

Synthesis and Physical Chemical Characterization of The Potassium 11-Tungstovanado(IV) Phosphate Anion

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Polyoxometalates constitute a large class of compounds that are remarkable for their molecular and structural versatility as well as their interesting and diverse properties, which makes them more and more popular in many fields such as catalysis, biology, medicine, magnetism, photochemistry and material science [1, 2].

The new $K_5[(VO)PW_{11}O_{39}].10H_2O$ complex was synthesized by alternative methods and investigated by chemical and thermogravimetric analysis, X-ray diffraction and spectroscopic (FT-IR, UV-Vis, ESR) methods.

The FT-IR spectrum in KBr pellets shows the following vibration bands: 1622 cm^{-1} (δ_{H-O-H}), 1089 and 1063 cm^{-1} ($\nu_{as}P-O_i$), 964 cm^{-1} ($\nu_{as}W-O_t + \nu_{as}V-O$), 889 cm^{-1} ($\nu_{as}W-O_c-W$), 798 and 735 cm^{-1} ($\nu_{as}W-O_e-W$), 679 and 594 cm^{-1} ($\delta_{W-O_c,e-W}$), which are characteristic for polyoxometalates.

The two absorption bands in the electronic spectrum at 13500 and 15500 cm^{-1} are due to d-d transitions and correspond to so-called bands I and II in normal oxovanadium complexes. The absorption bands at 20000 and 25000 cm^{-1} which are responsible for the intense color of the anion are assigned to the heteronuclear intervalence charge-transfer transitions $V(IV) \rightarrow W(VI)$.

The powder EPR spectrum (Fig. 1) of the complex obtained in the X band at room temperature contains eight hyperfine components, both in the perpendicular and parallel bands.

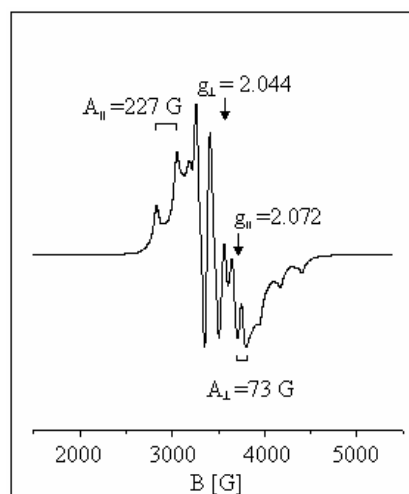


Fig. 1: The EPR spectrum of the $K_5[(VO)PW_{11}O_{39}].10H_2O$ complex

Single-crystal X-ray diffraction shows that the complex crystallizes in the triclinic system and belongs to the $P3(2)$ 21 space group with two molecules per cell.

[1] M.T. Pope, A. Müller, *Angew. Chem. Int. Ed. Engl.* 30 (1991) 34.

[2] A. Müller, F. Peters, M.T. Pope, D. Gatteschi, *Chem. Rev.* 98 (1998) 238.