Thermal decomposition of ammonium hexachloroosmate

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Structural changes of \((\text{NH}_4)_2[\text{OsCl}_6]\) occurring during thermal decomposition in a reduction atmosphere have been studied in situ using combined energy-dispersive X-ray absorption spectroscopy (ED-XAFS) and powder X-ray diffraction (PXRD). According to PXRD, \((\text{NH}_4)_2[\text{OsCl}_6]\) transforms directly to metallic Os without the formation of any crystalline intermediates but through a plateau where no reactions occur. XANES and EXAFS data by means of Multivariate Curve Resolution (MCR) analysis show that thermal decomposition occurs with the formation of an amorphous intermediate \([\text{OsCl}_4]^x\) with a possible polymeric structure. Being revealed for the first time the intermediate was subjected to determine the local atomic structure around osmium. The thermal decomposition of hexachloroosmate is much more complex and occurs within a minimum two-step process, which has never been observed before.

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