The HK University of Science and Technology

Department of Chemistry

SS Learning and Teaching Strategies for the Chemistry and Combined Science (Chemistry Part) Curricula Series: (1) Colorimetry (2) Nanomaterials (3) Liquid Crystal

Lab Manual

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Basic Safety & Waste Disposal Procedures

Personal Safety

Safety goggles must be **worn** at all times in the labs. ***Contact lenses should not be worn*** in the lab because chemicals and particulates can get caught behind them, causing severe eye damage.

Lab gown must be worn in the labs where an unexpected chemical spill may expose you to the risk of injury. The following clothing is **not permitted** in the labs unless covered by protective clothing:

- Open-toed shoes, sandals or other uncovered footwear; clothes that expose above the ankles;
- Eating, chewing gum, and drinking in the lab.
- Untied long hair, dangling jewelry, loose clothing, and anything else that may get caught in equipment, or dipped in chemicals.

• ***Never work alone or unsupervised in the labs.*** Work only during the scheduled laboratory periods and perform only authorized experiments.

- Wash your hands, arms, and then face, with soap and water as soon as possible after leaving the lab.
- If you are uncertain about any safety aspect of an experiment, please ask your TA.

Lab Safety

• ***Make sure you know the exact locations of the safety features of the lab***; e.g., eyewash fountains, safety showers, chemical spill kits, fire extinguishers, fire alarms, fire blankets.

• ***Whenever possible, do not deal with incidents on your own***. Your TA, the lab instructor and the technician are all trained to respond to the sort of incidents that may occur in this lab; e.g., chemical spills, cuts, burns, fires, medical emergencies, etc.

• ***Keep your work area clean and organized*** to reduce the possibility of accidents. Know what you are doing and don't be careless.

• ***Avoid unnecessary exposure to chemicals***. Never pipette by mouth. Never taste or inhale a chemical on purpose. Wear gloves when directly working with hazardous chemicals. Use hoods when appropriate.

• ***Take appropriate precautions***. Keep flammables away from hot plates and open flames. Wear gloves when using toxic, carcinogenic, or other hazardous chemicals. Take care with corrosive acids and bases. Always pour concentrated acid slowly into water (never water into acid). Read the ***Safety Issues*** section at the beginning of each experiment.

• ***Be informed***. Material Safety Data Sheets (MSDS) summarize known hazards associated with every chemical are available from the lab.

• Chemicals & equipment may not be removed from the lab without permission from the Lab Instructor.

Disposal of Chemical Waste

It is very important to properly dispose the chemical waste you generate. Follow these guidelines and dispose of your waste properly, to avoid adding to the contamination of our environment.

• ***Generate as little waste as possible.*** It is expensive to have hazardous waste removed and disposed. Don't prepare more of a chemical than you expect to use.

• ***Never return unused portions of chemicals to the reagent bottle.*** At the end of your experiment, unused reagent must be disposed of as waste, so don't pour out more than you need.

• ***Don't discard chemicals down the sink or in the wastebasket***, unless you are explicitly told that it's okay to do so. Most of your chemicals will pose a threat to the environment if disposed improperly.

• ***Place chemical waste only in the appropriate container***. Often, more than one waste container is provided to separate certain chemicals for safety or easier disposal. Pay attention to the *Waste Disposal* information for each experiment in this lab manual, and use the waste containers indicated. If you cannot find a waste bottle labeled with your particular chemical, ask your TA where to dispose it.

• ***Fill in the appropriate waste inventory sheet***. There is separate inventory sheet for each waste container. Use it to record the chemical(s), concentrations and volume you dispose.

• ***Do not over-fill a waste container***. Tell the technician the bottle is getting full and they will replace it.

• ***Use the clearly marked GLASS containers to dispose of broken glass and Pasteur pipettes***. Do not place broken glass in the sink or wastebasket, to avoid serious injury to an unsuspecting person.

• ***Use the clearly marked WASTE SOLIDS wide-mouth bottles to dispose of waste solids.*** Waste solids include solid chemicals, filter paper, and weighing paper.

• ***If you realize that you disposed of a chemical in the wrong container***, use the waste inventory list provided by the waste container to let us know.

• ***If you have waste whose identity you can't recall***, you can often test your waste, e.g., with litmus paper, to deduce its identity. Do not add unidentified waste to the waste bottles. You will force us to categorize the entire waste container as "unknown" which becomes extremely expensive to identify and dispose of.

Colorimetry-Determination of Sunset Yellow (428nm) in *Fanta soda* and *Lucozade*

Chemical hazard notes

 ***Glacial acteic acid is corrosive to skin and tissue. The vapour is very irritating to lungs.***

 ***Ethanol is flammable.***

 ***Ammonia solution can lead to serious eye damage***

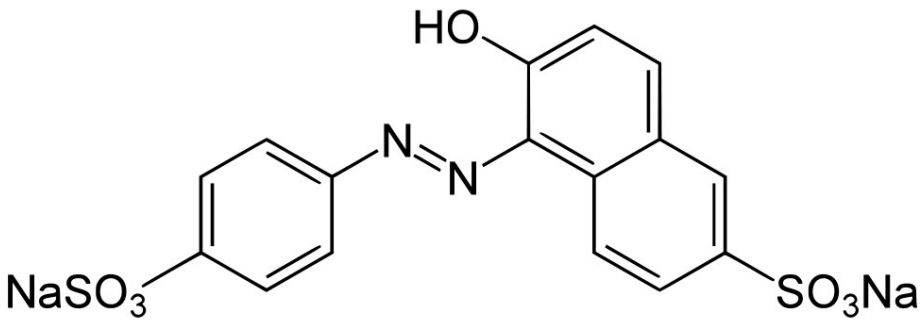
Disposal of wastes

Organic reagents and solvent must be disposed to appropriate waste solvent bottle.

Theory

Food coloring (colouring) is any substance that is added to food or drink to change its color. Food coloring is used both in commercial food production and in domestic cooking. Due to its safety and general availability, food coloring is also used in a variety of non-food applications, for example in home craft projects and educational settings.

Sunset Yellow FCF (also known as **Orange Yellow S**, **FD&C Yellow 6** or **C.I. 15985**) is a colourant that may be added to foods to induce a colour change. It is denoted by E Number E110, and has the capacity for inducing an allergic reaction.



It may be found in orange squash, orange jelly, marzipan, Swiss roll, Irn-Bru, apricot jam, citrus marmalade, lemon curd, sweets, hot chocolate mix and packet soups, trifle mix, breadcrumbs and cheese sauce mix and soft drinks. Specifically it can be found in the capsules of DayQuil (in high concentrations), some extra strength Tylenol,

Astro peach yogurt (potentially others), fortune cookies, some red sauces, certain pound cakes, snack chips and other yellow, orange, and red food products.

Sunset Yellow is often used in conjunction with E123, Amaranth, in order to produce a brown colouring in both chocolates and caramel

Chemicals and Apparatus

Sunset yellow
polyamide
diluted acetic acid
10% ammonia solution
95% ethanol
Fanta soda
Lucozade

Colorimeter
486 nm wavelength filter
Cuvettes
6 × 50 mL volumetric flasks with stoppers
Filter funnel
50 mL beaker
Pipette filler
Dropper teat
Dropper
Glass rod

Procedure

<u>NB</u>: (A) Label all flasks and container; (B) Wear your safety glasses.

Experimental Procedure

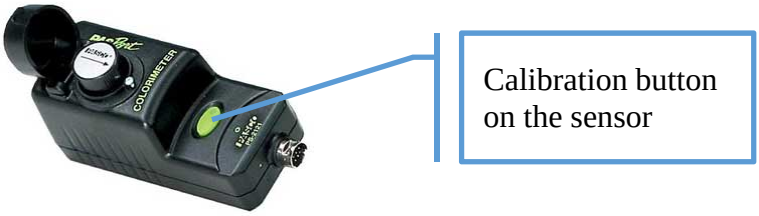
A. Preparation of the Datalogger

<u>Computer Setup</u>

1. Connect the USB Link Interface to the computer's USB port.
2. Connect the colorimeter to the USB Link interface.
3. Double click to launch the software **"DataStudio"**. The software would automatically identify the colorimeter.
4. Click **Setup**.
5. Under **Sample Rate**, select **1** for value and **Hz** for units. Select "**blue (486nm) absorbance**" for measurement.

<u>Colorimeter Calibration</u>

1. Fill a cuvette 3/4 full with deionised water and close the cap.
2. Insert the cuvette into the cuvette holder of the colorimeter and close the lid tightly.
3. Press the green Calibration button on the sensor. The light illuminates to indicate calibration is in progress.



4. Wait for the light to turn off and then remove the cuvette.

B. Measurement and Constructing Calibration curve

1. With the standard Sunset Yellow provided (308 μM) (139 μg/mL), a series of diluted standard solution is prepared according to the following table:

Reagents	Flask A	Flask B	Flask C	Flask D	Flask E
Sunset Yellow	1 mL	2 mL	3 mL	4 mL	5 mL
Conc. of Sunset Yellow					
Total volume	50 mL	50 mL	50 mL	50 mL	50 mL

2. Fill a cuvette 3/4 full with the solution of known concentration, starting from the lowest concentration. Wipe the outside of the curvette with a tissue to make sure that it is clean and dry.

3. Place the curvette in the sample compartment of the colorimeter and close the lid.
4. Click the Start button to begin recording the absorbance.
5. Click the Stop button when the absorbance stabilizes. Write down the data on the result table.
6. Repeat steps above, for each solution of known concentration making sure that you maintain the order from lowest to highest concentration.
7. Plot a graph of absorbance versus concentration for the series of standards.

C. Sample preparation

1. Degas Fanta soda drink/Lucozde using ultrasonic bath or leave the sample in air for 2 hours before use.
2. 30 mL of sample solution is measured to a beaker and 2 drops of 50% acetic acid is added.
3. A column (dropper) packed with polyamide (4 cm height) is prepared and the packing polyamide is activated by flushing with 50% acetic acid.
4. Sample solution is loaded into the column.
5. After the deposition of colorant on the top of the column, it is being washed with diluted acetic acid (0.1 M, 30 mL).
6. Few drops of 10% ammonia solution are added onto the polyamide.
7. The adsorbed colorant is eluted with ethanol, and the solution is collected using a beaker.
8. Steps 4 to 5 are repeated once.
9. The solution is evaporated to dryness over a hot water bath.
10. Few drops of 10% ammonia and ethanol are added to redissolve the residue.
11. Sample is transferred into a volumetric flask F and the absorbance of the sample is collected.
12. From the graph, obtain the concentration of Sunset Yellow in the sample.

Simplified Method

1. Degas Fanta soda drink/Lucozde using ultrasonic bath or leave the sample in air for 2 hours before use.
2. Pipette 5 mL of sample solution to a volumetric flask, and dilute the solution to 50mL solution using deionized water.
3. The absorbance of the sample is collected.
4. From the graph, obtain the concentration of Sunset Yellow in the sample.

Copy this table into your practical report book

Absorbance of solution in flask A	=
Absorbance of solution in flask B	=
Absorbance of solution in flask C	=
Absorbance of solution in flask D	=
Absorbance of solution in flask E	=
Absorbance of solution in flask F	=
Concentration of Sunset Yellow in the sample	=

Nanomaterials - Octadecanethiol Monolayer on Silver

Chemical hazard notes

 ***Potassium Hydroxide is corrosive to skin and tissue.***

 ***Ethanol is flammable.***

 ***Ammonia solution can lead to serious eye damage***

Disposal of wastes

Organic reagents and solvent must be disposed to appropriate waste solvent bottle.

Theory

Procedure developed by George Lisensky based on the Tollens' Test and the well-known self-assembly of thiol monolayers (SAM) on gold surfaces.

In this experiment, the aldehyde group in glucose reduces $Ag(NH_3)_2^+$ to Ag metal. This silver is then coated with a self-assembled monolayer of octadecanethiol, making a non-polar surface that allows water beading up.

Chemicals and Apparatus

Materials for 25 students

- 0.8 M KOH (Dissolve 0.22 g KOH in 5 mL of water.)
- Octadecanethiol solution in absolute ethanol
- Active silver ion solution, $Ag(NH_3)_2^+$
  - 0.1 M silver nitrate (Dissolve 0.17 g $AgNO_3$ in 10 mL of water.)
  - 15 M ammonia (Concentrated aqueous ammonium hydroxide.)

Petri dish
Microscope slide
Dropper teat
Dropper
Hair dryer
Forceps

Reference

http://www.youtube.com/watch?v=SvgEd71nHUg

Preparation:

Active silver ion solution:

1. Add concentrated ammonium hydroxide dropwise to 10 mL of 0.1 M silver nitrate solution until the initial precipitate just dissolves.
2. Mix with a glass stir rod. Add 5 mL of 0.8 M KOH solution; a dark precipitate will form. Add more aqueous ammonia dropwise until the precipitate just redissolves. This "active silver" solution should be used within an hour of preparation.
3. Dispense from a dropper bottle. To avoid the formation of explosive silver nitride, discard any remaining active solution by washing down the drain with plenty of water.

Glucose solution:

0.5 M glucose or dextrose (Dissolve 0.90 g in 10 mL of water. Dispense from a dropper bottle.) Sugar or sucrose does not work.

Alkanethiol solution:

Add a very small amount (just barely visible) of a long-chain alkanethiol, such as octadecanethiol, to 20 mL of absolute ethanol. Dispense from a dropper bottle.

Procedure

NB: (A) Wear your safety glasses.

Experimental Procedure

1. Place a clean microscope slide in a Petri dish. Place 4 large drops of a 0.5 M glucose solution on the microscope slide.
2. Add 12 large drops of the active silver ion solution. Gently agitate to mix the solution. Wait several minutes while the solution darkens and a grayish precipitate forms.
3. A silver mirror is also forming on the slide, though it may be obscured by the precipitate formed.
4. Use water from a wash bottle to wash off the precipitate and reveal the silver mirror. *Avoid contact with the solution since it will stain your hands.* Remove the slide from the Petri dish and rinse the silver mirror with water with the aid of forceps.
5. With the use of hair dryer, dry the surface of the slide.
6. Cover only part of the silver surface with a few drops of octadecanethiol solution. One way to do this is to rest the slide at an angle. Allow the ethanol to evaporate at room temperature (no hair dryer), leaving behind an octadecanethiol monolayer with the sulfur atoms bound to the silver and the hydrocarbon tails pointing away. This effectively coats the surface with a monolayer of hydrocarbons.
7. Add water dropwisely to the coated surface, observe the difference between glass surface and the coated surface.

Nanomaterials - Octadecanethiol Monolayer on Silver – Data Sheet

Experimental Data and Results

	Actions	Observations and Conclusion
1.	Attraction of water drops to the monolayer coated surface?	
2.	To the silver surface and the glass, do water drops spread out or bead up?	
3.	Is the water attracted more to the plain glass, to the silver, or to the octadecanethiol coated silver?	

Liquid Crystal- Properties of Liquid Crystal Display-Disassembly of a Liquid Crystal Watch

Theory

Liquid crystals are commonly used as displays for electronic devices such as watches, calculators, televisions. This is the result of the unusual optical and electrical properties of liquid crystals. The long thin liquid crystal molecules cause light to travel at different speeds along the molecular axis and perpendicular to that axis. This leads to their ability to rotate the plane of polarized light.

When the current is off, the liquid crystal molecules in all segments of the panel are precisely aligned. In this case, the panel appears silvery because light passes through both polarizers, reflects off the mirrored surface, and then passes back through both polarizers.

When the current is on, the liquid crystals are twisted, losing the initial alignment of the molecules. The polarized light is no longer aligned with the second polarizer and that segment of the display will appear black against a silver background.

Chemicals and Apparatus

An inexpensive LCD watch (not necessarily functioning)
Small screwdriver
Forceps
600 mL beaker
9-Volt battery and battery snap
Hot water bath
Cup or beaker
Thermometer
Polarizing filter
Scissor
Heater

Reference:

http://www.youtube.com/watch?v=6cEWISobgu8

Procedure

Experimental Procedure

1. Remove the front or back plate to access the interior. Your watch may be slightly different from the one described here. If screws are removed, place them in a small container for safe keeping since tiny parts can be easily lost.
2. Remove the tiny screws holding the printed circuit board and battery retainer to the white plastic inner case. Remove the electrically conducting pad and the LCD panel.
3. Place the polarizing filter on the top of the LCD panel, turn the filter to 90^{o}, record the observation.
4. Turn the filter to further 90^{o}, 180^{o} and 270^{o}, record all observations.
5. Examine the LCD panel and find the contact area of the panel on the bottom of the wider glass plate. Use a battery snap connected to 9-V battery. Hold one of the leads against the contact area at one end of the panel and rub the other lead along the contact area to address various segments of the LCD panel. Record the observable changes on the panel.
6. Press the LCD panel by finger/forceps, record the observation under pressure.
7. Obtain 300 mL of water to the 600 mL beaker, heat the water gradually with stirring.
8. Monitor the temperature of the water using thermometer.
9. With the help of the forceps, insert the LCD panel into the water bath. Record the temperature of liquid phase transition.
10. Lift the LCD panel from hot water, dry with a tissue paper.
11. Remove the top polarizer of the panel by carefully prying up one corner and then gently peeling it away.
12. (This step is not totally reversible) Cut the polarizer in half with scissors. Place one half back on the panel in its original orientation. Place the other half back after rotating 90^{o}.
13. Use a battery snap connected to 9-V battery. Hold one of the leads against the contact area at one end of the panel and rub the other lead along the contact area to address various segments of the LCD panel. Record the changes with reference to step 5.

Liquid Crystal-Properties of Liquid Crystal Display – Data Sheet

Experimental Data and Results

	Actions	Observation(s)
1.	Turn the polarizing filter on the top of the LCD panel	
2.	Rub the panel by battery	
3.	Press the panel	
4.	Increase the temperature	
5.	Reverse the position of the polarizer	