Preparation of 2,5-Dimethyl-1-Phenylpyrrole

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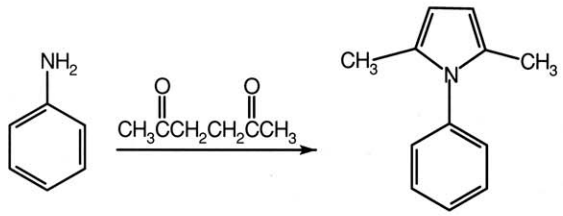
Preparation of heterocyclic compounds has been the subject of few undergraduate experiments. There are many different cyclization reactions that produce nitrogen heterocycles, with the Paal–Knorr synthesis being one of the most general. In this reaction, a 1,4-dicarbonyl compound is heated with ammonia or a primary amine to give a pyrrole.



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The condensation of aniline with 2,5-hexanedione to give 2,5-dimethyl-1-phenylpyrrole (**1**) is an example of a Paal–Knorr synthesis that can easily be carried out in a 3-h undergraduate organic laboratory. The procedure for this experiment was adapted to microscale from previously published procedures (*1*, *2*).

Experimental

Synthesis of 2,5-Dimethyl-1-phenylpyrrole

To a round-bottomed flask fitted with a reflux condenser, add 186 mg (2.0 mmol) of aniline, 228 mg (2.0 mmol) of 2,5-hexanedione, 0.5 mL of methanol, and 1 drop of concentrated HCl. Heat the mixture to reflux for 15 min, and then add it to 5.0 mL of 0.5 M HCl, which is kept cool in an ice bath.

The crystals were collected by suction filtration and recrystallized from 1 mL of 9:1 methanol/water. The yield was 178 mg (52%).

Analysis Data

The product had the following properties.

FT–IR (CCl_4): 3068, 2921, 1600, 1499, 1440, 1402, 1322, and 695 cm^{-1}.

^{13}C NMR ($CDCl_3$): δ = 13.05, 105.64, 127.64, 128.81, 129.06, and 139.00.

^{1}H NMR ($CDCl_3$): δ = 2.04(s,6H), 5.91(s,2H), 7.22(m,2H), and 7.44(m,3H).

mp = 48 °C (lit. 50–51 °C).

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Literature Cited

1. Wolthuis, E. *J. Chem. Educ.* **1979**, *56*, 343–344.
2. Wolthuis, E.; VanderJagt, D.; Mels, S.; De Boer, A. *J. Org. Chem.* **1965**, *30*, 190–193.

A Simple and Inexpensive Gas Trap or Gas Inlet for Microscale Work

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Many chemistry experiments (both microscale or macroscale) require the use of a gas inlet or a gas trap. A simple and inexpensive device can be constructed using an Eppendorf ultramicropipet tip instead of the syringe needle suggested in several laboratory manuals (*1–3*)(Fig. 1). These tips cannot be pulled through the septum as would be the case if simple plastic tubing were used and do not have the disadvantages of syringe needles (cost, handling, and disposal). The lower half of the tapered tip is cut off to prevent the build-up of excess pressure in the reaction vessel. The pipet tip is carefully placed through a rubber septum that has been pierced previously. Any debris that enters the plastic tip from the septum during the insertion process can be blown out. The narrow end of the tip can be attached to a suitable length of plastic tubing by sliding the tip into the tubing (Fig. 2). If a stronger seal is desired or if the tubing is somewhat smaller than the shaft it is to be placed over, the tubing can be heated several seconds using a heat gun before and after the tubing has been placed onto the tip. These devices can be assembled ahead of time and reused. In our laboratories, they have been reused as many as 10 times without any serious problems. If a larger opening is desired a larger pipet tip inserted into a thermometer adapter can be used. Numerous brands of disposable pipet tips for use on 200–1000 µL pipeters will fit into the thermometer adapters and offer an inexpensive alternative to glass tubing or commercial inlet adapters.

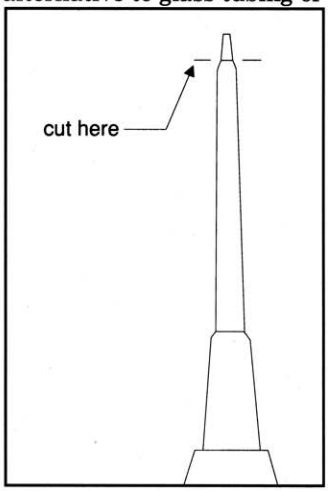

Figure 1. Preparing the ultramicropipet tip.

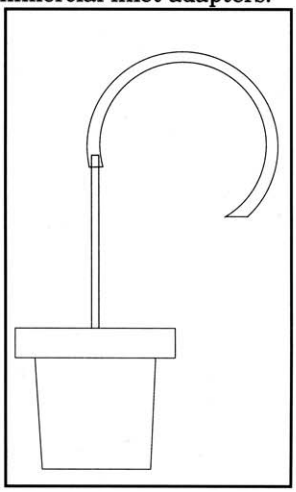

Figure 2. Attaching the tubing.

(*Continued on page A314*)

Literature Cited

1. Mayo, D. W.; Pike, R. M.; Butcher, S. S.; Trumper, P. K. *Microscale Techniques for the Organic Laboratory*; John Wiley and Sons, Inc.: New York, 1991.
2. Mayo, D. W.; Pike, R. M.; Butcher, S. S. *Microscale Organic Laboratory*; John Wiley and Sons, Inc.: New York, 1989.
3. Williamson, K. L. *Macroscale and Microscale Organic Experiments*; D. C. Heath and Co.: Lexington, MA, 1989.

Microscale Filtration

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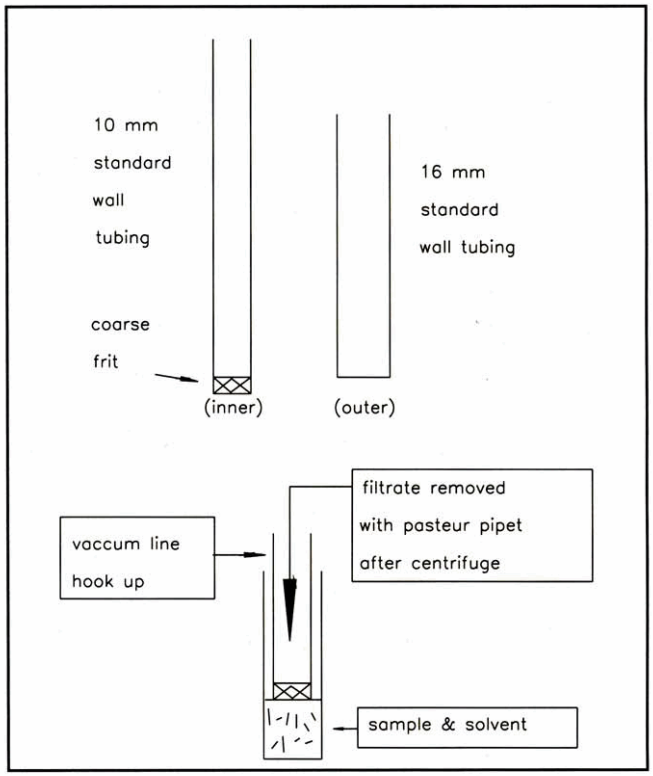
The use of devices such as the Craig tube or filtered Pasteur pipets, for the microscale recrystallization of solids can be quite imposing and frustrating for the average organic chemistry lab student. We would like to offer an alternative filtration device similar to one constructed of polymeric materials[1] used in the biological sciences. This device has been constructed of glass fitted with a coarse frit to avoid the use of polymeric material that might not be compatible with some organic solvents.

The device in the figure, consisting of a 10 mm × 127 mm glass tube closed with a coarse frit that fits snugly in the 16 mm × 95 mm glass tube[2] works quite effectively. The material to be recrystallized is placed in the outer tube with the appropriate solvent and brought to boil as in a normal recrystallization. Once the crystals are regenerated the inner tube is placed into the outer tube and the apparatus is centrifuged for a few minutes. The mother liquor forced into the inner glass tube can be removed easily with a Pasteur pipet leaving the purified material in the outer tube. The tubes are separated and the filtered material is allowed to dry.[3] To dry the product more quickly, the inner tube can be attached to an aspirator or vacuum line to reduce the pressure and remove the solvent residue not removed by the filtration process.



Filtering tube.

A variety of sample sizes and solvent volumes can be accommodated by varying the length and diameter of the tube. The cost for each set is about $11, but if large numbers of tubes are ordered the cost would surely be less. This system offers the advantage of being more durable and more easily handled by the student than the Craig tubes.

[1]Curtin Matheson Scientific, Inc. (CMS #265-256, manufacture #ES13229E).

[2]Our thanks to Tina Miller and Ron Harness of Custom Lab Glass Service (Spencer Chemistry Bldg., 5100 Rockhill Road, Kansas City, MO, 64110) for their fabrication of the filtering tube.

[3]Some residue solvent remains in the tube.