DETERMINATION OF 4-NITROBIPHENYL AND 4-AMINOBIPHENYL BY CONTROLLED-POTENTIAL COULOMETRY, DIFFERENTIAL PULSE POLAROGRAPHY AND DIFFERENTIAL PULSE VOLTAMMETRY*

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Conditions were sought for the determination of 4-nitrobiphenyl by controlled-potential coulometry based on its reduction on a mercury pool electrode and for its polarographic determination on a dropping mercury electrode, with detection limits of $0.7 \, \mu mol \, l^{-1}$ for TAST polarography and $0.06 \, \mu mol \, l^{-1}$ for DP polarography. The reduction of this compound on a glassy carbon electrode was employed for its determination with detection limits of $10 \, \mu mol \, l^{-1}$ for DC voltammetry on a rotating electrode and $1 \, \mu mol \, l^{-1}$ for DP voltammetry on a stationary electrode. For 4-aminobiphenyl, its oxidation on a glassy carbon electrode was employed for the determination; the detection limits were $10 \, \mu mol \, l^{-1}$ for DC voltammetry on a rotating electrode and $0.5 \, \mu mol \, l^{-1}$ for DP voltammetry on a stationary electrode.

Carcinogens that cause malignant growth by their interactions with cellular macromolecules are attracting interest over the world. 4-Aminobiphenyl (4-ABP) is among substances positively recognized as carcinogens by the International Agency for Research on Cancer[1] and 4-nitrobiphenyl (4-NBP), which can be reduced enzymatically to 4-ABP, is a suspect human carcinogen. Because of their carcinogenity, methods for the determination of these substances in trace quantities also in complex, particularly biological, matrices are increasingly demanded.

4-ABP is determined most sensitively by gas chromatography using the electron capture detector, which exhibits a detection limit of 0·4 ng for this substance transformed into the pentafluoropropionic acid derivative[2], and 0·2 ng for the substance after its bromination[3]. As little as 2 ng of 4-ABP in 1 mg of 2-aminobiphenyl can be isolated by thin layer chromatography, extracted from the thin layer and determined fluorometrically[4], and 2 μg of 4-ABP can be detected on a thin layer with antimony pentachloride[5]. The spectrofluorometric determination of this compound is highly sensitive and selective[6]; less sensitive and selective is its spectrophotometric determination based on its coupling with 8-amino-1-naphthol-5,7-disulphonic[7] or 2--naphthol-3,6-disulphonic[8] acid. A radioimmunoassay method has been developed

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for the determination of picomole quantities of acetylated metabolite of 4-ABP in urine, which can serve as a measure for human exposition[9]. Of electrochemical methods, only the determination of higher quantities of 4-NBP by classical polarography has been reported[10]. The present work is therefore aimed at the study of analysis of 4-NBP and 4-ABP based on their electrochemical reduction or oxidation, respectively. We found controlled-potential coulometry a convenient method for the determination of 4-NBP in higher amounts; this approach was applied to the standardization of the stock solutions which were employed for plotting the calibration graph for the substantially more sensitive polarographic and voltammetric methods. Trace quantities of 4-NBP can be well determined by differential pulse polarography (DPP). Trace amounts of 4-ABP were determined by differential pulse voltammetry (DPV) on a glassy carbon electrode, a method which is also suitable for the determination of a number of other carcinogenic aromatic amines[11,12].

EXPERIMENTAL

Reagents

Stock solutions of 4-NBP and 4-ABP (1 mmol l^{-1}) in methanol or in glacial acetic acid were prepared by dissolving the requisite amounts of the substances (Merck, Darmstadt) in redistilled solvent. Standard Britton–Robinson buffers were prepared conventionally[13]. Water was distilled twice in a quartz apparatus before use. All chemicals were of reagent grade purity.

Apparatus

The polarographic and voltammetric curves were recorded on a PA 2 polarographic analyzer interfaced to an XY 4103 recorder (Laboratorní přístroje, Prague) in the three-electrode connexion. Polarographic measurements were made at room temperature using a polarization rate of 5 mV s^{-1} and with a controlled drop time of 1 s and mercury column height 36 cm unless stated otherwise. The dropping mercury electrode parameters, at $h = 36$ cm, were $m = 2.83$ mg s^{-1} and $t = 2.82$ s in 0·1M-KCl at 0 V vs S.C.E. The voltammetric measurements were performed using an RDE glassy carbon rotating disc electrode (Laboratorní přístroje, Prague) in the three-electrode connexion at a polarization rate of 5 mV s^{-1}. Cyclic voltammograms were obtained with a HMDE 1 hanging mercury drop electrode (Laboratorní přístroje, Prague); the maximum drop size obtainable by opening the valve for 160 ms was used, the polarization rate was 100 mV . . s^{-1}. A saturated calomel reference electrode and a platinum wire auxiliary electrode were invariably employed. The controlled-potential coulometry experiments were made on an OH 404 coulometric analyzer (Radelkis, Budapest) with digital integration of the charge passed and automatic compensation for residual current. The cathode was mercury, the anode was an OH 935 platinum electrode (5 cm^2 area), separated by a glass frit; a saturated calomel reference electrode was used. The solution was stirred with an electromagnetic stirrer. Oxygen was removed by nitrogen purging. The nitrogen purification line consisted of an alkaline solution of sodium anthraquinone 2-sulphonate and solutions of chromium(II) ions in dilute hydrochloric acid in contact with zinc amalgam. The acidity was measured with a PHM 62 pH-meter (Radiometer, Copenhagen) equipped with a glass indicator electrode and a calomel reference electrode.

Data Handling

All linear relations were subjected to linear regression processing[14] on a TI-59 programmable calculator (Texas Instruments, USA). Logarithmic analysis of the polarographic waves was also performed on this calculator. The detection limit was determined according to Skogerboe and Grant[15] as the $tS_{y,x}/a$ value, where $S_{y,x}$ is the standard deviation of the experimental points from the calibration straight line, a is the slope of this straight line and t is Student's coefficient, for which the tabulated values[14] at the 99% confidence level were taken with respect to the number of points (n) used for constructing the calibration plot.

Procedures

Controlled-potential coulometry. To a 200 ml coulometric vessel were added 50 ml of Britton–Robinson buffer at pH 8·6 or 50 ml of 0·1M phosphoric acid and 40 ml of methanol, and the solution was nitrogen purged while the preelectrolysis at a chosen constant potential was started. In about 15 min the residual current decreased to less than 0·2 mA and then remained constant. The parameters for the automatic compensation of the residual current then were set and 10·00 ml of methanolic solution containing 1—7 mg of 4-NBP was added to the bubbled solution. The electrolysis was stopped after the electrolytic current decreased down to the residual current value before the addition of analyte. The charge passed was determined by current integration.

Polarography and voltammetry. Ten ml of the solution of a suitable composition (buffer or other supporting electrolyte, methanol, depolarizer) was freed from oxygen by 10 min nitrogen purging, and its polarographic curve was recorded. The same procedure was used for voltammetry except that 50 ml of solution was taken. The mixed aqueous–methanolic system was used because of the sparing solubility of the substances in pure aqueous solutions.

RESULTS AND DISCUSSION

Determination of 4-Nitrobiphenyl by Controlled-Potential Coulometry

As follows from a comparison of the polarographic wave heights for 4-NBP and azobenzene (see later), four electrons are exchanged in alkaline solutions and six in acid solutions. This was confirmed by controlled-potential coulometry. The accuracy and reproducibility of determination are documented by data of Table I, which indicate that alkaline solutions suit better for the analysis.
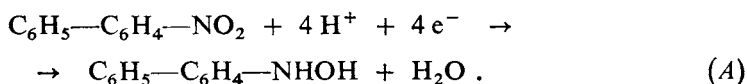
TAST Polarography of 4-Nitrobiphenyl

The dependences of current I_{lim} and potential $E_{1/2}$ on pH in solutions containing 50% (V/V) Britton–Robinson buffer and 50% (V/V) methanol are given in Table II. The $E_{1/2}$ value shifts to more negative values with increasing pH over the pH $3-12$ range. A well-developed wave is observed at pH > 7 whereas two ill-resolved waves appear at pH < 7. The height of the first wave is pH-independent whereas the second wave, observable in acid solutions only, decreases with increasing pH. The slope of the dependence of $E_{1/2}$ on pH is 58 mV for the first wave. The best-developed

waves were obtained at pH 2·6 and pH 8·6, which thus can be recommended for the determination.

The dependences of the logarithm of the limiting current on the logarithm of the flow rate m, at various electronically controlled drop times t and various mercury column heights h, were measured in 50% (V/V) Britton–Robinson buffer–50% (V/V) methanol solutions at pH 8·65. The slopes of the log I_{lim} vs log m dependence $(0·66 \pm 0·02$, which is about 2/3) and the log I_{lim} vs log t dependence $(0·17 \pm 0·01$, which is about 1/6), confirm the validity of the relation $I_{lim} \sim m^{2/3}t^{1/6}$ and thereby the diffusion nature of the limiting current.

The slope in the logarithmic analysis of the polarographic waves of 4-NBP (Table II) indicates that the process is irreversible. This is also confirmed by cyclic voltammetry on a hanging mercury drop electrode, the anodic peak being absent from the cyclic voltammogram of this compound (Fig. 1).

For establishing the number of exchanged electrons, the wave height of 4-NBP was compared with that of azobenzene in the same medium (a two-electrode exchange is known[16] to take place with azobenzene on a mercury electrode). It follows from this comparison (Fig. 2) that at pH 8·65, 4-NBP is reduced with the exchange of four electrons, which corresponds to the formation of the hydroxylamine,

$$C_6H_5\!-\!C_6H_4\!-\!NO_2 \; + \; 4\,H^+ \; + \; 4\,e^- \; \rightarrow$$

$$\rightarrow \; C_6H_5\!-\!C_6H_4\!-\!NHOH \; + \; H_2O \; . \qquad\qquad (A)$$

In a system of 50% (V/V) 0·1M-H$_2$SO$_4$–50% (V/V) methanol the total wave height for 4-NBP is three times higher than that for azobenzene (Fig. 2), which indicates a

TABLE I

Accuracy and reproducibility of determination of 4-nitrobiphenyl by controlled-potential coulometry. A: $E = -800$ mV, medium: Britton–Robinson buffer pH 8·62–methanol 1 : 1; $n = 4$. B: $E = -500$ mV, medium: 0·1M phosphoric acid–methanol 1 : 1; $n = 6$

Added mg	Found[a], mg		Standard deviation, mg		Time of reduction, min	
	A	B	A	B	A	B
0·996	0·994	0·986	0·01	0·02	30	40
1·992	1·983	1·973	0·02	0·03	40	60
4·980	4·963	4·946	0·05	0·02	50	90
6·972	6·967	6·954	0·05	0·02	60	130

[a] Average of 7 replicate determinations for which the standard deviation is also calculated.

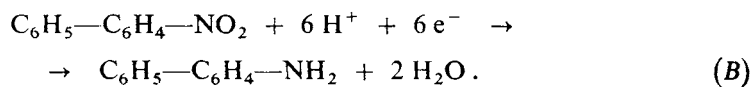six-electron exchange according to Eq. (B),

$$C_6H_5—C_6H_4—NO_2 + 6H^+ + 6e^- \rightarrow$$
$$\rightarrow C_6H_5—C_6H_4—NH_2 + 2H_2O . \qquad (B)$$

TABLE II

Dependence of $E_{1/2}$ and I_{lim} on pH for 4-nitrobiphenyl in 50% (V/V) Britton–Robinson buffer––50% (V_i/V) methanol solutions. Depolarizer concentration 100 μmol l^{-1}

pH	$E_{1/2}$ mV	I_{lim}[a] μA	$α$[b] mV	pH	$E_{1/2}$ mV	I_{lim}[a] μA	$α$[b] mV
2·61	−251	68·0	34·6	6·78	−453	69·0	48·6
		100·0				77·0	
2·86	−252	70·0	28·5	8·08	−534	70·0	45·9
		105·0		8·60	−582	70·0	43·2
4·00	−312	68·0	39·6	9·28	−628	70·0	49·7
		94·0		10·02	−679	70·0	49·5
5·15	−361	69·0	39·8	10·65	−723	70·0	53·8
		92·0		12·15	−748	70·0	32·8
6·02	−406	70·0	41·2				
		85·0					

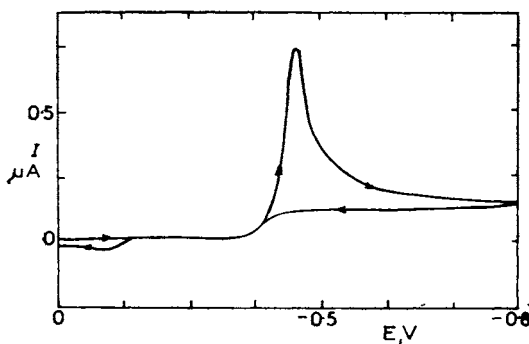[a] The bottom figure pertains to the 2nd wave where a pair of data is given; [b] slope of logarithmic analysis.

FIG. 1

Cyclic voltammogram for 4-nitrobiphenyl (100 μmol l^{-1}) on a hanging mercury drop electrode. Medium: 50% (V/V) Britton–Robinson buffer – 50% (V/V) methanol, pH 8·65

The concentration dependence was measured in solutions of 50% (V/V) Britton–Robinson buffer $-$ 50% (V/V) methanol at pH 2·65 and pH 8·65 over the region of $1 - 100\ \mu mol\ l^{-1}$. The detection limits in the acid and alkaline solutions were 1 and $0·7\ \mu mol\ l^{-1}$, respectively. Polarograms of 4-NBP at pH 8·65 and at the lowest determinable concentrations are shown in Fig. 3. The calibration straight line parameters are given in Table III.
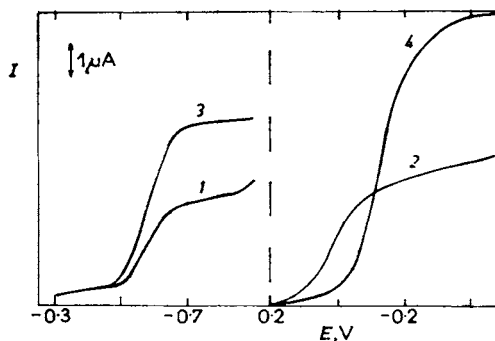

FIG. 2

Polarographic waves of azobenzene (1, 2) and 4-nitrobiphenyl (3, 4) at pH 8·6 (1, 3) and pH 1·4 (2, 4). Depolarizer concentration 100 $\mu mol\ l^{-1}$
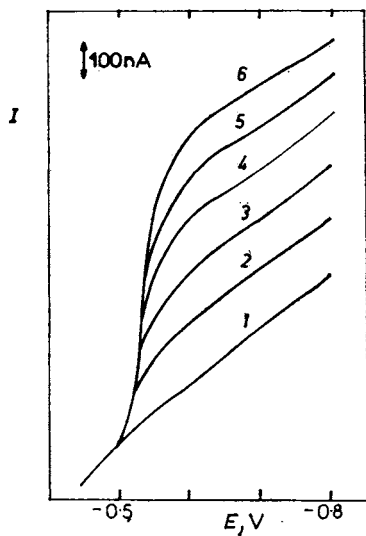
FIG. 3

TAST polarograms of 4-nitrobiphenyl in 50% (V/V) Britton–Robinson buffer $-$ 50% (V/V) methanol solutions at pH 8·65. Depolarizer concentration ($\mu mol\ l^{-1}$): 1 0, 2 2, 3 4, 4 6, 5 8, 6 10

TABLE III

Parameters of the calibration curves for the determination of 4-nitrobiphenyl and 4-aminobiphenyl: slope (a) and its standard deviation (S_a), intercept (b) and its standard deviation (S_b), correlation coefficient (r) and standard deviation of experimental points from the regression straight line ($S_{y,x}$)

pH	Concentration μmol l^{-1}	$a(S_a)$ μA l mol^{-1}	$b(S_b)$ μA	r	$S_{y,x}$ μA
4-NBP in Britton–Robinson buffer – methanol 1 : 1, TAST technique
2·6	10 — 100	$8·89 . 10^4 (1·7 . 10^2)$	$2·83 . 10^{-2} (1·0 . 10^{-2})$	0·9985	$1·5 . 10^{-2}$
8·6	10 — 100	$4·67 . 10^4 (1·8 . 10^2)$	$-3·50 . 10^{-2} (1·1 . 10^{-2})$	0·9942	$1·6 . 10^{-2}$
2·6	1 — 10	$1·06 . 10^5 (3·4 . 10^1)$	$7·00 . 10^{-3} (2·1 . 10^{-3})$	0·9959	$3·1 . 10^{-3}$
8·6	1 — 10	$6·10 . 10^4 (1·4 . 10^2)$	$-1·20 . 10^{-3} (8·9 . 10^{-4})$	0·9978	$1·3 . 10^{-3}$
4-NBP in Britton–Robinson buffer – methanol 1 : 1, DPP technique
2·6	10 — 100	$1·84 . 10^5 (2·0 . 10^3)$	$3·87 . 10^{-1} (1·3 . 10^{-1})$	0·9995	$1·9 . 10^{-1}$
8·6	10 — 100	$1·66 . 10^5 (1·7 . 10^3)$	$-7·00 . 10^{-2} (1·1 . 10^{-1})$	0·9996	$1·6 . 10^{-1}$
2·6	1 — 10	$1·77 . 10^5 (7·0 . 10^2)$	$5·20 . 10^{-2} (4·5 . 10^{-3})$	0·9999	$6·6 . 10^{-3}$
8·6	1 — 10	$1·61 . 10^5 (5·5 . 10^3)$	$-2·73 . 10^{-2} (3·4 . 10^{-2})$	0·9953	$5·0 . 10^{-2}$
2·6	0·1 — 1	$1·66 . 10^5 (3·4 . 10^3)$	$-3·47 . 10^{-3} (2·1 . 10^{-3})$	0·9983	$3·1 . 10^{-3}$
8·6	0·1 — 1	$1·52 . 10^5 (6·2 . 10^3)$	$-7·73 . 10^{-3} (3·9 . 10^{-3})$	0·9933	$5·6 . 10^{-3}$
4-NBP in acetic acid–water 1 : 1, DCV technique
1·45	100 — 500	$1·02 . 10^5 (1·1 . 10^3)$	$-5·00 . 10^{-1} (3·7 . 10^{-1})$	0·9998	$3·5 . 10^{-1}$
4-NBP in acetic acid–water 1 : 1, DPV technique
1·45	20 — 100	$3·44 . 10^4 (3·5 . 10^2)$	$6·75 . 10^{-2} (2·3 . 10^{-2})$	0·9998	$2·2 . 10^{-2}$
1·45	2 — 10	$4·38 . 10^4 (1·5 . 10^3)$	$8·50 . 10^{-3} (9·7 . 10^{-3})$	0·9983	$9·3 . 10^{-3}$
4-NBP in Britton–Robinson buffer – methanol 1 : 1, DPV technique
7·8	20 — 100	$1·04 . 10^5 (8·7 . 10^2)$	$1·30 . 10^{-1} (5·7 . 10^{-2})$	0·9999	$5·5 . 10^{-2}$
7·8	2 — 10	$9·88 . 10^4 (3·8 . 10^3)$	$-2·25 . 10^{-2} (2·5 . 10^{-2})$	0·9978	$2·4 . 10^{-2}$
4-ABP in 0·1M-HCl–methanol 1 : 1, DCV technique
1·4	100 — 500	$2·05 . 10^4 (7·6 . 10^2)$	$-8·00 . 10^{-1} (2·5 . 10^{-1})$	0·9979	$2·4 . 10^{-1}$
4-ABP in acetic acid–methanol 1 : 1, DPV technique
3·0	200 — 1000	$1·59 . 10^4 (4·7 . 10^1)$	$6·25 . 10^{-2} (3·1 . 10^{-2})$	0·9986	$3·0 . 10^{-2}$
3·0	20 — 100	$1·50 . 10^4 (2·4 . 10^2)$	$1·19 . 10^{-1} (1·6 . 10^{-2})$	0·9983	$1·5 . 10^{-2}$
3·0	2 — 10	$1·51 . 10^4 (3·2 . 10^2)$	$7·75 . 10^{-3} (2·1 . 10^{-3})$	0·9993	$2·0 . 10^{-3}$

Differential Pulse Polarography of 4-*Nitrobiphenyl*

The pH dependences of the peak current I_p and potential E_p are given in Table IV. At pH $2-12$, E_p shifts to negative values with increasing pH. The slope of the dependence of E_p on pH is 60 mV for the first peak and 164 mV for the second peak which appears in strongly acid solutions only.

The concentration dependence was measured in 50% (V/V) Britton–Robinson buffer–50% (V/V) methanol at pH 2·6 and pH 8·6 over the region of 0·1 −100 µmol l^{-1}. The detection limits were 0·12 µmol l^{-1} in the acid region and 0·06 µmol l^{-1} in the alkaline region. Polarograms for the lowest concentrations are shown in Fig. 4.

TABLE IV

Dependence of E_p and I_p on pH for 4-nitrobiphenyl in 50% (V/V) Britton–Robinson buffer − 50% (V/V) methanol solutions. Depolarizer concentration 100 µmol l^{-1}, pulse height 100 mV

pH	$E_p{}^a$ mV	$I_p{}^a$ µA	pH	E_p mV	I_p µA
2·60	− 190	19·7	6·89	− 440	16·7
	− 370	3·5	8·05	− 510	16·0
3·81	− 270	18·1	8·60	− 560	15·8
	− 560	2·1	9·12	− 600	15·5
4·99	− 330	18·4	10·03	− 650	14·5
	− 770	0·6			
6·04	− 390	17·4	12·03	− 780	13·5

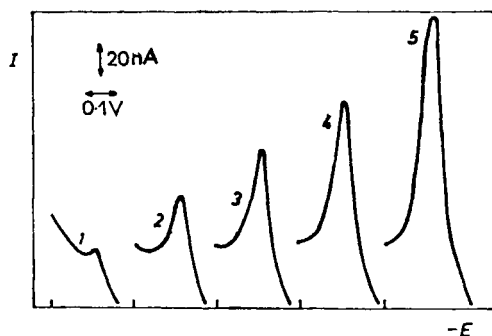^{a} The bottom figure pertains to the 2nd peak where a pair of data is given.

FIG. 4

Differential pulse polarograms of 4-nitrobiphenyl in 50% (V/V) Britton–Robinson buffer – 50% (V/V) methanol solutions at pH 8·6. Pulse height 100 mV, starting potential $-0·4$ V, depolarizer concentration (µmol . l^{-1}): *1* 0·1, *2* 0·3, *3* 0·5, *4* 0·7, *5* 0·9

DC *Voltammetry on a Rotating Glassy Carbon Disc Electrode*

The DC voltammograms were measured at 2 000 rpm in 50% (V/V) Britton–Robinson buffer – 50% (V/V) methanol solutions at analyte concentrations of 100 µmol l^{-1}. Cathodic reduction was studied for 4-NBP and anodic oxidation for 4-ABP. The I_{lim} and $E_{1/2}$ values are given in Table V in dependence on pH. Well-developed waves were obtained for 4-NBP in buffered solutions at pH 7·8, and even better-developed ones were observed in acetic acid–water $(1 : 1)$ mixture in which the depolarizer is well-soluble. For 4-ABP the best-developed waves were obtained in 0·1M-HCl–methanol $(1 : 1)$ solution.

The effect of rotation speed was also examined. The dependences of I_{lim} on $\omega^{1/2}$ both for the reduction of 4-NBP in acetic acid–water 1 : 1 solution and for the oxidation of 4-ABP in 0·1M-HCl–methanol 1 : 1 are linear. The high current intercept is indicative of adsorption of the analytes on the electrode used.

The concentration dependences were measured at 2 000 rpm in acetic acid–water $(1 : 1)$ solutions for 4-NBP and in 0·1M-HCl–methanol $(1 : 1)$ solution for 4-ABP over the region of $100 - 500$ µmol l^{-1}. The linear shape of the dependences confirms the validity of Levich's equation. Their slopes are 10·25 µA l mol^{-1} for 4-NBP and 2·05 µA l mol^{-1} for 4-ABP. As compared with DPV (see below), DCV exhibits considerable noise so that the limit of detection for the two substances is about 10 µmol l^{-1}.

TABLE V

Dependence of $E_{1/2}$ and I_{lim} on pH for reduction of 4-nitrobiphenyl and oxidation of 4-amino-biphenyl on a rotating electrode at 2 000 rpm in 50% (V/V) Britton–Robinson buffer – 50% (V/V) methanol solutions. Depolarizer concentration 100 µmol l^{-1}

	Reduction of 4-NBP			Oxidation of 4-ABP	
pH	$E_{1/2}$ mV	I_{lim} µA	pH	$E_{1/2}$ mV	I_{lim} µA
2·80	−0·64	16·00	2·90	0·73	14·25
3·70	−0·67	18·00	4·18	0·67	13·50
4·70	−0·71	19·25	5·34	0·66	13·50
5·75	−0·74	19·75	6·40	0·65	12·75
7·77	−0·75	21·25	7·69	0·62	12·60
8·40	−0·75	12·50	8·37	0·61	12·00
9·16	−0·75	10·00	9·40	0·59	12·45
10·02	−0·75	8·75	10·64	0·60	10·35
11·80	−0·75	6·25	10·95	0·60	9·54
			12·45	0·55	8·40

Differential Pulse Voltammetry on a Stationary Glassy Carbon Disc Electrode

Preliminary experiments showed that DPV on a stationary electrode is more sensitive than DCV on a rotating electrode. The dependences of the DPV peak height on the concentration of 4-NBP were therefore measured in 50% (V/V) Britton–Robinson buffer – 50% (V/V) methanol solutions at pH 7·8 and in acetic acid–water 1 : 1 solutions over the region of $1-100\ \mu mol\ l^{-1}$. The detection limits in the two systems were 1 and 0·9 $\mu mol\ l^{-1}$, respectively.

For the anodic oxidation of 4-ABP, the supporting electrolyte curve in the presence of hydrochloric acid is too steep at high gains. 0·1M-Acetic acid–methanol 1 : 1 solution was therefore used; well-developed and evaluable peaks were obtained even at low concentrations. The DP voltammograms were measured at depolarizer concentrations of $1-100\ \mu mol\ l^{-1}$. The detection limit was 0·5 $\mu mol\ l^{-1}$.

The calibration curves for the approaches used are summarized in Table III, in which the sensitivities of the various methods can be seen. It is an advantage of the glassy carbon electrode that the two carcinogens can be determined on it simultaneously. For their determination in biological materials, however, the two substances will have to be prior separated by extraction or by thin layer chromatography.

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