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# The Preparation of o-Fluorobenzoic Acid. An Elementary Organic Laboratory Experiment

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## ABSTRACT

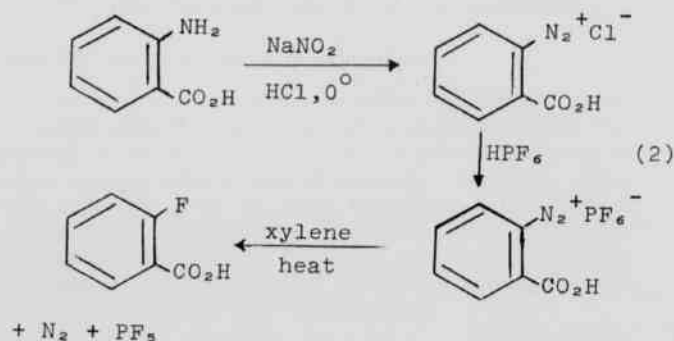
An experiment designed for organic chemistry students at the sophomore level is presented. The experiment, which involves no special equipment and which employs only inexpensive reagents, demonstrates the conversion of anthranilic acid to o-fluorobenzoic acid via the modified Schiemann Reaction.

Although most elementary organic texts illustrate the introduction of fluorine into aromatic systems by the Schiemann Reaction,<sup>1,2</sup> (equation 1) no experiments are generally available for the student laboratory application of this reaction.



Rutherford and co-workers<sup>3</sup> observed that replacement of fluoboric acid (HBF<sub>4</sub>) with hexafluorophosphoric acid (HPF<sub>6</sub>) in the above reaction resulted in improved overall yields of aromatic fluorides. The most outstanding yield increase (+52%) was in the case of the conversion of anthranilic acid to o-fluorobenzoic acid.

We have found that the general procedure employed by Rutherford is, with some modification, suitable for the preparation of o-fluorobenzoic acid in the elementary organic laboratory. The overall reaction scheme is illustrated by equation 2.



The experiment is conveniently performed in two laboratory periods. Diazotization of anthranilic acid and subsequent isolation of the o-carboxybenzenediazonium hexafluorophosphate is performed in the first period, and the second period is utilized for the thermal decomposition of the diazonium salt. The decomposition step proceeds smoothly, and students find it an interesting chemical transformation to observe.<sup>4</sup>

## Experimental

To a solution of anthranilic acid (10.3 g, 0.75 mole) dissolved in water (53 ml) and conc HCl (7.5 ml) in a 400 ml beaker cooled to -5° (a ppt of the hydrochloride results) is added NaNO<sub>2</sub> (6.1 g in 20 ml H<sub>2</sub>O dropwise from a separatory funnel) over a 30 min period with manual stirring. Hexafluorophosphoric acid<sup>5</sup> (25 ml of 65%) is added in one portion from a polyethylene graduate cylinder to the cold, now gelatinous, diazotized mixture. After allowing the resulting slurry to stand at ice bath temperature for 1/2 hr, the white solid is suction filtered, washed on the filter pad with ice water (150 ml), sucked dry, and air dried until the next laboratory period (mp. 125-129°, dec; yield 13-15 g).

Decomposition is carried out in a standard taper 500 ml, round-bottomed, one-necked flask containing 200 ml xylene and equipped with a condenser and inverted funnel gas trap<sup>6</sup> leading to a beaker of dilute NaOH solution. The mixture is heated with a small flame and with manual agitation until decomposition of the salt begins (evolution of white phosphorus pentafluoride fumes. WARNING: PF<sub>5</sub> is intensely irritating to skin, eyes and mucous membranes. Inhalation may cause pulmonary edema. Students should be thoroughly warned of this danger, each apparatus individually inspected by the instructor and the decomposition performed in an efficient fume hood). As decomposition ceases, the xylene is heated to boiling for 5 min to expel the excess gases. The reaction mixture is cooled to room temperature, extracted with three 50-ml portions of 5% NaOH

solution, and the aqueous extracts are poured with stirring into a slush of ice (100 g) and conc HCl (100 ml). The precipitated acid is suction filtered, washed on the filter with ice water (150 ml), dried, and recrystallized from methylcyclohexane (15 ml/g) using a steam cone. The yield of light yellow solid is 4-5 g, (40-50% from anthranilic acid), mp 120-122° (lit<sup>2</sup> mp 124-125°).

#### References

1. Balz, G., and Schiemann, G., *Chem. Ber.*, **60**, 1186 (1927).
2. Roe, A. in "Organic Reactions," Vol. V, J. Wiley and Sons, Inc., New York, 1949, p. 193.
3. Rutherford, K., Redmond, W., and Rigamonti, J., *J. Org. Chem.*, **26**, 5149 (1961).

4. Unlike some aromatic diazonium salts which have been reported to be shock sensitive, diazonium fluoborates and hexafluorophosphates have been found to be remarkably stable in this respect. Furthermore, the success of their use in synthesis is based upon their stability and ability to be smoothly decomposed.
5. Obtainable from the Ozark Mahoning Co., Tulsa, Oklahoma. This reagent will etch glass on prolonged contact. However, we have observed no glassware damage if students thoroughly wash all glassware in previous contact with the acid.
6. c. f. Dannley, R. and Crum, J., "Experimental Organic Chemistry." The Macmillan Co., New York, 1968, p. 29.

## Physics And Environmental Studies

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#### ABSTRACT

To further implement the course curriculum for the Earth Science major and to constructively channel the current interest and concern with the environmental problems, the Department of Physical Science at the University of Arkansas at Monticello introduced a course entitled Environmental Studies this past spring.

As it was necessary to offer both a general education Elementary Physics course in addition to the new course, it was decided to combine the two courses and use an approach similar to that of Edwin Marston, Queens College, Flushing, New York.

Problems of conservation, pollution, and environmental quality were considered by the class whose rank ranged from freshman to senior with widely diverse backgrounds. Physics with minimal math was incorporated sporadically as needed.

The basic structure of class organization allowed students of the Environmental Studies class to present panel discussions for the other students to participate in through comments, questions, and answers.

The process of implementing the planned Earth Science major curriculum began two years ago at Arkansas A&M College, now the University of Arkansas at Monticello. The program, which is one of the offerings of the Department of Physical Science, gained several majors and minors and matured rapidly.

This past fall it was realized that the single person devoted to the earth science and geology area could not offer all of the general and advanced courses currently needed. As finances would not permit the addition of another faculty member at that time and as there was one member on leave for the academic year, faculty time was at a premium.

A general education physics course was to be offered and an additional upper level course for those majoring or minoring in Earth Science was needed for the fall semester. It was, therefore, decided to introduce one of the previously formulated courses, Environmental Studies, at this time. It was further agreed that the two courses, Elements of Physics and Environmental Studies, would be combined and taught together. The temporary combining of these two courses was the apparent solution to the situation previously indicated.

This approach was not altogether unique as Dr. Edwin Martson, Queens College, Flushing, New York, had described an innovative course in an article in the