Lab Notes

Effective Cooling System in Bulb-to-Bulb Distillation

Bulb-to-bulb distillation is a useful laboratory technique for the small-scale purification of liquid substances. In this microscale, short-path technique, a small quantity of material is redistilled using several glass bulbs connected in line. In some Kugelrohr models, heating is carried out by a built-in oven, and control of both temperature and stirring speed is performed by an integrated electronic controller. The flask containing the product to be distilled and the collecting bulbs are connected to a rotary device (**Figure 1**).

Traditionally, cooling of the collecting bulbs has been carried out with dry ice in acetone (-78˚C), especially when working with low-boiling substances or when distilling under vacuum. This cooling technique has several drawbacks:

a) Acetone is usually applied by a piece of cotton held with tweezers and may accidentally drip on the Kugelrohr apparatus and attack the plastic cover. It may also drip inside the housing, through the control buttons, and damage the electronic circuit boards. Acetone can also dissolve the plastic parts of the front panel and lead to the fusion of the buttons to the housing.

b) Acetone, because of its low flash point (-18 ˚C), could ignite if it contacts the electrical system since the rotor can generate sparks.

c) From an operational point of view, the ground joints, which connect the glass bulbs, usually are not lubricated with vacuum grease to avoid contaminating the final distillate during its recovery. As a result, and even though the bulb-to-bulb seal is usually good, a reabsorption of acetone through the joints could happen principally when the distillation is carried out under vacuum. In this case, contamination of the distillate with acetone could become a problem, especially when dealing with low-boiling substances.

To avoid these problems, we have designed and used extensively a new, safe, and simple cooling system. In this system, the distillation bulbs are cooled with dry ice without the use of acetone. As containers of solid dry ice (coolers), we use cheap and readily available plastic or cardboard cylindrical tubs of 4 to 5 cm in diameter and 10 cm in length (**Figure 2a**).

To adapt the cylindrical container to the shape of the cooling bulb and to increase the cooling surface, it is recommended that two circular cuts be made on opposite sides of the bottom of the cylinder (**Figure 2b**).

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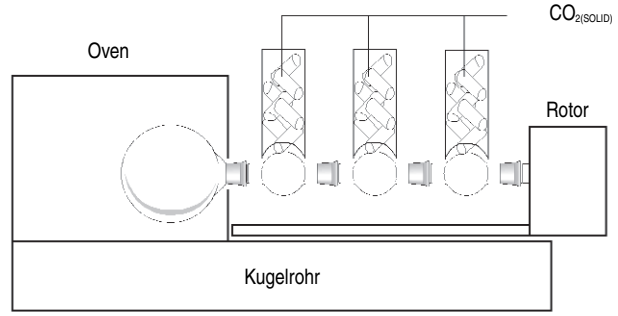

Figure 1: Bulb-to-bulb distillation apparatus with a cooling system of open cylinders filled with dry ice.

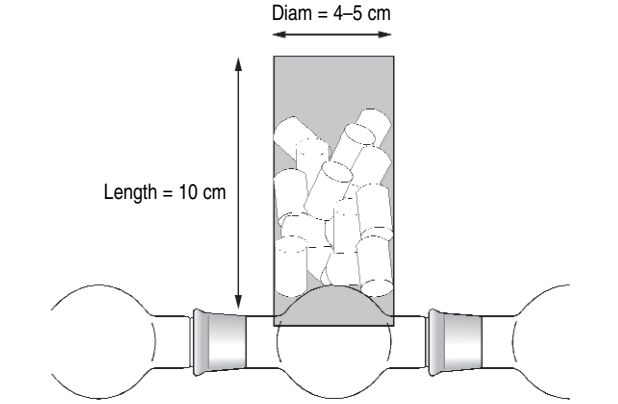

Figure 2(a): Detail of the cylindrical dry-ice container of the cooling system.

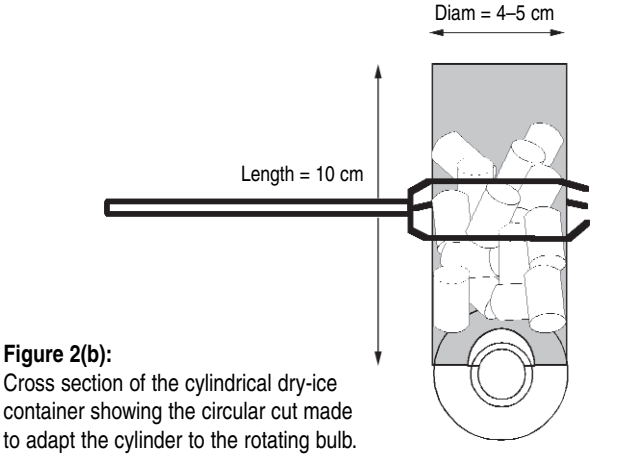

Figure 2(b):
Cross section of the cylindrical dry-ice container showing the circular cut made to adapt the cylinder to the rotating bulb.