

MICRO-BOILING POINT DETERMINATION

1. Preparing Sample
 - a. In a very small (1 mL) test tube place
 - i. About 0.25 mL liquid
 - ii. Open end of a capillary tube
 - b. Attach test tube to thermometer
 - i. Use a small black rubber band attached at upper portion of test tube
 - ii. Liquid in the test tube must be centered with mercury bulb of thermometer
2. Thiele Tube Procedure
 - a. Thiele tube
 - i. Fill with glycerin to level 1 cm above top of upper side-arm joint
 - ii. Attach to ring stand using utility clamp
 - (1) Place directly above base of ring stand
 - b. Suspend thermometer from ring stand using
 - i. Thermometer adapter turned upside-down (rubber portion down)
 - ii. Utility clamp (attach to rubber portion of adapter only)
 - iii. Suspend thermometer assembly above Thiele tube such that
 - (1) Thermometer bulb/sample are completely submerged in glycerin
 - (2) Rubber band must be 1 cm above glycerin
 - (3) Do not seal Thiele tube opening with thermometer adapter
 - c. Heat lower arm of Thiele tube
 - i. Use Bunsen burner
 - (1) WARNING: most volatile liquids are flammable
 - ii. Heat middle of lower arm
 - iii. Move heat source as necessary
 - (1) Use slow sweeping movements as necessary
 - iv. Heat until the liquid begins to boil
 - v. Turn heat source (Bunsen burner) OFF
 - vi. Observe the bubbles emitted from the open end of the capillary tube
 - (1) Micro-boiling point determination
 - (a) Watch for cessation of bubbles
 - (b) Liquid begins to go up into the capillary tube
 - (i) Determination made exactly at this point
 - (c) Determine bp to 1st decimal place
 - d. To repeat the micro-boiling point determination on the same sample
 - i. Remove liquid from the open end of the capillary tube by shaking
 - ii. Reinsert empty capillary tube into the liquid in the bottom of the test tube
 - iii. Repeat the above heating and cooling sequence
 - (1) It is not necessary to cool to room temperature before 2nd determination
 - (2) Only necessary that glycerin be below boiling point of sample
3. Water Bath Procedure
 - a. A water bath may be substituted for the Thiele tube if bp is less than 100°C
 - b. Water bath may be heated using Bunsen burner or hotplate