Supplementary Material (ESI) for Chemical Communications

Novel Cerium(IV) Heteropolyoxotungstate Containing Two Types of Lacunary Keggin Anions

Supporting Information

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Single-Crystal X-ray Diffraction: Details on Crystal Structure Solution and Refinement

A suitable single crystal was mounted on a glass fibre using perfluoropolyether oil.[1] Data were collected at 180(2) K on a Nonius Kappa charge coupled device (CCD) area-detector diffractrometer (Mo K_α graphite-monochromated radiation, $\lambda = 0.7107$ Å), equipped with an Oxford Cryosystems cryostream and controlled by the Collect software package.[2] Images were processed using the software packages of Denzo and Scalepack,[3] and the data were corrected for absorption by using the empirical method employed in Sortav.[4] The largest crystal from several batches was a very small yellow block (minimum dimension 0.05 mm) and diffracted weakly at high angles. Applying a cut-off at 1.01 Å $2\theta = 20.58°$ resolution results in a good number of reflections observed at the 2σ level and a good merging *R* value. Collection of higher-angle data is only likely to be possible using a synchrotron or a rotating anode source. However, given the quality of the structure refinement using this data set, efforts in this direction are not warranted. The structure was solved by the direct methods of SHELXS-97,[5] and refined by full-matrix least squares on F^2 using SHELXL-97.[6] Atoms were directly located from difference Fourier maps and refined, when possible, with anisotropic displacement parameters. O-atoms have been refined with independent isotropic displacement parameters, U_{iso}. Several crystallisation water molecules [O(12W), O(13W), O(14W), O(15W), O(16W), O(18W) and O(19W)] were found to be severely affected by thermal disorder and were modelled with fixed occupancy rates of 50%. H-atoms could not be located from difference Fourier maps, but have been included in the empirical formula.

References

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