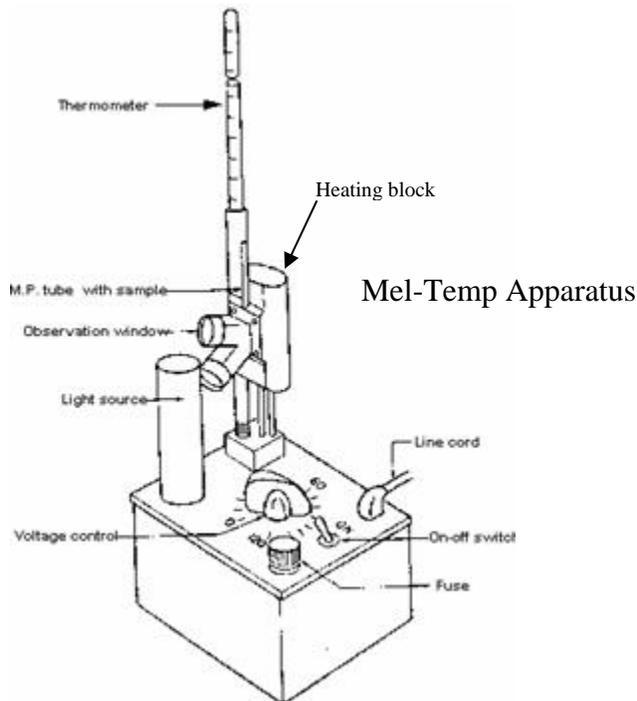


INSTRUCTIONS FOR THE MEL-TEMP MELTING POINT APPARATUS

JMH, 5/2006



http://spot.pcc.edu/~gbackes/CH222/Labs222.W05/Exp.12.Synthesis.Aspirin_files/image016.jpg

Safety Precautions

1. Never insert a room temperature thermometer into a hot Mel-Temp. It may shatter.
2. Do not leave a Mel-Temp on if you are not actually using it, or leave it running unattended.
3. Do not heat the Mel-Temp beyond the upper temperature of the thermometer.
4. Do not touch the heating block – it gets very HOT!

General Information

1. Use no more than 2 mm of sample in the capillary tube. A larger sample may lead to non-uniform heating, and poor results.
2. It is virtually impossible to determine precisely a melting *point* (i. e. 123 °C). Rather, chemists actually determine a melting *range* (i. e. 122 – 124 °C). The first temperature in the melting range is recorded when the first signs of liquid droplets appear in the sample, and the second temperature is recorded when the entire sample becomes liquid.
3. Sometimes, the following may be observed:
 - i. The sample will “settle” in the melting point tube
 - ii. Solvent will condense on the cooler portions of the tubeIt is important to note that these phenomena are **not** melting.

INSTRUCTIONS FOR THE MEL-TEMP MELTING POINT APPARATUS

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I. Establishing an **Approximate** Melting Point

The Mel-Temp apparatus is the most efficient way to quickly establish an approximate melting point for substances whose melting point is unknown. An approximate melting point can be taken using a rate of temperature rise of about 7 – 10 °C/min.

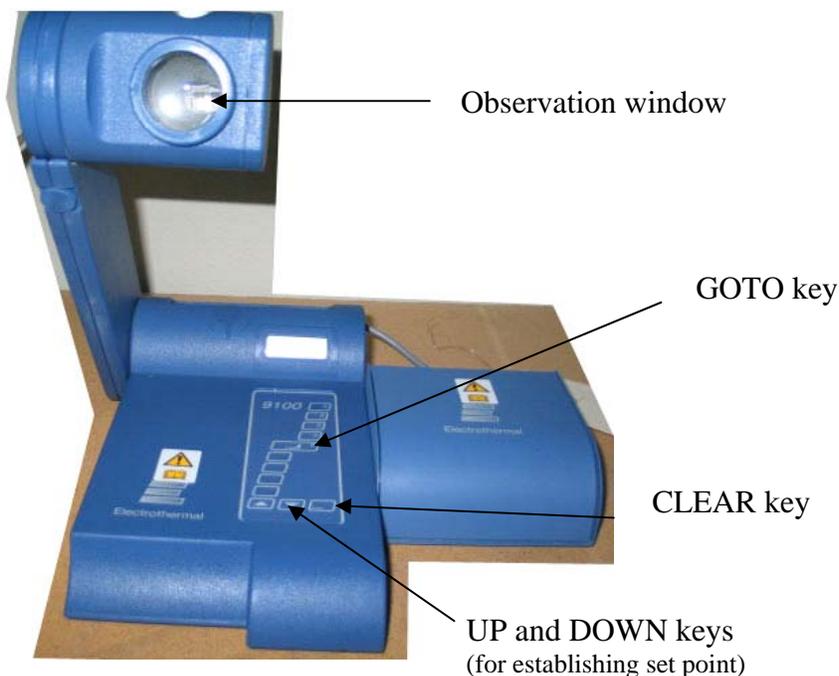
1. Make sure a thermometer is inserted into the Mel-Temp. If not, place the bulb end of a thermometer (with an upper temperature of at least 250 °C) in the opening.
2. Turn on the apparatus using the on/off switch
3. Insert melting point capillary tube into the holder
4. Turn the voltage control knob to achieve a rate of temperature rise of about 7 – 10 °C/min.
5. Observe the approximate melting range of the sample by looking through the observation window. If the rate of temperature rise slows to less than 5 °C/min, turn the voltage control knob to a higher value to increase the rate.
6. When finished, turn the voltage control knob to “0” or turn the unit off.

II. Establishing a **Precise** Melting Point

Once an approximate melting point has been established (or if the melting point of the substance is known from the literature) the Mel-Temp apparatus can be used to find the precise melting range for the substance. To establish a precise melting point, a rate of temperature rise of 1 – 2 °C/min must be used.

1. Make sure a thermometer is inserted into the Mel-Temp. If not, place the bulb end of a thermometer (with an upper temperature of at least 250 °C) in the opening.
2. Turn on the apparatus using the on/off switch
3. Turn the voltage control knob so as to establish a starting temperature about 5 °C below the approximate melting point found from part I (or about 5 °C below the literature value for the substance)
4. Insert melting point capillary tube into the holder.
5. Adjust the voltage control knob so as to establish the rate of temperature rise to be 1 – 2 °C/min
6. Observe the precise melting range of the sample by looking through the observation window. If the rate of temperature rise slows to less than 0.3 °C/min, turn the voltage control knob to a higher value to increase the rate.
7. When finished, turn the voltage control knob to “0” or turn the unit off.

Electrothermal 9100



http://labtrader.com/equipment_images/CT1105059_larger.jpg

Safety Precautions

1. Do not leave an Electrothermal IA9100 on if you are not actually using it
2. Do not leave the Electrothermal IA9100 running unattended.

General Information

1. Use no more than 1 mm of sample in the capillary tube. A larger sample may lead to non-uniform heating, and poor results.
2. It is virtually impossible to determine precisely a melting *point* (i. e. 123 °C). Rather, chemists actually determine a melting *range* (i. e. 122 – 124 °C). The first temperature in the melting range is recorded when the first signs of liquid droplets appear in the sample, and the second temperature is recorded when the entire sample becomes liquid.
3. Sometimes, the following may be observed:
 - i. The sample will “settle” in the melting point tube
 - ii. Solvent will condense on the cooler portions of the tubeIt is important to note that these phenomena are **not** melting.

INSTRUCTIONS FOR THE ELECTROTHERMAL IA9100 MELTING POINT APPARATUS

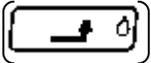
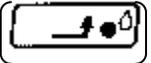
JMH, 5/2006

I. Establishing a Precise Melting Point

Once an approximate melting point has been established using an Mel-Temp or similar piece of equipment (or the melting point of the substance is known from the literature), the Electrothermal IA9100 apparatus can be used to find the precise melting range for the substance. The Electrothermal IA9100 automatically regulates the rate of temperature rise to be 1 °C/min – which is the optimum value for determining precise melting points.

1. Turn on the apparatus using the on/off switch on the back of the power supply. If the power supply is already on, press the CLEAR button  at least 4 times.
2. Enter the starting temperature (the “set point” – usu. 5 °C below the measured approximate melting point or literature value).

NOTE: The arrowed UP key  raises the set point in increments of 10 °C, while the arrowed DOWN key  lowers the set point in increments of 1 °C. Use the appropriate combination of these keys to set the starting temperature. For example, to enter a set point of 107 °C, press the UP key  11 times followed by pressing the DOWN key  3 times:

3. Go to the set point by pressing the GOTO key . When the set point temperature is reached, the left hand L.E.D. on the GOTO key will illuminate  and three “beeps” will sound to indicate that the temperature has stabilized.
4. Insert a melting point capillary tube containing a sample into the holder.
5. Initiate the ramp (1 °C/min) by pressing the GOTO button. The left hand L.E.D. will go out and the right hand L.E.D. will illuminate .
6. Observe the precise melting range of the sample by looking through the observation window.
7. When finished, press the CLEAR button  at least 4 times or turn the unit off.