# **LIGHT ENERGY**



# SOLAR CELL BASED ON ARTIFICIAL PHOTOSYNTHESIS

15<sup>TH</sup> MAY 2008 - TEST 2

- TASK SHEET -

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#### 1. GENERAL DIRECTIONS

- Write your names and all your personal data in the frame at the right corner of the first page. Do not forget to note the number of your laboratory station.
- You have 4 hours to finish the test. Manage your time wisely.
- There are .....pages of Test and .... pages of Answer Sheet.
- Write answers and calculations within the designated box.
- Additional samples or supplies will be provided with 2p penalty for each item (except distilled water).
- No additional explanations will be provided.
- Volume measurements using a burette should be with precision of ±0.05 mL.
- The use of correction fluid and programmable calculators is prohibited.
- Use only black or blue pen.
- You may go to the restroom with permission.
- After finishing the test, place all sheets (Test and Answer Sheets) in the envelope provided and seal.
- Remain seated until instructed to leave the room

# 2. RULES TO BE FOLLOWED IN LABORATORIES

- Wear safety goggles, protective gloves and lab coat, during the entire duration of your stay in the laboratory.
- For filling a pipette, use the pipette filler bulb provided.
- Follow safety and risk phrases.
- Do not sniff reagents.
- Dispose used chemicals in the plastic bottle labeled "DISPOSABLE".
- Discard used test tubes and broken glasses in the "Waste Basket".
- It is not permitted to eat or drink in the laboratory.
- Do not move from your place and do not borrow any chemicals or instruments from the other competitors. If you need any kind of help do not hesitate to ask the lab assistant.
- Penalty points will be applied for the violation of safety rules or for any damaged glassware or broken instruments.
- Always follow the lab assistant's directions.

# 3. LIST OF CHEMICALS

Reagent	Quantity	Placed in	Labeled
Titanium Dioxide suspension	1mL	Capped bottle	TiO <sub>2</sub> Suspension
Pomegranate juice +10% distilled water	25mL	Amber glass bottle	Pomegranate juice
lodide electrolyte	15mL	Plastic dropper bottle	KI / lodine electrolyte
Sodium thiosulphate	70mL	Amber glass bottle	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> 0.1M
lodine/potassium iodide solution in ethylene glycol	60 mL	Amber glass bottle	KI <sub>3</sub> C <sub>x</sub> (CH <sub>2</sub> OH) <sub>2</sub>
Ethanol	200 mL	Plastic wash bottle	ETHANOL
Isopropanol	100 mL	Plastic wash bottle	ISOPROPANOL
Distilled water	500mL	Plastic wash bottle	Distilled Water
Starch solution	10mL	Dropper bottle	Starch indicator
Hydrochloric acid	20mL	Dropper bottle	HCl 1M
Ammonia solution	20mL	Dropper bottle	NH <sub>3</sub> 0.5%
Aluminium chloride	10g	Plastic vial	AICI <sub>3</sub>

# **4. APPARATUS AND SUPPLIES**

Item	Quantity
Lab coat	1
Safety goggles	1
Protective gloves	1
Digital multimeters	2
Hot air gun	1
Glass cylinder adapted to hot air gun	1
Halogen lamp	1
Scotch (3M) adhesive tape	1
Conductive glass plates	2
Binder clips	4
Distilled water wash bottle	1
Graphite pencil	1
Ethanol wash bottle	1
Isopropanol wash bottle	1
Glass rod	1
100mL measuring cylinder	1
Tong	1
forceps	1
Petri dish	2
Petri dish covers	2
10 mL glass pipette	1
50 mL Burette	1
150 mL Erlenmeyer flask	1

Plastic Pasteur pipettes	2
Pipette filler	1
Cotton sticks (buds)	3
Soft paper tissues	1 box
Ruler	1
Marker	1
White sheet of paper	2

# 5. Safety regulations, S-phrases, R-phrases

Titanium dioxide	R-none S:22-25-36/37
Ethanol	R:11 S:7-16
Isopropanol	R:11 S:7-16
Glycol	R:10-20/21/22 S:53-45
Sodium thiosulphate	R-none S-none
Iodine	R20/21 S:23-25
Starch	R-none S-none
Ethylene glycol	R: 22-26-10-20/21/22 S: 53-45

# Risk phrases (R)

R 10	Flammable.
R 11	Highly flammable.
R 22	Harmful if swallowed
R 26	Very toxic by inhalation

# Combination of risk phrases(R)

**R20/21** Harmful by inhalation and in contact with skin.

**R20/21/22** Harmful by inhalation and in contact with skin and if swallowed.

# Safety phrases (S)

S 7	Keep container tightly closed.
S 16	Keep away from sources of ignition - No smoking.
S 22	Do not breathe dust.
S 23	Do not breathe gas/fumes/vapor/spray (appropriate wording to be specified by
	the manufacturer).
S 25	Avoid contact with eyes.

In case of accident or if you feel unwell, seek medical advice immediately (show the label

where possible).

**S53** Avoid exposure-obtain special instructions before use.

# Combination of safety phrases (S)

**S 36/37** Wear suitable protective clothing, gloves and eye/face protection.

#### **EXPERIMENT 1:**

#### NANOCRYSTALLINE DYE SENSITIZED SOLAR CELL CONSTRUCTION PROCEDURE

- **A. INTRODUCTION**
- B. DEPOSITION OF TiO<sub>2</sub> FILM
- D. TiO<sub>2</sub> SINTERING
- E. TiO<sub>2</sub> STAINING WITH ANTHOCYANIN & CARBON COATING GLASS PLATE
- F. SOLAR CELL ASSEMBLY

#### A. INTRODUCTION

The sun provides our planet with a staggering amount of energy. Green plants convert solar energy through photosynthesis to biomass with a typical yearly average efficiency of less than 0.3%.

Today solar electricity is a steadily growing energy technology and solar cells have found markets in a variety of applications ranging from consumer electronics and small scale distributed power systems to centralized megawatt scale power plants.

Despite the abundance and versatility of solar energy, we use very little of it to directly power human activities. Solar electricity accounts for a minuscule 0.015% of world electricity production, and solar heat for 0.3% of global heating of space and water.

Direct utilization of solar radiation to produce electricity is close to an ideal way of utilizing nature's renewable energy flow. With photovoltaic cells, power can be produced near the end user of the electricity, thus avoiding transmission losses and costs. The solar panels themselves operate without noise, toxic and greenhouse gas emissions and require very little maintenance. Furthermore, the huge theoretical potential and the very high practical potential of solar electricity make it attractive for large-scale utilization.

Despite significant developments over the past decades the high cost of solar cells has remained a limiting factor for the utilization of solar electricity at a large scale. The standard silicon solar cell technology has matured to a stage where costs reductions are mostly foreseen only by the economies of scale. Cost calculations of thin film photovoltaic technologies on the other hand place them more or less in the same level with standard silicon technology. There is therefore a prevailing need for the development of new materials and concepts for photovoltaic conversion, to lower the price of solar cells.

The general trend of nanotechnology has recently emerged also in the field of photovoltaic energy conversion. Development of material engineering in the nanometer scale has generated new photovoltaic materials and systems that could potentially lead to realization of low-cost solar cells in the future. These materials include for example different types of synthetic organic materials and inorganic nanoparticles and nanoparticle systems. The solar cells based on these

materials are called organic solar cells or molecular solar cells. In the process, chemistry has emerged as a new key science alongside with physics in the development of new photovoltaic devises.

The most well known and studied unconventional photovoltaic system is the dye sensitized nanostructured solar cell (DSSC) developed by Professor Grätzel (Lausanne, Switzerland) in 1991. At the moment this unique photoelectrochemical solar cell based on a TiO<sub>2</sub> nanoparticle photoelectrode sensitized with a light-harvesting organic dye, is on the verge of commercialization offering an interesting alternative for the existing silicon based solar cells as well as for the thin film solar cells. At the same time research activity as well as industrial interest around this technology is growing fast.

# Operation principle of the dye sensitized solar cell (DSSC)

Dye sensitized solar cells are novel solar cells that scientists are developing. These cells have a lot of potential because they can be made with low-cost materials and manufactured at a low cost. Dye sensitized cells can work effectively in low lighting conditions, such as cloudy skies, where traditional cells lose some of their energy. Also, traditional models lose energy to heat. Dye sensitized solar cells are less susceptible to losing energy to heat.

A dye sensitized solar cell consists of two conducting glass electrodes in a sandwich arrangement, (see Figure 6). Each layer has a specific function in the cell. The glass electrodes are transparent which allows the light to pass through the cell. The tin dioxide coating is a transparent, conductive layer. The titanium dioxide serves as a holding place for the dye. The dye molecules (artificial or natural) collect light and produce excited electrons, which cause a current in the cell. The iodide electrolyte layer acts as a source for electron replacement. The bottom conductive layer is coated with a graphite carbon laye,r which serves as a catalyst.

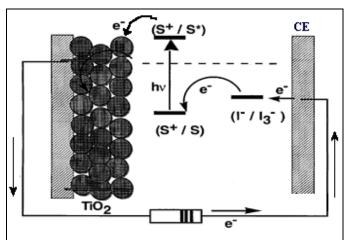


Figure 1

Dye sensitized solar cells produce electricity through electron transfer. Sunlight passes through the conductive glass electrode (see Diagram below). The dye absorbs the photons of light and one of the electrons in the dye goes from the ground state to an excited state. This is referred to as photoexcitation. The excited electron jumps to the titanium dioxide (TiO<sub>2</sub>) layer and

diffuses across the film. The electron then reaches the conductive electrode, travels through the wire, and reaches the counter electrode. The dye molecule, having lost an electron to the titanium dioxide, is now oxidized, which means it has one less electron than before. The dye wants to recover its initial state so it seeks to obtain an electron. It obtains this electron from the iodide electrolyte ( $\overline{I}$ ) and the dye goes back to the ground state. This causes the iodide to become oxidized. When the original lost electron reaches the counter electrode, it gives the electron back to the electrolyte ( $\overline{I}_3$ ) (see Figure 1).

$$S + light \rightarrow S^*$$
  
 $S^* + TiO_2 \rightarrow e^- (TiO_2) + S^+$   
 $e^- (T\iota O_2) + C.E. \rightarrow T\iota O_2 + e^- (CE) + electrical energy$   
 $S^+ + 3/2 I^- \rightarrow S + 1/2 I_3^-$   
 $1/2 I_3^- + e^- (C.E) \rightarrow 3/2 I^- + CE$ 

S: dye molecule

S\*: excited dye

S<sup>+:</sup> oxidized dye

**CE:** counter electrode

# B. DEPOSITION OF TiO<sub>2</sub> FILM

- 1. Obtain and clean two conductive glass plates (2.5cm x 2.5cm) by rinsing them in ethanol and use a digital multimeter, set to ohms, in order to check which side of the glass is conductive; the reading should be between 10 to 30 ohms.
- 2. Orient one glass plate with the conductive side up. This plate will be coated with the TiO<sub>2</sub> suspension. Turn over another glass plate, so that the conductive side is face down. Place it adjacent to the glass slide that is to be coated. When the assembly is completed, one glass plate will be conductive side up and the other with its conductive side down (keep track of the plate that is conductive side up). At this stage, the second piece of glass merely aids in the coating process.
- **3.** Apply two pieces of Scotch (3M) adhesive tape (6-7 cm in length) to the top faces of the glass plates, in order to mask a strip NO MORE than 1 mm wide on the two longer edges. (See Figure 2).

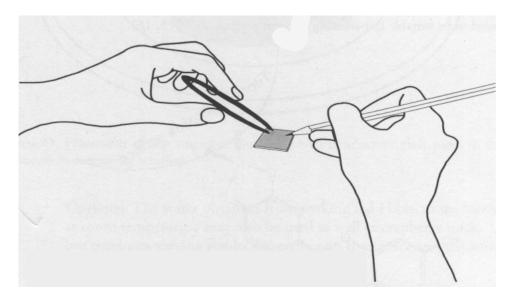


Figure 2: Orientation of conductive glass plates, masking of plates and application of Titanium dioxide to the surface of the conductive plate

**4.** Apply another piece of adhesive tape along the top of the glass to be coated so as to mask a 4 to 5 mm strip. The three pieces of tape should extend from the edge of the glass to the table in order to secure the plates to the table.

This tape controls the thickness of the  $TiO_2$  layer, forming a 40-50  $\mu m$  deep channel for the  $TiO_2$  suspension. The tape also masks a strip of the conductive glass so that an electrical contact can later be made.

- **5.** To coat the glass, a thin line (or three drops) of the TiO<sub>2</sub> suspension is uniformly applied to the edge near the tape of the conductive-side-up glass using a plastic Pasteur pipette.
- 6. Within five seconds after application of the TiO<sub>2</sub> suspension, slide (**DO NOT ROLL**) a clean glass rod (held horizontally) over the plate to spread and distribute the material (see Figure 3). The most successful technique for achieving a uniform film is to use a rapid sweeping motion of the rod towards the bottom of the setup and then back over the film in the opposite direction. Repeat if needed without lifting the rod.

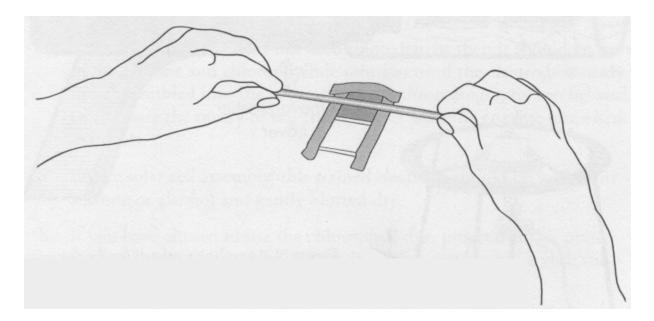


Figure 3: A rapid sweeping motion of the glass rod is used to coat titanium dioxide suspension on the masked conductive glass plate.

7. If the coating looks non-uniform, then the material can be wiped off the plate and the glass rod wiped with a dampened tissue and the deposition procedure repeated. After deposition of the TiO<sub>2</sub> suspension, carefully remove the tape. Place the plate in a Petri dish without touching its face (should be picked up at the edges) and cover it. Allow the TiO<sub>2</sub> film to dry for one minute. Wash and dry the plate that was conductive side down and clean glass rod.

# C. TiO<sub>2</sub> SINTERING

- **1.** Anneal the TiO<sub>2</sub> film on the conductive glass plate using the hot air gun provided. The heating of the film should take place in the hood.
- 2. Transfer carefully using tweezers the conductive glass (Titanium dioxide side up) within the horizontal glass tube (see Figure 4) and set air gun switch to stage 1 (upwards). The air temperature reaches 450 °C and titanium dioxide anneals and sinters by heating for 30 minutes.
- 3. Hint: While waiting for the sintering to be completed, carry out EXPERIMENT 2
- **4.** After annealing is completed, allow the glass plate to slowly cool within the glass cylinder to room temperature. This will take approximately fifteen minutes.

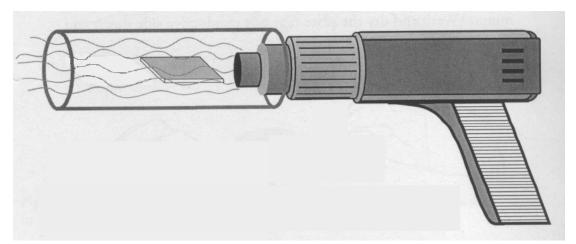


Figure 4: The film is placed inside a glass tube furnace for annealing of the titanium dioxide film on the conductive glass.

**5.** Store the plate using tweezers in a clean Petri dish for later use and cover it.

# D. TiO<sub>2</sub> STAINING WITH ANTHOCYANIN & CARBON COATING GLASS PLATE

1. Transfer the cooled TiO<sub>2</sub>-coated glass plate and place it (face down) into a Petri dish having 20mL anthocyanin (pomegranate juice) solution. Soak the TiO<sub>2</sub>-coated glass plate for 10 minutes in the dye. If any of the white colour of the TiO<sub>2</sub> can be seen upon viewing the stained film from either side of the glass plate, then the film should be placed back in the dye for an additional 5 minutes. Adsorption of anthocyanin to the surface of TiO<sub>2</sub> and complexation to Ti (IV) sites is rapid.

<u>Hint:</u> While soaking the titanium coated plate in the anthocyanin solution carry out experiment 3

Do not remove the glass plate from the pomegranate solution until you are ready to assemble the solar cell in the next section.

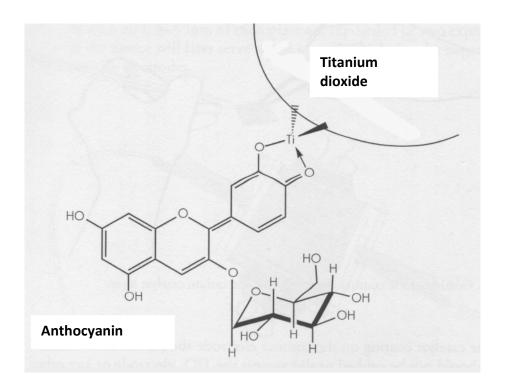


Figure 5: The dye molecule must possess carbonyl ( - C=O ) or hydroxyl ( -OH) groups capable of complexing (chelating) to  $TiO_2$ 

2. While the TiO<sub>2</sub> electrode is being stained in the pomegranate juice, the carbon-coated counter electrode can be made from the other conductive (2.5cm x 2.5cm) glass plate. Clean your second glass plate (the one that is not soaking in pomegranate juice) by rinsing it in ethanol and then drying with a soft tissue—use the same technique as with cleaning a pair of eyeglasses. Once you have cleaned the glass plate, do not touch the face of the plate, as the oils in your hand will contaminate the surface.

- Use a digital multimeter, set to ohms, in order to check which side of the glass is conductive; the reading should be between 10 to 30 ohms.
- **3.** Hold the conductive glass plate by the edges or with tweezers. Using a graphite (carbon) pencil provided, apply a carbon uniform film to the entire conductive side of the plate. Be careful not to miss any spots. This thin carbon layer serves as a catalyst for the electron transfer resulting in the triiodide to iodide regeneration reaction. No tape is required for this electrode, and thus the whole surface is coated with the catalyst (see Figure 6).

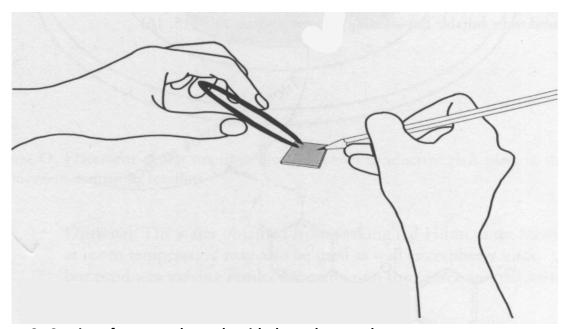


Figure 6: Coating of counter electrode with the carbon catalyst

**4.** The catalyst coating on the counter electrode should not be touched. It should not be rubbed or slid against the  $TiO_2$  electrode or any other surface. The counter electrode should be picked up at the edges and carefully placed where it is desired.

#### **E. SOLAR CELL ASSEMBLY**

- **1.** Using tweezers remove glass plate (which is stained dark purple) from the pomegranate juice and rinse it with distilled water, and then with ethanol.
- 2. Place the plate on a paper tissue with the film side up. Gently press the TiO<sub>2</sub> with another dry tissue (repeat drying procedure using ethanol).
- 3. It is important to dry the stained glass plate and to remove the water from within the porous TiO<sub>2</sub> film before the iodide electrolyte solution is applied to the film. One way to ensure the TiO<sub>2</sub> film is dry is to repeat above procedure with isopropanol.
- **4.** Place the dried and stained electrode on a flat surface so that the TiO<sub>2</sub> film is face up; the carbon-coated counter electrode is placed on top of the TiO<sub>2</sub> film such that the conductive side of the counter electrode faces the TiO<sub>2</sub> film. To avoid excessive exposure of the stained film to air, this step should be completed within 1 minute.
- **5.** Gently lift the counter electrode and offset the two plates so that all of the TiO<sub>2</sub> is covered by the carbon-coated counter electrode, and the uncoated 4-5 mm strip of each glass plate is exposed (see figure 7).

At each end, 4-5 mm of each plate is exposed. The two exposed sides of the device will later serve as the contact points for the negative and positive electrodes.

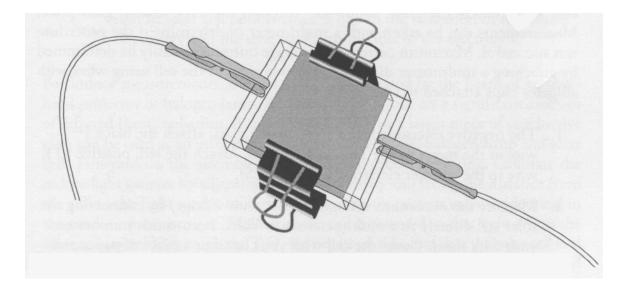


Figure 7: Assembled dye sensitized solar cell. The two glass plates are offset so that the uncoated portion of the  $TiO_2$  plate is exposed. A portion of the catalyst coated plate will also be exposed. Light enters the assembly through the  $TiO_2$  side of the cell.

**6.** Carefully pick up the assembly while it is in this orientation. Place two binder clips on the longer edges to hold the plates together.

- 7. The iodide electrolyte solution consists of KI mixed with I<sub>2</sub> in ethylene glycol. Carefully place two drops of this liquid I<sub>3</sub><sup>-</sup> solution at one edge of the plates. Keeping the plates sandwiched together, alternately remove and replace each binder clip. This creates a small space between the plates into which the solution is drawn by capillary action. Continue alternating between the clips until all of the stained area is contacted by the electrolyte.
- 8. Wipe off the excess electrolyte from the exposed areas of the glass using cotton sticks (buds) dampened with ethanol and finally with dry tissues. It is important that the electrolyte is completely removed from the two exposed sides of the cell.

# **EXPERIMENT 2: VOLUMETRIC DETERMINATION OF IODINE IN ELECTROLYTE SOLUTION**

#### **INTRODUCTION**

The dye sensitized solar cell electrolyte is a  $I_2/$  KI ( $I_3^-$ ) solution in ethylene glycol solvent. The ionic chemical equation of the reaction between sodium thiosulphate,  $Na_2S_2O_3$  and  $I_3^-$  is given below.

$$I_3^- + 2 S_2 O_3^{2-} \rightarrow 3I^- + S_4 O_6^{2-}$$

#### **INSTRUCTIONS**

- 1. Prepare and fill the burette with standard solution of sodium thiosulhate solution Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.
- **2.** Record the initial reading of your burette in the answer sheet.
- **3.** Using a pipette, transfer an aliquot of 10mL of the solution of iodine in ethylene glycol into a clean Erlenmeyer flask.
- **4.** Titrate, swirling the conical flask until a yellow colour appears. Add 2mL starch indicator and 20 mL distilled water and continue titration until the blue black colour disappears and the solution becomes colourless.
- **5.** Record the final reading of your burette in the answer sheet.
- **6.** Repeat the titration at least three times.
- **7.** Complete the table in the answer sheet.
- 8. Calculate the molar concentration of I<sub>3</sub> in the electrolyte.

# **Experiment 3: Chemical properties of anthocynin**

Place 1mL of pomegranate anthocinin solution into a beaker and add 9 mL of distilled water to it (solution A). Transfer 1mL of the diluted solution into each of 4 test tubes. In addition 1 drop of HCl is added to the solutions of  $T_1$ ,  $T_3$  and  $T_4$ . % drops of ammonia solution are added to  $T_3$ . Using a small spatula add some crystals of aluminium chloride to  $T_4$ .

Complete the table in your answer sheet

# **EXPERIMENT 4:** The electrical output characteristics of the solar cell