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Technical Notes

A Practical Device for the Transfer of Liquid Reagents at Low Temperature

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Abstract:

A convenient and practical device designed for cooling and transferring air-sensitive reagents at low temperature during laboratory syntheses is described. The setup can be easily constructed from a cannula and a cooling/insulating sheath with an optional circulating chiller.

Transfer of reagent at low temperature between processing vessels is easily achieved at pilot plant or production scale using insulated or refrigerated lines. However, this becomes a formidable task to achieve in the laboratory as reagents tend to warm up during the cannulation process. Jacketed addition funnels/flasks are quite cumbersome and offer limited help. Consequently, there exists a disconnect between benchtop and plant scale process development involving temperature-sensitive reagents. The elaborate setup for the transfer of thermally unstable reagents reported recently by Mortier et al.[1] prompted us to disclose a simple and practical device we have developed and have been using in these laboratories to address the problems associated with the cannulation of cold reagents at laboratory scale.

A standard double-tipped needle was encased in a flexible plastic or rubber tube fitted with a T-adapter at each end. The tips of the needle were allowed to protrude from the tubing through rubber septa fitted on the straight arms of the T-connectors (Figure 1). For less demanding tasks, for example, cannulation of reagent at > −5 °C, the side arms of the connector were capped with rubber septa, and the tubing provided adequate insulation for the cold liquid being transferred. For more demanding situations where the temperature needed to be strictly controlled, the side arms of the adapters were connected to a supply of chilled fluid, for example, from a commercially available refrigerating circulator. The reagent could be either prechilled or transferred from a room temperature reservoir and cooled to the desired cryogenic temperature in the cannula during the transfer. In cases where a circulating chiller is not available, cooling fluid could be led slowly from a funnel through the tubing to achieve the same purpose. Alternatively, the reagent to be transferred could be maintained at low temperature by passage of the cannula through a septum-capped funnel filled with, for example, a 2-propanol−dry ice mixture, atop the receiving flask and/or the reservoir (Figure 2). Optionally, the temperature of the exiting reagent could be monitored by Teflon-taping a thermocouple to the tip of the needle inside the receiving flask. Decreased solubility of reagents at lower temperature should be taken into consideration to prevent clogging.

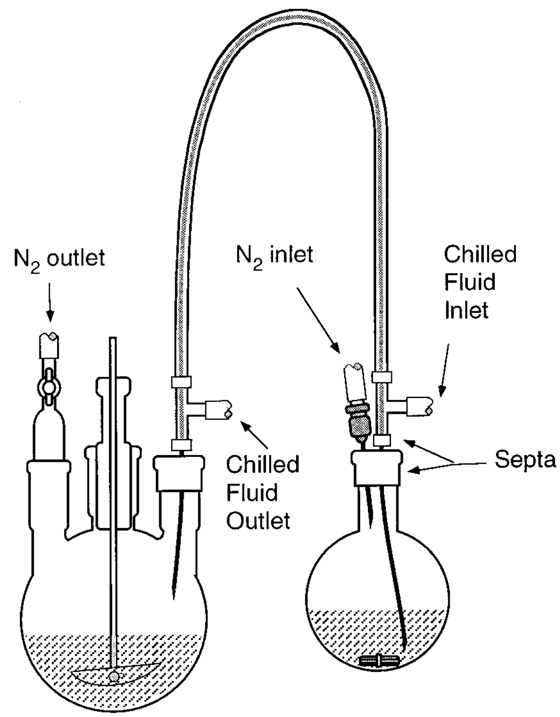

Figure 1.

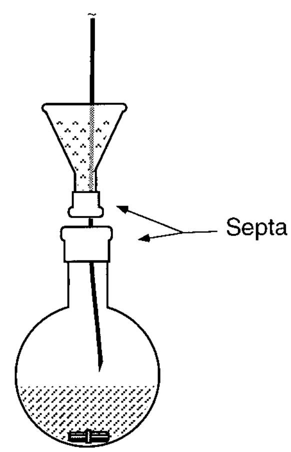

Figure 2.

(1) Mortier, J.; Vaultier, M.; Cantegril, R.; Dellis, P. *Aldrichimica Acta* **1997**, *30*, 34.

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This simple setup does not require any special custom-made glassware and is not limited by flask size or shape, stirrer type, or hood space. The in situ cooling allows for the addition of cold reagent directly from a measuring container at room temperature. Additionally, this device minimizes the contact between the cooling bath liquid and the reagent, a risk commonly associated with these operations.

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