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The use of a gold electrode for the determination of amphetamine derivatives and application to their analysis in human urine

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Abstract: The catalytic abilities of a gold electrode were tested for the quantitative determination of amphetamine (A) and 3,4-methylenedioxy-*N*-methylamphetamine (MDMA) standards by their oxidation using cyclic voltammetry (CV). The values of the oxidative currents of A and MDMA standards at 0.80 V *vs.* SCE in 0.05 M $NaHCO_3$ at a scan rate of 50 mV s^{-1} were linear functions of the concentration in range of 110.9–258.9 μM and 38.7–229.2 μM, respectively. Square wave voltammetry (SWV) revealed a linear increase of current with the concentration of MDMA (range 30.9–91.6 μM), which enabled the quantitative determination of amphetamine derivates. SWV analysis was also successfully performed in spiked urine samples. A and MDMA in the presence of sucrose and as a content in illegally produced tablets were also determined. The voltammetric determinations of A and MDMA derivatives using CV and SWV at gold a electrode are rapid, selective and simple procedures and their accuracy was confirmed with a reference method, high performance liquid chromatography (HPLC). The analysis of spiked urine samples offers an additional possibility for the rapid detection of A and MDMA in human urine.

Keywords: amphetamine derivatives; cyclic voltammetry; square wave voltammetry; spiked urine samples.

INTRODUCTION

The abuse of amphetamine type stimulants (ATS) is on the rise worldwide. According to UNODC data, the number of ATS users is larger than the number

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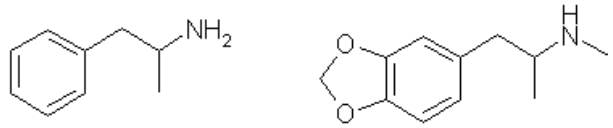
of heroin and cocaine users combined.[1,2] Amphetamine (Scheme 1a) and methamphetamine belong to the *β*-phenyl ethylamine structure sympathomimetic drugs that were utilized as psycho stimulants, antidepressants and appetite suppressants.[3,4] 3,4-Methylenedioxy-*N*-methylamphetamine (Scheme 1b) can induce euphoria, and diminished anxiety.



a **b**

Scheme 1. Chemical structures of a) amphetamine and b) 3,4--methylenedioxy-*N*-methylamphetamine.

There is an increasing interest in the development of rapid, selective and sensitive methods for the identification and quantification of A and MDMA in illegal market samples. This has been realized using a variety of methodologies: chromatographic techniques, such as HPLC and gas chromatography, capillary electrophoresis and infrared spectroscopy.[4,5]

Electro-analytical techniques have become powerful tools in modern analytical chemistry for the determination of amphetamine-type drugs.[6–11] The electrochemical oxidation mechanism of amphetamine-like compounds has not been clarified.[12,13]

A significant contribution to the understanding of the oxidative behavior of amphetamine derivatives in different buffer systems employing cyclic, differential pulse and square-wave voltammetry using a glassy carbon electrode was recently published. Primary amines oxidize at potentials higher than those allowed by the potential window of the glassy carbon electrode and a quantitative electroanalytical method was developed and successfully applied to the determination of MDMA in seized samples and in human serum.[13]

The oxidative behavior and determination of amphetamine derivatives on a gold electrode has not hitherto been reported. Taking into account the results obtained on glassy carbon electrode,[13] the testing of gold electrode for the oxidation of amphetamine like drugs is interesting and promising.

The aim of this work was to investigate the use of a gold electrode in the quantitative determination of A and MDMA standards, their content in illegally produced tablets and in spiked urine samples in 0.05 M $NaHCO_3$ by cyclic voltammetry. Furthermore, the voltammetric behavior of A and MDMA standards in the model systems, *i.e.*, in mixtures with sucrose, was studied. A standard analysis of MDMA was also performed by square wave voltammetry and the method was applied on spiked urine samples. The accuracy of the quantitative determination of amphetamine derivatives was confirmed with a reference method, HPLC.

EXPERIMENTAL

Materials

Amphetamine sulfate and 3,4-methylenedioxy-*N*-methylamphetamine hydrochloride were obtained from UN (Lipomed, Switzerland). Illegal amphetamine products (IAP) contain caffeine and quinine and illegal MDMAs product contain microcrystalline cellulose and lactose.[2] HPLC grade acetonitrile, methanol, ammonium acetate, sodium bicarbonate, sulfuric acid, sucrose, dichloromethane, sodium dihydrogenphosphate ($NaH_2PO_4 \cdot H_2O$), H_3PO_4 and $NaHCO_3$ (*p.a*. purity) were produced by Merck. Diethylamine, analytical grade, and ammonium hydroxide, 25 %, were produced by J. T. Baker. All the solutions were prepared with water (18 MΩ cm) obtained from a Millipore system.

Cyclic voltammetry

Standard equipment was used for the cyclic voltammetry measurements. The employed three-electrode electrochemical cell was described in detail previously.[14,15]

Polycrystalline gold served as the working electrode, a gold wire was used as the counter electrode and a saturated calomel electrode as the reference electrode. Polycrystalline gold (Pine rotating disc, used as stationary electrode, surface area 0.500 cm^2) was polished with diamond paste, cleaned with a mixture of water (18 MΩ cm) and sulfuric acid and further cleaned with deionized water (18 MΩ cm) in an ultrasonic bath. All the potentials are given *vs*. the SCE. Prior to the addition of A and MDMA, the electrolyte was deoxygenated by purging with nitrogen. All the experiments were performed at room temperature.

Square wave voltammetry

Square wave voltammetry (SWV) measurements were performed using an Autolab potentiostat–galvanostat (Metrohm, ECO Chemie, The Netherlands). The operating parameters were: step size 2 mV, pulse size 25 mV, frequency 8 Hz and scan rate 15 mV s^{-1}.

Preparation of the standard solutions for the analysis of A and MDMA as a content of a solid dosage form

Ten tablets were weighed and then the average mass per tablet was determined. The tablets were ground to a fine powder in a mortar. The required amount from the crushed tablets powder was dissolved in 10 cm^3 of deionized water by sonication for 5 min and filtered into a 100 cm^3 volumetric flask. The residue was washed three times with 0.05 M $NaHCO_3$ and the volume was completed to the mark with the same solvent. The obtained concentrations were checked by HPLC.

Preparation of urine samples

To 1 cm^3 of urine sample, 0.1 cm^3 of 25 % ammonium hydroxide and 5 cm^3 of dichloromethane were added. The samples were mixed on a mechanical shaker for 20 min and centrifuged at 3000 rpm for 10 min. After centrifugation, the organic layer was separated and evaporated in a stream of air. The dry extracts were reconstituted in methanol and analyzed by the HPLC–UV method at 200.5 nm.[15]

Preparation of standard solutions for the urine analysis

Stock standard solutions of A and MDMA tablets were prepared by dissolving 10 mg of tablet into 10 cm^3 of methanol and stored at –4 °C. Other concentrations of amphetamine as a content of tablets were made by diluting the stock standard solutions with methanol. Calibration samples were prepared by adding A and MDMA solution in blank (“drug-free”) human

urine. The calibration curves for urine spiked by A and MDMA were obtained by plotting their peak areas for the concentrations range 110.9–258.9 μM.

Equipment and chromatographic conditions

Characteristics of the HPLC instrument: HPLC Agilent 1100 (variable wavelength detector); column: Zorbax C18; mobile phase: A (85 %) – 1 % ammonium acetate, 2.5 % diethylamine in deionized water and B (15 %) - acetonitrile; column temperature 35 °C, wave length: 257 nm and injection volume 0.045 cm^3.

RESULTS AND DISCUSSION

Cyclic voltammetry of amphetamine

In order to avoid the influence of organic molecules (by their direct oxidation–reduction or adsorption on the gold electrode) either as a solvent or a component of the buffer solution in the electrolyte and hence to obtain only the anodic oxidation of A and MDMA, 0.05 M $NaHCO_3$ was chosen as the supporting electrolyte.[14,15] With a pH value of 8.4, this electrolyte is in accordance with the physiological pH value and the carbonate–bicarbonate buffer system of blood.[4] The polycrystalline gold electrode was previously selected as the optimal working electrode for the examination of pharmaceutical compounds (macrolide antibiotics)[14,15] and as such, was now selected as a possible suitable catalyst for the oxidation of amphetamine derivatives.

The selected concentration range for the CV determination of A (1.1–2.6 μM) is in complete accordance with the range of concentrations found in human body liquids (urine and blood)[1,3,4] during human sample testing.

The tested concentrations of the amphetamine standard, continuously added in the same experiment, are presented in Fig. 1 (full lines). For the five concentrations, the amphetamine oxidation began at a potential 50 mV more negative than the potential at which gold oxide formation occurs. The cyclic voltammograms show the apparent oxidative reaction with maximum current values covering the whole range in the area of oxide formation. It seems that amphetamines were strongly adsorbed on the gold electrode and that the gold oxide enabled their oxidation. It was recently published that the electro-oxidation of isomeric butylamines at a gold electrode in contact with an alkaline electrolyte solution was catalyzed by the gold oxide layer.[16] As expected, the gold oxide layer was also the catalyst in the electro-oxidation of A in contact with 0.05 M $NaHCO_3$. At higher concentrations of amphetamines, the flat plateau of the oxidative currents became sharper. An A concentration of 258.9 μM was the limiting one. With further increases in the concentration, the currents decreased. In the reverse sweep, the reduction peak of the gold oxides in the presence of amphetamine was diminished because of the reduction of A and the products of its oxidation formed in the forward sweep. The current values of this peak were not a linear function of its concentration, as was observed previously[16] for isomeric butylamines.

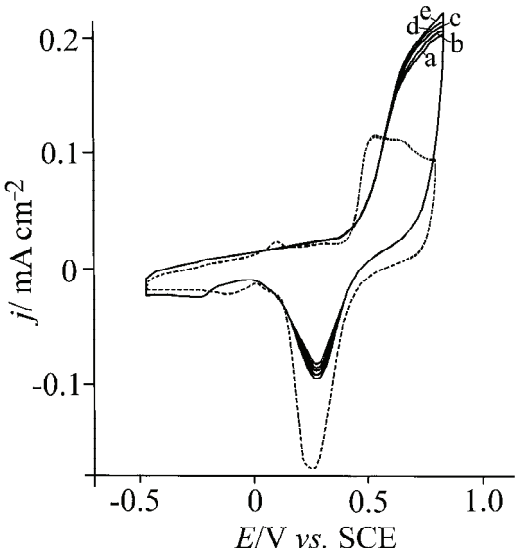


Fig. 1. Cyclic voltammogram of the gold electrode in 0.05 M $NaHCO_3$ (dashed line) and in a presence of amphetamine standard (full line) a) 110.9, b) 147.9 μM, c) 184.9 , d) 221.9 and e) 258.9 μM.
Sweep rate: 50 mV s^{-1}.

The value of the oxidative current of the amphetamine standard at 0.80 V *vs.* SCE in 0.05 M $NaHCO_3$ at the scan rate of 50 mV s^{-1} was a linear function of the concentration in a range of 110.9–258.9 μM. This linearity is presented in Fig. 2. The mentioned linear relationship corresponded to the equation:[17]

$$j_{pa} / \text{mA cm}^{-2} = 0.1112\ (\pm 0.0010) + 0.0002\ (\pm 0.000005) c / \mu\text{M} \quad (1)$$
$$r = 0.9993$$

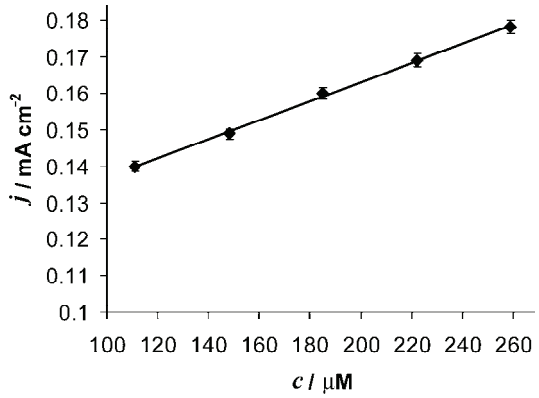


Fig. 2. Linear dependency of the anodic currents of amphetamine on its concentration, obtained from the data presented in Fig. 1.

Such equations were previously applied in the analysis of azithromycin, roxithromycin and midecamycin on a gold electrode.[14,15,18] After the first cycle, the anodic currents of the electro-oxidation of A strongly decreased with the cycling, which was attributed to poisoning of the electrode surface by the adsorption of their amine group. The most probable explanation is that amphetamine

molecules are adsorbed on the gold electrode surface in the potential range preceding the oxidation as was previously shown for isomeric butylamines.[16] This adsorptive behavior enabled a reproducible catalytic behavior of gold electrode in second sweep. After the first sweep, before the addition of the subsequent concentration, the gold electrode was prepared as is described in the Experimental.

The complete study of the MDMA dependency on pH value of electrolyte (from pH 1 to 14) is given elsewhere[13] with the suggestion that MDMA is electrochemically active on glassy carbon electrode in the range from pH 8 to 13. A is completely inactive.

In order to achieve better developed oxidation peak of A on the gold electrode, different pH values were tested in a suggested range, at pH 9 and 13. At both pH values the oxidation peak of A is less pronounced than at pH 8.

Cyclic voltammetry of 3,4-methylenedioxy-N-*methylamphetamine hydrochloride*

The tested concentrations of the MDMA standard, continuously added in the same experiment, are presented in Fig. 3 (full lines). The cyclic voltammograms show an apparent oxidative reaction, with a sharp maximum at the end of the examined oxide region and maximum current values at 0.80 V. Contrary to amphetamine, the oxidation of MDMA began at 0.1 V at the gold electrode surface, 350 mV before oxide formation. This suggests that for MDMA, the gold electrode acted as a catalyst and its molecules were not strongly adsorbed, as in the case of amphetamine. As was observed for A and isomeric butylamines,[16] the reduction peak of the gold oxides decreased markedly in the presence of MDMA.

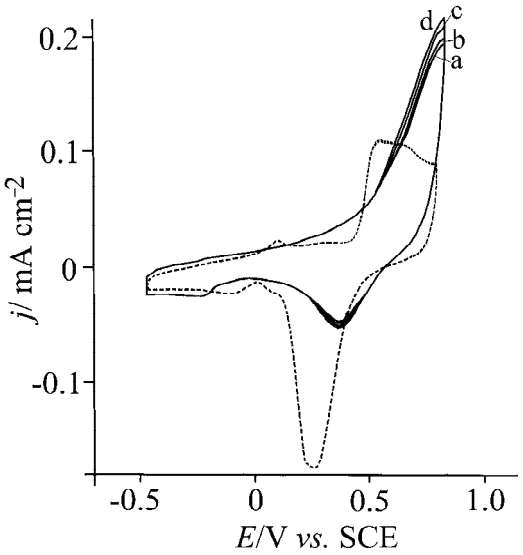


Fig. 3. Cyclic voltammogram of the gold electrode in 0.05 M $NaHCO_3$ (dashed line) and in a presence of MDMA standard (full line) a) 38.7, b) 77.1, c) 153.7 and d) 229.2 μM. Sweep rate: 50 mV s^{-1}.

The value of the oxidative current of MDMA standard at 0.80 V *vs*. SCE in 0.05 M $NaHCO_3$ at a scan rate of 50 mV s^{-1} was a linear function of the concentration in a range of (38.7–229.2 μM). This linearity is presented in Fig. 4. The obtained linear relationship corresponded to the equation:

$$j_{pa} / \text{mA cm}^{-2} = 0.1899\ (\pm 0.0005) + 0.0001\ (\pm 0.000007)c / \mu\text{M} \quad (2)$$
$$r = 0.9990$$

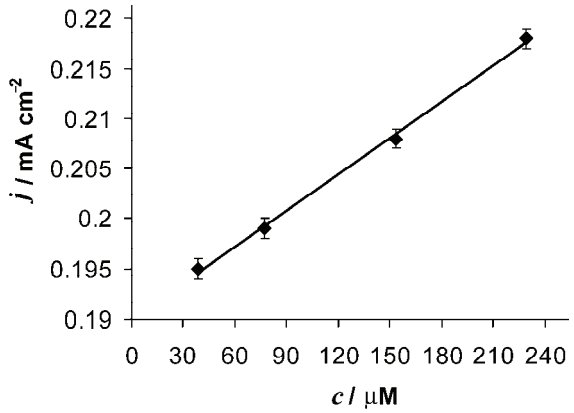


Fig. 4. Linear dependency of anodic peak currents of MDMA on its concentration, obtained from the data presented in Fig. 3.

For the determination of MDMA, only the first cycle was recorded and it is presented for all the examined concentrations. After the first cycle, the anodic currents of MDMA electro-oxidation decrease with cycling but to a smaller extent than in a case of A, which was attributed to poisoning of the electrode surface by the adsorption of its amine group. After the first sweep, before the addition of the subsequent concentration, the gold electrode was prepared as described in the Experimental.

The all tested A and MDMA concentrations are analyzed and confirmed by HPLC[5] during the electrochemical experiment and the obtained linearity was in accordance with the linearity presented in Figs. 2 and 4. The data obtained by HPLC (chromatograms) are not presented herein.

Determination of A and MDMA in illegal tablets by cyclic voltammetry

In the illegally produced samples, the basic excipient is powdered sucrose. A model experiment was performed in which samples of A and MDMA were mixed with sucrose in a concentration most often found in illegal samples (concentration: A/sucrose followed the relationship 1/6). The model samples exhibited the same electrochemical behavior as those presented in Figs. 1 and 3.

Compared to Fig. 3, the samples of MDMA mixed with sucrose exhibited an additional anodic peak (from 0.1 V to 0.5 V), which was attributed to sucrose. The oxidation of added sucrose did not influence the electro-oxidation of MDMA.

Illegal A and MDMA products containing microcrystalline cellulose, lactose and quinine were analyzed in the same manner as was presented for the A (Fig. 1) and MDMA (Fig. 3) standards. Before the electrochemical analysis, all the samples were analyzed by GC-MS and their content was confirmed as previously published.[2] The obtained cyclic voltammograms were the same as those presented in Figs. 1 and 3. The collected data are listed in Table I.

TABLE I. Application of the proposed method to the determination of A and MDMA in illegal tablets; mean of six experiments

Preparation	Amount found, mg ± *RSD* / %	
	Proposed method	Reference method
A tablets	30±3.7	24.9±1.2
MDMA tablets	60±1.7	60±1.1

The presence of microcrystalline cellulose, lactose and quinine has no influence on the catalytic properties of gold oxide in the A and MDMA determinations and cyclic voltammograms were the same as those presented in Figs. 1 and 3.

The presence of caffeine disturbed the determination of A and MDMA in the sense that the gold oxide was also a catalyst for caffeine electro-oxidation and A, MDMA and caffeine undergo the oxidation processes at the same potential values.[19] A method for caffeine separation before the electrochemical analysis should be developed in order to avoid synergetic effects. The cyclic voltammogram of illegal A tablets containing caffeine is presented in Fig. 5 (for a concen-

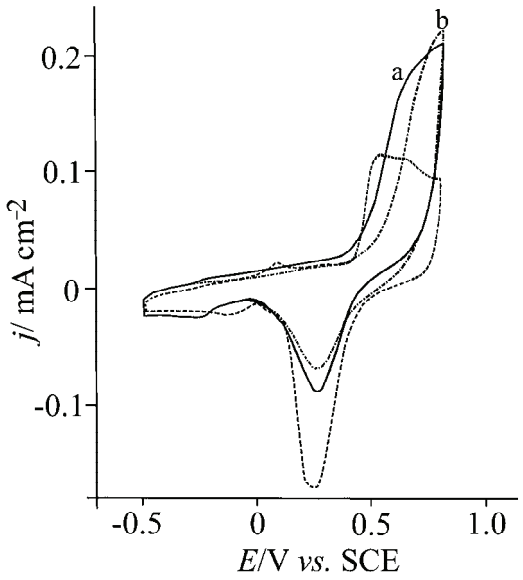


Fig. 5. Cyclic voltammogram of the gold electrode in 0.05 M $NaHCO_3$ (dashed line) and in the presence of a) amphetamine standard (full line), 184.9 μM, and b) amphetamine tablet (dashed–dotted line) 184.9 μM. Sweep rate: 50 mV s^{-1}. The concentration of A in the tablet was determined by HPLC.

tration of 184.9 μM). The found amount of A in the tablets using cyclic voltammetry (from Fig. 5) was 20 % higher in the presence of caffeine than was determined for the standard, as can be seen from Table I. The same effect was observed for MDMA.

Determination of A in urine samples using cyclic voltammetry

The urine samples spiked with amphetamine tablets (in the concentration range: 110.9–258.9 μM) were analyzed in a same manner as was presented for A.

The determination of A in the spiked urine samples was also realized by the standard addition method at two different concentration levels (110.9 and 184.9 μM). Four determinations were performed at each concentration level (Table II). The mean recoveries for the two concentrations were 98.85 and 97.36 % with relative standard deviations of 0.141 and 1.226, respectively.

TABLE II. Determination of amphetamine in spiked urine samples using the CV method

Taken concentration, μM	Recovery, %		*SD* / %		*RSD* /%	
	CV	HPLC	CV	HPLC	CV	HPLC
110.9	98.25	99.85	0.138	0.100	0.141	0.110
184.9	97.36	99.87	1.201	0.750	1.226	0.805

Different analytical methods are usually combined and compared in drug analysis[20,21] and statistical comparison of the results obtained with cyclic voltammetry and HPLC for the two concentrations are presented in Table II.[22]

Square wave voltammetry of MDMA

Being a fast and sensitive technique with a low detection limit, square wave voltammetry was tested as a possible method for the quantitative determination of A and MDMA on a gold electrode. Analysis of the A standard showed strong adsorption of the molecules on the surface of the gold electrode in the potential range preceding the oxidation,[16] blocking the surface and preventing its determination by the SW method. Contrary to A, SW voltammograms for different concentrations of the MDMA standard were recorded in 0.05 M $NaHCO_3$ in the potential range from 0 to 1.1 V at a scan rate of 15 mV s^{-1}. Before each scan, the compound was accumulated at the electrode surface at 0.1 V for 220 s. The square wave anodic stripping voltammograms for different concentrations of MDMA are presented in Fig. 6. Each voltammogram is characterized by a well-defined peak at approximately 0.7 V that was attributed to the oxidation of adsorbed MDMA. The current of the anodic stripping peak exhibited a linear dependence on the MDMA concentration as shown in Fig. 7.

The value of the oxidative peak of the MDMA standard in 0.05 M $NaHCO_3$ is linear function of the concentration in a range of (30.9–91.6 μM). This line-

arity is presented in Fig. 7. The obtained linear relationship corresponded to the equation:

$$j_{pa} / \text{mA cm}^{-2} = 0.0052\ (\pm 0.0003) + 0.00017\ (\pm 0.000006) c / \mu\text{M} \qquad (3)$$
$$r = 0.9991$$

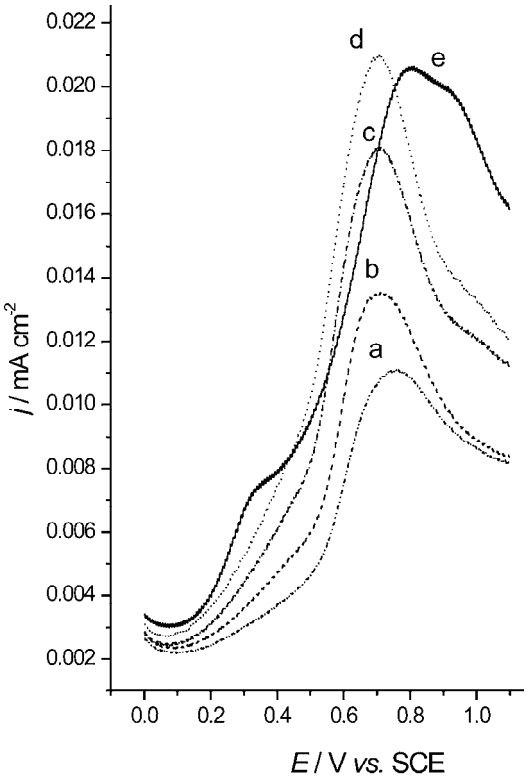


Fig. 6. Square wave anodic stripping voltammograms at the gold electrode in 0.05 M $NaHCO_3$ in a presence of MDMA standard, a) 30.9, b) 46.4 , c) 76.5, d) 91.6 and e) 91.6 μM spiked with urine. Accumulation time: 220 s at E = 0.1 V; step size 2 mV, pulse size 25 mV, frequency 8 Hz and scan rate 15 mV s^{-1}.

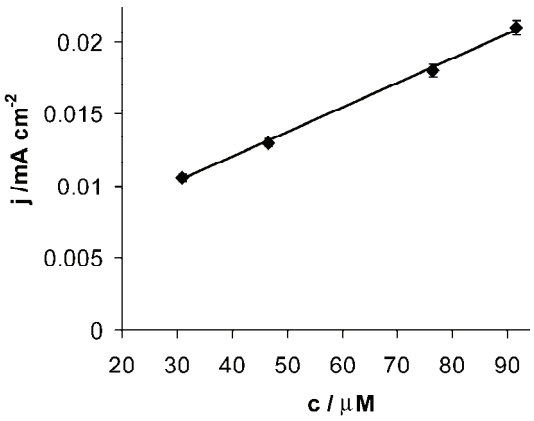


Fig. 7. Linear dependency of anodic currents of MDMA on concentration, obtained from the data presented in Fig. 6.

Determination of MDMA in urine samples using square wave voltammetry

The urine samples spiked with MDMA standard were analyzed as well and Square wave anodic stripping voltammograms of spiked urine samples for the set of concentrations of MDMA presented in Fig. 6 are the same as those observed in the absence of urine. The voltammogram for the highest tested concentration of MDMA, 91.6 μM, in Fig. 6 shows that a small shoulder that appeared at 0.35 V, attributed to the presence of the urine of healthy volunteers, did not disturb the oxidation of MDMA and had no influence on the value of the oxidation peak. The peak could be slightly shifted to positive potential values (as is presented in Fig. 6) but it does not occur with all spiked urine samples and depended on the urine content, which is common in clinical praxis. The results obtained revealed that SWV could be successfully applied for the quantitative determination of MDMA in urine.

The determination of MDMA in spiked urine samples was also performed by the standard addition method at two different concentration levels (76.5 and 91.6 μM). Four determinations were performed at each concentration level (Table III). The mean recoveries for the two concentrations were 98.32 and 98.46 % with relative standard deviations of 0.138 and 1.134, respectively.

A statistical comparison of the results obtained using cyclic voltammetry and HPLC for the two concentrations is presented in Table III.

TABLE III. Determination of MDMA in spiked urine samples using the SWV method

Taken concentration, μM	Recovery, %		*SD* / %		*RSD* /%	
	SWV	HPLC	SWV	HPLC	SWV	HPLC
76.5	98.46	99.72	1.118	0.764	1.134	0.316
91.6	98.32	99.77	0.120	0.102	0.138	0.120

CONCLUSIONS

A gold electrode as a good catalyst for amine-type molecules was successfully employed for the quantitative determination A and MDMA standards *via* their voltammetric oxidation.

The value of the oxidative current of A and MDMA standards, obtained by cyclic voltammetry at 0.80 V *vs.* SCE in 0.05 M $NaHCO_3$ at the scan rate of 50 mV s^{-1}, was a linear function of concentration in the range 110.9–258.9 μg cm^{-3} for A and 38.7–229.2 μg cm^{-3} for MDMA.

The quantitative determination of amphetamine derivatives in solid dosage form and in spiked urine samples was successfully realized using cyclic voltammetry.

The determination of an A standard in spiked urine samples was performed by the standard addition method employing cyclic voltammetry. The mean reco-

veries for the two concentrations were 98.85 and 97.36 % with relative standard deviations of 0.141 and 1.226, respectively.

Square wave voltammetry was successfully applied for quantitative determination of MDMA standard (30.9–91.6 μM) in solution and in spiked urine samples. The determination of MDMA with SWV in spiked urine samples was also performed by the standard addition method. The mean recoveries for the two concentrations were 98.32 and 98.46 % with relative standard deviations of 0.138 and 1.134, respectively.

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ИЗВОД

ОДРЕЂИВАЊЕ ДЕРИВАТА АМФЕТАМИНА И ПРИМЕНА У АНАЛИЗИ ХУМАНОГ УРИНА НА ЕЛЕКТРОДИ ОД ЗЛАТА

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Каталитичка својства електроде од злата су тестирана за квантитативно одређивање амфетамина (A) и 3,4-метилендиокси-*N*-метиламфетамина (MDMA) стандарда. Електроксидација A и MDMA је праћена цикличном волтаметријом (CV). Вредност оксидативног пика A и MDMA стандарда је линеарна функција концентрација у опсегу 110,9––258,9 μM (A) и 38,7–229,2 μM (MDMA). Волтаметрија са правоугаоним импулсима (SWV) је показала линеарну зависност струја од концентрација за MDMA стандард (у опсегу: 30,9–91,6 μM) као и у спајкованим узорцима хуманог урина. Успешно је анализиран и садржај A и MDMA у илегално произведеним таблетама. Волтаметријско одређивање A и MDMA деривата уз помоћ CV и SWV на електроди од злата је брза, селективна и једноставна процедура. Анализа спајкованих узорака урина нуди додатну могућност за брзу детекцију A и MDMA у хуманом урину.

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