

*Supporting Information*

# Enhancing STEM Education by Integrating Research and Teaching in Photochemistry: An Undergraduate Chemistry Laboratory in Spectroscopy and Photochemistry

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## Section S1. Laboratory manual.

# Experiment: Extraction, Characterization and Performance of Sunscreens

## Procedure:

### Part I: Sample Preparation

1. Obtain two **clean, dry** centrifuge tube from your instructor.
2. Weigh between 0.20-0.30 g of sunscreen lotion in your tubes. Be sure to record the mass of the sample in your report sheet or laboratory book.
3. Place a label on the tube: the label should report your name and sample number.
4. Using a pipette, add 2.00 mL of hot water to your sunscreen lotion in each test tube.
5. Close each tube with the cap provided and shake the sunscreen lotion vigorously; a cloudy solution (suspension) should be formed. If available, place the sample in the vortex and shake at high speed for 2-3 minutes.
6. Open the cap and use a pipet to add a 10.00 ml aliquot of isopropanol to the each sunscreen-water mixture. The solution should now appear clear, but some precipitate may be observed.
7. Replace the cap on the tubes and shake the samples vigorously again. If available, place the samples in the vortex and shake at high speed for additional 2-3 minutes.
8. If available, use an ultrasonicator to help dissolution of sunscreen active ingredients. Use a clamp to secure each test tube and then lower them into the water in the sonicator bath. Set the timer to ultrasonicate the sample for 10 minutes.
9. Repeat step 6 and 7 once more.
10. Place the test tubes in the centrifuge. Centrifuge on high for 5 minutes. This operation will encourage the undissolved material to be settled at the bottom of the tube. The supernatant fluid (liquid above the precipitate) will appear *mostly* clear.
11. If centrifuge is not available, place the test tubes in your draw to store overnight. The undissolved material will settled at the bottom of the tube and the solutions will be ready to be analysed during the next laboratory session.

### Part II: TLC analysis

12. For the following analysis only the supernatant fluid will be sampled. **Take care not to sample the undissolved material/precipitate.**
13. Obtain a **clean, dry** 100 mL beaker from your drawer.
14. Obtain the mobile phase from the instructor. This has been prepared for you and is made up of a hexane and acetone mixture (15:2)
15. Place ~10 mL of mobile phase in the beaker. Cover with plastic wrap and set aside. This will serve as the development chamber.
16. Obtain at least one TLC plate from your instructor. Be sure not to touch the silica gel surface with your fingers as this can contaminate the surface.
17. Prepare the TLC plate by drawing an horizontal line at ~1 cm from the bottom of the plate (use a **pencil, do not use a pen**). Be sure not to scrape any silica off the plate during this operation. Place three equally distanced circles or crosses along this line.
18. Using a capillary, place a small amount of sample solution on each of the three circles/crosses on the TLC plate. Once the solvent dries, you will not be able to see the sample unless the

TLC plate is placed under the UV lamp. You may need to repeat this procedure 3-4 times to make sure that enough sample is deposited on the TLC plate.

19. Bring the TLC plate to the instructor and ask for the short wave UV lamp.
20. Place the TLC plate under the UV lamp. You should be able to clearly see sample deposited on the circles/crosses; if not, more sample needs to be placed on the plate.
21. Place the TLC plate in the developing chamber containing the mobile phase. Complete this action with care; the sides of the TLC plates should not touch the walls of the beaker. The plate should be placed as vertical as possible.
22. Cover the beaker with the plastic wrap.
23. The mobile phase will start moving upwards along the TLC plate. Do not move or disturb the chamber during this time.
24. Remove TLC plate from the development chamber when the solvent front is about 1 cm below the top edge of the plate. QUICKLY draw a line to mark the distance travelled by the mobile phase before it dries invisible.
25. You will not be able to see any spots on the TLC plate at this point.
26. Bring the TLC plate to the instructor and ask for the short wave UV lamp. Place your TLC plate under the UV light.
27. Circle all visible spots on the TLC plate using a pencil.
28. Calculate the retention factors for each spot you identified on your TLC plate.

### **PART III: UV-Vis characterization**

29. Isopropanol is your solvent. Obtain a standard 10.0 mm quartz cuvette from your instructor and fill it with Isopropanol.
30. Place the cuvette in the spectrophotometer and take a blank using the solvent.
31. Obtain a 5 ml volumetric flask. Add about 5-10 drop of your sample (only supernatant fluid) to the flask and dilute with isopropanol. This dilution is necessary since the starting solution is too concentrated and direct analysis will not give reliable results.
32. Place the cuvette in the spectrophotometer and record an absorption spectrum of your sample: the maximum absorbance at the near UV should be equal or lower than 1. If the absorbance is above 1 (saturation), further dilution with isopropanol is required. If the absorption is below 0.3 – 0.4, it is advisable to add more sample to the volumetric flask and repeat the analysis.
33. Export the absorption spectrum as a .csv file (to open in Excel or equivalent program) for your report.

### **PART IV: UV Photophysics**

34. Once step 30 -33 are completed, place the LED light source appropriately to irradiate the sample. Be sure that the light is aligned so as to go straight through the sample holder. If in doubt, ask the instructor to check the alignment of the light source. **Use caution; DO NOT look** directly at the light emitted by the LED. Always make sure that the light source is OFF when looking directly at the bulb.
35. Obtain a stopwatch.
36. Switch the LED light on while starting the stopwatch at the same time.
37. Irradiate the sample for 60 minutes (or the time advised by the instructor), acquiring one absorption spectrum every two minutes. Make sure to save the data at regular intervals.
38. Export the absorption spectra as a .csv file (to open in Excel or equivalent program) for your report.

## Section S2. The TLC results

Each TLC plate contains a triplicate of a sample. Three plates were developed for a total of nine data point for each ‘spot’. Below we report the  $d_m$  and  $d_s$  values as well the corresponding  $R_f$ . The average  $R_f$  values ( $\bar{R}_f$ ) and standard deviation ( $\sigma$ ) are also reported.

### Sample 1

Values	Trial 1			Trial 2			Trial 3		
$d_m$	3.45			3.70			3.70		
$d_s$ (spot 1)	0.85	0.80	0.90	1.15	1.15	0.90	1.70	1.60	1.60
$R_f$ (spot 1)	0.25	0.23	0.26	0.31	0.31	0.24	0.46	0.43	0.43

$\bar{R}_f$	$\sigma$
0.33 (spot 1)	0.09

### Sample 2

Values	Trial 1			Trial 2			Trial 3		
$d_m$	3.30			3.90			3.60		
$d_s$ (spot 1)	1.25	1.25	1.25	1.30	1.25	1.30	1.40	1.35	1.35
$d_s$ (spot 2)	1.50	1.50	1.45	1.60	1.55	1.60	1.70	1.60	1.60
$R_f$ (spot 1)	0.38	0.38	0.38	0.33	0.32	0.33	0.39	0.38	0.38
$R_f$ (spot 2)	0.45	0.45	0.44	0.41	0.40	0.41	0.47	0.44	0.44

$\bar{R}_f$	$\sigma$
0.36 (spot 1)	0.03
0.44 (spot 2)	0.02

### Sample 3

Values	Trial 1			Trial 2			Trial 3		
$d_m$	3.30			3.35			3.35		
$d_s$ (spot 1)	1.80	1.75	1.75	1.70	1.60	1.65	1.55	1.55	1.55
$d_s$ (spot 2)	2.05	2.00	2.05	1.95	1.90	1.90	1.85	1.85	1.85
$d_s$ (spot 3)	2.60	2.60	2.60	2.65	2.75	2.60	2.50	2.45	2.50
$R_f$ (spot 1)	0.55	0.53	0.53	0.51	0.48	0.49	0.46	0.46	0.46
$R_f$ (spot 2)	0.62	0.61	0.62	0.58	0.57	0.57	0.55	0.55	0.55
$R_f$ (spot 3)	0.79	0.79	0.79	0.79	0.82	0.78	0.75	0.73	0.75

$\bar{R}_f$	$\sigma$
0.50 (spot 1)	0.03
0.58 (spot 2)	0.03
0.78 (spot 3)	0.03