

## DISTILLATION: SEPARATION OF A 2-METHYLPENTANE / HEPTANE MIXTURE

*Work in Pairs (availability of GC)*

In this exercise a 1:1 mixture of 2-methylpentane and heptane will be separated by ***fractional distillation*** using the Hickman-Hinkle spinning-band still head. The percent composition of each of four fractions will be determined by gas chromatography.

Assemble the distillation apparatus pictured in the Pavia Laboratory Manual, 4<sup>th</sup> Ed. on page 723 (Figure 15.10) using a 3 mL conical vial. Make sure that the spinning band is vertical and that it rotates freely when the stirrer is turned on. With a 1 mL measuring pipet, add 1 mL 2-methylpentane and 1 mL of heptane to a 3 mL conical vial. Reassemble the apparatus. Be sure the bulb of the thermometer is below the collection well of the still head. Insulate the lower neck of the still head by wrapping it with a small amount of cotton. Place a *loose tent* of aluminum foil below the collar of the still head to reflect the heat away from it. **DO NOT PLACE THE FOIL ON THE SURFACE OF THE STIRRER/HOTPLATE.**

Set the spinning band to a low rate of rotation as the pot is gently heated to boiling (a hot plate setting of about 5 to 6). Once ***reflux*** is observed in the neck of the still head, increase the rate of rotation to maximum. You will need to increase the heat as the distillation proceeds (a hot plate setting of about 7 to 8). Adjust the rate of heating so that the ring of reflux is maintained at the bulb of the thermometer.

Collect the first ***fraction of distillate*** (about 0.5 mL) by withdrawing the ***condensate*** through the still head side arm with a Pasteur pipet, place it into a labeled and tared small vial, then immediately cap it. Record the internal temperature at the time of the collection. Collect a second and third fraction in the same manner. Allow the system to cool to room temperature. The ***pot***, containing the fourth “fraction” (condensed vapor) should be removed, capped, and labeled. If any condensate collects in the still head collar, collect it as an additional fraction.

Analyze each fraction by gas chromatography. Use a 1  $\mu$ L injection. Normalize the chromatogram if necessary. Report the % composition of each fraction.