Supplementary Information

Covalent Organic Framework Encapsulated Multi-Walled Carbon Nanotubes for Ultrasensitive Electrochemical Determination of Lead Ions in Water and E-Cigarette Samples

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S1. Modification and preparation of electrodes

Initially, the glassy carbon electrode (GCE) was polished using 0.5 μm and 50 nm alumina (Al_2O_3) powders to achieve a mirror-like finish. Following this, ultrasonic cleaning was conducted with ethanol, followed by rinsing with deionized water. After that, the electrode surface was dried with nitrogen. Subsequently, the electrode surface was dried using nitrogen. The ground Tp-Bpy-COFs@MWCNTs were then weighed to 0.5 mg and placed into a 1.5 ml vial containing 950 μL of deionized water and 50 μL of Nafion, following optimized conditions to create the dispersion solution. This prepared solution underwent ultrasonic treatment for 20 minutes to ensure even dispersion. The uniformly dispersed droplets were then deposited onto the surface of the glassy carbon electrode (GCE) to fabricate Nafion@Tp-Bpy-COF@MWCNTs-GCE. The preparation process for other modified electrodes followed a similar approach.

S2. Electrochemical measurement

All tests for lead ions were conducted using an RST5000 electrochemical workstation and a 100 mL electrolyzer, employing square wave stripping voltammetry (SWASV) in a HAc-NaAc buffer. To prepare the e-cigarette digestion solution, 0.3-0.4 g of e-cigarette liquid is first weighed and placed in a polytetrafluoroethylene tank. Simultaneously, a blank sample is prepared by adding 5 mL of nitric acid and 2 mL of hydrogen peroxide. Following the microwave digestion procedure outlined in the standard, the reaction mixture is heated. After digestion, the tube is removed, and the digestion solution is transferred to a 50 mL

reagent bottle. The inner tank is rinsed with water three times, and the washing solution is combined and adjusted to the appropriate volume. Finally, the sample is tested using the GB5009.9-2014 conditions.

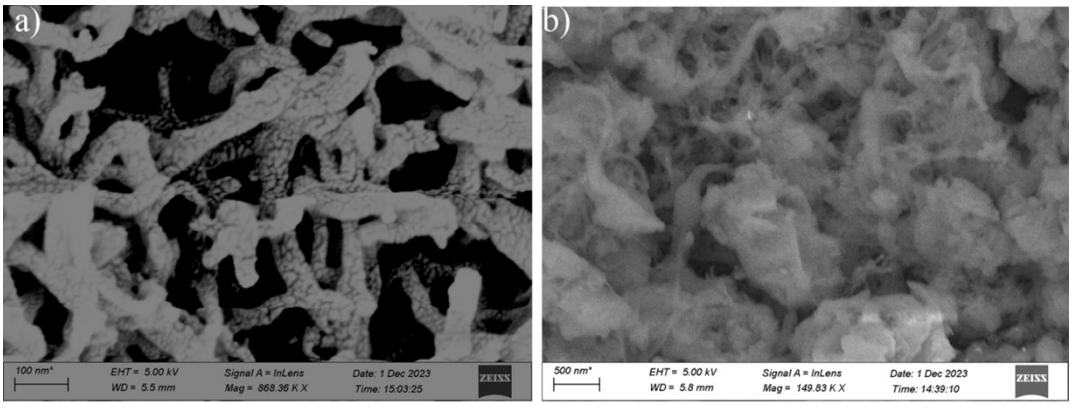

Fig. S1. (a-b) SEM image of Tp-Bpy-COFs@MWCNTs.

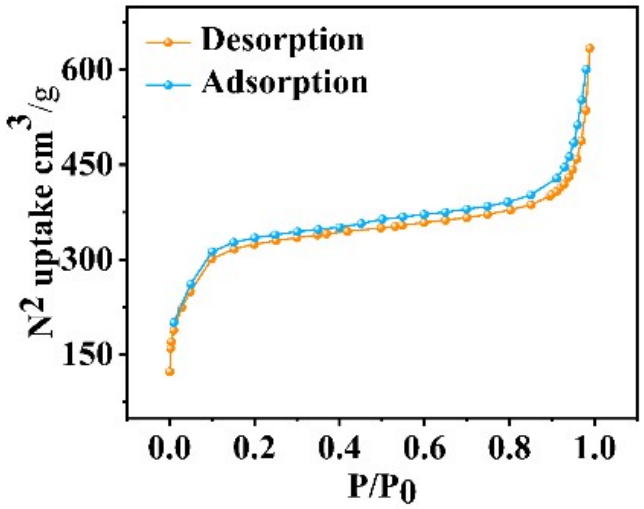

Fig. S2. BET image of Tp-Bpy-COFs@MWCNTs.

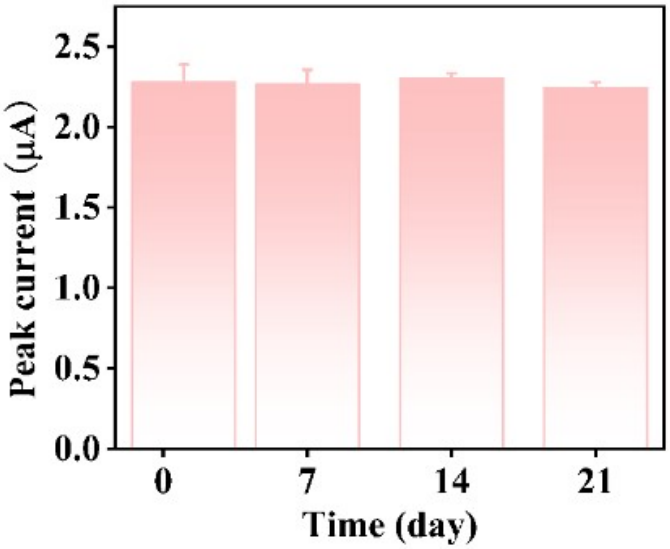

Fig. S3. Stability of Tp-Bpy-COFs@MWCNT/Nafion/GCE.

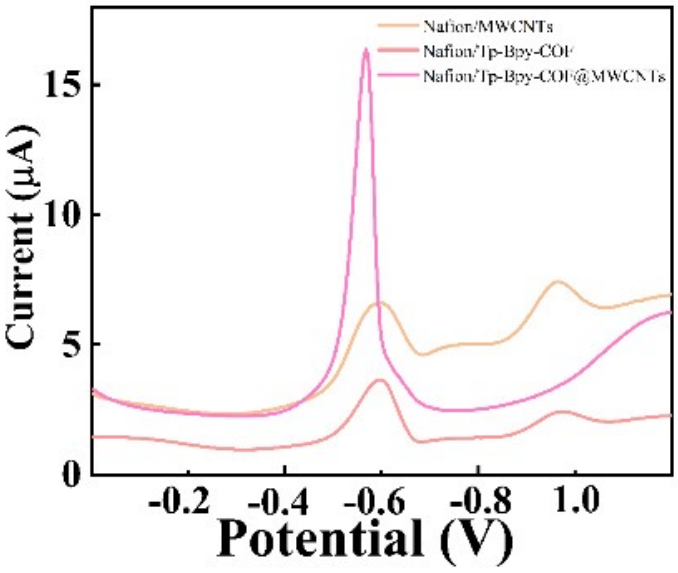

Fig. S4. Comparison of SWASV Performance for Naftion/COF@MWCNTs, Naftion/COF, and Naftion/MWCNTs.

Table. S1. Material name abbreviation

Full name of material	Abbreviation
Graphene oxide	rGO
Chitosan	CS
$Na_9[BiW_9O_{33}]\cdot 16H_2O$	BiW_9
Nanoporous carbon	NPS
1,3,6,8-pyrenetetrasulfonic acid sodium salt-functionalized	PyTS
Carbon nanohorns	CNHs
Polyaniline	PANI
Laser scribed graphene	LSG
Electrochemically reduced graphene oxide	ErGO
Cellulose graphene	CSG
BC-Au electrode and ionic liquid carbon paste electrode	CILE
Graphene	G

Table. S2. Comparison of Detection Methods for Pb^{2+} Analysis

Detection Method **Comparison**	**Electrochemical** **Method（SWASV）**	**AAS**	**ICP-MS**
Equipment Cost	Low	Medium	High
Detection Time	Simple operation, rapid detection	Requires complex preprocessing, long detection time	Requires high technical support, long detection time
Portability	Suitable for on-site detection	Laboratory detection	Laboratory detection
Sensitivity	Up to 10^{-9} M (depending on material modification)	Up to 10^{-6} M	Up to 10^{-12} M