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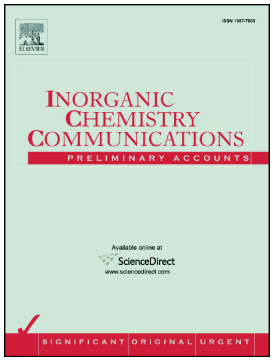

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ACCEPTED MANUSCRIPT

Synthesis and crystal structure of hexacerium(IV) cluster-containing Keggin polyoxometalate trimer

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ABSTRACT

A novel hexacerium(IV) cluster-containing polyoxometalate (POM), $K_{18}[\{Ce_6(\mu_3\text{-}O)_4(\mu_3\text{-}OH)_4(OAc)_2\}(A\text{-}\alpha\text{-}SiW_9O_{34})_2(\alpha\text{-}SiW_{11}O_{39})]\cdot 37H_2O$ (**$K_{18}Ce_6$-POM**), was unexpectedly obtained by a reaction of the potassium salt of open-Dawson POM, $K_{13}[\{K(H_2O)_3\}_2\{K(H_2O)_2\}\alpha,\alpha\text{-}Si_2W_{18}O_{66}]\cdot 19H_2O$ (**K-open POM**), with $(NH_4)_2[Ce(NO_3)_6]$, in a 0.5 M KOAc/HOAc buffer solution (pH 4.8) at 80 °C, and unequivocally characterized by X-ray crystallography, ^1H, ^{13}C, and ^{29}Si NMR spectroscopy, FTIR spectroscopy, complete elemental analysis, and TG/DTA. X-ray crystallography revealed that the monolacunary Keggin unit, $[\alpha\text{-}SiW_{11}O_{39}]^{8-}$, and the two trilacunary Keggin units, $[A\text{-}\alpha\text{-}SiW_9O_{34}]^{10-}$, are connected by a central Ce_6-oxo/hydroxide cluster moiety, $[Ce_6(\mu_3\text{-}O)_4(\mu_3\text{-}OH)_4(OAc)_2]^{10+}$, to form a trimeric Keggin polyanion, $[\{Ce_6(\mu_3\text{-}O)_4(\mu_3\text{-}OH)_4(OAc)_2\}(A\text{-}\alpha\text{-}SiW_9O_{34})_2(\alpha\text{-}SiW_{11}O_{39})]^{18-}$ (**Ce_6-POM**). ^1H, ^{13}C, and ^{29}Si NMR spectra in D_2O suggested that the **Ce_6-POM** was obtained as a single species and its molecular structure was maintained in solution.

Keywords:
Polyoxometalate/ Cerium(IV)/ Cluster / Oligomer / ^{29}Si NMR / X-ray structure analysis

Polyoxometalates (POMs) are discrete metal oxide clusters that are of current interest as soluble metal oxides with applications in catalysis, medicine, and materials science[1]. The preparation of POM-based materials is therefore an active field of research. Lacunary POMs are vacant derivatives of the saturated POM structures. They can be viewed as multidentate inorganic ligands, which can coordinate to transition metal ions. The lacunary sites of these POMs can encapsulate a variety of transition metal ions, resulting in the formation of multi-transition metal aggregates, and unstable

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