A 2D/2D NiCo-MOF/Ti₃C₂ heterostructure for

simultaneous detection of acetaminophen, dopamine and

uric acid by differential pulse voltammetry

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Fig. S1 SEM image and the corresponding elemental mapping images of NiCo-MOF/Ti $_3C_2$.



Fig. S2 SEM images of (a) CCE and (b) NiCo-MOF/Ti $_3C_2$ /CCE.



Fig. S3 (a) The N₂ adsorption-desorption curves and (b) the corresponding pore size distribution of NiCo-MOF and NiCo-MOF/Ti₃C₂.



Fig. S4 CV curves of (a) NiCo-MOF/Ti₃C₂/CCE, (c) Ti₃C₂/CCE, (e) NiCo-MOF/CCE and (g) CCE recorded in 0.1 M KCl containing 1 mM K₃[Fe(CN)₆] at scan rates varied from 0.02 to 0.30 V s⁻¹; (b, d, f and h) the dependence plots of peak current vs. square root of scan rate (v^{1/2}).



Fig. S5 (a) Plot of ψ vs. $\Delta E_p \times n$ for different electrodes under disparate scan rates (30, 40, 60, 80, 100 mV/s); (b) the relationships of ψ vs. 32.80 v^{-1/2}



Fig. S6 (a, c and e) CVs on NiCo-MOF/Ti₃C₂/CCE in 0.1 M PB with different pH values (5.0 to 9.0) containing 20 μM AP, DA and UA; (b, d and f) the plots of peak current vs. pH value, and the peak potential vs. pH value.



Fig. S7 (a, d and g) CV curves of NiCo-MOF/Ti₃C₂/CCE in 0.1 M PB (pH 7.4) containing 30 μ M AP, DA and UA recorded at different scan rates (40 to 200 mV s⁻¹);

(b, e and h) the linearity plots of I_p vs. $(\upsilon^{1/2})$ and (c, f and i) E_p vs. log $\upsilon.$



Fig. S8 Crystal structure of NiCo-MOF and structure of AP, DA and UA.



Fig. S9 The DPV curves of five successive measurements with the same electrode and six different measurements with disparate batches of electrodes.



Fig. S10 The DPV curves of NiCo-MOF/Ti $_3C_2$ /CCE toward (a) AP (200 μ M), (b) DA (100 μ M) and (c) UA (100 μ M) within 14 days.



Fig. S11 (a) The current response of NiCo-MOF/Ti₃C₂/CCE toward 20 μ M AP, DA UA within 14 days; (b) SEM image of NiCo-MOF/Ti₃C₂/CCE after electrochemical



Fig. S12 (a) EIS of NiCo-MOF/Ti₃C₂/CCE after immersing in FBS solution under different dilution ratios.



Fig.S13 (a) DPV curves in diluted serum containing standard solution DA(0, 7, 30 μM), UA(0, 18, 50 μM) and AP (0, 4,70 μM); (b) DPV curves in diluted urine containing standard solution DA(0, 90, 200 μM), UA (0, 80, 180 μM) and AP (0, 85, 250 μM).

The procedures of actual samples detection are as follows:

(i) The serum was purchased from KEJING Biology Co. Ltd, and got urine from normal people. Before the experiment, the serum and urine were centrifuged to obtain the supernatant, and then diluted 100 times (by volume) with 0.1 M PB (pH 7.4).

- (ii) Pipette 10 mL of the diluted samples into vial to proceed DPV analysis by a three-electrode system, and then the amounts of DA and UA were determined according to the linear equation of analyte concentration vs. current response in PB.
- (iii) The standard addition method is used next to assess the practicability of the fabricated biosensor. The known amount of DA, UA and AP were added into the diluted samples, and were measured their DPV curves. The current response was compared with the standard curve in PB to calculate the recovery.

| Eletrodes | Techniques | Li | LOD (µM) | | | Ref. | | |
|--|------------|----------|----------|----------|-------|-------|-------|-----------|
| | | AP | DA | UA | AP | DA | UA | |
| K ₂ Fe ₄ O ₇ -GCE | DPV | _ | 1-140 | _ | _ | 0.22 | _ | 1 |
| Au NPs@3D GR/ITO | DPV | _ | 0.1–60 | 0.1–60 | _ | 0.1 | 0.1 | 2 |
| HKUST-1/GCE | DPV | 12.5–275 | 12.5–175 | _ | 0.092 | 0.11 | _ | 3 |
| Pt/CeO ₂ @Cu ₂ O-CPE | DPV | 0.5-160 | 0.5-160 | _ | 0.091 | 0.079 | _ | 4 |
| MoS ₂ -TiO ₂ /rGO/SPE | DPV | 0.1–125 | _ | _ | 0.046 | _ | _ | 5 |
| MIPs/pThi/NPG | DPV | _ | 0.3–100 | _ | 0.1 | _ | _ | 6 |
| CNCo/GCE | DPV | _ | _ | 2–110 | _ | _ | 0.83 | 7 |
| Cu/Cu _x O NPs/PGE | DPV | _ | 0.3–53 | _ | _ | 1.07 | _ | 8 |
| MIP/NPGL | DPV | _ | 2-180 | 5-160 | _ | 0.3 | 0.4 | 9 |
| NiCo-MOF/Ti ₃ C ₂ /CCE | DPV | 0.01-400 | 0.01-300 | 0.01-350 | 0.008 | 0.004 | 0.006 | This work |

 Table S1 The reported methods towards AP, DA and UA detection compared to

NiCo-MOF/Ti₃C₂/CCE.

| | Analytes | $I_{l}/\mu A$ | $I_2/\mu A$ | $I_3/\mu A$ | $I_4/\mu A$ | $I_5/\mu A$ | $I_6/\mu A$ | RSD/% |
|------------|----------|---------------|-------------|-------------|-------------|-------------|-------------|-------|
| The same | AP | 118.77 | 121.07 | 124.14 | 119.46 | 120.92 | - | 1.50% |
| electrode | DA | 204.65 | 208.87 | 204.27 | 203.04 | 204.19 | - | 0.97% |
| | UA | 65.41 | 68.63 | 66.94 | 67.25 | 68.24 | - | 1.68% |
| Different | AP | 161.79 | 165.24 | 162.17 | 164.70 | 163.17 | 161.33 | 0.90% |
| electrodes | DA | 125.52 | 123.76 | 125.44 | 121.46 | 124.68 | 123.60 | 1.11% |
| | UA | 77.21 | 74.61 | 77.29 | 73.99 | 75.30 | 72.15 | 2.40% |

Table S2 The reproducibility of NiCo-MOF/Ti₃C₂/CCE.

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