Freezing Point Depression

The purpose of this experiment is to observe one of the colligative properties of solutions: freezing point depression, the amount by which the freezing point is lowered due to the addition of a known amount of solute to a given amount of a particular solvent. The magnitude of the freezing point lowering (ΔT_f) depends on the nature of the solvent and the concentration of solute species:

$$\Delta T_f = -i\, K_f\, m$$

In this model: K_f is the molal freezing point depression constant of the solvent (in °C × kg solvent/mol solute), i is the number of solute atoms/ions/molecules per formula unit (van't Hoff factor, unitless), and m is the molality of the solution (in mol solute/kg solvent). The lowering of the freezing point is the difference in the freezing point of the pure solvent and that of the solution. (Recall that at a material's freezing point its solid and liquid phases are in equilibrium.)

In this experiment paradichlorobenzene is used as the solvent because of its convenient melting point and its large K_f value. Since paradichlorobenzene is particularly susceptible to super-cooling, its freezing point is best determined by the use of cooling curves. During super-cooling the liquid will cool below the equilibrium freezing point before any solid forms, and then the temperature will increase back toward the equilibrium temperature. Examples of typical cooling curves for a pure solvent and a solution are given at the end of the procedure.

Procedure:
Work with a partner to collect data (Steps 1-6). The analysis of the data is to be done individually. Each pair of students should obtain from the designated point in the lab: a digital thermometer and a numbered unknown.

You will collect (a) two sets of time-temperature data for the determination of the freezing point of the solvent, (b) two sets for the determination of the freezing point of a dilute solution of the unknown, and (c) two sets for a more concentrated solution. Using these six sets of data you will plot six cooling curves and from the freezing points determined, calculate the molecular weight of the unknown.

1) Set up your burner and tripod, and begin heating water in a 400mL beaker. To set up your pure solvent, on the top-loading balance weigh about 12.5 grams of paradichlorobenzene to the nearest 0.01g. Put it in a large test tube from your drawer. Clamp the test tube so that it is immersed in the hot water bath. After the solid has melted, carefully raise the test tube well out of and away from the water bath, dry it with a paper towel, and insert the thermometer. With constant gentle mixing, monitor the temperature. When the temperature reaches about 60°C, begin recording the temperature every 30 seconds. Continue the time-temperature readings until the system becomes difficult to mix. (Suggestion: **record time and temperature in a tabular form**)

Caution: Stir gently and be careful not to damage the test tube or thermometer.

2) Using the hot water bath, reheat the solid until it has melted. Raise the test tube out of the water bath, dry it, and collect a second set of time-temperature data (from about 60 °C to the point where it is mostly solid).

3) Weigh about 1.5 grams of the unknown, to the nearest 0.01g.

4) Re-melt the solid in the water bath. Add the weighed unknown sample, and dissolve it in the solid by mixing thoroughly (Solution 1). When the temperature drops to about 60°C, start collecting time-temperature data as was done with the pure solvent. As before, reheat the system, and collect a second set of data for Solution 1.

5) Weigh a second approximately 1.5-gram sample of the unknown, to the nearest 0.01g. Re-melt the same solution you've been using, and add the second weighed sample to produce Solution 2. [**Note: The mass of solute in this solution is the sum of the two approximately 1.5-gram amounts you've added**.] Collect two sets of time-temperature data for Solution 2.

6) To clean up after completing the experiment: Carry your test tube with thermometer to the hood; heat the contents until they have melted; use the thermometer and your glass stirring rod to get as much solid as possible into the liquid; quickly pour the melt into the labeled container. Use a minimum amount of acetone from the wash bottle to dissolve any remaining solid and pour this into the container.

After completing the procedure but before leaving lab, write in your notebook a brief statement (two to three sentences) on the quality and reasonableness of the data you collected. Note what you might do differently if you performed the lab again.

For the lab report:
For each trial, plot time on the x-axis and temperature on the y-axis. Draw a straight line to represent the cooling before the liquid begins to solidify and a second straight line through the points during solidification. Extend both lines so that they cross. This intersection is the best estimate of the freezing point of the liquid. Plot all six sets of data. (Some CHM 112 instructors may want all 6 sets on one graph, offset as shown below. Listen to the specific directions given to your class).

Calculate the molecular weight (g/mol) of the unknown. Make two calculations, one from data for Solution 1 and one from data for Solution 2. For each solution use the average of the two freezing points (from the two cooling curves) to determine the freezing point depression for the solution. The value of K_f for paradichlorobenzene is 7.1 kg solvent °C/mol of solute. [Hint: In order to calculate the molecular weight of the solute, you will first need to determine the molality of each solution.]