The Microscale Laboratory

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A Microscale Synthesis of Mauve

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In 1856, William Henry Perkin accidentally discovered mauve when he was trying to synthesize the antimalarial drug quinine. Although it is no longer used as a dye, the synthetic purple dye was quickly accepted by society at the time. It can be argued that mauve's synthesis almost single-handedly began the synthetic organic chemicals industry.

Although Perkin originally started with (impure) allyltoluidine, a different ultimate synthesis from aniline and toluidine isomers was devised (*1*). Even a small "lab-scale" synthesis requires a volume of 5 L of solution and ultimately yields only 2–4 g of mauve as the final product. We have reduced the scale of the synthesis so it can be performed as a microscale organic chemistry experiment. Because the scaling down ultimately yields very tiny amounts of solid product, we also suggest an alternate method for isolating larger, more visibly obvious amounts of the final product.

Experimental Procedure

CAUTION: Petroleum ether is very flammable. Evaporations should be performed under hoods, and no open flames should be present in the lab. Disposal of all chemical wastes should follow standard laboratory procedures.

Place 2.3 mL of water in a 5-mL conical vial and add:

- 52 μL of aniline
- 60 μL of *o*-toluidine
- 122 mg of *p*-toluidine
- 600 μL of 2 N sulfuric acid

Stir using a large spin vane until the reactants have dissolved, heating gently if necessary. After solution, add 30 mg of potassium dichromate in 160 μL of water

Stir for 2 hours. Very soon after the addition of $K_2Cr_2O_7$, the solution will turn an obvious purple. At the end of the reaction time, use a Pasteur filter pipet to draw off the liquid portion, which can be discarded.

Transfer the solid to a ceramic filter with a seated filter paper already in place. Using gentle suction filtration, wash the dark solid with distilled water until the washing is clear. Dry the remaining solid in an oven at ~110 °C for 30 min. Then wash the solid with petroleum ether until the washings are clear; dry again for 10 min at 110 °C.

Wash the remaining solid with a 25% methanol/water solution until the solution runs clear, being very careful to not contaminate the resulting solution. Evaporate this aqueous/alcoholic solution, transferring to a 5-mL conical vial as soon as the total volume allows. After evaporation is complete, add 300 μL of 100% methanol to the remaining solid, shake to dissolve any soluble materials, and use a filter pipet to transfer the liquid to a clean 3 mL vial. Carefully evaporate the liquid in a conical vial until its has a volume of ca. 30 μL or less. As the solution volume gets smaller, the purple color should grow more intense. This final methanol solution contains the ultimate product, mauve (~2 mg yield).

An alternate conclusion to this experiment would be to have all students in the class combine their methanol solutions, and have the teaching assistant or professor evaporate the combined solutions into a larger volume of a more concentrated solution. On this larger scale, the purple color of the mauve can get very intense and is very obvious. A small piece of cotton cloth can be wetted with the solution, rinsed in water, and dried; the dyed cloth makes a nice denouement to the experiment.

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Literature Cited

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