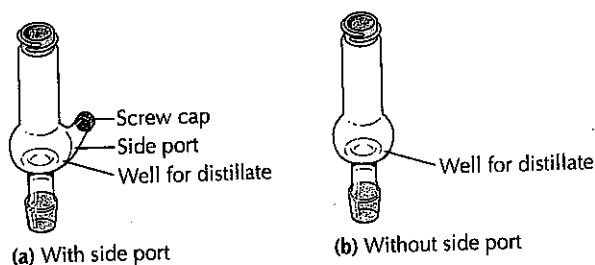


Using a Hickman Distilling Head

Another type of standard taper microscale distillation apparatus consists of a Hickman distilling head (Figure 13.11) and a 3-mL or 5-mL conical vial or a 10-mL round-bottomed flask. The Hickman distilling head also serves as the receiving vessel, an arrangement that considerably reduces the holdup volume. Vapors condense on the upper portion of the Hickman still and drain into the bulbous collection well. One version of the Hickman still has a port at the side for easy removal of the condensate (Figure 13.11a).

FIGURE 13.11
Hickman distilling heads. The condensate collects in the well at the bottom of the head in both versions.



Grease is not used on ground glass joints of microscale glassware because its presence could contaminate the product.

It may be necessary to wrap the distillation vial loosely with glass wool to prevent rapid heat loss, but do not wrap the well of the Hickman distilling head.

Setting up the apparatus. To carry out a microscale distillation, select a conical vial or 10-mL round-bottomed flask appropriate for the volume of liquid to be distilled; the vessel should be no more than two-thirds full. Use a Pasteur pipet to place the liquid in the vial and add a magnetic spin vane or a boiling stone. Attach the Hickman distilling head to the vial with a screw cap and O-ring. Usually an air condenser or a water-cooled condenser (for particularly volatile liquids) is placed above the Hickman distilling head to minimize the loss of vapor (Figure 13.12). Clamp the assembled apparatus at the Hickman distilling head, and place the vial in an aluminum heating block. If you are using a spin vane, turn on the magnetic stirrer.

Carrying out the distillation. Begin heating the aluminum block slowly to a temperature 20° – 30° C above the boiling point of the liquid being distilled. Position a thermometer inside the condenser and the Hickman distilling head, with the top of the thermometer bulb aligned with the bottom of the head's collection well, as shown in Figure 13.12. Clamp the thermometer firmly above the condenser.

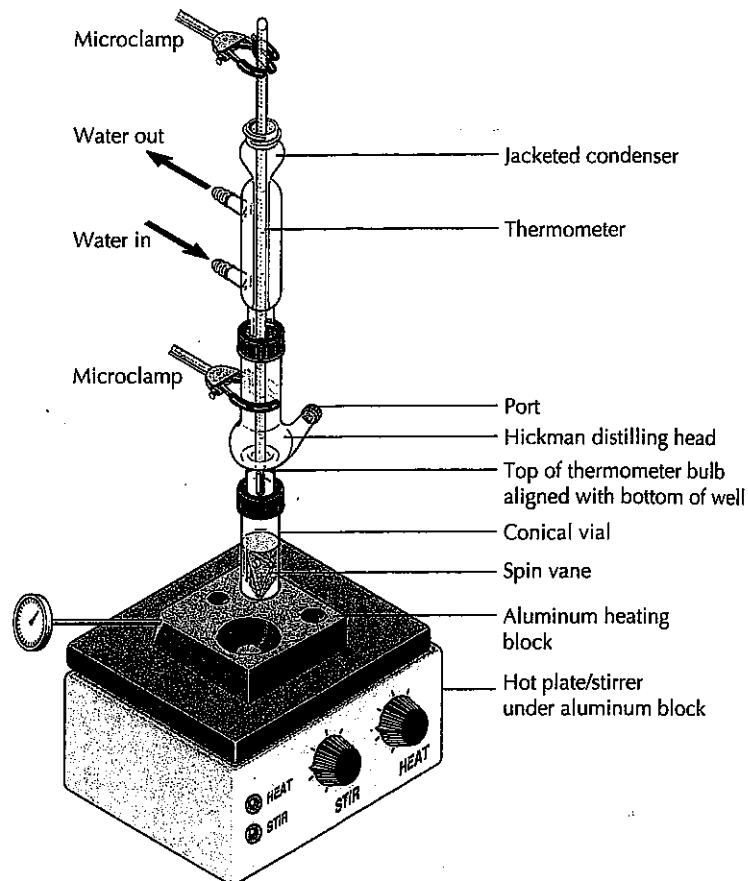


FIGURE 13.12
Standard taper apparatus for a microscale distillation using a Hickman distilling head with a side port.

Removing the distillate. After the liquid in the vial boils, you should see a ring of condensate slowly moving up the vial and into the Hickman distilling head. The temperature observed on the thermometer rises as the vapor reaches the thermometer bulb. You may also see the upper neck of the Hickman distilling head become wet and shiny as the vapor condenses and begins to fill the well. The distillation must be done at a rate slow enough to allow the vapor to condense and not evaporate out of the condenser.

The collection well has a capacity of about 1 mL, so the distillate may need to be removed once or twice during a distillation. Open the port and quickly remove the distillate with a clean Pasteur pipet. Alternatively, withdraw the distillate using a syringe inserted through the plastic septum in the screw cap of the port.