



Lead hexacyanoferrate(II) tetrahydrate: Crystal structure, FTIR spectroscopy and thermal decomposition studies

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ABSTRACT

Pb₂[Fe(CN)₆]·4H₂O was synthesized by mixing aqueous solutions of lead(II) nitrate and potassium ferrocyanide. Its crystal structure was solved *ab initio* from synchrotron powder X-ray diffraction data using the direct methods and refined by the Rietveld method. Thermal analysis (TGA and DTA), Fourier Transform Infrared Spectroscopy (FTIR) and scanning electron microscopy (SEM) studies were also used for the solid state characterization of this compound and its decomposition products. The complex crystallizes in the monoclinic crystal system, space group *P*2₁/*n*. The Fe²⁺ cation is octahedrally coordinated to six cyano groups, and the Pb²⁺ cation is penta-fold coordinated to three N atoms from C≡N ligands and two O atoms from coordinated water molecules. The most important peculiarity of the structure of this complex is the occurrence of water bridges linking two neighboring Pb²⁺ cations. An unusual Fe–C≡N–Pb(H₂O)₂–Pb–N≡C–Fe linkage alternates with a usual Pb–N≡C–Fe one. Zeolitic water molecules are also observed in the structure; they are located in small channels in the structure and they are hydrogen bonded to coordinated water molecules forming a cumulus. Coordinated water and zeolitic water molecules in this complex can be removed without affecting the hexacyanometaallate framework. The thermal decomposition in air to produce Pb₃Fe₂O₅ and PbO as final products has been studied by the thermogravimetric and differential thermal analysis, FTIR spectroscopy and laboratory powder X-ray diffraction. The crystallite size and morphology of the complex and its thermal decomposition products were evaluated by SEM.

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