Supplementary Material

Method: CME performance

Electroanalysis was performed in a 5 mL conventional electrochemical cell containing 3 mL of 0.1 mol L-1 KCl support electrolyte in methanol/water, 1:24 (v/v). The potential values ranged from 0.80 V to 1.30 V, the scan speed was 100 mV s-1, and the equilibrium potential was 0.8 V, applied for 5 s. Experiments were performed with 7.96 μmol L-1 MDMA or without MDMA. Glassy carbon was analyzed in the same conditions for comparative purposes. For each tested CME, sequential cyclic voltammetry cycles (n = 6) with an equilibrium potential of 0.8 V for 5 s were employed. CME performance was checked by analyzing the peak potential (Ep) and peak current (Ip). Standard deviation was assessed by analyzing the mean Ep and Ip of the electrodes. The relative standard deviation of Ep (RSD Ep%) and the relative standard deviation of Ip (RSD Ip%) were calculated. Statistical analyses were performed with the software Microsoft® Excel 365. For statistical evaluations, the Test F was employed; 95% of confidence interval was considered. Differences in inter-variances occurred when F < Fcritic (0.05). Next, the Student's t test was employed by assuming that both groups had the same variance and considering a 95% confidence interval. Results were considered significantly different when $p < 0.05$. The best electrode was selected for the subsequent analyses.

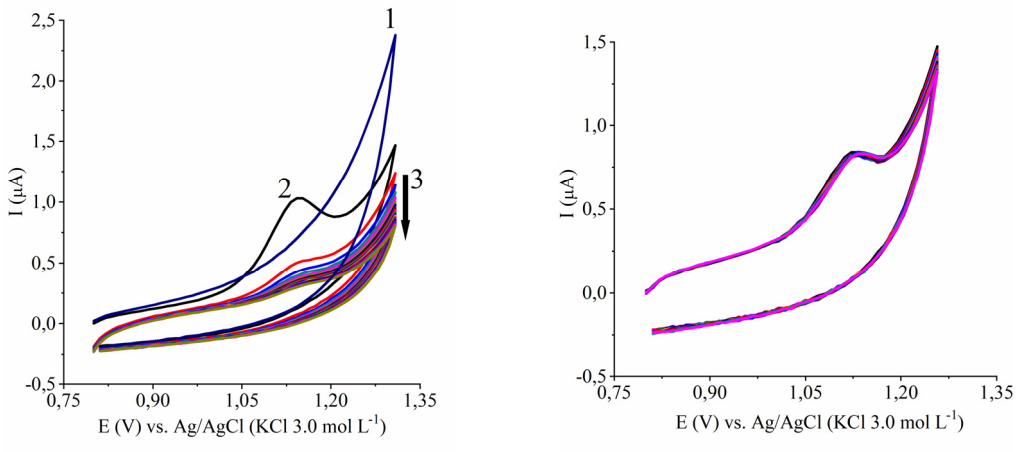
Method: Pre-concentration time evaluation

Electroanalysis was performed in a 5 mL conventional electrochemical cell containing 3 mL of 0.1 mol L^{-1} KCl support electrolyte in methanol/water, 1:24 (v/v). Experiments were performed with 5.17 μmol L^{-1} MDMA, and the blank was analyzed without MDMA. The current signal of CME 1.5 was evaluated with or without pre-concentration time. The potential values ranged from 0.80 to 1.30 V (and from 1.30 V to 0.80 V), the scan speed was 100 mV s^{-1}, and pre-concentration was performed at 0.8 V for 0, 5, 60, 180, 270, 360, 480, 540, or 600 s for each cycle. Ip was measured, and the results were compared with the difference in the signal obtained for a pre-concentration time of 5 s, as a percentage. This parameter was named Ip addition.

Supplementary Figures

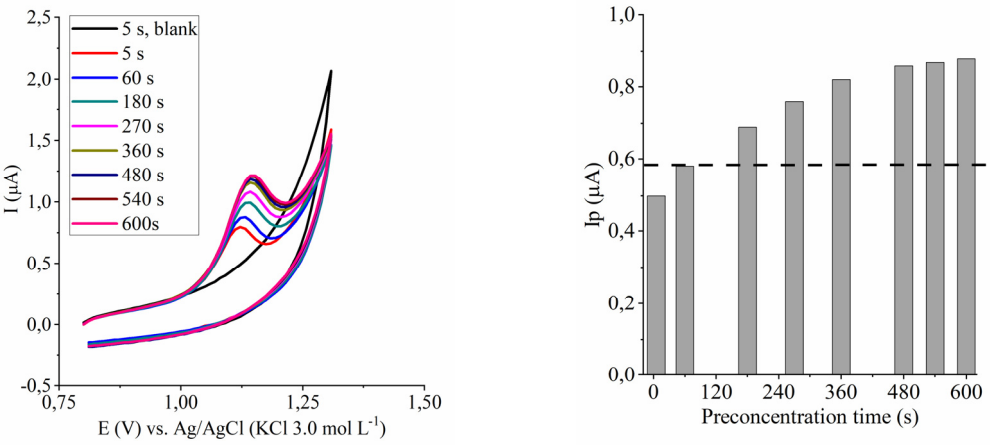
Figure S1. Seized ecstasy samples.

Figure S2. Pre-concentration time study: cyclic voltammetry employing CME 1.5 (polymer % (v/v)). (**a**) Twenty consecutive cycles: 1—Blank: pre-concentration time of 60 s. 2—1st cycle. Bulk with MDMA: pre-concentration time of 60 s. 3—2nd to 20th cycle. Bulk with MDMA: without pre-concentration time. (**b**) Twenty non-consecutive cycles: 1st to 20th cycle. Bulk with MDMA: pre-concentration time of 60 s.



(**a**) (**b**)

Figure S3. Evaluation of pre-concentration time: (**a**) voltammograms recorded with different pre-concentration time (5 − 600 s); (**b**) graph of the peak current as a function of pre-concentration time; (**c**) table of the peak current and the increase in the peak current, in percentage, compared to the pre-concentration time of 5 s.



(**a**) (**b**)

Pre-concentration time (s)	Ip (µA)	Addition (%)
5	0.50	---
60	0.58	16
180	0.69	38
270	0.76	52
360	0.82	64
480	0.86	72
540	0.87	74
600	0.88	76

(c)

Figure S4. Colorimetric results of the Marquis test, sulfuric acid test, Simon's test, and Simon's test with acetone. From 0 to 48 h.