A Practical Setup for the Transfer of Thermally Unstable and/or Insoluble Reagents

How often have you encountered the problem where cannulation of a reagent is impractical because of its thermal instability and/or insolubility?

To alleviate the problem, we have found the following apparatus ideally suited for the transfer of thermally unstable and/or insoluble compounds (Figure 1). The design and use of this piece, to the best of our knowledge, is novel. The apparatus consists of a glass cooling bath A and a round bottom flask B. Attached to A is a valve, a female ground glass joint which is connected to B, and a male ground glass joint which can be connected to a 50–1000 mL angled two- or three-neck, round-bottom flask (C). The valve consists of a male joint with a threaded Teflon® nut and corresponding external glass screw threads. Flask B is constructed by attaching a male ground glass joint to the bottom of a 50–1000 mL angled two- or three-neck, round-bottom flask.

Cooling bath A, which has a slot in it to allow easy attachment, is connected to B containing the selected liquid. Teflon sealing tape and a Delrin® plastic joint clip provide a tight seal. An electric motor clamped above B rotates a specially designed vertical rod equipped with a paddle and two extendable blades (Figure 2). It is essential to use a paddle that forms a good fit to the bottom of the female ground glass joint of B.

Agitation in flask C is achieved by placing an egg-shaped magnetic stirring bar large enough to stir the reaction mixture effectively. Flask C is clamped over the top of a magnetic stirrer whose flat top allows cooling or heating baths to be placed upon it. Temperatures inside B and C may be monitored by inserting a thermometer or a thermocouple through one of the necks. An inert atmosphere may be established through the use of rubber septa and syringe needles.

Flask B can be used for the preparation of lithium reagents at -78 to 25 °C; an addition funnel can be incorporated for large-scale work. Once the lithium reagent is prepared, the valve is opened and the organolithium reagent flows into flask C containing a solution of the appropriate electrophile.

Our laboratory has employed this apparatus extensively during the preparation of 1,1,2- and 1,2,2-trisubstituted 1,2-dihydroanthracenilenes via the stereospecific 1,4-addition of organolithium reagents to unprotected 1- and 2-naphthalenecarboxylic acids at low temperature.


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“Please Bother Us.”

Professor Hinze at Wake Forest University kindly suggested that we offer these zwitterionic surfactants. They have been utilized for the extractive separation of hydrophilic proteins, steroids, and vitamin E. Their lack of absorbance in the ultraviolet region and their inducement of phase separation at moderate temperatures are two of the advantages that allow them to be used for heat-sensitive compounds such as vitamin E.


47,260-3 N,N-Dimethyl-N-[3-sulfooxy]propyl]-1-nonanaminium hydroxide, inner salt, 98%.
47,258-1 N,N-Dimethyl-N-[3-sulfooxy]propyl]-1-decanaminium hydroxide, inner salt, 98%.

Naturally, we made these useful surfactants. It was no bother at all, just a pleasure to be able to help.

Do you have an innovative short-cut or unique laboratory hint you’d like to share with your fellow chemists? If so, please send it to Aldrich (attn: Lab Notes, Aldrichimica Acta). For submitting your idea, you will receive a complimentary, laminated periodic table poster (Cat. No. Z15,000-2). If we publish your Lab Note, you will also receive an Aldrich periodic table turbo mouse pad (Cat. No. Z24,409-0). It is Teflon®-coated, 8½ x 11 in., with a full-color periodic table on the front. We reserve the right to retain all entries for future consideration.


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