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SHORT COMMUNICATION

CRYSTAL STRUCTURE OF $(Cy_2NH_2)_2MoO_4 \cdot 2H_2O$

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Abstract: The structure of this salt consists of a 3D structure. The anion interacts with NH_2 group of the cation and H_2O molecules respectively through N-H---O and O-H---O hydrogen bonds. The anion MoO_4^{2-} is almost perfectly tetrahedral. The four values of the lengths of Mo-O bonds are all equal to 1.7613 Å (12).

Keywords: *crystal structure, dicyclohexyl ammonium, hydrogen bonds, molybdate, 3D structure*

INTRODUCTION

Targeting new coordination complexes and adducts using alkyl-or aryl ammonium salts which are generally soluble in organic solvents is one of our main axis of research since decades [1- 4]. In this dynamic we have initiated here the study of the interactions between Cy_2NH and MoO_3 in water which has yielded the studied salt X-ray structure of which has been determined in this work.

EXPERIMENTAL

Synthesis

Mixing Cy_2NH with MoO_4H_2 in water precipitation occurs. The precipitate was stirred around two hours and filtered off. The solution, submitted to a slow solvent evaporation affords crystals suitable for X-ray work.

Crystal data and structure refinement

Empirical formula: $C_{24}H_{52}MoN_2O_6$; Formula weight: 560.62; Crystal system: Tetragonal; Space group: I_{42d}; *a* (Å): 12.61070(10); *b* (Å): 12.61070(10); *c* (Å): 17.3035(3); β (°): 90; *V* (Å^3): 2751.77(66); *Z:* 4; ρ_{calc} (mgm^3): 1.353; μ(Mo-k$_\alpha$) (mm^{-1}): 0.515; F(000): 1200; Refl'ns collected: 22497; Independent refl'ns [R(int)]: 1576 [0.0408]; Refl'ns observed (>2σ): **6266**; Absorption correction: Semi-empirical from equivalents; Max., min. transmission: 0.9503, 0.9268; Refinement method: Full-matrix least-squares on F^2; Goodness-of-fit: 1.117; Final R indices [I>2σ(I)]: 0.0180, 0.0441; R indices (all data): 0.0202, 0.0449; Largest diff. peak and hole (eÅ^3): 0.457,-0.506; program(s) used to refine structure: *SHELXL97*, *ORTEP-3 for Windows*, *WinGX* [5-7] CCDC deposition number: 856380.

RESULTS AND DISCUSSION

Its structure consists of tetrahedral MoO_4^{2-} anion with equal Mo-O distances of 1.7613 Å (12). Each oxygen interacts with a water molecule through O-H---O hydrogen bonds and a NH_2 group through N-H----O hydrogen bonds. When comparing the Mo-O bond distances in this work with Mo=O and Mo-O bonds distances in $(NBu_4)_2[(Ph_2Sn)_2(\mu\text{-}OH)_2(\mu\text{-}MoO_4)_2]\cdot 4CH_2Cl_2$ [8], while distances half way between Mo=O and Mo-O distances were expected, the bonds in $(Cy_2NH_2)_2MoO_4\cdot 2H_2O$ appear longer, indicating the strength of the NH---O (1.854 Å) and OH---O (2.054 Å) hydrogen bonds. This molybdate salt is isostructural with $(Cy_2NH_2)_2WO_4\cdot 2H_2O$ [9].

Selected bonds: Mo-O(1) 1.7613(12); Mo-O(1)#11.7613(12); Mo-O(1)#2 1.7613(12); Mo-O(1)#31.7613(12); N(1)-C(1) 1.5052(19); N(1)-C(1)#4 1.5052(19); N(1)-H(1) 0.91(2); C(1)-C(6)1.525(2); C(1)-C(2)1.531(2); C(1)-H(1A) 1.0000;

Selected angles: O(1)-Mo-O(1)#2 110.44(8); O(1)#1-Mo-O(1)#2 108.99(4); O(1)-Mo-O(1)#3108.99(4); O(1)#1-Mo-O(1)#3 110.44(8); O(1)#2-Mo-O(1)#3108.99(4); C(1)-N(1)-C(1)#4118.10(18); C(1)-N(1)-H(1)109.6(13); C(1)#4-N(1)-H(1)107.2(13);

Symmetry transformations used to generate equivalent atoms: y, -x+1, -z+1; -x+1, -y+1, z; -y+1, x, -z+1; x, -y+3/2, -z+5/4.

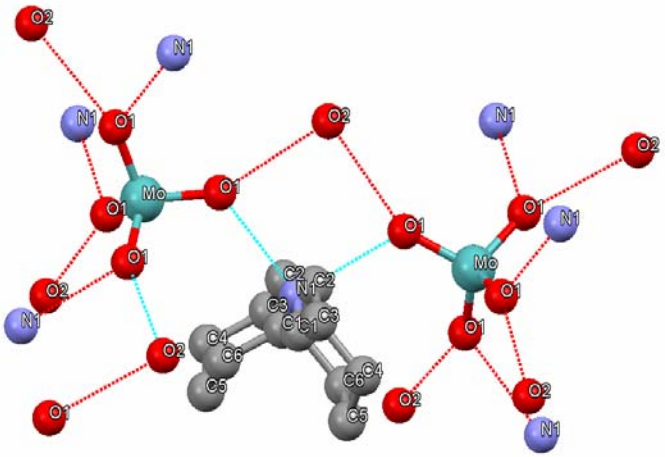

Figure 1. *Crystal structure of* $(Cy_2NH_2)_2MoO_4 \cdot 2H_2O$

CONCLUSION

The x-ray structure of $(Cy_2NH_2)_2MoO_4 \cdot 2H_2O$ consists of a central molybdate surrounded by four cations and four water molecules. Two oxygen atoms belonging to two different molybdates are connected by one bridging cation and one bridging water molecule through NH---O and OH---O hydrogen bonds leading to a 3D structure.

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