Supporting Information

A Novel Sandwich-Type Polyoxometalate Compound with Visible Light Photocatalytic H₂ Evolution Activity

Zhenyu Zhang\textsuperscript{a}, Qipu Lin\textsuperscript{a}, Shoutian Zheng,\textsuperscript{b}

Xianhui Bu\textsuperscript{b}, Pingyun Feng\textsuperscript{a}*  

\textsuperscript{a}Department of Chemistry  
University of California,  
Riverside, CA 92521, U. S. A  
Fax: (+1)951-8274713; Tel: (+1)951-8272042

\textsuperscript{b}Department of Chemistry and Biochemistry  
California State University, Long Beach  
1250 Bellflower Boulevard  
Long Beach, CA 90840, USA

*To whom correspondence should be addressed. E-mail: pingyun.feng@ucr.edu
Single crystal analysis of 1

The data were collected on Bruker APEX II diffractometer equipped with a fine focus, 2.0 kW sealed tube X-ray source (MoKα radiation, λ = 0.71073 Å) operating at 50 kV and 30 mA. The crystal data: Triclinic, P-1, a = 13.6358(16)Å, b = 20.7414(18)Å, c =16.0530(24)Å, α=100.093(2)°, β=90.703(2)°, γ=101.961(2)°, V = 4367.2(8) Å³, Z=1, Dc= 4.391 g cm⁻¹, 21615 measured reflections, R1= 0.0584 for 16067 reflections (I > 2σ(I)), GOF =0.997. The structure was solved by direct methods and refined by full-matrix least-squares method based on |F²| using the SHELXTL 5.1 programs suite. All non-hydrogen atoms were refined with anisotropic displacement parameters.
Figure S2. Simulated XRD pattern(a) and measured XRD pattern of 1. ICP elemental analysis(The PerkinElmer OPTIMA 2000 ICP optical emission spectrometer) indicated the ratio of K: Sn: Si: W in compound 1 is 10.38:3.88:2.00:17.37 which support the purity and the composition of the compound 1.

Figure S3. FT-IR spectrum of compound 1. The peaks at 993, 937, 890 and 789 cm\(^{-1}\) can be attributed to \(\nu\)(Si-O) and \(\nu\)(W-O).
Figure S4. TGA curve of compound 1.

Figure S5. UV-Vis spectra of 1 before (black) and after (red) 5 runs of the photocatalytic reactions with 0.5% Pt nanoparticles as co-catalyst in 20% methanol solution.
Figure S6. Time course of H₂ evolution from photocatalytic reaction (>400nm) on compound 2.

Figure S7. Time course of H₂ evolution from photocatalytic reaction (>400nm) on 3% Pt loaded Ta₂N₅ in 20% methanol solution. Ta₂N₅ was prepared according to literature (G. Hitoki, A. Ishikawa, T. Takata, J.N. Kondo, M. Hara and K. Domen, Chem. Lett. 2002, 7, 736)