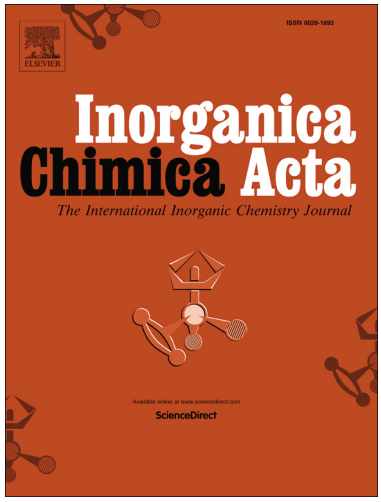

Research paper

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Synthesis of hexavalent molybdenum formo- and aceto-hydroxamates and deferoxamine via liquid-liquid metal partitioning.

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Abstract

We report a new method of crystal growth and synthesis based on liquid-liquid partitioning that allows for isolation and in-depth characterization of molybdenyl bis(formohydroxamate), **Mo-FHA**, molybdenyl bis(acetohydroxamate), **Mo-AHA**, and molybdenyl deferoxamine, **Mo-DFO**, for the first time. This novel approach affords shorter crystal growth time (hourly timeframe) without sacrificing crystal size or integrity when other methods of crystallization were unsuccessful. All three Mo complexes are characterized in solution via FTIR, NMR, UV-vis, and EXAFS spectroscopy. Mo-AHA and Mo-FHA structures are resolved by single crystal X-ray diffraction. Using the molybdenyl hydroxamate structural information, the speciation of Mo in a siderophore complex (Mo-DFO) is determined via complimentary spectroscopic methods and confirmed by DFT calculations. ESI-MS verifies that a complex of 1:1 molybdenum to deferoxamine is present in solution. Additionally, the Mo solution speciation in the precursor organic phase, $\mathbf{MoO_2(NO_3)_2HEH[EHP]_2}$ (where HEH[EHP] is 2-ethylhexylphosphonic acid