

MS41-P2 New peroxo-compounds of Mo(VI). Completeness of structural research by powder diffraction studies

Wieslaw Lasocha^{1,2}, Alicja Rafalska-Lasocha¹, Adrianna Borowiec¹

1. Faculty of Chemistry, Jagiellonian University, Ingardena 3, 30-060 Krakow, Poland

2. Institute of Catalysis PAS, Niezapominajek 8, 30-239 Krakow, Poland

email: lasocha@chemia.uj.edu.pl

The new peroxo- compounds of Mo(VI), W(VI), or V(V) are important in pharmacy, oxidation processes, removal of impurities and many other applications. Synthesis of new peroxo- compounds provides materials for scientific research, and allows finding new materials having certain oxygen content, given stability, and other tailored properties. In this project a group of molybdenum and pyridine carboxylic acids (pimelic acid and nicotinic acid) compounds were investigated. These compounds exhibit varying stability, from a few minutes (with a tendency to explosive decomposition) to several months. Due to the extremely low stability, part of the obtained compounds can be investigated solely immediately after the synthesis process. Chemical syntheses of these compounds were carried out at low temperatures. Due to their good solubility in water the excess of sediment reagents was used to precipitate them. In these syntheses both pyridine-carboxylic acids and their N-oxides were used. In the presented research we have obtained and determined crystal structures of 5 new compounds, two of them were examined only by powder diffractometry techniques. Only the simultaneous application of single crystal and powder diffraction methods enabled the structural characterization of the family peroxo compounds formed by isomeric pyridine carboxylic acids. The structural studies were performed with the use of global optimization methods (parallel tempering [1]) and laboratory diffractometers. In near future the obtained compounds will be tested in the reactions of catalytic oxidation of hydrocarbons.

Crystallographic data for some investigated compounds: (formula, sg, abc,abg, Vol.).

1. $\text{Na}[\text{MoO}(\text{O}_2)_2 \cdot \text{ONC}_5\text{H}_4\text{COO}] \cdot 2\text{H}_2\text{O}$; C2/c; 25.070, 6.315, 14.588 Å, 92.76°, 2307 Å³
2. $\text{K}[\text{MoO}(\text{O}_2)_2 \cdot \text{ONC}_5\text{H}_4\text{COO}]$; Cc; 7.496, 17.692, 7.5605, 101.45°, 982.68 Å³
3. $\text{NH}_4\{(\text{C}_5\text{H}_4\text{NOCOO})\text{MoO}(\text{O}_2)_2\}$; P-1, 6.169, 6.314, 12.764, 77.99, 81.45, 86.47°, 480.66 Å³
4. $\text{K}_2[\text{MoO}(\text{O}_2)_2 \cdot \text{ONC}_5\text{H}_4\text{COO}]_2$; P-1, 9.138, 10.132, 6.908, 95.66, 110.41, 74.34°, 577.18 Å³

Reference *J. Appl. Cryst.* **35** (2002), 734-743, V. Favre-Nicolin and R. Cerny

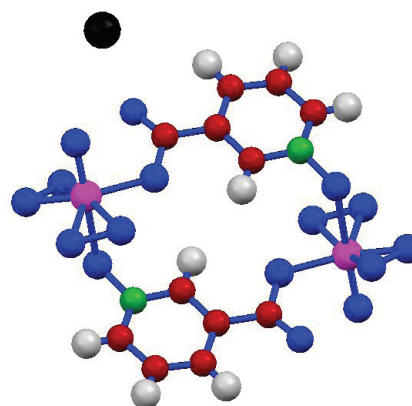


Figure 1. Potassium cation and cyclic anion of oxodiperoxomolybdate nicotinic acid N-oxide salt (4). Colors indicate: magenta Mo, green N, blue O, black K, brown C, gray H.

Keywords: peroxomolybdates, powder diffraction, global optimization techniques