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LETTERS TO THE EDITOR

Synthesis and Study of Manganese Hexamolybdocobaltate

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Previously we suggested a procedure for preparing copper hexamolybdocobaltate [1] and studied this compound by X-ray phase and structural, as well as thermogravimetric analysis. In the present work we prepared manganese hexamolybdocobaltate (**I**) of the composition $Mn[CoMo_6O_{18}(OH)_6]$, which can be applied as a catalyst in organic synthesis.

$$\underset{\textbf{III}}{(NH_4)_3[CoMo_6O_{18}(OH)_6]} \longrightarrow \underset{\textbf{II}}{H_3[CoMo_6O_{18}(OH)_6]}$$

$$\xrightarrow[-3CH_3COOH]{Mn(CH_3COO)_3} \underset{\textbf{I}}{Mn[CoMo_6O_{18}(OH)_6]}.$$

Compound $Mn[CoMo_6O_{18}(OH)_6]$ (**I**) was prepared by an exchange reaction between hexamolybdocobaltic acid (**II**) and manganese acetate (**III**) in water at room temperature. The mixture of the solutions was evaporated in a dessicator over KOH. After several weeks compound **I** precipitated as light violet crystals. The crystals were filtered off, washed with water and ethanol, dried, and recrystallized from water.

Hexamolybdocobalic acid (**II**) was obtained from ammonium hexamolybdocobaltate by ion-exchange chromatography on a KU-2-8 resin (8% divinylbenzene).

Ammonium hexamolybdocobaltate (II) was synthesized by a modified procedure [2]. A hot solution of 53 g of ammonium paramolybdate in 200 ml of water, acidified with nitric acid to pH 3, was added a solution of 25 g of cobalt nitrate in 100 ml of water, after which 25 ml of 18% hydrogen peroxide was added dropwise. The mixture was heated for 3 h on a water bath. After cooling to room temperature, compound **II** precipitated as green crystals within a day. The crystals were washed with water and recrystallized from water.

To prove individuality and purity of compound **I** and to obtain crystallographic data, its X-ray phase analysis was performed. The X-ray powder patterns were indexed using the Powder-2 program package. Retrieval from the PCPDFWIN database showed that compound **I** is individual, contains no possible admixtures, and relates to a monoclinic singony with the following unit cell parameters: *a* 18.6394, *b* 3.4220, *c* 17.2499 Å, β 97.280°; *V* 1091.39 Å^3. The picnometric density of the compound, determined by the Syromyatnikov procedure is dexp 3.015 g cm^{-3}, number of formula units $Z = 1$.

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