

Freezing Point Diagram

Introduction

This experiment shows how one determines the temperature-composition diagram for a two-component system. The system studied will be naphthalene-biphenyl. This particular mixture forms no solid solution or compounds. The addition of impurity to each of the pure components decreases the freezing point so that two curves are obtained which intersect at the eutectic point.

The procedure will consist of obtaining cooling curves for the pure substances and a number of their mixtures. A cooling curve is constructed by melting a sample, then allowing it to cool, measuring the temperature at regular intervals. When only melt is present, there is a constant cooling rate. As the solid begins to form, the system remains at a constant temperature until the melt is completely converted to solid. The eutectic composition is that at which two solids crystallize out in a ratio equal to that of the melt, and the cooling curve obtained would have the same characteristics as that of a pure substance. The eutectic temperature is the melting point of such a mixture.

Supercooling often occurs in this system. The cooling curve will show a momentary dip below the mixture's freezing point, then will revert back up to the freezing point. This is usually helpful in determining the freezing point, provided the supercooling is not too pronounced. Excess supercooling may be avoided by seeding the melt with **very** small crystals of the solid.

Procedure

Both biphenyl and naphthalene have characteristic odors, so as much of the work as possible should be performed in a hood. The cooling tubes (25×200 mm Pyrex test tubes), in their air jackets, should dip well below the surface of the water in the large beaker. This should prevent crystallization of the melt along the upper edges of the tube before the rest of the melt solidifies, provided the total melt is kept below 25 g. The water temperature should be maintained at about 5 °C below the freezing point of the melt.

Obtain cooling curves (record the temperature over 15 second intervals, plot temperature versus time) for the pure substances and mixtures containing 15, 30, 45, 60, 75, and 90% biphenyl by weight. Record the temperature at which crystallization is first observed (this may be helpful in analyzing the cooling curves).

Calculations

Plot the cooling curves and determine the freezing points of the pure substances and the mixtures. Plot the temperature-composition (**mole fractions**) diagram: the two freezing curves should meet at the eutectic temperature. The accepted values for the eutectic temperature and composition are 39.4 °C and 60% biphenyl by weight.