[MS24-P23] Crystal structure studies of a few new octamolybdates.

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Molybdates of amines are a very large and interesting group of compounds, with a large number of possible structures and unusual, though difficult to predict properties, synthesis conditions are still far from fully predictable.
Recently, we synthesised a number of new octamolybdates. These compounds are formed in a few hours, in an acidified aqueous solution containing appropriate amine and molybdic acid (H_2MoO_4 . H_2O). The amines used in the synthesis were: 4-ethylaniline, 4-propylaniline and 4- butylaniline.
The single crystal investigations were performed using a Bruker-Nonius Kappa-CCD diffractometer, radiation MoKα. Structure solution and refinement were carried out with use of SHELXS, SHELXL and WINGX software [2,3].
The investigated compounds clearly tend to form layered structures with distinct separation of inorganic components (octamolybdate anions) and organic groups (cations R-Ph-NH_3^+; Ph – denotes phenyl, R-ethyl, propyl, butyl radicals). As might be expected, we observe disorder in the side groups (the alkyl chains) in organic cations. Compound no. {**1**} is similar to 4-methylanilinium octamolybdate [1], previously investigated in our group, whereas the compounds {**2**} and {**4**} are isostructural.
The obtained compounds are octamolybdates of known types of anions. The compounds {1,2,4} are typical beta-octamolybdates, while the octamolybdate of 4-propylanilinium {3} is an octamolybdate of the gamma type. Two tetragonal pyramids observed in this type of anions are converted into octahedra through the direct connection of one oxygen atom of the carboxyl group to the Mo atom.
The names of the investigated compounds and their formulas; a,b,c, α,β,γ, volume, space groups:
{1} tetrakis(4-ethylanilinium) octamolybdate dihydrate: $Mo_8O_{26}.4\{NH_3\text{-}C6H_4\text{-}C_2H_5\}$. $2H_2O$; 9.8533(2), 27.7217(4), 9.9166(2) [Å], 90.0, 111.406(2), 90.0 [º], 2521.88(7) [Å^3], $P2_1/n$.
{2} tetrakis(4-propylanilinium) octamolybdate monohydrate: $Mo_8O_{26}.4\{NH_3\text{-}C_6H_4\text{-}C_3H_7\}$. H_2O; 10.6714(2), 14.4880(3), 19.3376(4) [Å], 70.781(1), 75.669(1), 79.247(1) [º], 2717.2(1) [Å3], P-1.
{3} hexakis(4-propylanilinium) di(acetato)-octamolybdate: $[Mo_8O_{26}\ (CH_3COO)_2]$. $4\{NH_3\text{-}C_6H_4\text{-}C_3H_7\}$; 20.9858(7), 12.1212(2), 15.0835(3) [Å], 90.0, 91.211(2), 90.0 [º], 3836.0(2) [Å3], $P2_1/c$.
{4} tetrakis(4-buthylanilinium) octamolybdate: $Mo_8O_{26}.4\{NH_3\text{-}C_6H_4\text{-}C4H_9\}$. H_2O; 10.5427(2), 14.6047(3), 20.6901(4) [Å], 70.207(1), 84.408(1), 80.400(1) [º], 2952.7(1) [Å3], P-1.
We also plan to conduct a series of tests for chemical compounds obtained in this study, such as investigations of their thermal decomposition processes (DSC/DTG) and the study of their catalytic properties (e.g. in oxidation processes). Research is also planned to complete phase diagram R-Ph-NH_3 - MoO_3 - H_2O. These studies will be directed towards obtaining the penta-and trimolybdates of relevant amines.

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