

## Formation of the $\text{Al}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$ by the solution method

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The literature survey has shown, that in the  $\text{Al}_2\text{O}_3\text{-V}_2\text{O}_5\text{-WO}_3$  system  $\text{Al}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$  phase forms [1]. It crystallizes in a tetragonal system and is isostructural with  $\text{Fe}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$  [2,3]. Its unit cell parameters are following:  $a=b=1.9487$  nm,  $c=0.36706$  nm. This compound adopts  $\text{M-Nb}_2\text{O}_5$  structure and is a potential component for fabrication of electrodes in lithium batteries and catalysts.  $\text{Al}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$  melts at  $910^\circ\text{C}$  with depositing two solid products, i.e.  $\text{Al}_2(\text{WO}_4)_3$  and  $\text{WO}_3$ .  $\text{Al}_2(\text{WO}_4)_3$  crystallizes in an orthorhombic system and is the only compound forming in the system  $\text{Al}_2\text{O}_3 - \text{WO}_3$ . On the other hand in the  $\text{Al}_2\text{O}_3\text{-V}_2\text{O}_5$  system triclinic  $\text{AlVO}_4$  forms, melting incongruently at  $745^\circ\text{C}$  with deposition of  $\alpha\text{-Al}_2\text{O}_3$  as a solid product. Until now the  $\text{Al}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$  phase was obtained as a result of reaction between oxides. The aim of this work was to obtain this phase by the solution method.

The reactants used for the preparation of  $\text{Al}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$  by the solution method were  $\text{Al}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$  a.p. (Chempur, Poland),  $\text{NH}_4\text{VO}_3$  a.p. (POCH Gliwice, Poland) and  $(\text{NH}_4)_6\text{W}_{12}\text{O}_{39}\cdot x\text{H}_2\text{O}$  (Aldrich, Germany). Water solutions of metal precursors weighed in the appropriate proportions were mixed together with energetic stirring. Such obtained solution of an orange color was evaporated to dryness at  $60^\circ\text{C}$  on an electric heating plate. The dark orange solid obtained after evaporation was calcined at  $190^\circ\text{C}$ ,  $300^\circ\text{C}$ ,  $500^\circ\text{C}$ ,  $600^\circ\text{C}$  and  $750^\circ\text{C}$  in one hour cycles. The temperatures of calcinations were selected basing on the results of TGA-DTA investigations of obtained solid. The products obtained after each heating stage were investigated with the help of XRD and UV-Vis-NIR measuring techniques.

The TGA curve of the dark orange solid product obtained after water evaporation to dryness has a complex shape. A series of processes quickly following one another, accompanied by a considerable mass loss, begins at  $110^\circ\text{C}$  and ends at  $500^\circ\text{C}$ . On the other hand on the DTA curve were recorded three endothermic effects and two exothermic ones. The mass loss process (TGA curve) can be divided into four stages. The first stage starts at  $110^\circ\text{C}$  and ends at  $220^\circ\text{C}$  and is accompanied by the endothermic effect, whereas the second stage commencing at  $250^\circ\text{C}$  and ending at  $320^\circ\text{C}$  is accompanied by exothermic one. The third stage with onset temperature at  $320^\circ\text{C}$  is connected with relatively low mass loss. The fourth stage of the process is very short and runs with small mass loss. It starts at  $460^\circ\text{C}$  and ends at  $500^\circ\text{C}$  and is accompanied by exothermic effect recorded on DTA curve. In the powder diffraction pattern of the dark orange product obtained after evaporation of water was recorded the set of diffraction lines. Part of them were attributed to  $\text{NH}_4\text{NO}_3$ , the by-product of the synthesis. Powder diffraction pattern recorded after heating stage at  $190^\circ\text{C}$  does not contain diffraction lines characteristic for  $\text{NH}_4\text{NO}_3$ , what implies that the first mass loss stage is connected with the decomposition of  $\text{NH}_4\text{NO}_3$ . Powder diffraction patterns recorded after subsequent heating stages were complex and contained broadened diffraction lines. In the diffractogram of preparation heated at  $500^\circ\text{C}$  for 60 minutes a set of diffraction reflections corresponding to the  $\text{Al}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$  was recorded. The intensity of diffraction lines characteristic for  $\text{Al}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$  gradually increased with the increase of sintering temperature. After calcination at  $750^\circ\text{C}$  the powder diffraction pattern of sample contained besides lines characteristic for  $\text{Al}_8\text{V}_{10}\text{W}_{16}\text{O}_{85}$  only low-intensity reflections which could be attributed to impurities.

[1] P. Tabero, *J. Therm. Anal. Calorim.*, **101** (2010) 561-6.

[2] I. Rychłowska-Himmel, P. Tabero, *J. Therm. Anal. Calorim.*, **65** (2001) 537-543.

[3] J. Rychłowska-Himmel, *J. Therm. Anal. Calorim.*, **60** (2000) 173-7.