

LETTERS
TO THE EDITOR

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

O. M. Trokhimenko

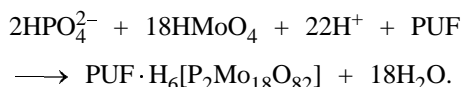
Shevchenko Kiev National University, Kiev, Ukraine

Received October 6, 2003

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 polyurethane foam from aqueous solutions of alkali metal phosphates and molybdates, acidified with mineral acids.



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 prosopus(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 $5 \times 10^{-2}\text{M}$ aqueous solution of acid **I** contains one signal with $\delta_{\text{P}} -2.40$ ppm, what agrees with the data in [5] for aqueous $\text{P}_2\text{Mo}_{18}\text{O}_{62}^{6-}$.

18-Molybdo-2-phosphate acid. A solution of 31 g of $\text{Na}_2\text{MOO}_4 \cdot 2\text{H}_2\text{O}$ 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 [$m_s/m_{\text{P(V)}}$ 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.

REFERENCES

1. Talismanov, S.S. and Eremenko, I.L., *Zh. Neorg. Khim.*, 2003, vol. 72, no. 7, p. 627.
2. Pop, M.S., *Geteropoli- i isopolioksometalaty* (Heteropoly- and Isoxopolyoxometallates), Novosibirsk: Nauka, 1990.
3. Dmitrienko, S.G. and Zolotov, Yu.A., *Usp. Khim.*, 2002, vol. 71, no. 2, p. 180.
4. Schill, E., *Iso- und Heteropolyverbindungen. Handbuch der präparativen anorganischen Chemie in drei Bänden herausgegeben von Georg Brauer*, Stuttgart: Thieme, 1981, vol. 3, p. 1776.
5. Petersson, L., Andersson, I., and Ohmann, L.-O., *Inorg. Chem.*, 1986, vol. 25, no. 26, p. 4726.