Synthesis and calorimetric investigation of unstable β-glycine

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Glycine, $(NH_2)CH_2COOH$, forms three polymorphs. The α ($P2_1/n$) and γ ($P3_1$) forms remain unchanged under normal conditions. The β polymorph ($P2_1$) is unstable and its properties were not adequately investigated. This contribution reports new data on the synthesis of the β-glycine and its transformation into α-glycine.

The crystals of β-glycine were prepared from a saturated solution of glycine in a mixture of water and concentrated acetic acid (volume ratio of 5:1). Then the solution was filtered and acetone was poured in a ratio of 1:1. Just after the pouring the solution became turbid. Not waiting for a precipitate to appear, the solution was filtered. The filter occured to be full of the needle-like crystals of β-glycine. The samples were characterized by X-ray diffraction.

Calorimetric measurements were performed using DSC-111 (SETARAM, standard aluminum crucibles, temperature range from 300 to 450 K at a heating rate of 1 K/min, sample mass 181.91 mg). Eight runs with the same sample were carried out. The results of the first run differ from those of the rest runs. The results of the seven runs reproduced with high accuracy, 5 mJ/gK. As compared to the heat capacity, this value was about 0.3 %. After completion of the calorimetric measurements, the sample was investigated by means of X-ray powder diffraction again. The crystals were found to transform into α-phase completely. Thus, the difference between the calorimetric results of the first run and the rest runs is the difference between the heat capacity of the β-glycine at transformation into α-glycine and the heat capacity of α-glycine. Over the temperature range 310 to 340 K, the difference in the heat capacity was nearly constant, about 15 mJ/gK. We also measured the heat capacity of α-glycine, and of γ-glycine. Heat capacity of α-glycine in a temperature range 310 - 340 K fits to the polynomial $Cp(mJ/gK) = 63 + 4.173T$. Thus, for that temperature range, the heat capacity of β-glycine is about 1 % less than that of α-glycine. At 340 K, exothermal peak appears that can be explained as a start of the β→α transformation. The transformation was found to be due to the recrystallization, i.e. mass transfer. The recristallization rate depends on the temperature and increases with heating. The exothermic process increases with increasing temperature reaching the maximum value at 390 K. Then the exothermic reaction ceases. At 415 K the heat capacity measured during the first run becomes equal to those at next runs. The transformation has finished. Total heat release during the β→α transformation is about 200 J/mol.

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