SUNSCREENS: PREPARATION AND EVALUATION

INTRODUCTION

Had a great day at the beach? Boating? Hiking? Working in the garden? Skiing? Did you get a sunburn? Did you use some protection? Did you use a sunscreen?

The sun, our nuclear furnace at the center of our solar system, produces a wide range of electromagnetic radiation, some of which sustains life on our planet. Although we are most aware of the visible light from the sun, the only light we can see with the unaided eye, there are a number of types of light we cannot see. One of these is infrared light, which is responsible for heating our planet. Another is ultraviolet light which is responsible for sunburn and suntan and increases the risk of basal cell carcinoma and malignant melanoma. Ultraviolet light is artificially divided into three ranges:

UVA is radiation in the 320-400 nm range
UVB is radiation in the 290-320 nm range
UVC is radiation in the 100-290 nm range

UVC is totally blocked by the ozone layer in the upper atmosphere of the Earth. The ozone layer blocks some of the UVB and all of the UVA passes through the ozone layer. Generally, UVB has been blamed for sunburn, but some studies indicate that UVA may also cause skin damage.

Mild sunburn is a first-degree radiation burn which produces redening of the skin with accompanying pain. Generally, as the skin heals, redness may persist and the outer layers of the epidermis will peel within a week with accompanying itching. Prolonged exposure can result in a second-degree burn which is characterized by blistering of the skin and more severe pain. Third-degree sunburn is rare.

One of the body's defenses against UV radiation is the production of melanin, a pigment, that results in darkening of the skin. An individual's response to UV radiation and melanin production is dependent on skin color and other genetic factors. Even if an individual has dark skin, or whose skin readily produces melanin when exposes to UV radiation, may still experience sunburn as a result of high intensity of UV radiation and an extended length of exposure.

Sunscreens are cosmetic formulations that block UV rays. Sunscreens are assigned sun protection fators, or SPF, ratings that are supposed to indicate the level of protection from UV radiation. The SPF rating is a multiplier that tells how long one can safely remain in the sun. For example, if an individual whose unprotected skin becomes sunburned in 5 minutes, then an SPF-15 sunscreen should allow that individual to stay in the sun for 15 times 5 minutes, or 75 minutes, without burning.

Some active ingredients in sunscreens are:

Benzyl salicylate and salicylate derivitaves. One of the first sunscreen agents. It provides UVB protection, but not UVA. It is not soluble in water and can be used in waterproof formulations. It is often used in combination with other ingredients. One of the derivative compounds is known as homosalate.

Benzyl cinnamate and cinnamate derivatives. Another early sunscreen agent. It is an effective UVB blocker, but is not waterproof. Often found in combination with other ingredients.

PABA (p-aminobenzoic acid). This compound was extensively used in many formulations, however, it was not water soluble and needed to be used in alcohol-based solutions, it would discolor fabrics, and many individuals experienced or developed allergic reactions to it. Most sunscreen lotions are now PABA free.
Butyl methoxydibenzoylmethane and related compounds. Also known as Parsol 1789 and Parsol A is an effective UVA blocker. Oxybenzone is a related compound.

Zinc oxide and titanium dioxide are two inorganic compounds that are insoluble in most liquids. These block the UV radiation because their preparations are opaque to light. Sunscreen lotions containing these are normally white opaque ointments on the skin.

Each of the active ingredients provides an SPF factor related to its concentration in the sunscreen. Increasing the concentration of the ingredient should also increase the SPF rating of the sunscreen.

In Part I of this experiment, we will prepare a sunscreen lotion varying the concentration of some of the active ingredients in the formulations. In Part II, we will evaluate the effectiveness of several sunscreens using a Spectronic 20 spectrophotometer in the 320 to 400 nm range and determine the SPF rating of the laboratory prepared sunscreen lotion. Although this UV radiation is in the UVA range, general trends in UV absorption can be observed allowing the sunscreen lotions to be uantitatively compared. (Note: depending on the age and model of the Spectronic 20, you may only be able to measure down to 330 or 340 nm.)

PART I: PREPARATION OF A SUNSCREEN

MATERIALS NEEDED:

Cetyl alcohol (also known as 1-hexadecanol)
Benzophenone-3 (also known as oxybenzone)
Ethylhexylmethoxycinnamate (also known as octyl methoxycinnamate)
Stearic acid
Glycerin
Triethanolamine
Water, distilled or deionized
Stearyl dimethicone silicate crosspolymer and Cyclopentasiloxane (Wacker-Belsil RPG 33, Wacker-Chemie)
Beaker, 150 mL
2 Beakers, 400 mL
Thermometer, 110°C
Stirring rod
Water bath
Beaker tongs

SAFETY PRECAUTIONS:

Wear approved eye protection in the laboratory at all times.

The chemicals used in this procedure are normally used in cosmetic formulations and do not present any direct skin contact hazards. In the event of skin contact, wash your hands with soapy water.

DISPOSAL:

All materials this experiment should be disposed in the proper waste containers provided in the laboratory.

PROCEDURE:

This experiment is performed as a class project. Student groups will prepare a single formulation of a sunscreen lotion asigned from Table 1. Data and results from all individuals and/or lab groups will be combined for comparison.

Place a 150-mL beaker on a balance and weigh it. Weigh the quantities of cetyl alcohol, benzophenone-3, ethylhexylmethoxycinnamate, stearic acid, glycerin, and stearyl dimethicone silicate crosspolymer called for in your assigned formulation from Table 1 into the 150-mL beaker. Heat the beaker with the organic mixture in a water bath until all the ingredients have melted. **Note:** Cosmetic ingredients should not be melted over a direct flame or high heat because they may scorch or decompose if they are heated much above the boiling point of water.

Measure 78 g of water into a 400-mL beaker. Add 1.0 g of triethanolamine to the water. Stir. Heat the water solution to a temperature of 80° to 85°C.

After the water solution has reached a temperature between 80° and 85°C, remove it from the heat and slowly pour the melted cetyl alcohol, benzophenone-3, ethylhexylmethoxycinnamate, stearic acid, glycerin, and stearyl dimethicone silicate crosspolymer mixture into the water a little at a time, stirring constantly. It may be helpful to hold the 400-mL beaker using a pair of beaker tongs. (Note: If the “organic mixture” has solidified, heat briefly in the water bath to remelt it.) If you pour too fast or if you do not stir, your emulsion will be lumpy or the mixture may not fom an emulsion. Continue stirring until you have a smooth, uniform paste. Label the beaker and set the sunscreen cream aside to cool. You will need this sunscreen mixture for Part II of this experiment.

After the sunscreen mixture has cooled, rub a small amount onto you forearm. Describe the texture, and spreadability of the lotion. Does it dissolve into the skin in a reasonable amount of time? Does it leave the skin feeling oily or greasy?

Using tap water, wet the area of your skin where you applied the sunscreen lotion. Does it appear to wash off?

How does the sunscreen lotion you prepared compare with a commercial sunscreen lotion?

REFERENCE AND ACKNOWLEDGEMENT

This sunscreen formula appeared in the August 2003 issue of Cosmetics & Toiletries magazine.

The author wishes to acknowledge Dr. Lisa Mangos, Mary Kay, Inc., for her assistance in obtaining sunscreen formulation information.

Table 1. Components of a Sunscreen Lotion

Ingredient	Formulation 1	Formulation 2	Formulation 3	Formulation 4	Formulation 5	Formulation 6	Formulation 7	Formulation 8
Cetyl alcohol	2.0 g	2.0 g	2.0 g	2.0 g	2.0 g	2.0 g	2.0 g	2.0 g
Benzophenone-3	1.5 g	1.5 g	1.5 g	1.5 g	—	3.0 g	4.5 g	4.5 g
Ethylhexylmethoxy-cinnamate	1.5 g	—-	3.0 g	4.5 g	1.5 g	1.5 g	1.5 g	4.5 g
Stearic acid	4.0 g	4.0 g	4.0 g	4.0 g	4.0 g	4.0 g	4.0 g	4.0 g
Glycerin	2.0 g	2.0 g	2.0 g	2.0 g	2.0 g	2.0 g	2.0 g	2.0 g
Stearyl dimethicone silicate crosspolymer	10.0 g	10.0 g	10.0 g	10.0 g	10.0 g	10.0 g	10.0 g	10.0 g
Triethanolamine	1.0 g	1.0 g	1.0 g	1.0 g	1.0 g	1.0 g	1.0 g	1.0 g
Water, distilled or deionized	78.0 g	78.0 g	78.0 g	78.0 g	78.0 g	78.0 g	78.0 g	78.0 g

Note: Concentrations of ethylhexylmethoxycinnamate are limited to a maximum level of 7.5% in a sunscreen formulation.

PART II: EVALUATION OF SUNSCREENS

MATERIALS NEEDED:

Sunscreen lotion, prepared in Part I of this experiment
Assorted sunscreen lotions such as SPF 5; SPF 8 or 10; SPF 15; SPF 25 or 30; SPF 40 or 50 (Two different brands of sunscreen, of the same SPF, should be available for comparison purposes. Original containers of each sunscreen should be available in the laboratory.)
n-proply alcohol
2 beakers, 100 mL
glass stirring rods
Spectronic 20
3 Cuvettes

SAFETY PRECAUTIONS:

Wear approved eye protection in the laboratory at all times.

n-propyl alcohol is flammable. Avoid flames or spark sources.

n-propyl alcohol vapors may be irritating to the eyes and repiratory system. Work in a well ventilated area.

n-proply alcohol does not present any direct skin contact hazards. In the event of skin contact, wash your hands with soapy water.

DISPOSAL:

All materials this experiment should be disposed in the proper waste containers provided in the laboratory.

PROCEDURE:

This experiment is performed as a class project. Data and results from all individuals and/or lab groups will be combined for comparison.
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You will be evaluating the sunscreen you prepared in Part I of this experiment and your instructor will assign you or your lab group at least one commercial sunscreen to evaluate.

The Spectronic 20 spectrophotometer should be turned on, set to 400 nm, and allowed to warm up for 15 minutes.

Prepare a 0.10% solution (w/v) of your assigned commercial sunscreen lotion in n-propyl alcohol. Weigh 0.050 g of sunscreen lotion into a 100 mL beaker. Add 50.0 mL of n-propyl alcohol and stir to dissolve the lotion. Label the solution with the brand name of the sunscreen product and its SPF rating. Following the same procedure, prepare a second 0.10% solution of the sunscreen lotion you prepared in the laboratory in n-proply alcohol.

Fill a cuvette approximately ¾ full with the 0.10% solution of the commercial sunscreen. Record the brand name and SPF rating of the sunscreen. You may label the cuvette near the top using a waterproof marker or small piece of laboratory tape.

Fill a second cuvette ¾ full with the 0.10% solution of the laboratory prepared sunscreen lotion. This is your blank solution. You may label the cuvette near the top using a waterproof marker or small piece of laboratory tape.

Fill a third cuvette ¾ full with n-propyl alcohol. This is your blank solution. You may label the cuvette near the top using a waterproof marker or small piece of laboratory tape.

Place the cuvette with the n-propyl alcohol in the cuvette holder of the Spectronic 20 and close the cell compartment cover. Zero the instrument.

Remove the blank and insert the cuvette containing the commerical sunscreen solution sample. Record the absorbance at 400 nm.

Remove the sunscreen solution sample, and insert the cuvette containing solution of the laboratory prepared sunscreen lotion. Record the absorbance at 400 nm.

Remove the sunscreen solution sample. Change the wavelength reading of the Spectronic 20 to 390 nm. Place the blank in the sample holder, close the cover, and zero the instrument.

Remove the blank and insert each of the cuvettes containing the sunscreen solution samples. Record the absorbances at 390 nm.

Continue to take readings at 10 nm intervals to 320 nm. Remember to zero the instrument using the blank solution at every wavelength before reading the sunscreen absorbance. Note: some Spectreonic may not give readings to 320 nm, if this occurs, stop at 330 nm.

After collecting all the data, graph the absorbance (y-axis) vs. the wavelength (x-axis) for each sunscreen sample using Excel on one of the laboratory computers. Save your data and graph files on the computer, as instructed by your instructor, and print out a copy of the graph.

Optional Extension Project: Determine the effect of aging on a sunscreen product. Compare the effectiveness of the same brand of sunscreen that is at least one year old with that of a sample that was recently purchased.

REFERENCE:

This experiment is based on *Analysis of Suntan Lotions*, contained in a package on Spectrophotometry from the Alabama Science in Motion project which was shared with teachers at ChemEd 2003.

EVALUATION OF SUNSCREENS

DATA AND RESULTS

Name(s): ____ Date ___________________

PART I: PREPARATION OF A SUNSCREEN

1. Which formulation did you use to prepare your sunscreen lotion?

2. Describe the texture, and spreadability of the sunscreen lotion you prepared.

3. Does the sunscreen lotion dissolve into the skin in a reasonable amount of time?

4. Does the sunscreen lotion leave the skin feeling oily or greasy?

5. Does the sunscreen lotion appear to wash off easily?

6. How does the sunscreen lotion you prepared compare with a commercial sunscreen lotion?

PART II: EVALUATION OF SUNSCREENS

Brand of sunscreen(s) and SPF used:

Laboratory prepared sunscreen lotion, Formulation no. ______ mass used: __________ g

Lotion #1: ____ mass used: __________ g

Lotion #2: ____ mass used: __________ g

Lotion #3: ____ mass used: __________ g

Lotion #4: ____ mass used: __________ g

Lotion #5: ____ mass used: __________ g

Wavelength nm	**Absorbance**					
	Formulation #_____	**Lotion #1**	**Lotion #2**	**Lotion #3**	**Lotion #4**	**Lotion #5**
400						
390						
380						
370						
360						
350						
340						
330						
320						

Attach a graph of absorbance vs. the wavelength of UV light for each suntan lotion you evaluated.

QUESTIONS: (You will need class data to answer some of these questions.)

1. What are the active ingredients in the commerical suntan lotion(s) you used?

2. Was your laboratory prepared suntan lotion effective in blocking UV radiation? Explain.
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3. Based on the absorbances of the sunscreen lotions tested in this experiment, what is the SPF of the sunscreen lotion you prepared in the laboratory? Explain.

4. How does the SPF value of the sunscreen lotion change with the different concentrations of benzophenone-3 and/or ethylhexylmethoxycinnamate? Explain.

5. Which of the two compounds, benzophenone-3 and ethylhexylmethoxycinnamate, is the most effective sunscreen ingredient? Explain.

6. Using the class data, which suntan lotion was the most effective in blocking UV radiation? Explain.

7 What is the active ingredient(s) in the most effective suntan lotion? What is the active ingredient(s) in the least effective suntan lotion?

8. Two different brands of suntan lotions with the same SPF rating were evaluated. How do these compare? Do they contain the same active ingredients?

9. As the SPF values of the different suntan lotions increase, does the effectiveness of the lotions increase in the same proportions?

10. The commercial suntan lotion that you evaluated has additional ingredients, other than the active ingredients. List those ingredients. Can you determine the functions of the additional ingredients?

11. Which of the suntan products that you tested would you use or recommend to others? Would you tell others to avoid particular products? Explain.