

# How to Use a Thiele-Dennis Tube

## Introduction

The purpose of this experiment is to become familiar with the Thiele tube which is used to measure melting points and to understand the value of using the melting point as a tool for indicating the purity of a mixture of compounds.

## Concepts

- Melting point
- Chemical purity

## Background

The *melting point* is defined as the temperature at which solid and liquid phases are in equilibrium. At the melting point, a solid will begin to melt into a liquid or, inversely, a liquid will begin to solidify. (This is also known as the *freezing point*.) The melting point of a material is one of the physical properties that can help identify a compound and is also a good indication of a compound's purity. A compound which is pure not only has a characteristic melting point but the process of melting occurs over a very narrow temperature range. In situations where the compound in question is not pure, the melting point is not as distinct. Even though the change is quick, a melting point usually refers to a temperature range and not a single melting point number. If a material is very pure, its melting point range is usually two degrees Celsius or less. A melting point range of greater than two degrees will usually indicate some impurities in the sample.

Thiele-Dennis tubes are designed to give excellent convection and heat transfer for melting point and boiling point determinations. The unique design creates convection currents when the oil inside the tube is heated, allowing the oil to flow continuously through the tube without stirring or shaking. The recommended heating fluid is silicone oil or vegetable oil. Fill the tube to the level shown—the oil will expand when heated.

## Materials

Silicone oil, 50 mL	Rubber band
Stearic acid, 5 g	Stopper, 2-hole, size 3
Bunsen burner	Support stand
Capillary tube, closed-end	Thermometer
Clamp	Thiele-Dennis tube
Graduated cylinder, 50 mL	

## Safety Precautions

*Remind the students never to handle hot glassware with bare hands; always use insulated gloves or beaker tongs. Do not use chipped capillary tubes; use only new tubes in good condition. Wear chemical-splash goggles, chemical-resistant gloves, and a chemical-resistant apron. Wash hands thoroughly with soap and water before leaving the laboratory.*

## Procedure

1. Measure out 50 mL of silicone oil and add to a Thiele tube. Clamp the oil-filled tube to a support stand.
2. Obtain a capillary tube and place a small amount of stearic acid in the tube open side down by tapping the tube to a surface with the stearic acid. Flip the capillary tube over and tap continuously open side up until the stearic acid reaches the bottom (see Figure 1). *Note:* The teacher may have to assist.
3. Carefully insert a thermometer into one of the holes in the rubber stopper.
4. Attach the capillary tube to the thermometer using a rubber band. The rubber band should be near the top of the tube so that it does not come in contact with the oil.
5. Place the thermometer/capillary tube assembly into the Thiele tube until the thermometer bulb and solid in the capillary tube are submersed in the oil. Do not let the rubber band come in contact with the oil (see Figure 2).



Figure 1.

## How to Use a Thiele-Dennis Tube *continued*

- Place the Bunsen burner about 3–4 inches below the Thiele tube. Heat the tube with a Bunsen burner.
- Carefully watch the capillary tube for the melting of the solid.
- When the solid begins to melt, record the temperature of the oil. That is the melting point.
- Allow the oil in the Thiele tube to cool to at least 50 °C before disassembling the apparatus.

## Disposal

Please consult your current *Flinn Scientific Catalog/Reference Manual* for general guidelines and specific procedures, and review all federal, state and local regulations that may apply, before proceeding. Stearic acid, along with the used capillary tubes may be disposed of according to Flinn Suggested Disposal Method #26a. It may be best to collect all the capillary tubes in one container and then dispose of all the glass in a manner that will ensure that no one will get cut.

## Connecting to the National Standards

This laboratory activity relates to the following National Science Education Standards (1996):

### **Unifying Concepts and Processes: Grades K–12**

- Evidence, models, and explanation
- Constancy, change, and measurement

### **Content Standards: Grades 9–12**

- Content Standard B: Physical Science, structure and properties of matter

## Tips

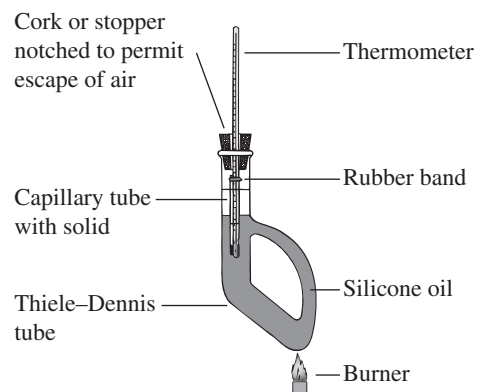
- This activity may also be used along with *Melting Point of a Mixture—Student Laboratory Kit* (Flinn Catalog No. AP1759) to enhance your melting point lesson.
- Other substances such as palmitic acid (P0129) or lauric acid (L0052) may be used for this activity.
- The melting point of pure stearic acid is 63 °C. Melting points and boiling points of organic compounds may be found in the *Merck Index*, *CRC Handbook of Chemistry and Physics*, and *Lange's Handbook of Chemistry*, etc. Melting points may also be found online by looking up Material Safety Data Sheets. Section 9 of the MSDS lists common physical and chemical properties, including melting point, boiling point, density, etc. Visit the Flinn Web site at [www.flinnsci.com](http://www.flinnsci.com) to download current MSDS for all Flinn chemicals.
- It is best to work in a room with low humidity. Wet crystals or crystals that have absorbed water from the atmosphere are difficult to load into the capillary tubes.

## References

This activity was adapted from *Flinn ChemTopic™ Labs*, Vol. 19, *Chemistry of Organic Compounds*; Cesa, I., Editor; Flinn Scientific: Batavia IL (2006).

Atkins, P. W. *Physical Chemistry*, 4th ed.; W. H. Freeman: New York, 1990; pp 196–197.

Russo, T.; Meszaros, M. W. *Vial Organic*; Flinn Scientific: Batavia, IL, 1996; pp 13–18.



**Figure 2.** Design and Use of a Thiele-Dennis Tube.

Materials for *How to Use a Thiele-Dennis Tube* are available from Flinn Scientific, Inc.

Catalog No.	Description
S0451	Silicone Oil
S0335	Stearic Acid, Reagent Grade, 100 g
GP7047	Tubes, Capillary, one end open
GP6090	Thiele-Dennis Melting Point Tube
AP6991	Melting Point Tube, Thiele, Economy Choice
AP8872	Ever-Safe <sup>®</sup> Thermometer

Consult your *Flinn Scientific Catalog/Reference Manual* for current prices.