

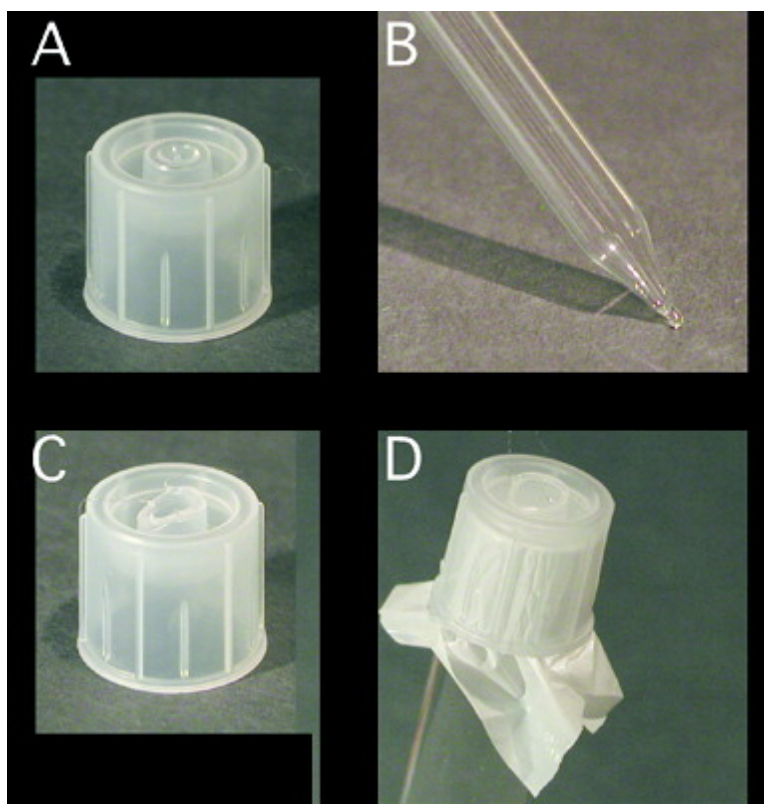
Report

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A Simple and Inexpensive Device for Removal of Solvent from a Large Collection of Sample Tubes

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Introduction. In most high-throughput chemical synthesis processes, whether it be combinatorial or parallel synthesis, one is faced with the task of having to remove solvent from a large number of containers, such as glass tubes or vials. Removal of solvent by evaporation is a necessary step following various synthetic workup or chromatographic procedures. Solvent removal from a collection of containers is typically carried out with a vacuum centrifuge in which centrifugal force is used to prevent mechanical sample loss due to violent solvent boiling or degassing (bumping). Although vacuum centrifuges that can accommodate hundreds of sample containers are commercially available, they are very expensive and require a large amount of laboratory space. We now describe a simple alternative to a vacuum centrifuge that can be assembled for less than a few thousand dollars and is capable of removing solvent from several hundred or more tubes.

Results and Discussion. The new device is shown in Figure 1, and a full description and construction information along with photographs is given as Supporting Information. It is a stainless steel chamber with an O-ring-sealed acrylic lid. The side of the box is fitted with a vacuum port and a four-electrode feed-through. Inside the box at its base is an acrylic heat insulator sheet and a layer of aluminum honeycomb, which serves as a heat-conducting rack for hundreds of test tubes. The honeycomb is heated by six canister heaters inserted at regular intervals in cells of the honeycomb block. Electrical current to these heaters is controlled by an inexpensive solid-state temperature control unit attached to the honeycomb block. A thermometer is inserted into the honeycomb block so that the block temperature can be set to the desired value. The vacuum port of the box is connected via rubber tubing to a refrigerated vapor trap, which is connected to a standard mechanical oil vacuum pump.

The key innovation of this vacuum box device is the use of a chemically inert, microporous cap that provides a barrier to mechanical loss of solvent due to bumping but

allows evaporative loss of gaseous solvent. The porous tube cap is shown in Figure 2 (additional details given in Supporting Information). A commercially available polyethylene push-top cap is used to hold in place a small sheet of microporous polypropylene. A hole in the top of the cap is bored with a flame heated glass rod. This microporous sheet has a thickness of 25 μm and contains a high density ($\sim 55\%$ porosity) of pores of dimension $\sim 0.2 \times \sim 0.05 \mu\text{m}$. It is sold under the commercial name Celgard. The typical use of this material is as a semipermeable membrane in lithium batteries, serving the same purpose as the agar salt bridge used in the typical freshman chemistry laboratory electrochemical cell. This membrane, being made of polypropylene, is hydrophobic and allows rapid diffusional passage of gaseous molecules such as small organics, oxygen, and noble gases, but typically excludes hydrophilic substances, including water. Celgard is sometimes used as a membrane to permit gas exchange between the enclosed aqueous solution and the surrounding atmosphere. However, under vacuum, water was found to readily pass through this membrane (see below). Thus, Celgard serves as a useful mechanical barrier to both hydrophobic and hydrophilic solvents, yet allows passage of any small molecule that enters the gas phase. Furthermore, polypropylene has excellent chemical resistance properties, and Celgard is an inexpensive material. The polyethylene push-top cap can be reused after washing, whereas the Celgard sheet is disposable.

Using the device described above and with a test tube rack temperature of 40 $^{\circ}\text{C}$, complete solvent removal from an array of 300 tubes (18-mm o.d.) filled two-thirds with either methanol/water (1/1) or acetonitrile/water (1/1) was achieved in less than 24 h using a standard oil vacuum pump and a low-temperature ($-95 \text{ }^{\circ}\text{C}$), high-capacity vapor trap. Other test tube sizes can be used with the appropriate choice of cell size in the honeycomb rack (see Supporting Information) Although an occasional drop of solvent was seen on the outer wall of the tube during the solvent evaporation period (due to passage of solvent through creases in the Celgard sheet), recovery of solute was $> 99\%$ in all 300 tubes. It is not recommended that the device be used to evaporate solvent from radiolabeled compounds, since the possibility of contamination of the chamber cannot be ruled out.

The solvent removal device described in this Report should be useful in any laboratory where solvent removal from a large array of tubes (or other containers) is required. The device can be easily constructed in most machine shops for a cost of less than \$2000 (materials and labor). An oil vacuum pump ($\sim \$1500$) and a low-temperature vapor trap ($\sim \$2500$) are also required. This is a small fraction of the cost of a vacuum centrifuge device capable of handling several hundred sample tubes.

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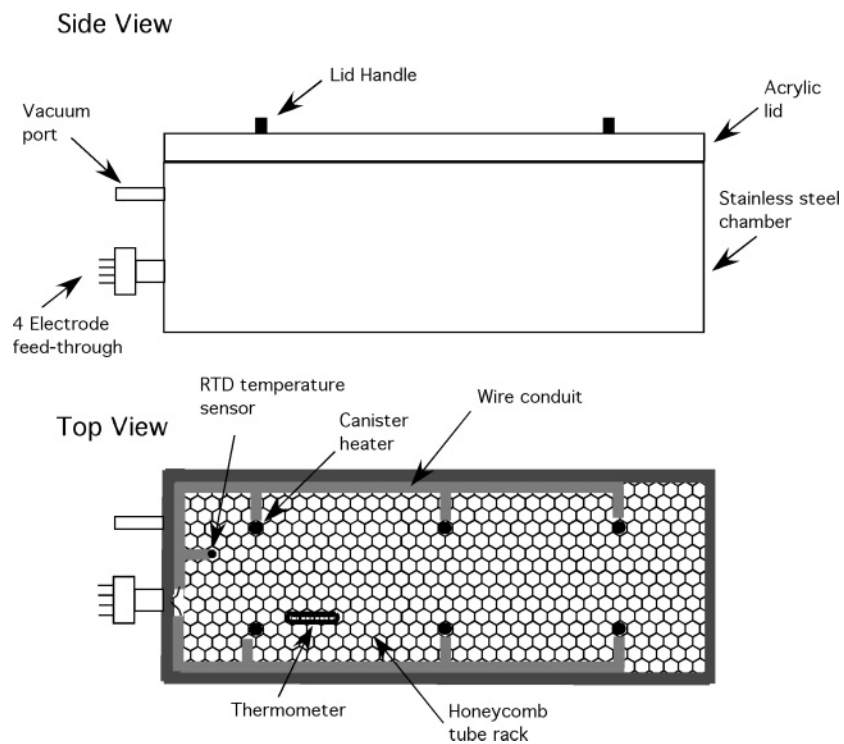


Figure 1. Schematic view of the vacuum box. A construction diagram and photographs are given in Supporting Information.

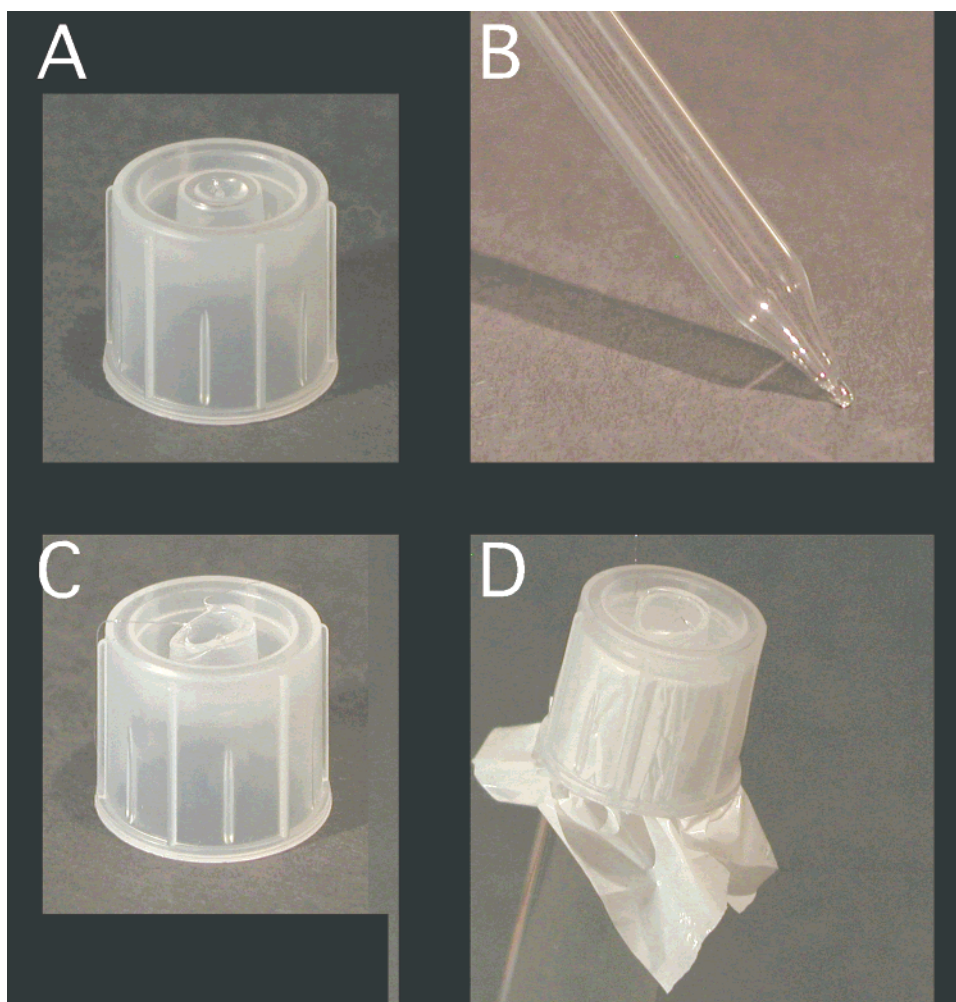


Figure 2. View of the porous cap. (Panel A) The polyethylene push-top cap; (panel B) a glass rod used to bore a hole in the push-top cap (made from a Pasteur pipet using a Bunsen burner); (panel C) push-top cap with small hole bored in top center with the flame-heated glass rod; (panel D) fully assembled cap showing a small square of white Celgard sheet held tightly in place with the push-top cap. A simple hand tool should be used to push the cap into position (to avoid hand injury). Additional details are given in Supporting Information.

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Supporting Information Available. Additional information as described in the text. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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