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LETTERS TO THE EDITOR

Synthesis of 18-Molybdo-2-Phosphate Acid by Sorption on Polyuretane Foam and Desorption with Acetone

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The interest in heteropolycomplexes is connected with their prospective use as effective catalysts, biologically active compounds, or new analytical reagents [1]. The known methods for preparing heteropolyacids, specifically 18-molybdo-2-phosphate acid (**I**), are two-stage, the first involving synthesis of their salts from the starting components and the second, isolation of free acids by extraction or ion exchange [2]. One more drawback of the known syntheses of acid **I** is a low yield of the target compound.

Heteropolyacids both in the oxidized and reduced form are effectively sorbed on polyurethane foams (PUF) [3], because the foams of different grades contain functional groups of organic solvents (ethers, esters, high-molecular amines) that effectively extract heteropolycomplexes.

We found that compound **I** is sorbed on polyuretane foam from aqueous solutions of alkali metal phosphates and molybdates, acidified with mineral acids.

$$2HPO_4^{2-} + 18HMoO_4 + 22H^+ + PUF \longrightarrow PUF \cdot H_6[P_2Mo_{18}O_{82}] + 18H_2O.$$

The sorbent is washed with distilled water to remove alkali metal cations, after which compound **I** is practically quantitatively extracted with acetone.

Hence, the proposed method permits to increase the prosporus(V)-based yield of 18-molybdo-2-phosphate acid from 7 to 70% compared to the method described in [4]. The synthesis is carried out in one stage for 3 h, omitting the intermediate preparation of ammonium salt of 18-molybdo-2-phosphate acid. Note that acid **I** cannot be desorbed with dry acetone from the surface of dry sorbent. Several percent of water in acetone is necessary to form structure **I** in solution. Instead of acetone, acetonitrile, dioxane, and some other water-miscible oxygen-containing solvents are suitable.

The ^{31}P NMR spectrum of a 5×10^{-2}M aqueous solution of acid **I** contains one signal with δ_P –2.40 ppm, what agrees with the data in [5] for aqueous $P_2Mo_{18}O_{62}^{6-}$.

18-Molybdo-2-phosphate acid. A solution of 31 g of $Na_2MOO_4 \cdot 2H_2O$ in 100 ml of water was treated with 0.8 ml of dilute (1 : 100) orthophosphoric acid and acidified with hydrochloric acid (1 : 1) to pH 1–2.5, after which 1 g of PUF [$m_s/m_{P(V)}$ 240] was introduced into the resulting mixture. Sorbent pieces were squeezed with a glass stick to remove air bubbles, and the mixture was magnetically stirred for 1 h. Sorbent pieces were then taken off, washed with two portions of distilled water, dried between two sheets of filter paper, and placed in 20 ml of acetone containing 3–5 vol% of water. Air bubbles were squeezed from the sorbent, and the mixture was stirred for 5 min. The sorbent was taken off, and the desorbate was left to stand in air until the acid began to crystallize. Yield 0.28 g (70%).

The NMR spectrum was recorded on a Bruker CXP-200 spectrometer (81 MHz) against 85% phosphoric acid.

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