Suggested Modifications to a Distillation-Free Solvent Purification System

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Suggested Modifications to a Distillation-Free Solvent Purification System

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The method usually used for drying and purifying organic solvents involves distillation from a suitable drying agent. The drying agents typically used include Li, Na, K, CaH_2, and $LiAlH_4$. While these methods provide solvents in purified, dry form, the use of such highly reactive drying agents can be quite dangerous for either the novice or the experienced researcher. Additionally, the maintenance of distillation equipment and the quenching of the spent drying agents can be somewhat laborious. Therefore, the development of methods that avoid such procedures is desirable.

In 1996, Grubbs and coworkers described a method of purifying a variety of organic solvents without distillation (*1*). Their method provides several advantages over distillation from a suitable drying agent. First, large quantities of solvent are available on demand. Second, since no extremely reactive reagents, no heat, and no electricity are utilized, this procedure is much safer than distillation. Finally, this method requires no quenching of the spent drying agent. Since many laboratory fires result from quenching distillation residues after use, this is a significant advantage. Their method, however, requires a substantial investment of laboratory space and research funds.

In an attempt to further improve the system reported by Grubbs and coworkers and render it more amenable to use in undergraduate laboratories, we made several modifications that have resulted in savings of not only space but also time and money. The general procedure is given below, and a complete description is available online.[W]

Procedure

All solvents (HPLC grade, 4-L bottles) are pre-dried by storing over activated 4-Å molecular sieves for at least 1 week. Solvent reservoirs are then filled via cannula with the pre-dried nitrogen-sparged solvent. During collection of solvent (just prior to use), solvent is passed under nitrogen pressure (~7 psi) through a single column of activated alumina, which effectively removes water.

Owing to the high cost of commercially available oxygen scavengers and the additional laboratory space required by a second column, we have omitted the use of a column filled with an oxygen scavenger to deoxygenate solvent. Instead, we degas solvents by sparging with nitrogen both before filling the solvent reservoir and after solvent collection, immediately before use. Between the column and the solvent collection vessel, the solvent passes through an in-line filter (2 or 7 μm), which removes any particulate matter. After collection, the solvent is sparged with nitrogen to ensure that it is free of dissolved oxygen. This method provides ultra-dry and O_2-free solvent and has eliminated the need for either plumbing directly into a glove box or the use of a vacuum manifold.

Discussion

The approximate cost of this system is $1200 per unit. This is approximately half of the cost of the previously described method (*1*), not including the cost of the nonrenewable and expensive oxygen scavenger. This method has been used successfully for the following solvents: pentane, hexanes, benzene, toluene and dichloromethane. Unfortunately, we have not been successful in purifying ethereal solvents (e.g., diethyl ether or tetrahydrofuran) by this method. A complete discussion of the problems encountered with such solvents is included in the full paper online.[W]

[W]Supplemental Material

A detailed description of the apparatus and its use, with diagrams, a list of hardware required, and further references and notes are available in this issue of *JCE Online*.

Literature Cited

1. Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518–1520.