Using Ice-Cooled Condensers in Chemistry Laboratory

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A variety of novel condensers have been described in this *Journal* including systems recirculating cooling water to prevent floods *(1, 2),* a wrap-around trough condenser using a variety of coolants designed for use with sublimation tubes *(3),* a one-piece apparatus combining a refrigerated dropping funnel and reflux condenser *(4),* as well as a high-efficiency condenser for low-boiling liquids *(5).* Air condensers have been described *(6)* as well as condensers that depend upon evaporation to overcome inadequate cooling from warm tap water *(7).*

The ice-cooled condenser described in this article is a versatile device made from readily available glass tubing and can be used for distillation or for refluxing. It was originally designed for use in college-level general chemistry or high school laboratories where water cooling is impractical or even impossible; however, it is also compatible with most microscale glassware kits and may be useful in organic labs. The ice-cooled accessory can be produced with either 14/10 or 14/20 ground glass joints, making it compatible with most commercially available microscale or small scale kits.

Design

The ice-cooled condenser can be used in setups for refluxing or for distillation (see Figure 1). The support is needed to prevent breakage of the inner tube when the ice is added. A section of rubber tubing is attached to the water outlet for drainage.

Materials Needed

All glass parts needed to make the ice-cooled condenser are listed in Table 1 with approximate quantities for the construction of one device.

Construction

The steps involved in making an ice-cooled condenser are described in detail below and shown in Figures 2, 3, and 4. The inner piece consists of the narrow inside tube and support brace. The outer piece is the large jacket (and water outlet).

Inner Piece

1. The 14/20 female (outer) joint (snap cut to a length of 35 mm) is sealed to the 12-mm standard wall tubing (see Figure 2a). The assembly is then cut to a length of 155 mm and the end is “flare” cut.
2. The brace, made of 5-mm solid glass rod, is attached just below the female joint and then cut to a length of 25 mm (Figure 2b).

Outer Piece

1. A segment of 51-mm standard glass tubing is sealed with a round bottom (Figure 3a).
2. A drip tip for the 14/20 male (inner) joint is cut at a 45° angle, then fire-polished (Figure 3b).

Table 1. Required Materials

Part	Size	Amount
Glass tubing (standard wall)	51-mm o.d.	8 in.
Glass tubing (standard wall)	12-mm o.d.	8 in.
Glass tubing (medium wall)	8-mm o.d.	3 in.
Glass rod	5 mm	2 in.
Outer (female) joint	14/20	1
Inner (male) joint	14/20	1

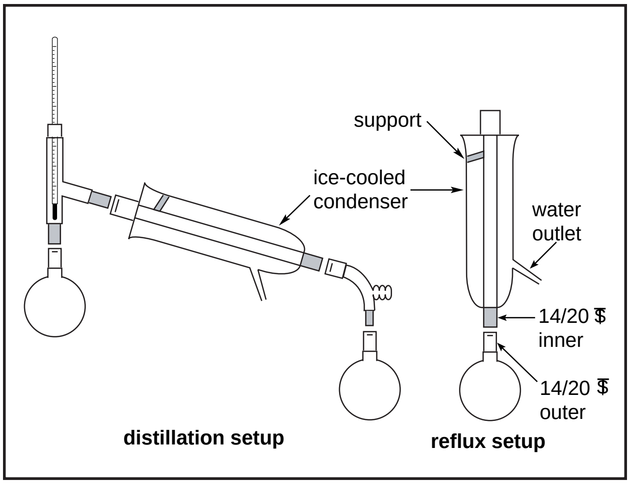

Figure 1. Ice-cooled condenser in reflux and distillation setups.

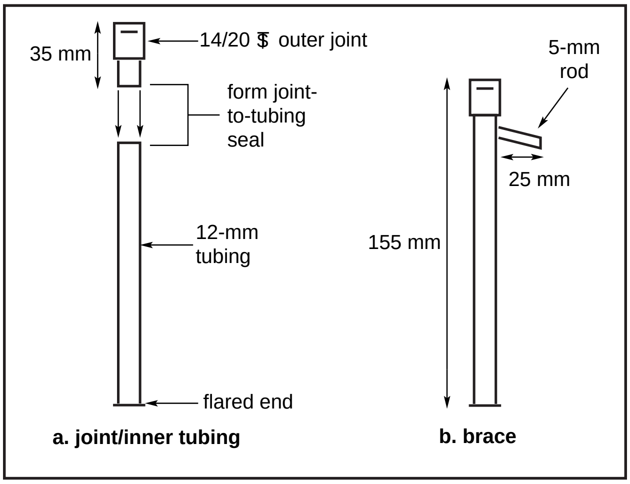

Figure 2. Construction of inner piece.

Assembly

1. The inner piece is positioned inside the outer piece, then sealed at the bottom as shown in Figure 4.
2. The male (inner) joint piece is sealed onto the bottom and a hole is blown to connect the joint to the inner piece.
3. While the apparatus is still hot, a hole is blown in the outer jacket (for the outlet tube). Medium wall glass tubing is sealed to the outer jacket at a 65° angle, and then is cut to a length of 1 in. and its end fire-polished.
4. The brace is sealed to outer jacket.
5. The apparatus is annealed first by flame then in a 1040 °F oven.

Ice-cooled condensers are commercially available from Glassblowers.com, Inc.[1]

Applications

Experiments using ice-cooled condensers and simple household materials are described briefly. One requires reflux and the other distillation.

Synthesis of Ethyl Salicylate from Aspirin

The ice-cooled condenser may be used in any experiment that involves refluxing such as the synthesis of ethyl salicylate from aspirins using household chemicals *(8)* and simple equipment (e.g., no round bottom flasks or water-cooled condensers). Previously the 2-h refluxing for the esterification step was done in an Erlenmeyer flask placed on a hot plate with a cold finger inserted. The cold finger (made from a test tube) had to be removed frequently in order to replenish the ice. An ice-cooled condenser (with a round bottom flask or inserted in a cork using an Erlenmeyer) would be much more convenient and requires less ice. The device holds about 130 g crushed ice. At a hot plate temperature sufficient to boil ethanol, an additional 100 g ice had to be added during the 2-h period.

The simplicity of the setup is ideal for college-level general chemistry or high school laboratory classrooms that may not be as well equipped for water-cooling as are most organic labs.

Separation of Acetone from Nail Polish Remover

Ice-cooled condensers also can be used in distillation setups (see Figure 1). Acetone-containing nail polish remover *(9)* was chosen because the acetone can be removed using a hot water bath as the heat source. The nail polish remover[2] also contains water, yellow dye, gelatin, fragrance, propylene carbonate, glycerol, and the dimethyl esters of adipic, succinic, and glutaric acids. Low-boiling acetone is readily removed by distillation.

When a 25-mL volume of the product was distilled for 30 min, 18 mL of clear liquid boiling at 55–57 °C was produced, leaving behind water and an oily residue in the distilling flask. The original quantity of ice used to pack the condenser did not have to be replenished during the distillation.

Ice-cooled condensers such as the one described here have been used in general chemistry laboratory classes at Drexel University and in Philadelphia high schools (Science in Motion[3]).

Hazards

Acetone and ethanol are flammable. Gloves and safety glasses should be worn.

Acknowledgment

We wish to thank Tom Cachaza, our talented glassblower, who patiently made one prototype after another and cheerfully answered our questions as we wrote the section on construction of the device. This work was supported by a grant from the Commonwealth of Pennsylvania, Department of Education (Science in Motion van project).

Notes

1. Glassblowers.com, Inc., Scientific Glassblowing Homepage. *http://www.glassblowers.com* (accessed Dec 2002).
2. Cutex regular nail polish remover.
3. Science in Motion is a program that transports equipment and expertise as requested by teachers in the school system. The Web address for the Philadelphia site is *http://www.philasim.org* (accessed Dec 2002).

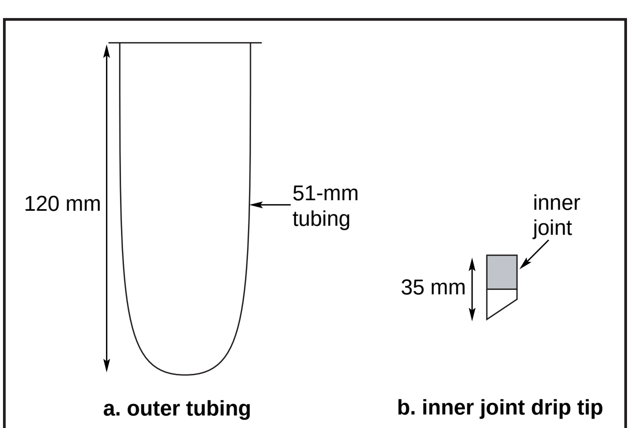

Figure 3. Construction of outer piece.

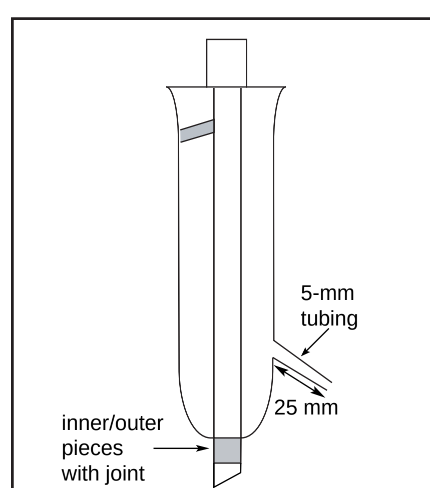

Figure 4. Assembly.

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