Melting-Point Determinations of Xenon Difluoride–Xenon Tetrafluoride Mixtures

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The melting point–composition diagram was determined for the binary system xenon difluoride–xenon tetrafluoride. The formation of the 1:1 compound was confirmed. The compound has a congruent melting point at 90.2°C. Melting temperatures were determined visually with a reproducibility of ±0.1°C.

In the system XeF_2–XeF_4 the triple points of both pure components (*5*) and the 1:1 adduct compound (*2*) were previously established. The characteristics of this system over the whole range of concentrations is now reported.

Experimental

Materials. XeF_2 was prepared by photosynthesis at room temperature (*4, 6*). The product was purified by pumping off the volatile impurities in vacuo at −78°C. Infrared spectra of the vapor showed no bands of possible impurities. The triple-point temperature of the sample was 129.1°C, compared to the published value of 129.03°C (*5*).

XeF_4 was prepared by thermal dissociation of xenon hexafluoride in the presence of sodium fluoride (*1*). The infrared spectra of the vapor showed no signs of XeF_2 or other impurities. The triple-point temperature of the sample was 117.1°C. The published value is 117.10°C (*5*).

Procedure. Mixtures were made by subliming in vacuo both pure fluorides, one after another, from two lightweight nickel containers (total weight about 40 grams each), into quartz capillaries. The capillaries were then sealed off, and the weight loss of the containers was determined. The quantities of the mixtures were about 100–400 mg. The pure fluorides were also filled in such tubes. Prior to the filling process, small magnetic stirrers were inserted in the capillaries, which were then baked in vacuo at 700°C.

The method of determining melting points was similar to that of Collett and Johnston (*3*). In a typical determination the capillary was fastened to a mercury-in-glass thermometer and immersed in a liquid bath in a transparent Dewar vessel. The mixture of two solids was first melted and then solidified again. The temperature of the bath was then carefully raised stepwise. The mixture of liquid and crystals in the tube was vigorously stirred with the small magnetic stirrer. The melting temperature was taken as the temperature at which the last crystal disappeared. This was usually reached in an hour or two. The liquid was allowed to crystallize again, and the measurement was repeated. In the same manner the melting points of the pure fluorides were determined.

The reproducibility of the melting temperatures was ±0.1°C. The temperature of the bath was constant to ±0.02°C. The thermometer used in these measurements was calibrated against a thermometer calibrated by Physikalische-Technische Bundesanstalt, Berlin.

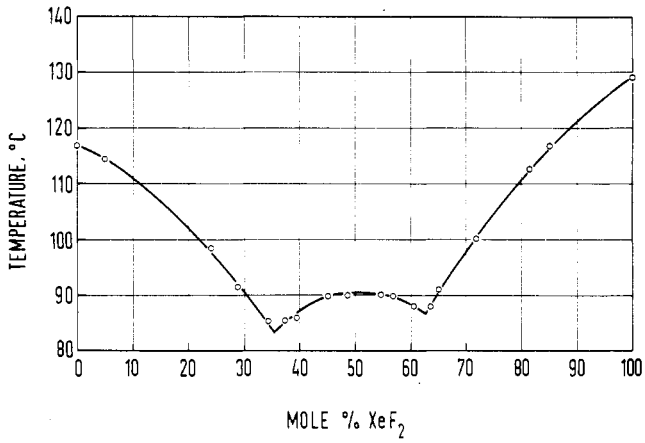

Figure 1. Melting point–composition diagram for XeF_2–XeF_4

Results

Figure 1 shows the melting point–composition curve for the XeF_2–XeF_4 system. The form of the curve confirms the formation of the 1:1 molecular compound, $XeF_2 \cdot XeF_4$. This compound was first found in the solid state by Burns et al. (*2*). The melting point of the compound is 90.2°C, as taken from the curve. The compound dissociates in the liquid state, as can be deduced from the flattened middle part of the curve. The coordinates of the two eutectics are 35.5 mol % XeF_2, 83.5°C and 62.8 mol % XeF_2, 86.7°C, respectively, obtained by extrapolation. The curve gives no evidence of other stable compounds between XeF_2 and XeF_4.

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