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# A SEARCH FOR NEW PHASES IN THE SYSTEM $Fe_2O_3$ - $V_2O_5$ - $WO_3$

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Studies on the three-component system $Fe_2O_3$-$V_2O_5$-$WO_3$ have shown the occurrence of a new, compound with molecular formula $FeVW_2O_{10}$. Its X-ray characteristics and its melting temperature, 865±10 °C, have been established.

The system $Fe_2O_3$-$V_2O_5$-$WO_3$ is of interest, particularly because of the catalytic properties of the components. One may expect that the phases arising in this system also possess catalytic properties. A literature survey suggests that the system $Fe_2O_3$-$V_2O_5$-$WO_3$ has not been studied previously. Hence, we have started a search for new phases in this system.

The structures and properties of the system components have been the subject of numerous studies and are well known [1-3]. The study of the two-component systems appears to be less advanced. Only the system $Fe_2O_3$-$V_2O_5$ is known to some extent, whereas the literature information on the two other systems is fragmentary and often inconsistent [5-8].

## Experimental

Analar $Fe_2O_3$ (VEB, GDR), analar $V_2O_5$ (POCh, Poland) and analar $WO_3$ (Fluka AG, Switzerland) were used in the experiments.

The samples were prepared from the appropriate oxides weighed in given proportions, the oxides being homogenized by grinding, then pastilled and calcined at given temperatures and in given time cycles. After each calcination cycle, the samples were slowly cooled down to ambient temperature, ground down, then analyzed by DTA and X-ray powder diffraction.

DTA was performed in air, in quartz crucibles, using a Paulik-Paulik-Erdey derivatograph (MOM, Budapest). All measurements were made

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under the same conditions, i.e. at 20-1000°, a heating rate of 10 deg/min, and a sample weight of 1000 mg. X-ray powder diffraction measurements on the preparations were carried out with a diffractometer of type $A_2$, with an a HZG-4 goniometer ($CoK_\alpha$ radiation). The phase compositions of the samples were established via the data included in the ASTM cards [9].

The compositions of the mixtures are shown in Table 1. The calcination conditions were as follows:

Preparation 1: 550° (24 h); 600° (24 h + 48 h); 650° (24 h + 48 h); 680° (48 h x 4).

Preparation 2: 550° (48 h); 600° (48 h x 2); 650° (48 h x 3); 670° (48 h x 3); 700° (4 h + 12 h).

Preparation 3: 550° (24 h); 600° (24 h); 650° (24 h); 700° (24 h x 3); 710° (24 h); 715° (24 h); 720° (24 h).

**Table 1** Compositions of the substrate mixtures and X-ray powder diffraction results on the preparations after the last calcination cycle

| No | Composition of initial mixture, mol % | | | X-ray powder diffraction |
|---|---|---|---|---|
| | $Fe_2O_3$ | $V_2O_5$ | $WO_3$ | |
| 1 | 25.00 | 25.00 | 60.00 | $FeVO_4$, $FeVW_2O_{10}$ |
| 2 | 20.00 | 20.00 | 60.00 | $FeVO_4$, $FeVW_2O_{10}$ |
| 3 | 16.67 | 16.67 | 66.66 | $FeVW_2O_{10}$ |

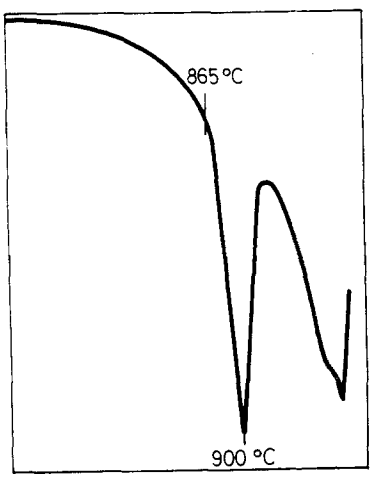


Fig. 1 DTA curve of $FeVW_2O_{10}$

**Table 2** Interplanar distances and relative intensities for the corresponding reflexions characterizing the phase $FeVW_2O_{10}$

| *d*, Å | *I*, % |
|---|---|
| 6.97 | 2 |
| 4.66 | 35 |
| 3.65 | 28 |
| 3.49 | 100 |
| 3.42 | 3 |
| 3.29 | 2 |
| 3.02 | 1 |
| 2.79 | 7 |
| 2.76 | 1 |
| 2.71 | 33 |
| 2.46 | 12 |
| 2.37 | 1 |
| 2.32 | 1 |
| 2.25 | 7 |
| 2.04 | 1 |
| 1.99 | 20 |
| 1.98 | 13 |
| 1.94 | 1 |
| 1.86 | 2 |
| 1.85 | 3 |
| 1.83 | 2 |
| 1.74 | 1 |
| 1.73 | 7 |
| 1.72 | 6 |
| 1.66 | 8 |

## Results and discussion

The X-ray powder diffraction results on preparations after the last calcination cycle are shown in Table 1. These result reveal that only the diffraction pattern of sample 3 after the last calcination cycle includes a set of undefined reflexions, which can be attributed neither to the substrates nor to the known phases which occur in the two-component systems making up this three-component system. This set of interplanar spacings and their relative intensities are presented in Table 2. Hence, X-ray powder diffraction implies that the components of the system $Fe_2O_3$-$V_2O_5$-$WO_3$ form a new phase, according to the following reaction:

$$Fe_2O_{3(s)} + V_2O_{5(s)} + 4\ WO_{3(s)} = 2\ FeVW_2O_{10(s)}$$

The DTA curve of this phase is shown in Fig. 1. The first endothermic effect, with a temperature start of $865 \pm 10^{\circ}$, is attributed to the melting of $FeVW_2O_{10}$. The mode of melting and the likely course of the reaction require further studies.

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**Zusammenfassung** — Einleitende Untersuchungen am Dreikomponentensystem $Fe_2O_3$-$V_2O_5$-$WO_3$ zeigten das Auftreten einer neuen, noch nicht publizierten Verbindung der Formel $FeVW_2O_{10}$. Die Verbindung wurde röntgenographisch beschrieben, ihr Schmelzpunkt beträgt $865 \pm 10^{\circ}C$.