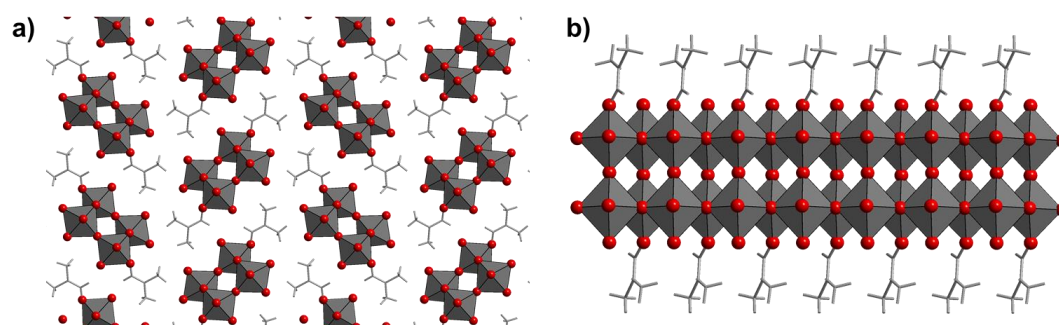


## Synthesis and structural control of one-dimensional molybdenum oxide chains

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Molybdenum oxides are known to exhibit diverse crystal structures and morphologies.<sup>1</sup> For example,  $\alpha$ -MoO<sub>3</sub>, a two-dimensional sheet-like structure, has been utilized as a catalyst material and electrode material through intercalation. However, conventional  $\alpha$ -MoO<sub>3</sub> faces several issues, such as a small surface area and the absence of micropores, when employed in catalytic materials. In this study, one-dimensional chain-like structures, resembling slices of a single sheet of  $\alpha$ -MoO<sub>3</sub>, were successfully synthesized and crystallized (**1**). X-ray crystallographic analysis revealed that **1** was crystallized in the primitive monoclinic  $P2_1/c$  with the short  $a$ -axis and was the bundle of unique one-dimensional MoO<sub>3</sub> subnanofibers along the  $a$ -axis (Figure 1). Each fiber was composed of two edge-sharing octahedral {MoO<sub>6</sub>} zigzag chains connected by corner-sharing Mo–O–Mo bonds, which was essentially the same structure obtained by cutting one layer of two-dimensional  $\alpha$ -MoO<sub>3</sub> sheet into 8.0 Å wide strips along [001] direction. Importantly, O atoms of *N,N*-dimethylformamide molecules were coordinated to Mo atoms to prevent fibers from further condensation into two-dimensional structures. Inspired by this unique structure of **1**,  $\alpha$ -MoO<sub>3</sub> was synthesized by calcination of **1** at different temperatures. Consequently, structural control of the material through calcination was possible, resulting in the preparation of unique oxidation catalysts with properties not present in conventional  $\alpha$ -MoO<sub>3</sub>.



**Figure 1.** a) Crystal structure of **1** along  $a$ -axis and b) fiber structure of **1**.

1) I. A. de Castro, R. S. Datta, J. Z. Ou, A. Castellanos-Gomez, S. Sriram, T. Daeneke, K. Kalantar-zadeh, *Chem. Mater.* **2017**, 29, 1701619.