

Supporting Information

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

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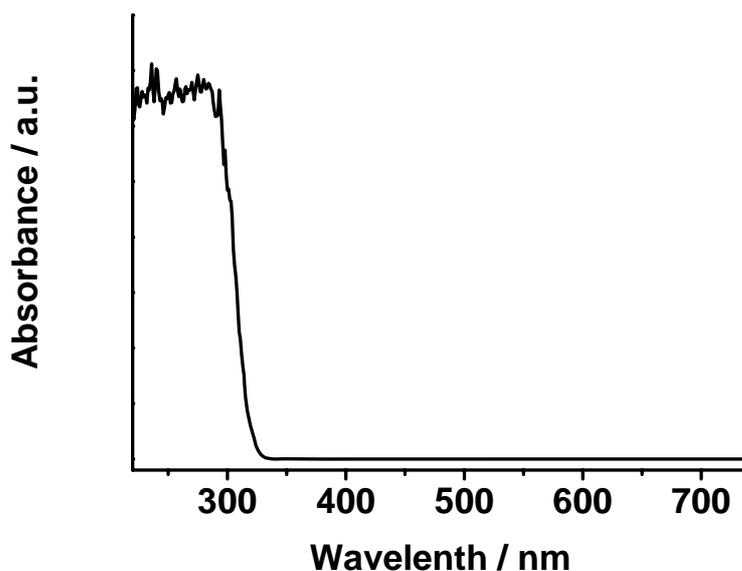


Figure S1. UV-Vis spectrum of Na₁₀[α-SiW₉O₃₄]·xH₂O

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 (MoK α radiation, $\lambda = 0.71073 \text{ \AA}$) operating at 50 kV and 30 mA. The crystal data: Triclinic, P-1, $a = 13.6358(16) \text{ \AA}$, $b = 20.7414(18) \text{ \AA}$, $c = 16.0530(24) \text{ \AA}$, $\alpha = 100.093(2)^\circ$, $\beta = 90.703(2)^\circ$, $\gamma = 101.961(2)^\circ$, $V = 4367.2(8) \text{ \AA}^3$, $Z = 1$, $D_c = 4.391 \text{ g cm}^{-3}$, 21615 measured reflections, $R_1 = 0.0584$ for 16067 reflections ($I > 2 \sigma(I)$), $\text{GOF} = 0.997$. The structure was solved by direct methods and refined by full-matrix least-squares method based on $|F^2|$ using the *SHELXTL 5.1* programs suite. All non-hydrogen atoms were refined with anisotropic displacement parameters.

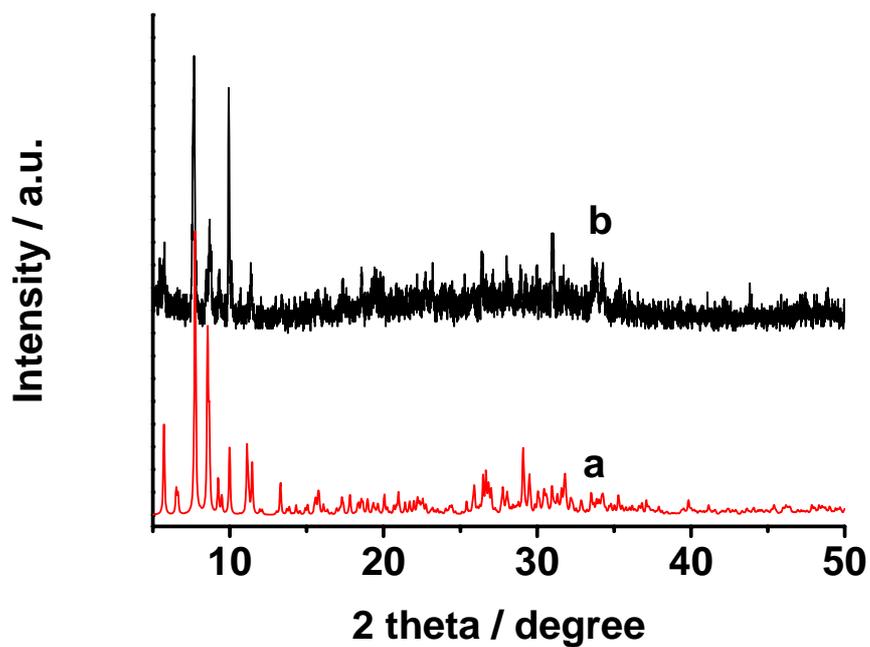


Figure S2. Simulated XRD pattern(a) and measured XRD pattern of **1**. ICP elemental analysis(The PerkinElmer OPTIMA 2000 ICP optical emission spectrometer) indicated the ratio of K: Sn: Si: W in compound **1** is 10.38:3.88:2.00:17.37 which support the purity and the composition of the compound **1**.

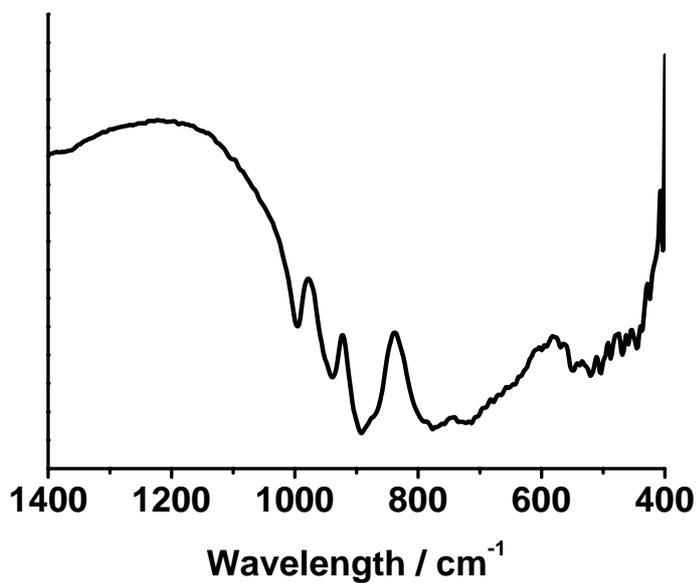


Figure S3. FT-IR spectrum of compound **1**. The peaks at 993, 937, 890 and 789 cm⁻¹ can be attributed to $\nu(\text{Si-O})$ and $\nu(\text{W-O})$.

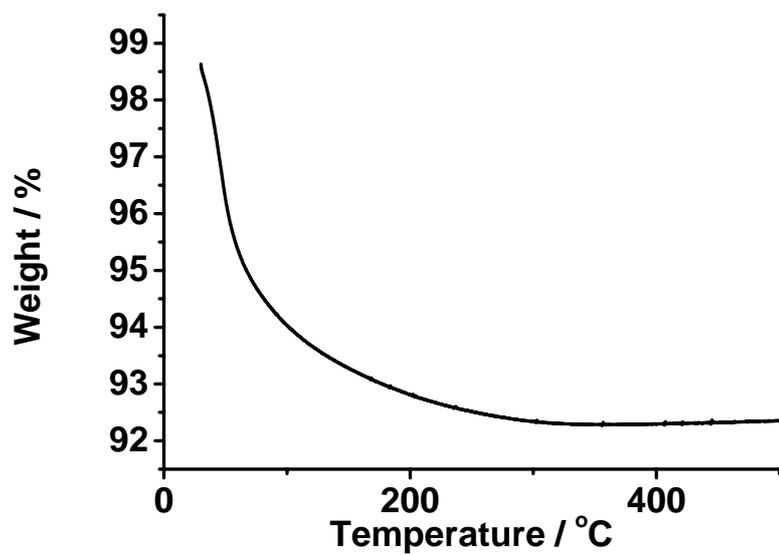


Figure S4. TGA curve of compound 1.

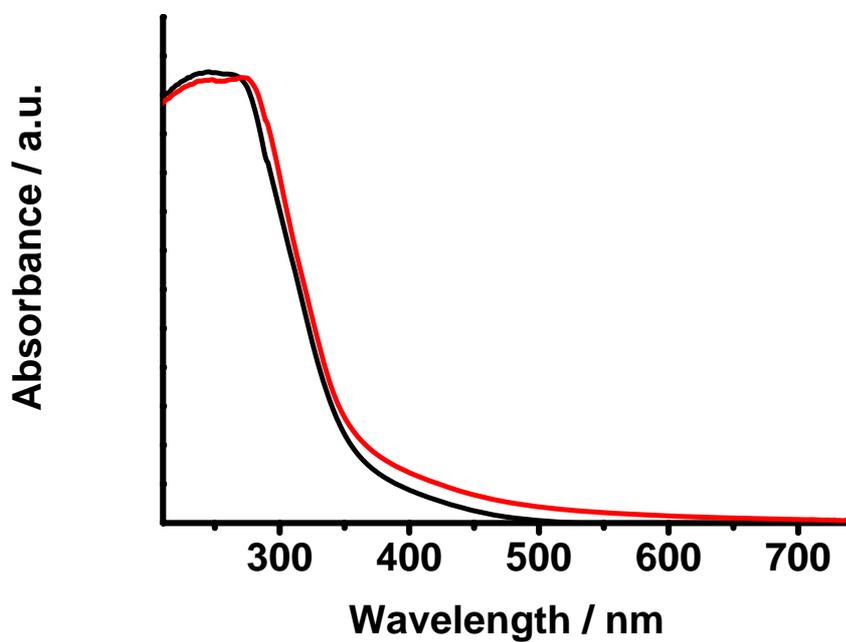


Figure S5. UV-Vis spectra of 1 before (black) and after (red) 5 runs of the photocatalytic reactions with 0.5% Pt nanoparticles as co-catalyst in 20% methanol solution.

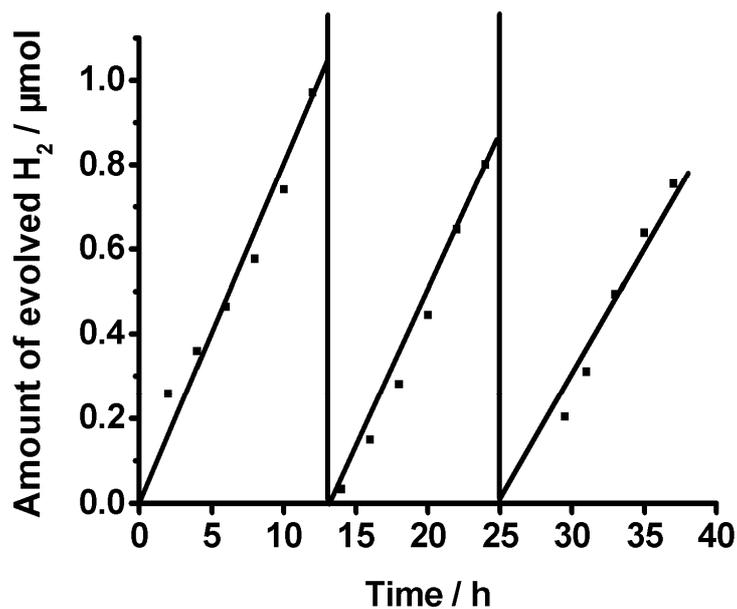


Figure S6. Time course of H₂ evolution from photocatalytic reaction (>400nm) on compound 2.

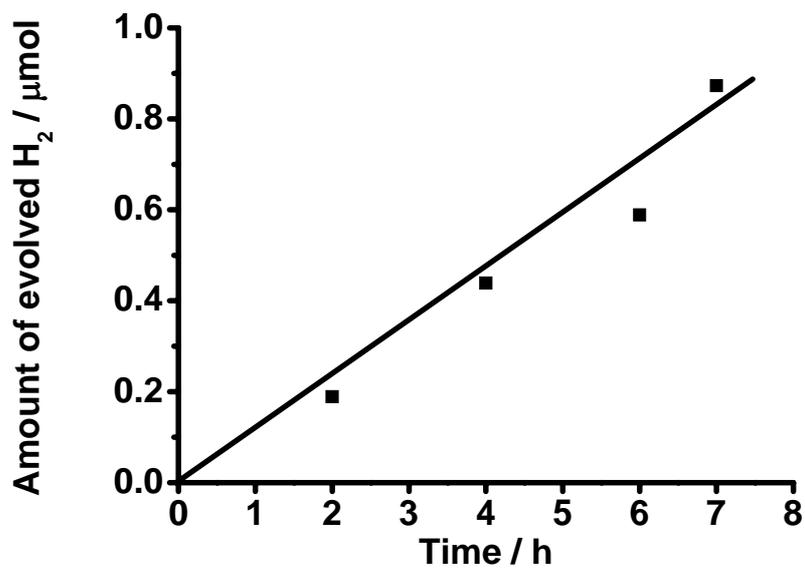


Figure S7. Time course of H₂ evolution from photocatalytic reaction (>400nm) on 3% Pt loaded Ta₃N₅ in 20% methanol solution. Ta₃N₅ was prepared according to literature (G. Hitoki, A. Ishikawa, T. Takata, J.N. Kondo, M. Hara and K. Domen, *Chem. Lett.* **2002**, 7, 736)