

**Voltammetric discrimination of skatole and indole at disposable screen  
printed electrodes**

*Supplementary Information*

José-María Maesa, Francesc-Xavier Muñoz-Pascual and Eva Baldrich\*

Institut Microelectrònica de Barcelona (IMB-CNM), CSIC,  
Campus Universitat Autònoma de Barcelona, 08193 – Bellaterra (Barcelona), Spain

\* Eva Baldrich

Address: Institut de Microelectrònica de Barcelona (IMB-CNM, CSIC). Campus  
Universitat Autònoma de Barcelona. Barcelona 08193 (Spain)

E-mail: [eva.baldrich@imb-cnm.csic.es](mailto:eva.baldrich@imb-cnm.csic.es)

Telephone: (+34) 93 5947700 (ext 2405)

Fax number: (+34) 93 58014 96

## **Materials and methods**

### *Chemical reagents and biocomponents*

Skatole (3-methylindole), indole, potassium nitrate ( $\text{KNO}_3$ ), potassium ferrocyanide ( $\text{K}_4\text{Fe}(\text{CN})_6 \cdot 3\text{H}_2\text{O}$ ), dimethylformamide (DMF), and carboxylic acid functionalized single-walled carbon nanotubes (SWCNT) were purchased from Sigma (Barcelona, Spain). Phosphate Buffered Saline pH 7.4 (PBS) was prepared using tablets from Invitrogen (Barcelona, Spain). Streptavidin-coated magnetic particles (MP, Dynabeads M-270, 2.8  $\mu\text{m}$  in diameter), were obtained from Invitrogen (Barcelona, Spain).

Because skatole and indole are not completely soluble in water, stock concentrated solutions were prepared in different organic solvents (ethanol, acetonitrile and DMF). It followed 1:10 dilution with supporting electrolyte (0.1 M  $\text{KNO}_3$ ) and serial dilution in a similar solution (e.g.: 0.1 M  $\text{KNO}_3$  supplemented with 10% of the corresponding organic solvent in order to prevent any matrix effect during electrochemical measurements). Optimal results were obtained using skatole solved in a 1/9 mixture of DMF /  $\text{KNO}_3$  0.1 M.

### *Electrochemical measurements*

Electrochemical measurements were performed using a CHI1000A multi-potentiostat (CH Instruments, Texas, USA).

The experiments were initially performed using a 10 mL glass cell, with a 3 mm diameter glassy carbon electrode (BAS) as a working electrode, an Ag/AgCl reference electrode (CH Instruments) and a graphite bar as counter electrode. In this case, the working electrode was polished using 1  $\mu\text{m}$  alumina suspension (Bluebhar) prior to

each measurement. Otherwise, measurements were carried out using carbon screen printed electrodes (cSPE) provided by Dropsens (Oviedo, Spain). These include a 4 mm working electrode and carbon and silver auxiliary and reference electrodes respectively. According to the provider's instructions, cSPE can be directly used and do not require activation or any additional treatment previous to measurement. SWCNT-modified cSPE and MWCNT-modified cSPE from the same provider were also assayed. In all cases, electrodes were characterised by cyclic voltammetry (CV) in 1 mM  $K_4Fe(CN)_6$  prepared in 0.1 M  $KNO_3$  at the beginning of each working day, and also between measurements in order to monitor their integrity (i.e.: absence of passivation).

#### *Modification of electrodes with SWCNT by magnetic entrapment*

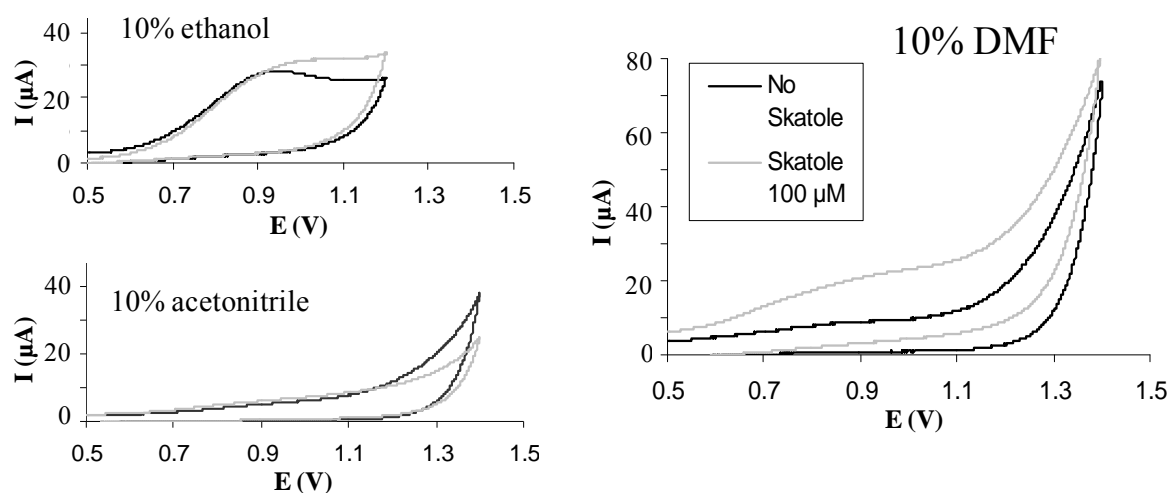
Alternatively, cSPE were modified with SWCNT by magnetic entrapment as previously reported<sup>1-2</sup>. Briefly, SWCNT were sonicated in sterile PBS at a final concentration of 0.3 mg mL<sup>-1</sup> until complete suspension. Streptavidin-coated MP were rinsed thrice with PBS and 10  $\mu$ L of the SWCNT suspension were then mixed with  $3.5 \times 10^6$  MP. Formation of SWCNT/MP complexes was observable by the naked eye. A magnet, placed immediately below the working electrode, promoted magnetic entrapment of the SWCNT/MP complexes. Subsequent electrochemical detection was done by depositing 50  $\mu$ L drops onto the electrode.

## FIGURES

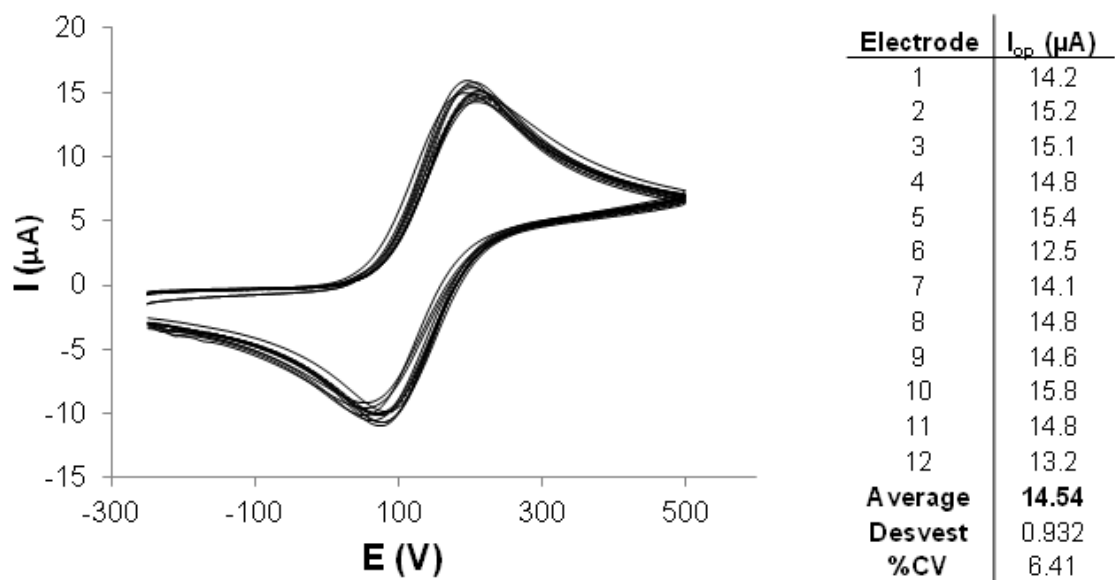
**Figure 1S. Electrochemical detection of skatole in different solvents.** Skatole was dissolved in pure ethanol, acetonitrile or DMF. It followed dilution 1:10 in 0.1 M KNO<sub>3</sub> and serial dilution in 0.1 M KNO<sub>3</sub> containing 10 % of the appropriate solvent. The alternative utilization of PBS and 0.1 M KCl as the supporting electrolyte did generate worse results (data not shown).

Electrochemical detection was performed by cyclic voltammetry (between 0.5 and 1.4 V vs. Ag/AgCl at 50 mV/sec) in a 10 mL electrochemical glass cell using a 3 mm diameter glassy carbon electrode as a working electrode, a Ag/AgCl reference electrode and a graphite bar as counter electrode.

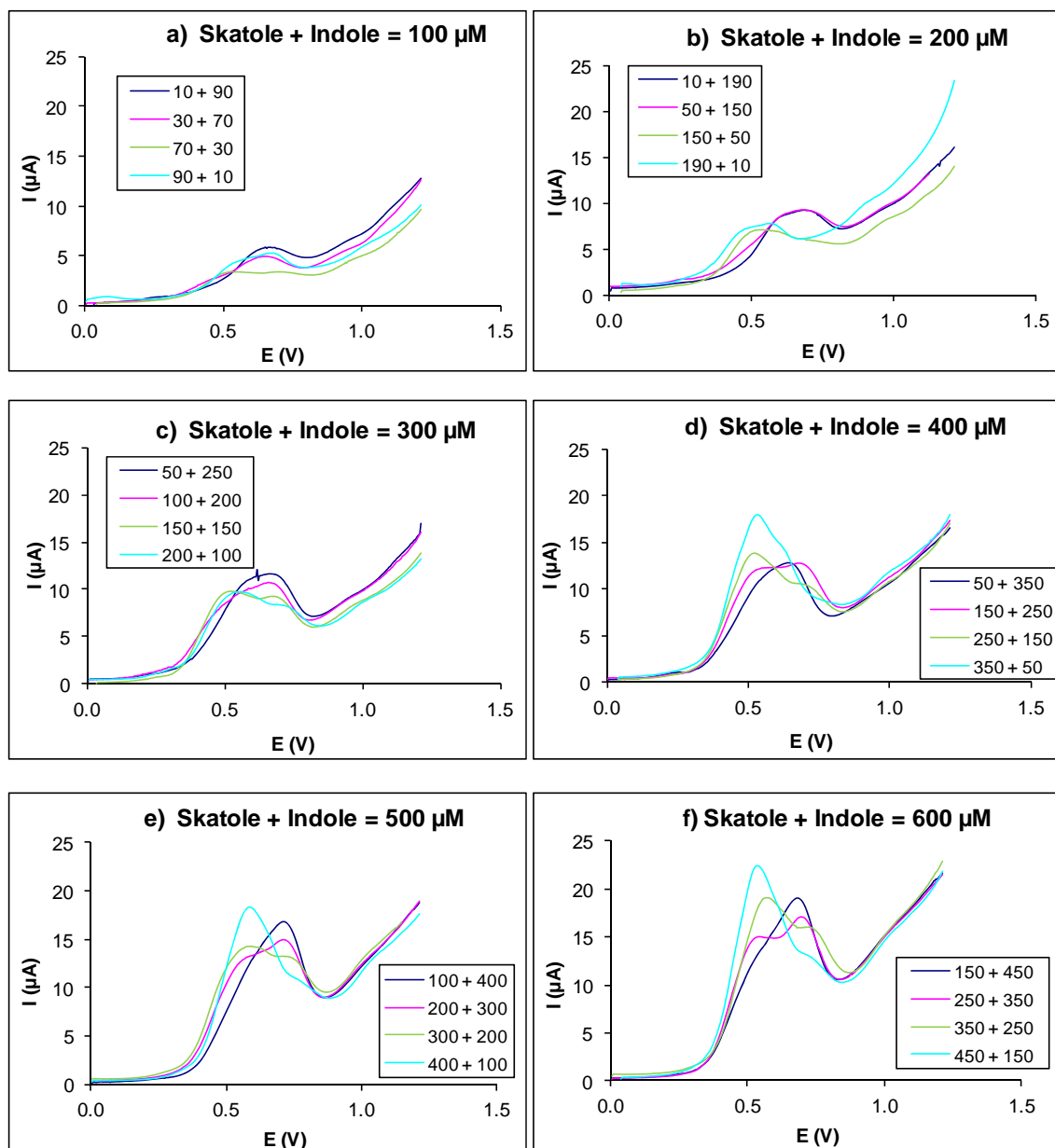
In the presence of ethanol, an oxidation wave appeared around 0.9 V vs. Ag/AgCl that masked the peak attributed to skatole. In acetonitrile, skatole detection was minimal. When DMF was used instead, skatole generated an oxidation wave around 0.9 V vs. Ag/AgCl, which height was proportional to the concentration of skatole present.



**Figure 2S. Characterisation of SPE.** Examples of the voltammograms independently obtained in 1 mM  $\text{Fe}(\text{CN})_6$  using 12 different carbon SPE. The table on the right summarized the oxidation peak currents registered.



**Figure 3S. Simultaneous detection of different concentrations of skatole and indole using cSPE.** Increasing concentrations of skatole and indole were mixed, corresponding to various molar ratios of the two molecules, and were assayed by DPV. Each graph shows the DPVs registered for 4 different molar ratios of the two molecules that summed the same final concentration.



## References

1. E. Baldrich and F. X. Muñoz, *Anal. Chem.*, 2011, **83**, 9244-9250.
2. E. Baldrich, R. Gómez, G. Gabriel and F. X. Muñoz, *Biosens. Bioelectron.*, 2011, **26**, 1876-1882.