SAMPLE PREPARATION

Preparation and handling of the alloy mixtures were carried out in a Vacuum Atmospheres Corporation argon glove box. Circulation of the argon through a purification train kept oxygen and water vapor concentrations at <1 p.p.m. Under these conditions, the liquid alloys showed no oxide formation after several hours in the box and showed only a slight oxide layer when left for several days.

Samples were prepared by weighing the sodium and rubidium inside the glove box on a top-loading Mettler P-160 single pan balance, which is accurate to ± 0.001 g. The samples were weighed into a nickel crucible and melted to form a liquid solution, which was then transferred into the freezing point apparatus. To conserve rubidium metal, some samples were prepared in a similar manner by dilution of an alloy sample of known composition with sodium or rubidium.

APPARATUS

The general design of the apparatus for making the freezing point measurements has been described.[5] Briefly, the sample is contained in a double-jacketed stainless steel sample tube. Coolant (usually liquid nitrogen) circulating through the outer jacket and a heater tape wound around the outer jacket provide the temperature variability needed to obtain cooling and warming curves. The inner jacket is connected to a vacuum/helium exchange gas system. The rate of cooling or warming can be controlled by varying the pressure of the heat exchange gas in this space as well as by varying the type of circulating coolant or the heater current. Rotary stirring is accomplished by driving a stainless steel stirrer tube with a variable speed motor through a worm gear. Temperatures are measured with a Leeds and Northrup platinum resistance thermometer, in combination with a Leeds and Northrup high precision resistance recorder. The thermometer passes down through the centre of the stirrer and into a thermometer well in the centre of the sample tube. It was calibrated by Leeds and Northrup Co. at the ice, steam, sulphur, and oxygen points. The calibration was checked by us at the ice point (273.150 K), the mercury freezing point (234.29 K), and the sodium sulphate decahydrate transition temperature (305.534 K) before, during, and at the conclusion of the measurements. In all cases, the values obtained agreed with the calibration to within 0.01 K. We estimate our temperature scale to be accurate to at least ± 0.02 K over the range of the experimental measurements. The apparatus is suspended inside the argon glove box so that all operations from preparation of sample to freezing point measurements are made with minimal chance of contamination of the metals with oxide.

RESULTS

FREEZING POINT MEASUREMENTS

Freezing points were determined over the entire composition range from time-temperature cooling and warming curves. "Clumping" of the soft solid metal as it was formed, made the melting points less reliable than the freezing points. The results are summarized in table 1 and fig. 1. The values are considered accurate to ± 0.15 K except for the five points on the steep portion of the curve immediately on the sodium-rich side of the eutectic. Here the uncertainty may be as high as $\pm 0.5°$. The time-temperature cooling and warming curves in this region gave only a small break at the melting point.

The eutectic point occurs at 268.65 ± 0.05 K and 0.821 mol fraction rubidium. From 0.0-0.4 mol fraction rubidium, the data are in reasonable agreement with the earlier data of Rinck [1] and of Goria.[2] However, between 0.4 mol fraction rubidium and the eutectic composition, the data of ref. (1) and (2) differ greatly from our values. The difference becomes largest around 0.7 mol fraction rubidium where the data of Rinck [1] and Goria [2] are 17 and 25 deg. lower, respectively. Even with the advantages of high precision resistance thermometry used in this study, it was difficult to determine