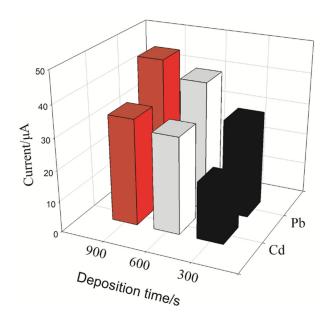
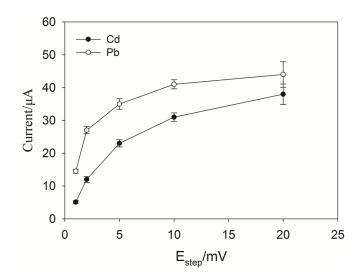
## **Supplementary Materials**

## **Preparation of SPAN Nanofibers**

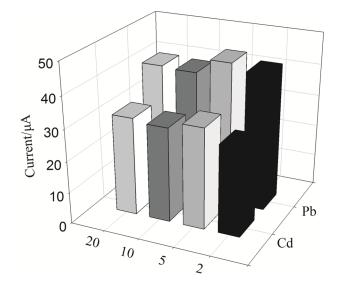
SPAN nanofibers were prepared according to the method provided by Zhang and co-workers [1]. Briefly, 0.11 g of cetyltrimethyl ammonium bromide (CTAB), 0.23 g of aniline (AN), 0.43 g of 2-aminobenzenesulfonic acid (ABS) were dissolved in 40 mL 0.15 M HCl solution. Then, 20 mL 0.06 M ammonium peroxydisulfate (APS) was added to the mixed solutions. The copolymerization reaction was performed under static conditions at 5 °C for 24 h. Finally, the resulting dark green precipitates were filtered, then, washed with ultrapure water and ethanol several times, and dried at room temperature.



**Figure S1.** Deposition time effect on peak height in a solution containing 300  $\mu$ g·L<sup>-1</sup> Bi<sup>3+</sup>, 10  $\mu$ g·L<sup>-1</sup> Pb<sup>2+</sup> and Cd<sup>2+</sup>, and 300 mM NaCl. Square wave anodic stripping voltammetry (SWASV) parameters: *Ebegin* = -1 V, *E*end = -0.4 V, *E*step = 0.010 V, *E*pulse = 0.02 V, *E*condition = -0.4 V, *E*deposition = -1 V, Frequency = 50 Hz, deposition time = 300–900 s, and equilibrium time = 20 s.



**Figure S2.** Optimization of step potential effect ( $E_{step}$ ) using a solution containing 300 µg·L<sup>-1</sup> Bi<sup>3+</sup>, 10 µg·L<sup>-1</sup> Pb<sup>2+</sup> and Cd<sup>2+</sup>, and 300 mM NaCl. SWASV parameters:  $E_{begin} = -1$  V,  $E_{end} = -0.4$  V,  $E_{step} = 0.001-0.02$  V,  $E_{pulse} = 0.02$  V,  $E_{condition} = -0.4$  V,  $E_{deposition} = -1$  V, Frequency = 100 Hz, deposition time = 600 s, and equilibrium time = 20 s.



**Figure S3.** Equilibrium time effect on peak height in a solution containing 300  $\mu$ g·L<sup>-1</sup> Bi<sup>3+</sup>, 10  $\mu$ g·L<sup>-1</sup> Pb<sup>2+</sup> and Cd<sup>2+</sup>, and 300 mM NaCl. SWASV parameters: *E*<sub>begin</sub> = -1 V, *E*<sub>end</sub> = -0.4 V, *E*<sub>step</sub> = 0.01 V, *E*<sub>pulse</sub> = 0.02 V, *E*<sub>condition</sub> = -0.4 V, *E*<sub>deposition</sub> = -1 V, Frequency = 100 Hz, deposition time = 600 s, and equilibrium time = 2–20 s.

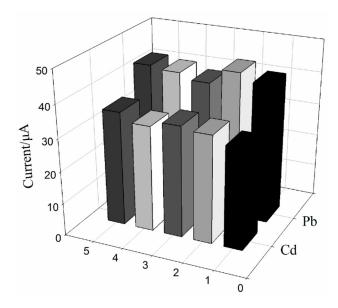


Figure S4. The reproducibility of proposed electrodes.

## Reference

1. Zhang, C.; Li, G.; Peng, H. Large-scale synthesis of self-doped polyaniline nanofibers. *Mater. Lett.* **2009**, 63, 592–594.



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