## Synthesis of Methylammonium Hepta-Monomolybdate and Its Transformation by Heat-Treatment

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## Abstract

methylammonium hepta-monomolybdate  $(CH_3NH_3)_8[Mo_7O_{24}-MoO_4]\cdot 4H_2O$ The was synthesized by the evaporation of solution resulting from the reaction of MoO<sub>3</sub> and methylamine. The solid crystallizes in the space group P-1, which is consists of [Mo<sub>7</sub>O<sub>24</sub>]<sup>6-</sup> anion, [MoO<sub>4</sub>]<sup>2-</sup> anion, lattice water molecule, and methylammonium cation. The [Mo<sub>7</sub>O<sub>24</sub>]<sup>6-</sup> anion is built up of seven condensed edgesharing MoO<sub>6</sub> octahedra. The crystal structure also contains co-crystallized monomolybdate [MoO<sub>4</sub>]<sup>2-</sup> anion with tetrahedral geometry as shown in Figure 1. A similar structure containing [Mo<sub>7</sub>O<sub>24</sub>]<sup>6-</sup> and [MoO<sub>4</sub>]<sup>2-</sup> with butan-1-ammonium (BuNH<sub>3</sub>) counter cation was also reported earlier [1]. The transformation of methylammonium hepta-monomolybdate was investigated by thermal treatment in the air. Based on powder X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR), formation of various methylammonium isopolymolybdate such as (CH<sub>3</sub>NH<sub>3</sub>)<sub>8</sub>[Mo<sub>10</sub>O<sub>34</sub>] and (CH<sub>3</sub>NH<sub>3</sub>)<sub>4</sub>[Mo<sub>8</sub>O<sub>26</sub>], and molybdenum oxide such as hexagonal MoO<sub>3</sub> and orthorhombic MoO<sub>3</sub> were confirmed. The thermal transformation was similar to that of (NH<sub>4</sub>)<sub>6</sub>[Mo<sub>7</sub>O<sub>24</sub>]·4H<sub>2</sub>O [2].

Keywords: Polyoxometalates, Molybdenum



Figure 1. Structure of (CH<sub>3</sub>NH<sub>3</sub>)<sub>8</sub>[Mo<sub>7</sub>O<sub>24</sub>-MoO<sub>4</sub>]·4H<sub>2</sub>O. Mo, light blue; O, red; C, grey; N, blue

References:

- [1] A. Wutkowski, B. R. Srinivasan, A. R. Naik, C. Schütt, C. Näther, and W. Bensch, "Synthesis, structure, and photochemistry of an organic heptamolybdate- monomolybdate," *Eur. J. Inorg. Chem.*, pp. 2254–2263, 2011.
- [2] T. N. Kovács, D. Hunyadi, A. L. A. de Lucena, and I. M. Szilágyi, "Thermal decomposition of ammonium molybdates," *J. Therm. Anal. Calorim,* pp. 1013–1021, 2016.