

B. Determination of Boiling Point

Set up a Thiele tube filled with oil as indicated in Figure 4-2. Examine the oil to make certain that it is *not cloudy or contaminated* with any substance. If the oil appears cloudy or contaminated, consult with the instructor about replacing the oil. The oil bath will be used to heat the boiling-point sample evenly. Remember that hot oil can spatter or ignite if heated too quickly or too strongly.

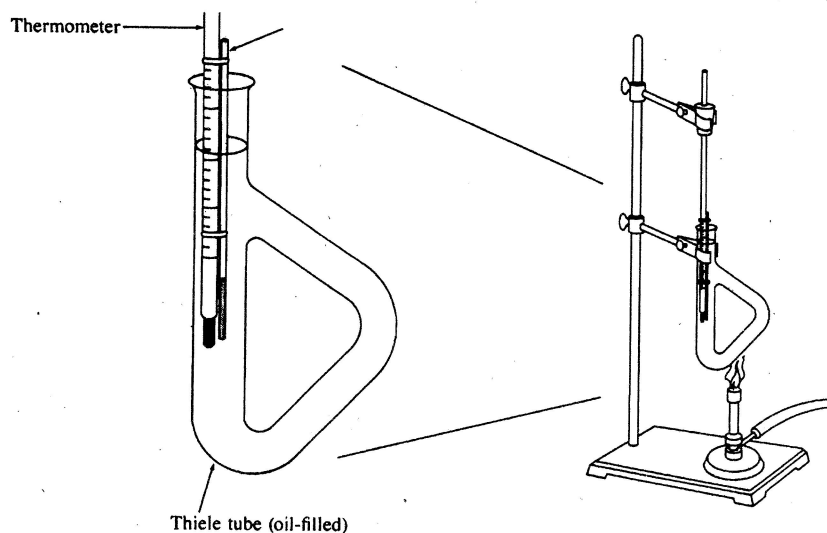


Figure 4-2. Thiele tube oil bath for determinations of boiling or melting point. Exercise caution when dealing with hot oil. Replace the oil if it appears cloudy. Make sure the thermometer and sample tube are dry when placed in the oil: moisture will cause the oil to spatter when heated.

Obtain a clean 10×75-mm semimicro test tube, which will contain the boiling-point sample. Attach the test tube to the lower end of your thermometer with two small rubber bands.

Obtain an unknown liquid for boiling-point determination from your instructor, and record the identification code number of the unknown in your notebook and on the lab report page. Transfer part of the unknown sample to the small test tube, until the test tube is approximately half full.

Obtain a melting-point capillary. If the capillary tube is open at both ends, heat one end briefly in a flame to seal it off. Using a glass file, carefully cut the capillary about 1 inch from the sealed end. Do *not* fire-polish the cut end of the capillary. Place the small portion of capillary *sealed end up* into the boiling-point sample in the test tube.

The capillary has a rough edge at the cut end, which serves as a surface at which bubbles can form during boiling. The capillary is filled with air when inserted, sealed end up, into the liquid, and the presence of this air can be used to judge when the vapor pressure of the unknown liquid reaches atmospheric pressure.

Lower the thermometer/sample into the oil bath so that the bulb of the thermometer and the sample are level with the upper branch of the Thiele tube's side arm. The Thiele tube is constructed in such a manner that when the side arm is heated, oil will circulate from the side arm into the main chamber. This makes it unnecessary to stir the oil bath.

Begin heating the side arm of the Thiele tube with a low flame so that the temperature rises by one or two degrees per minute. Watch the small capillary tube in the unknown sample while heating. As the sample is heated, air in the capillary tube will begin to bubble from the capillary. As the air bubbles from the capillary, it is gradually replaced by vapor of the unknown. As the liquid begins to boil, the bubbles coming from the