Electronic Supplementary Information (ESI)

Synthesis, crystal structure and magnetic properties of an oxalato-bridged $Re^{IV}Mo^{VI}$ heterobimetallic complex

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Preparation of compound 1.

The precursor $(PPh_4)_2[ReCl_4(ox)]$ was prepared according to the literature procedure described for $(AsPh_4)_2[ReCl_4(ox)]$ but using PPh_4^+ instead of $AsPh_4^+$ (*See* Ref. 7a of the main text). Compound **1** was prepared by pouring a solution of $(PPh_4)_2[ReCl_4(ox)]$ (54.7 mg, 0.05 mmol) in 25 cm^3 of acetonitrile into another containing 13.6 mg (0.05 mmol) of $MoCl_5$ in 25 cm^3 of ethyl acetate. X-ray suitable orange crystals of **1** were grown from the resulting orange-yellowish solution after three days by slow evaporation at room temperature. They were filtered off and washed with diethyl ether. The same compound was obtained by performing the synthesis under an argon atmosphere. Yield: *ca*. 52%. Found: C, 46.15; H, 2.97. Calc. for $C_{50}H_{40}Cl_6P_2O_6MoRe$ (**1**): C, 46.43; H, 3.12. The Re:Mo molar ratio in **1** was found to be 1:1 by X-ray microanalysis on a microcrystalline sample. This Re:Mo molar ratio was determined by means of a Philips XL-30 scanning electron microscope (SEM) equipped with an X-ray microanalysis system from the Central Service for the Support to Experimental Research (SCSIE) of the University of Valencia. IR (KBr pellet / cm^{-1}): bands associated to the oxalato ligand appear at 1656vs (ν_{asCO}) and 813s (δ_{OCO}). Compound **1** is soluble in common organic solvents such as acetone, acetonitrile or *N*,*N*-dimethylformamide at room temperature.

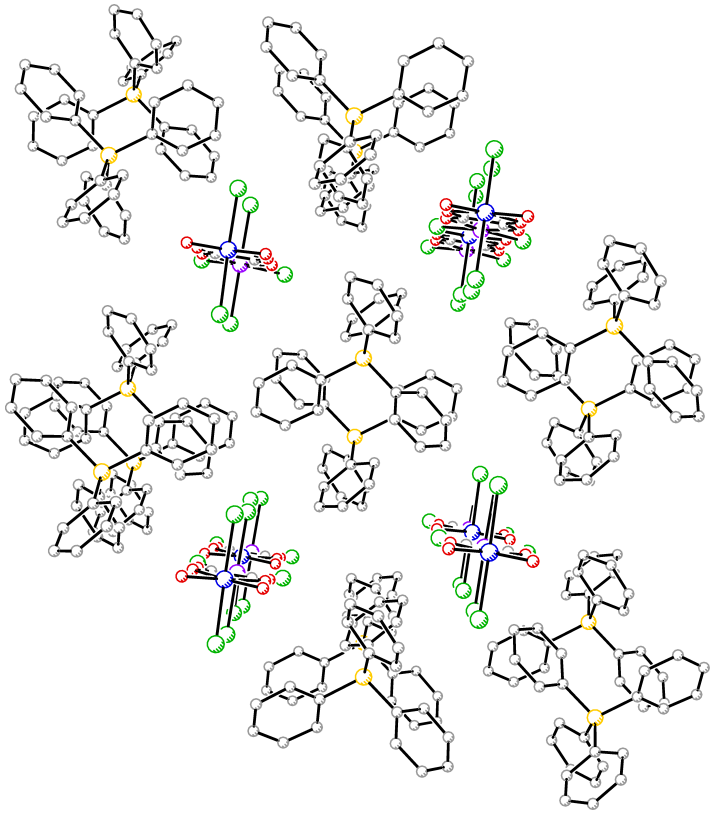

Figure S1. A view along the *b* axis of the packing in **1** showing the arrangement of the cations and anions.

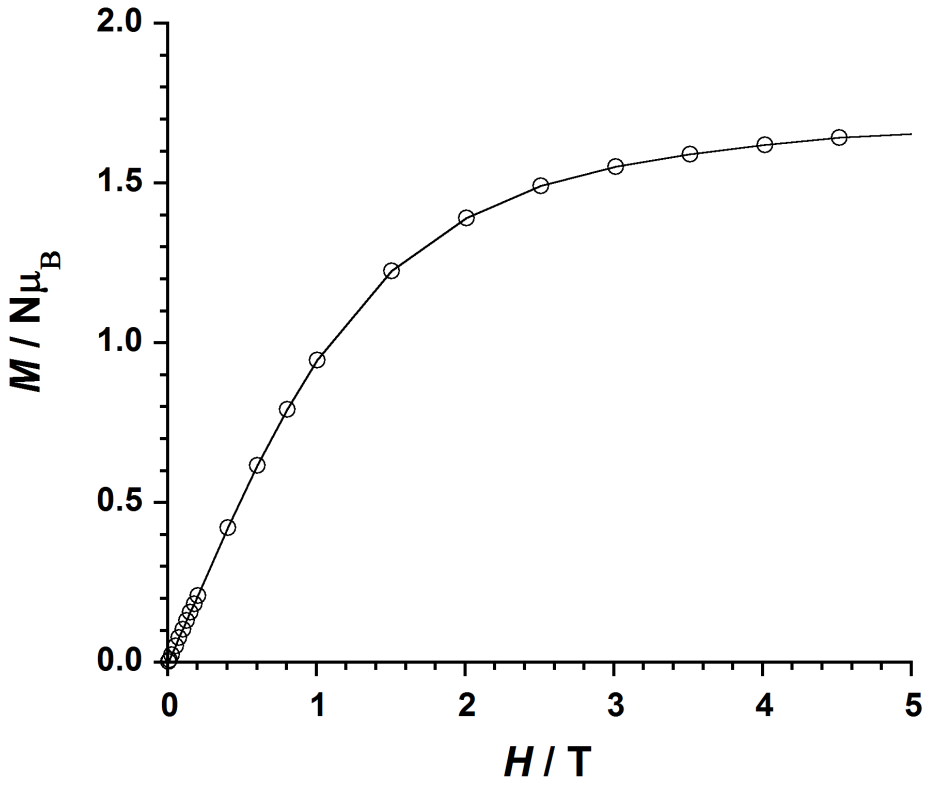

Figure S2. Field dependence of the magnetization at 2.0 K for **1** (the solid line is an eye guide).