clean) and, using an eyedropper, put a very small amount of blackberry juice into it Just enough to cover the entire bottom of the dish and form a pool a millimeter or two thick.
- 2. Take your completely cooled titanium dioxide coated glass slide and place it into the juice in the dish, with the titanium dioxide coated side face down. Make a note of the time (you will do something further with this in 10 minutes or so).
- **3.** While you are letting your titanium dioxide film soak in blackberry juice, take your other clean glass slide and identify the conductive side with a multimeter (ask an instructor for help); the resistance of the conductive side will be 10-30 ohms, while the non-conductive side will be too high to measure.
- 4. Having identified the conductive side, take the special graphite pencil that has been provided and coat the conductive side with graphite. The most effective technique is to use not just the point but the entire side of the pencil tip (as shown below), and to look at the reflection of the light off of the surface of the slide while doing this, as this will allow you to tell the difference between graphite-coated and uncoated areas. Make sure that the slide is *completely* coated in graphite, including the corners. Once the slide is coated, **be sure not to touch the coating with anything**. Hold it up by its edges only.

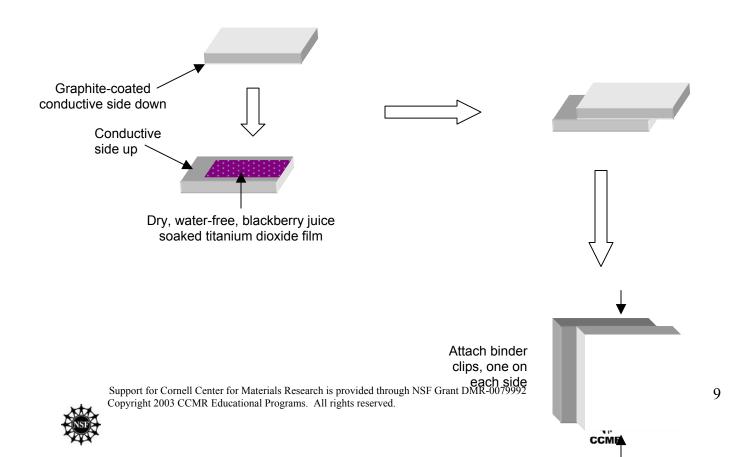
Slide is conductive side up	



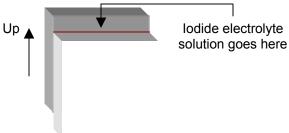
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- 5. By this time, your titanium dioxide coated slide should have soaked in the blackberry juice for at least 10 minutes. If not, wait until it has. Examine the slide from the back as it sits in the petri dish, and ensure that the titanium dioxide film now has a deep purple color throughout. If it does not, wait until it does. Once it does, carefully pick it up, handling it by the edges if you can and ensuring that you **do not touch the titanium dioxide film**. Examine the film from the other side (the side on which it is coated) in order to verify that it is deep purple throughout on that side as well. If not, put it back in the petri dish as before and let is soak longer, first adding more blackberry juice to the petri dish with an eyedropper if necessary.
- 6. The blackberry juiced soaked titanium dioxide film should now appear deep purple throughout when viewed from both sides, with no bright white spots remaining (ask an instructor if you are not sure whether your slide is OK). Holding the slide very carefully so as not to touch the titanium dioxide coating, gently but thoroughly wash the film with deionized water from a wash bottle Do this over the sink. Once you are done, repeat this process with ethanol from a wash bottle, ensuring that all of the water is rinsed away. It is very important at this point that no water remains in your film. Once you are done washing the film, put the slide with the film side face up on a clean paper towel, and use a Kim-Wipe to very gently blot it dry. This slide is now ready for use, and must be used very rapidly so as to avoid significant exposure to the air (which results in oxidation of the dye in the blackberry juice and loss of photosensitivity).
- 7. You must now construct the cell itself. Take the graphite-coated slide and identify the graphite-coated side. Place this side facedown on top of the dry, blackberry juice soaked, titanium dioxide coated side of the other slide, in such a way that the two slides are offset (see the diagram below; the titanium dioxide film should just be covered). Once you have done this, carefully pick up both slides, keeping them in contact and ensuring that they do not slide past each other, and use binder clips to attach the two.



8. Once your cell is clipped together, you will need to add the electrolyte solution. Holding the cell such that the one of the steps is facing upwards towards you, put one to two drops of the iodide electrolyte solution at the point where the two slides meet (see below). You will see that the solution is slowly drawn into the cell by capillary action. In order to ensure that the solution completely saturates the cell, loosen <u>one</u> of the binder clips, then clip it on again, then loosen the other binder clip and clip it on again. Repeat this process until the iodine solution has clearly saturated the entire cell, and then ensure that both clips are once more firmly attached. Use a cotton swab to remove any iodide solution that is present where you put the drops in the first place, or anywhere else along the edge of the cell. It is very important that all of the excess solution is removed.



9. Your solar cell is complete! Now, get a multimeter and use the alligator clip leads to attach your cell to the multimeter, in the positions shown below (make sure neither alligator clip touches both slides). Measure both the voltage and the current output of your cell in light and in darkness, and examine its behavior (ask an instructor to assist in this step).

