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Supporting Information

1. Crystal structure refinement

The structures were solved using the direct methods, completed by subsequent difference Fourier syntheses, and refined by full matrix least-squares procedures on F² with WinGX (1.70) program package (L. J. J. Farrugia, Appl. Crystallogr. 32, 837 (1999)). The CCDC data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. CCDC number 1451651 for **1**.

2. Titrimetric analysis of compound 1 with potassium permanganate

The molar concentration of potassium permanganate was 0.350mmol/L, which was calibrated by using the standard solution of sodium oxalate. Solution A: 0.4137g compound **1** was disolved in 0.2M H₂SO₄, and then the solution was transfered to 10 ml volumetric flask. 1.00 ml solution A, 1.00 ml 2M H₂SO₄ and 20 ml water were transfered to erlenmeyer flask, then titrated with standard potassium permanganate solution. In the parallel titrimetric experiments of three times, the consumed volume of potassium permanganate solution was 13.21 ml, 13.19 ml, and 13.22 ml, respectively, which corresponds to the ammount of Fe²⁺ at 0.0231 mmol, 0.0230 mmol, and 0.0231 mmol. Thus, the mean of [Fe²⁺]_{found} is 0.0231 mmol, which is in agreement with the [Fe²⁺]_{calc} (0.0231 mmol).

3. (GC/CNTs/Chitosan/POM) modified electrode.

The modified electrode was prepared as follow: The GCE was polished before each experiment with 1.0, 0.3 and 0.05 μ m α -Al₂O₃ powder, respectively, and rinsed with water between each polishing step. Then, washed successively with 1:1 nitric acid, acetone and water in ultrasonic bath and dried in air. 10 mg chitosan was dissolved in 10 ml 0.1 M acetic acid with magnetic stirring for some time and 5 mg oxidized CNTs were

dispersed in 10 ml 0.1 M acetic acid with ultrasonication for 15 min. The CNTs–chitosan composites were prepared by mixing the above two solutions by ultrasonic agitation over 30 min. Then, 6 µl of the resulting homogeneous solution was cast on the surface of cleaned GCE, dried at room temperature for 10 h. In order to form a uniform CNTs–chitosan composite film at the GCE surface, a beaker was covered over the electrode so that water can evaporate slowly. Before every measurement, the CNTs–chitosan modified electrode was activated in 0.25 M NaAc/HAc buffer solution by successive cyclic sweeps between 0.0 and 1.2 V until the voltammetric curve was stable. The activation treatments made the CNTs–chitosan modified GCE possess not only sensitive and stable electrochemical response, but also low memory effect. After that, the CNTs–chitosan coated electrode was dipped in 1 mM compound **1** (pH 5.45 NaAc/HAc buffer solution) for 20 min. Then, the electrode was rinsed and immersed in doubly distilled water for another 20 min to remove excess physical adsorbed compound **1**.



Fig. S1 IR spectrum of compound 1.

In Fig. S1, four characteristic sharp vibration peaks resulting from the $[SbW_9O_{33}]^{9-}$, namely v(W–Od), v(W–Oc), and v(W–Ob), appear at 933, 708, and 669 cm⁻¹ for **1**, the shoulder peak of 851 cm⁻¹ is attributed to v(Sb–Oa).



Fig. S2 Cyclic voltammograms of compound 1 in 0.25 M NaAc/HAc buffer solution.



Fig. S3 The CVs of the (GC/CNTs/Chitosan/POM) modified electrode in 0.25 M NaAc/HAc buffer solution containing (a) 0μ M (b) 0.5μ M (c) 1.0μ M (d) 2.0μ M and (e) 4.0μ M dopamine. Scan rate 50 mV s⁻¹.



Fig. S4 Current–time responses for the (GC/CNTs/Chitosan/POM) modified electrode at 0.3 V with the addition of 0.5 to 14.0 μ M dopamine. (a, compound **1**; b, [Sb₂W₁₈Fe^{III}₃O₇₆]⁸⁻)



Fig. S5 Current-time responses for the (GC/CNTs/Chitosan/POM) modified electrode at 0.3 V with the addition of 0.5 to 14.0 μ M dopamine.

The Fig. S5 shows that reused modified electrode has almost the same amperometric response in the dopamine solution. With the increase of the usage frequency, the modified electrode has a worse linear response.