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THERMOLYSIS OF ALKALI FLUOROALUMINATES

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High temperature X-ray and IR-spectroscopic investigations have been carried out to study reactions and structural transformations on thermal treatment of especially potassium fluoroaluminates. Samples like $KAlF_4$, $K_2AlF_5.H_2O$, a new acid fluoroaluminat K_2HAlF_6 and $Rb_2AlF_5.H_2O$ have been investigated. By means of high temperature X-ray measurements hydrolysis has been detected at temperatures above 250 °C connected with formation of K_3AlF_6 and Rb_3AlF_6. Besides hexafluoroaluminates a cubic or tetragonal phase - the last in the case of Rb-compound - is formed. These phases containing probably oxygen are intermediate products of hydrolysis. They couldn't be identified yet. Some bands in the IR-spectrum also indicate such species. After cooling K_3AlF_6 and Rb_3AlF_6 respectively and Al_2O_3 have been detected. Compared with $KAlF_4$, K_2HAlF_6 and $Rb_2AlF_5.H_2O$ dimension of hydrolysis in the case of $K_2AlF_5.H_2O$ is small. Contrary to investigations with greater amount of substance in high temperature X-ray measurements no dismutation of $K_2AlF_5.H_2O$ and $Rb_2AlF_5.H_2O$ into $KAlF_4$, K_3AlF_6 and $RbAlF_4$, Rb_3AlF_6 respectively has been found. The behaviour of the tetragonal K_2HAlF_6 on thermal treatment is different from the other fluoroaluminates. At 100 °C the whole amount of HF is lost and a condensation process takes place. A '$KAlF_4$'-phase with expanded lattice dimensions compared with normal $KAlF_4$ is formed. The IR-spectrum of these phase is similar to that of $KAlF_4$ but the splitting of ν_3-band is greater. In the range of 200–400 °C $KAlF_4$ with normal dimensions is yielded. Above 400 °C hydrolysis of $KAlF_4$ is observed.