

Supporting information to

Highly ordered surfactant micelles function as the extraction matrix for direct electrochemical detection of halonitrobenzenes at the ppb level

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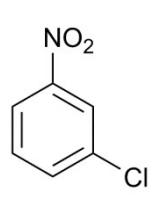
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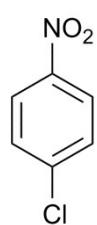
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S1. Molecular structures of studied HNBs



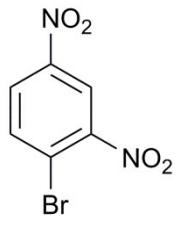
m-NCB



p-NCB



p-NBB



DNBB

Scheme S1. Molecular structures of studied HNBs

S2. TEM and SEM images of the OSM@SM/ITO electrode

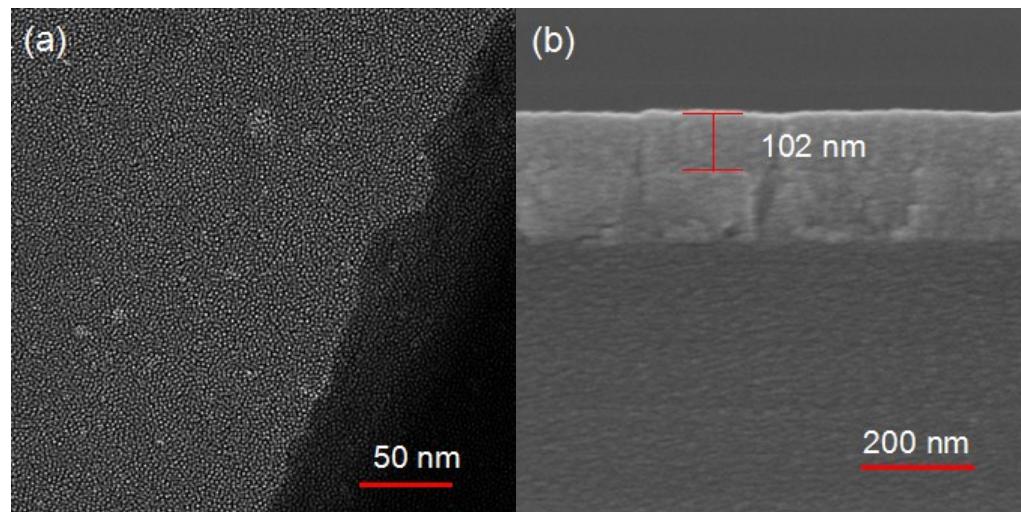


Fig. S1 (a) TEM image of OSM@SM film showing the mesopores as the bright spots. (b) Cross-sectional SEM image illustrating the OSM@SM layer on the ITO electrode surface with a thickness of ca. 102 nm.

S3. Electrochemical behaviors of the other three HNBs

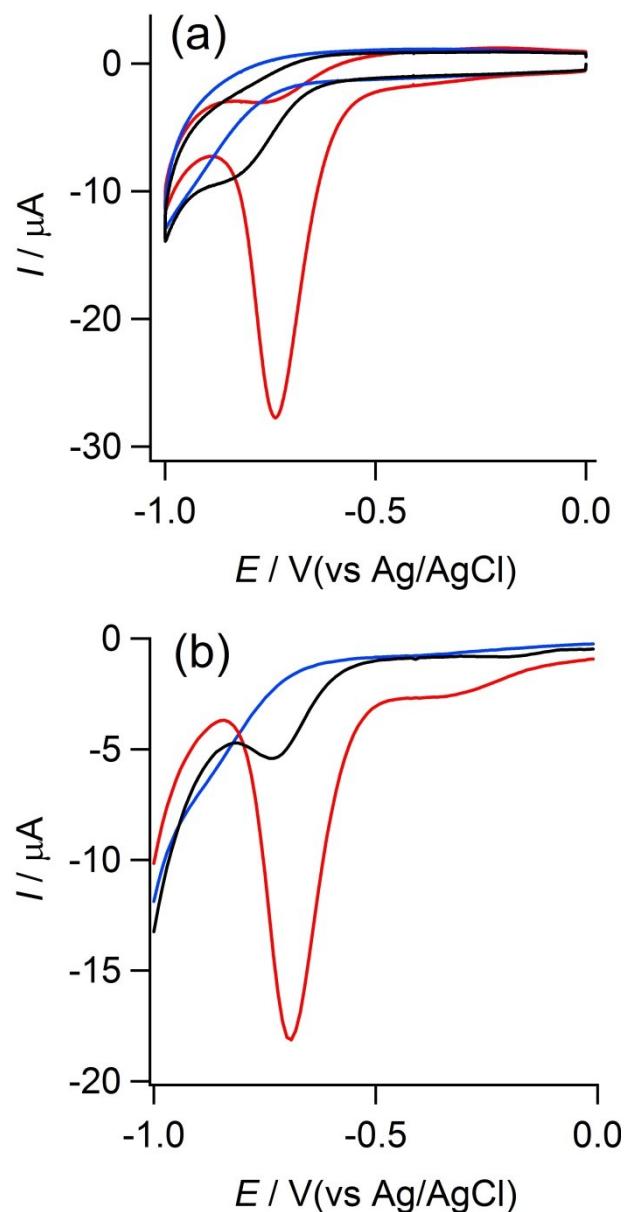


Fig. S2. CVs (a) and DPVs (b) of 1 ppm *p*-NCB at the bare ITO (black), SM/ITO (blue) and OSM@SM/ITO (red) electrodes in 0.5 M PBS (pH 7.0). The scan rate was 50 mV s⁻¹.

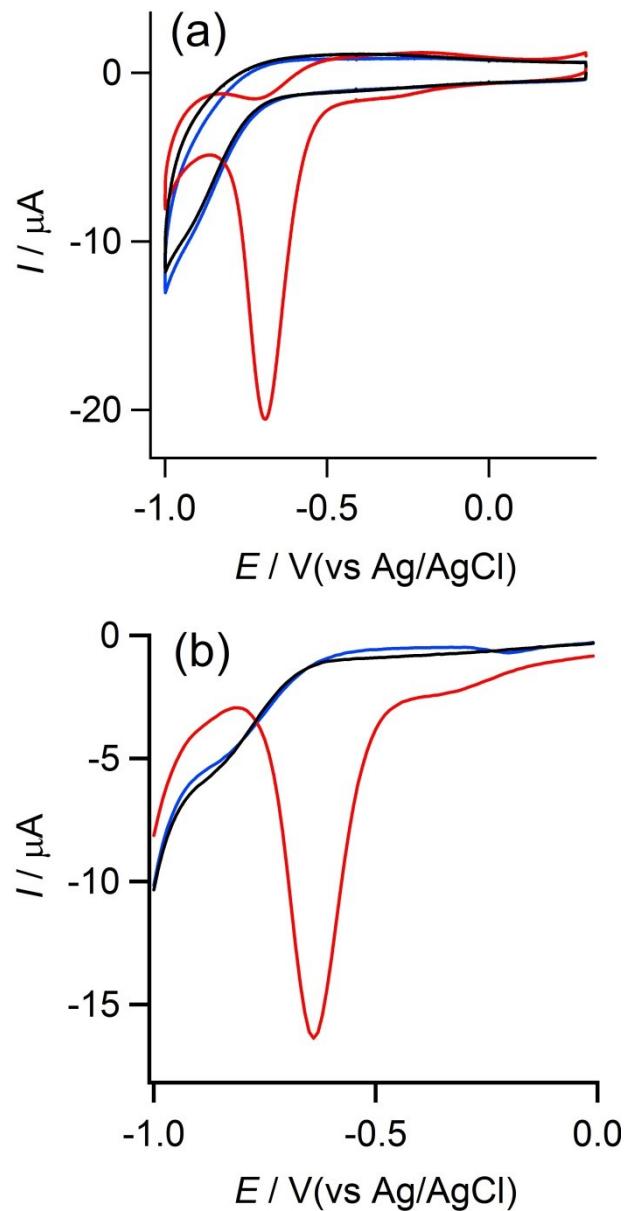


Fig. S3. CVs (a) and DPVs (b) of 1 ppm *p*-NBB at the bare ITO (black), SM/ITO (blue) and OSM@SM/ITO (red) electrodes in 0.5 M PBS (pH 7.0). The scan rate was 50 mV s⁻¹.

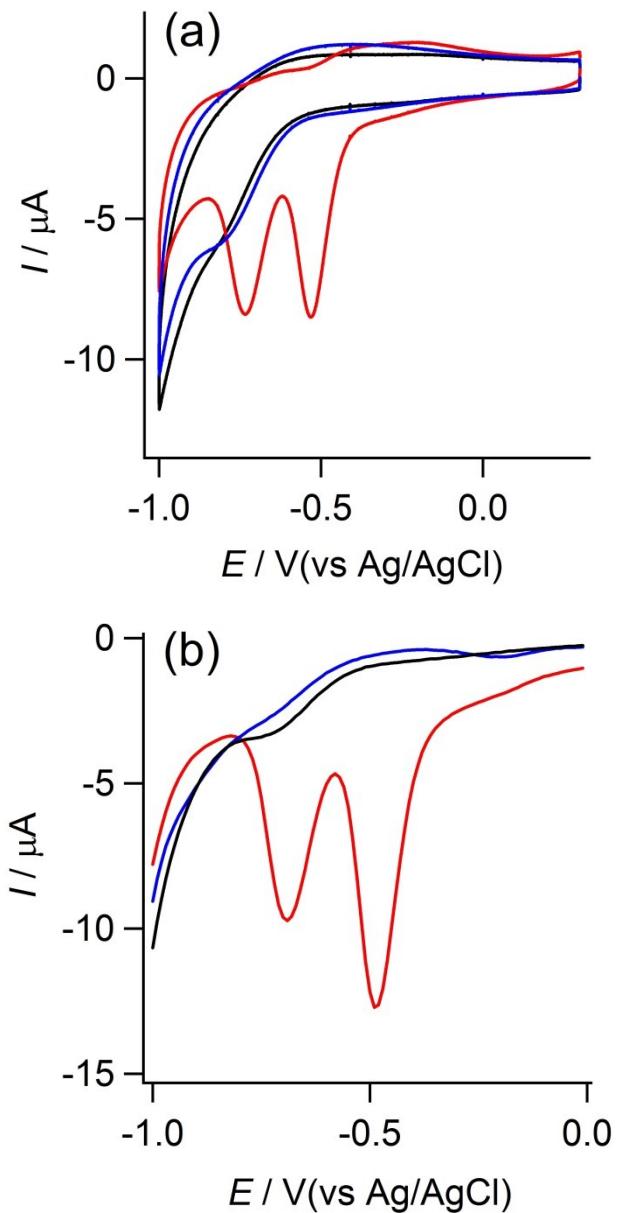


Fig. S4. CVs (a) and DPVs (b) of 1 ppm DNBB at the bare ITO (black), SM/ITO (blue) and OSM@SM/ITO (red) electrodes in 0.5 M PBS (pH 7.0). The scan rate was 50 $\text{mV} \text{ s}^{-1}$.

S4. The effect of the scan rate

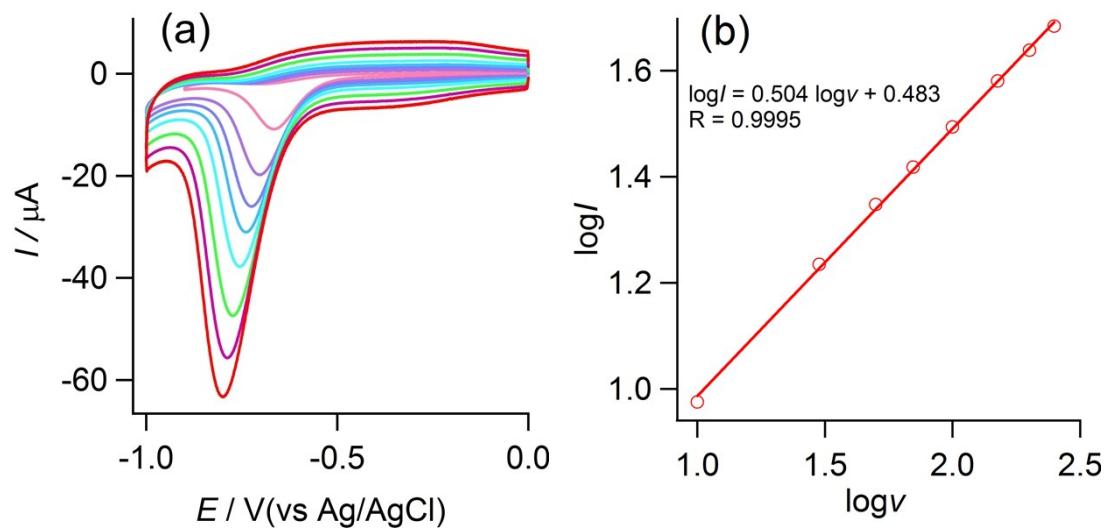


Fig. S5. (a) CVs of 1 ppm *m*-NCB at OSM@SM/ITO electrodes in 0.5 M PBS (pH 7.0) solution at different scan rates (from inner to outer: 10, 30, 50, 70, 100, 150, 200, 250 mV s⁻¹); (b) A double logarithmic curve of the reduction peak current and scan rate.

S5. Optimized conditions for HNBs detection

S5.1. Electrolyte solution

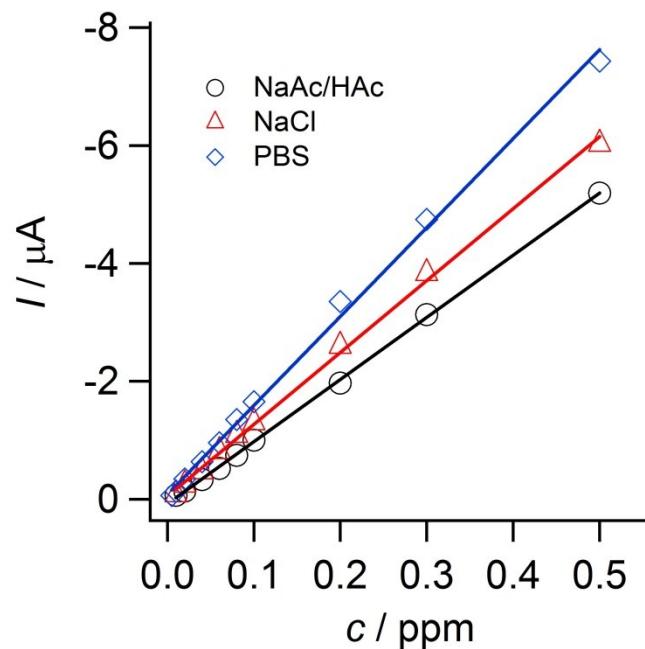


Fig. S6. Calibration curves for *m*-NCB detection at the OSM@SM/ITO electrode by DPV in different aqueous supporting electrolytes: 0.5 M NaCl, 0.5 M PBS (pH 7.0) and 0.5 M HAc/NaAc (pH 7.0). Before measurement, the preconcentration of *m*-NCB was performed by immersing the electrode in solutions under stirring for 1 min.

Table S1. Effect of the electrolyte solution on the detection of *m*-NCB.

| Electrolyte solution (0.5 M) | Lowest Detected Concentration (ppb) | Sensitivity ($\mu\text{A}/\text{ppm}$) | <i>R</i> |
|---------------------------------|--|---|----------|
| NaCl | 10 | 10.55 | 0.9997 |
| NaAc/HAc (pH=7) | 5 | 12.19 | 0.9990 |
| PBS (pH=7) | 10 | 15.08 | 0.9985 |

S5.2. Accumulation time

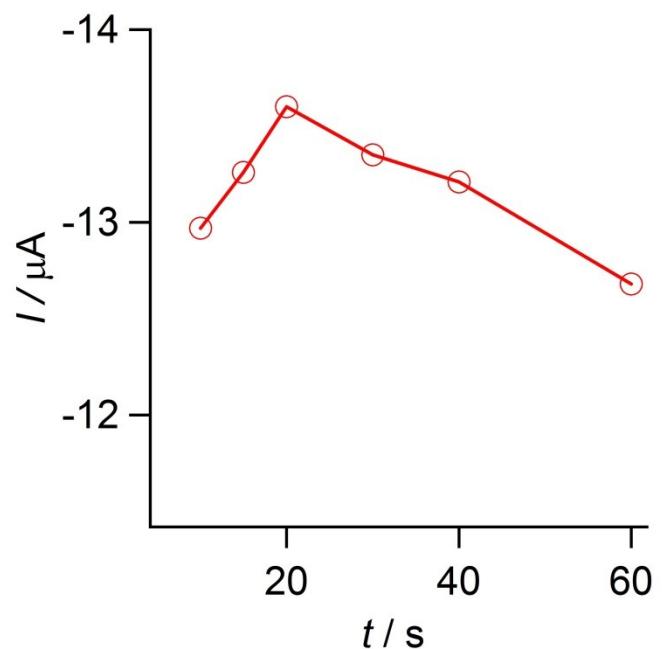


Fig. S7. Time-dependent plots at the OSM@SM/ITO electrode for 1 ppm *m*-NCB accumulated by stirring at 300 rpm.

S6. Detection of other three HNBs

When two or more reduction peak is observed, the signal of the most prominent reduction peak was selected for the quantitative analysis.

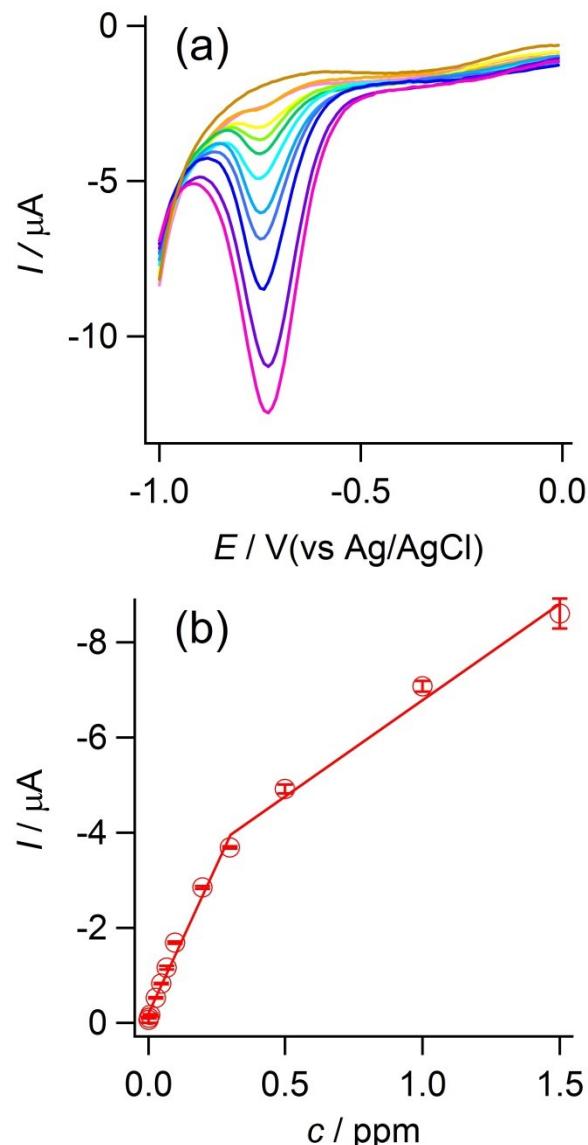


Fig. S8. (a) DPVs for various concentrations of *p*-NCB at the OSM@SM/ITO electrode in 0.5 M PBS (pH 7.0) solution. (b) The calibration curve of *p*-NCB. Error bars represent the standard deviations of three measurements.

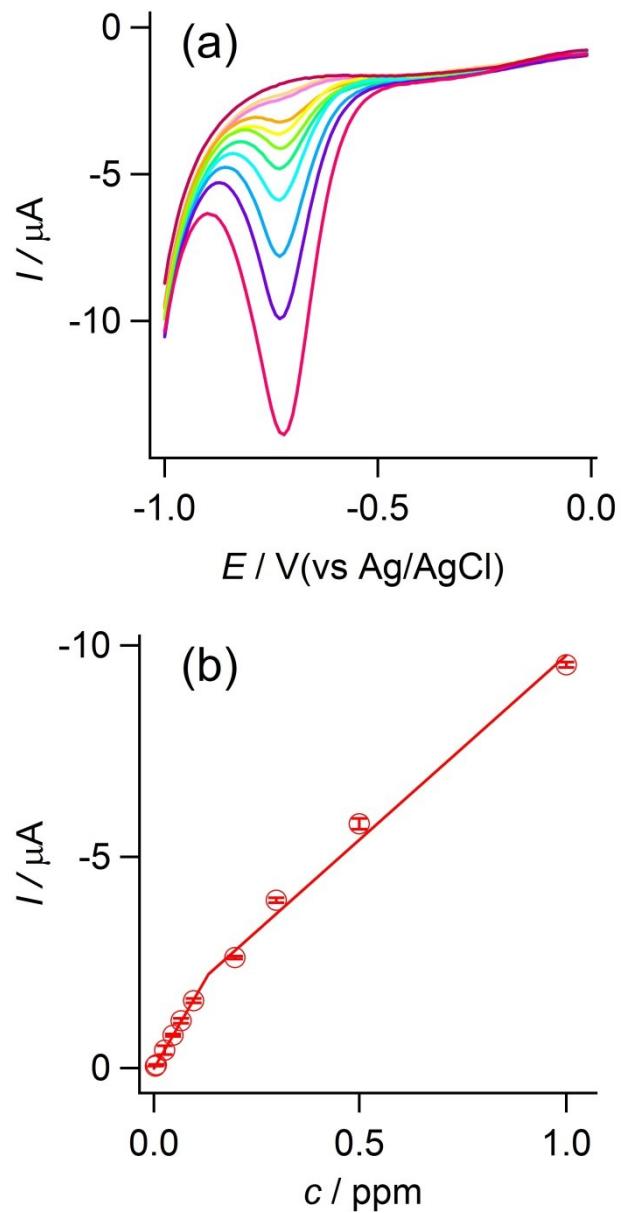


Fig. S9. (a) DPVs for various concentrations of *p*-NBB at the OSM@SM/ITO electrode in 0.5 M PBS (pH 7.0) solution. (b) The calibration curve of *p*-NBB. Error bars represent the standard deviations of three measurements.

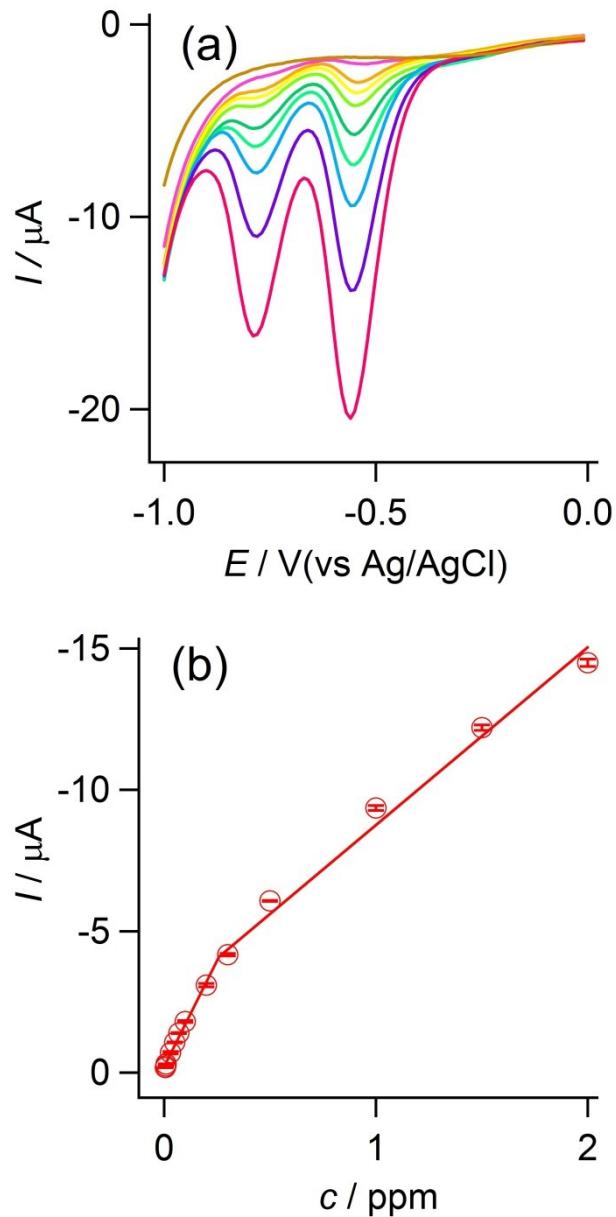


Fig. S10. (a) DPVs for various concentrations of DNBB at the OSM@SM/ITO electrode in 0.5 M PBS (pH 7.0) solution. (b) The calibration curve of DNBB. Error bars represent the standard deviations of three measurements.