

Green synthesis and properties of nickel terephthalate complex with 2,2'-bipyridine

Oxana V. Kharissova, Vladimir A. Zhinzhilo, Maria A. Chernomorova, Igor E. Uflyand, Idalia Gómez de la Fuente and Boris I. Kharisov

General.

IR spectra in the range 4000-400 cm^{-1} were recorded on a Nicolet 380 Fourier spectrometer in KBr pellets and Softspectra data analysis software.

Elemental analyses were carried out on a CHNOS vario EL cube analyzer (Elementar Analysensysteme GmbH, Germany). Nickel was determined on an energy dispersive X-ray fluorescence spectrometer «X-Art M» (Comita, Russia) or atomic absorption spectrometer «MGA-915» (Lumex, Russia).

XRD was performed using a Phywe XR 4.0 diffractometer with Cu-K α radiation (1.541874 Å). IR spectra were recorded on a Nicolet 380 Fourier spectrometer in KBr pellets in the range 4000-400 cm^{-1} .

SEM was performed on a ZEISS Crossbeam 340 with an accelerating voltage of 3 kV (Jena, Germany).

Synthesis.

Step 1. Water is used as a solvent; the resulting compound does not require lengthy and expensive washing and long-term drying. Sodium hydroxide (0.8 g, 0.02 mol) is dissolved in water (50 ml), and the resulting solution is heated to 85 °C. Terephthalic acid (1.6 g, 0.01 mol) is added with stirring, and a clear solution is formed. In another vessel, nickel(II) chloride hexahydrate (2.38 g, 0.01 mol) is dissolved in water (30 ml), and is heated to 85 °C. Both solutions are mixed, and the mixture is left to cool to room temperature until self-cooling, which caused formation of green precipitate. The precipitate is filtered off under suction, dried in air and then in dynamic vacuum at 110 °C for 8 h. After drying, 2.75 g (97.5%) of nickel terephthalate is obtained.

Step 2. Nickel terephthalate (2.82 g) was suspended in ethanol (20 ml), and 2,2'-bipyridine (1.56 g, 0.01 mol) was added at room temperature portionwise on stirring. During the addition of 2,2'-bipyridine, the formation of a bulky suspension and a significant growth of the viscosity of the solution are observed. The resulting substance is filtered off, washed with water, then with alcohol and dried in air for 3 h at room temperature, and then in a dynamic vacuum at 130 °C for 8 h. Anal. Found (%): C, 47.6; H, 4.2; N, 6.34; Ni, 13.3. Calculated for $\text{C}_{20}\text{H}_{12}\text{N}_2\text{NiO}_4$ (%): C, 47.8; H, 4.4; N, 6.2; Ni, 13.1. IR spectrum (ν , cm^{-1} , Figure S1): 3400, 1600, 1582, 1530, 1444, 1362, 912, 765. A broad band at 3400 cm^{-1} is attributed to the OH stretching vibration from a coordinated water molecule participating in a relatively strong hydrogen bond. Stretching vibrations $\nu_{\text{asym}}(\text{CO}_2)$ and $\nu_{\text{sym}}(\text{CO}_2)$ at 1530 and 1444 cm^{-1} , respectively, show that the terephthalate ligand adopts both monodentate and chelating coordination modes.

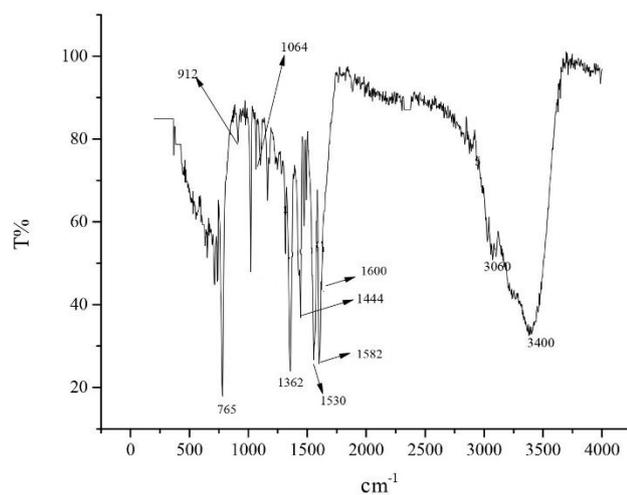


Figure S1 IR spectrum of nickel terephthalate-bipyridine complex.

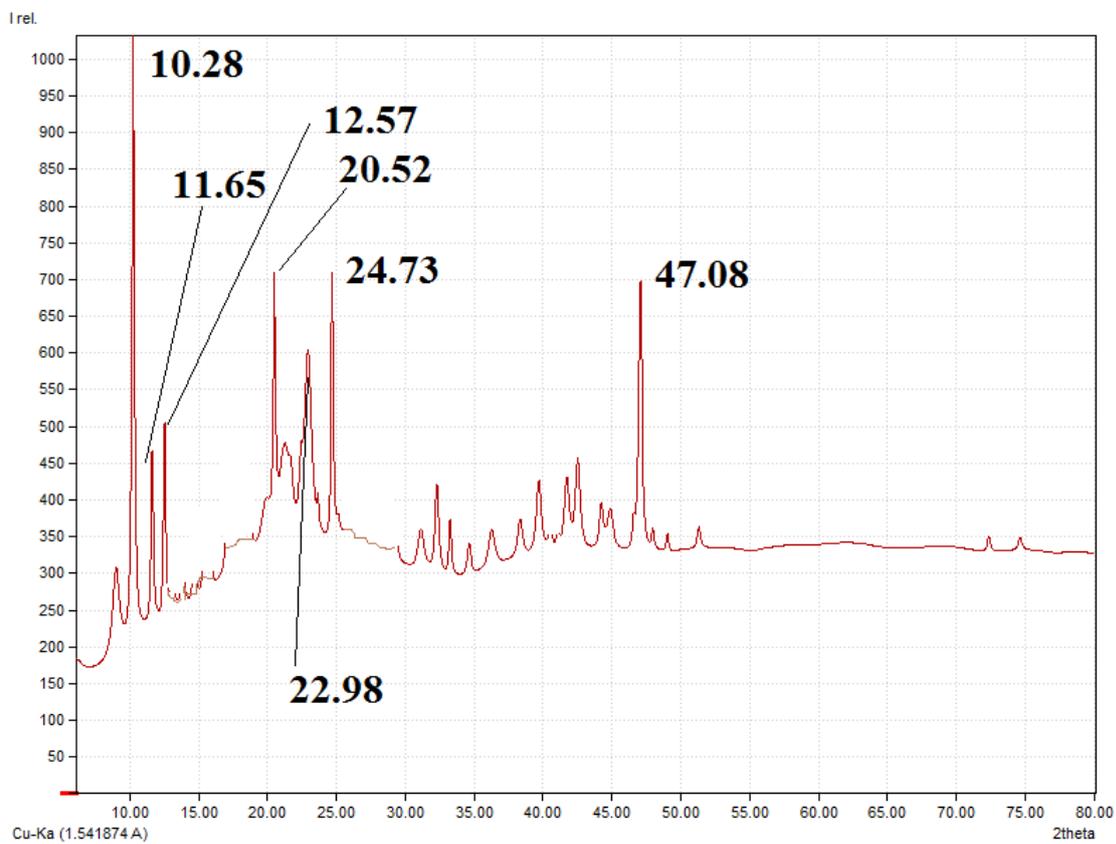


Figure S2 XRD profile of nickel terephthalate-bipyridine complex.