Fig. 32-1

Francis Bacon (1561 - 1626)
English philosopher
http://www.blupete.com/Literature/Biographies/Philosophy/Bacon.htm
http://oregonstate.edu/instruct/phl302/philosophers/bacon.html
http://www.ourcivilisation.com/smartboard/shop/patrickm/bacon/chap0.htm

Experiment 32

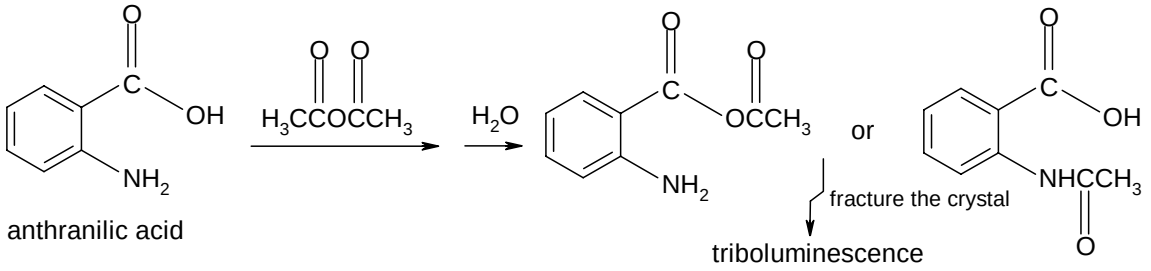
SYNTHESIS OF A TRIBOLUMINESCENT COMPOUND

Text Topics

Acylation reactions with anhydrides.

Discussion

The next time you are in a dark room or a cave with a companion, have the companion chew on a wintergreen hard candy. As the person chews with his/her mouth open, you should be able to observe the remarkable phenomenon of triboluminescence. For a some crystals including the wintergreen candy, the breaking of the crystal results in the emission of light. Even though triboluminescence was reported as early as 1605 by the great philosopher Francis Bacon, many questions regarding the cause of the emission still remain. One explanation is that the fracturing of the crystal results in an electrical discharge between the two separated parts. The energy of the discharge results in the electronic excitation of the compound and subsequent fluorescence. This experiment involves the attempted synthesis of a compound that exhibits triboluminescence.



As noted in the reaction scheme above, the reaction of anthranilic acid could lead to acylation of the carboxylic acid group or the amino group. You will run the reaction using either a traditional heating method or using a microwave oven. After isolation of the product, you will need to devise a method for determining the structure of your product and you will examine its triboluminescence.

Procedure

Traditional heating method. Set up a 25 mL round bottom flask with a reflux condenser. Add 2.0 g of anthranilic acid and 6 mL of acetic anhydride to the flask. Gently reflux the mixture for about 15 minutes and allow the solution to cool. Add 5 mL of water through the condenser to the solution and heat to the boiling point again. Subsequent cooling should lead to formation of crystals that you should be able to isolate by vacuum filtration. Wash the crystals with a small amount of ice-cold water and then with ice-cold methanol. Skip ahead to the purification procedure if purification is warranted. Determine the melting range and percent yield.

Microwave method. Add 1.0 g of anthranilic acid, 3 mL of acetic anhydride and a couple of boiling chips to a 100 mL beaker. Insert a stemless or powder funnel in the mouth of the beaker. Place the beaker in the middle of the turntable of a commercial microwave oven. Irradiate at full power for about 1 minute. The microwave should be turned off after the crystals dissolve and the mixture reaches the boiling point. Allow the solution to cool and add 4 mL of deionized water to the beaker. Microwave for about 10 seconds at full power. Carefully transfer the beaker to a 400 mL beaker that has been prelined and insulated with paper towels. Do not disturb during the cooling process. Crystals should form that you should be able to isolate by vacuum filtration. Wash the crystals with a small amount of ice-cold water and then with ice-cold methanol. Proceed to the purification procedure if warranted. Determine the melting range and percent yield.

Purification. Test your crystals in a dark room with a 360 nm black lamp. If the crystals fluoresce purple or slightly yellow, you should recrystallize from a 90% methanol/water mixture (this will require abut 0.5 mL of solvent/100 mg product). Once yellow or yellow-green fluorescence is attained you are ready to determine the structure of the compound and study its fluorescence.

Structure determination. This part of the procedure is left to you. However, the primary goal is to distinguish between the two options suggested on the first page of this experiment.

Triboluminescence. Crush the crytals between two small watch glasses in a dark room.

References

Erikson, J. *J. Chem. Educ.* **1972**, *49*, 688.
Baldwin, B. W.; Wilhite, D.M. *J. Chem. Educ.* **2002**, *79*, 1344 (and supplementary online material)

Prelaboratory Preparation - *Experiment 32*

First, be sure to list all the goals of the experiment. Prepare a table for insertion of useful and observed data such as molecular mass, mass, moles, melting points and percent yields and recoveries. Develop a plan for determining the structure of the product. Your primary goal will be to determine which of the possible products given on the first page of this experiment has the properties consistent with those you observe for your product.

Observations

Report all relevant observations including, masses and melting ranges, appropriate spectra and characteristics of the fluorescence. If a fluorescence spectrometer is available for use, determine the fluorescence spectrum of the compound.

Conclusions

This section should include the following:

1. Were the goals of the experiment achieved? Explain your answer.
2. What was the identity of your product and did it agree the properties of one of the predicted products? Explain your answer.
3. What was the percent yield and how could the percent yield and recoveries have been improved?
4. Did the fluorescence resemble the triboluminescence? If so, what information does this give you about the mechanism of triboluminescence?
5. Did this reaction fit any of the reactions included on the *Reaction-Map of Organic Chemistry* in ***Appendix C***? If so, include the reaction number in your report and explain your answer.