Eur. J. Solid State Inorg. Chem.
t. 31, 1994, p. 979-984

Synthesis and characterization of ferric nitroprusside

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(E. K., received July 1, 1994 ; accepted October 10, 1994.)

ABSTRACT.- Ferric nitroprusside has been synthesized in several ways: in solution, tribochemically and by ozonization of solid ferrous nitroprusside. The purer samples are obtained by the reactions of Ag^+ nitroprusside with ferric chloride and Ba^{2+} nitroprusside with ferric sulfate. The compound has a formula $Fe_2[Fe(CN)_5NO]_3.9H_2O$ and a cubic fcc structure with a_o of 10.217(4) Å. The Mössbauer and IR spectra are interpreted.

Introduction

Ferrous nitroprusside has been well studied by IR, Mössbauer, XRD and thermogravimetric techniques [1-7]. However, ferric nitroprusside has only been obtained by Seifer et al. [8] who reported two different compounds depending on the synthesis procedure. If $H_2[Fe(CN)_5NO]$ is neutralized with $Fe(OH)_3$, the solid has a formula $Fe_2[Fe(CN)_5NO]_3.9H_2O$ while if it is obtained from $Na_2[Fe(CN)_5NO].2H_2O$ and $FeCl_3$ solutions, the stoichiometry is $Fe[Fe(CN)_5NO]Cl.0.5H_2O$. No spectroscopic or crystallographic data for these solids was reported [8].

In this communication we report the synthesis of ferric nitroprusside by different paths and discuss the IR, Mössbauer, XRD and thermogravimetric data of the solids obtained.

Experimental

IR spectra were recorded in a Phillip FT-IR spectrometer in Nujol mulls between CaF_2 windows and in KBr pressed disks. XRD powder patterns were taken in a HZG-4 diffractometer (Carl Zeiss, Jena) using monochromated CuKα radiation. The hydration degree was determined thermogravimetrically using a MOM-Q-1500 equipment. Mössbauer spectra were recorded at room temperature with ^{57}Co in Rh source using a constant acceleration spectrometer (MS 1101 from Mösstech) in the transmission mode. The Mössbauer parameters, isomer shift δ, quadrupole splitting Δ, line width Γ and absorption line area A were obtained using an iterative least square minimization algorithm with Lorentzian line shapes.

ISSN 0992-4361/94/11/$4.00/© Gauthier-Villars