THE DETERMINATION OF N,N-DIMETHYL-4-AMINOAZOBENZENE AND ITS REDUCTION PRODUCTS BY ANODIC VOLTAMMETRY AT A GLASSY CARBON ELECTRODE*

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The oxidation of N,N-dimethyl-4-aminoazobenzene at a rotating and stationary glassy carbon disk electrode in a medium of 40% (v/v) acetic acid was studied in the framework of a systematic study of methods for determining the efficiency of the destruction of the genotoxic derivatives of this substance; conditions were found for the determination using direct current and differential pulse voltammetry. Analogous conditions were used to determine aniline, N,N-dimethylaniline and N,N-dimethyl-*p*-phenylenediamine as model substances representing possible products of the reductive splitting of genotoxic azocompounds. The determination limit of the method is less than $1 \,.\, 10^{-5}$ mol l^{-1} and its selectivity is completely satisfactory for controlling the efficiency of the destruction.

N,N-dimethyl-4-aminoazobenzene is well known chemical carcinogen[1]. For this reason, its polarographic behaviour[2] and its possible chemical destruction[3] for the decontamination of laboratories and laboratory waste have been studied in detail. The best means of destroying this substance seems to be reduction with powdered zinc, followed by oxidation of the aromatic amines formed with permanganate. An essential part of this method for the destruction of genotoxic substances is an analytical method to control its effectiveness. Consequently, this work describes the determination of N,N-dimethyl-4-aminoazobenzene based on its anodic oxidation at a glassy carbon electrode. A medium of 40% (v/v) acetic acid containing sulphuric acid (c = 1 mol l^{-1}) was employed which is optimum for the destruction of the test substances. The polarographic determination of N,N-dimethyl-4-aminoazobenzene is not very sensitive in this strongly acid medium, as it is reduced at very positive potentials (about +300 mV vs a saturated calomel electrode), where the dissolution of mercury is important. In addition, it could be expected that anodic

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differential pulse voltammetry would be useful to determine not only the initial azo compound but also aromatic amines formed by the reductive splitting of the test substance. This prediction was confirmed in this work using aniline, N,N-dimethylaniline and N,N-dimethyl-*p*-phenylene diamine as model substances.

In contrast to the polarographic reduction of the derivatives of N,N-dimethyl-4-aminoazobenzene, studied in detail in a number of works[2,4,5], far less attention has been devoted to their electrochemical oxidation. An orientative study has been carried out of the oxidation of some derivatives of N,N-dimethyl-4-aminoazobenzene at a glassy carbon electrode in a search for optimal conditions for their electrochemical detection in combination with high performance liquid chromatography[6]. The oxidation of N,N-dimethyl-4-aminoazobenzene at a platinum RDE and a large-area platinum grid was studied with special consideration of the oxidation products and its usefulness as a model of metabolic transformation[7]. The use of the anodic oxidation of 4-aminoazobenzene for its determination using DPV or direct current voltammetry (DCV) at a glassy carbon stationary (SDE) or rotating (RDE) disk electrode is described in ref.[8].

The anodic oxidation of aromatic amines formed in the reductive splitting of the derivatives of N,N-dimethyl-4-aminoazobenzene is discussed in detail in a monograph[9] and a number of references to works dealing with its oxidation at RDE's can be found in the review[10]. We have employed the anodic oxidation of these aromatic amines to demonstrate their formation in the reduction of azo compounds by potentiostatic coulometry on a large-area mercury electrode[11]; however, quantitative evaluations were not carried out.

EXPERIMENTAL

Reagents

The stock solutions of N,N-dimethyl-4-aminoazobenzene (Research Institute for Organic Synthesis, Pardubice-Rybitví), aniline (Lachema, Brno), N,N-dimethylaniline (Lachema, Brno) and N,N-dimethyl-*p*-phenylenediamine (Lachema, Brno) ($c = 1 \,.\, 10^{-2}$ mol l^{-1}) were prepared by dissolving exactly weighed amounts of the solid substance in 50 ml of anhydrous acetic acid and diluting to 100 ml with redistilled water. The stock solution of N,N-dimethyl-4-aminoazobenzene ($c = 1 \,.\, 10^{-3}$ mol l^{-1}) in methanol was prepared by dissolving an exactly weighed amount of the substance in p.a. solvent and diluting to 100 ml. The purity of the N,N-dimethyl-4-aminoazobenzene was controlled using TLC (ref.[12]) and HPLC (ref.[6]) and the concentration of the stock solutions was determined titanometrically[13]. The remaining chemicals were of p.a. purity (Lachema, Brno) and the water was doubly distilled in a quartz apparatus.

Apparatus

The voltammetric measurements were carried out using the PA 4 polarographic analyzer with the XY 4103 recorder (both from Laboratorní přístroje, Prague). Work was carried out using a three-electrode system with a platinum foil auxiliary electrode and saturated calomel reference electrode, to which all the potential values are referred. The working electrode was the RDE 1 rotating disk electrode (Laboratorní přístroje, Prague). The body of this electrode consisted of a teflon cylinder with a diameter of 12 mm, with an electroactive part consisting of a glassy carbon disk with a diameter of 3 mm. Where not stated otherwise, a polarization rate of 5 mV . . s^{-1}, modulation amplitude of 100 mV and interval between pulses of 1 s were employed.

Procedure

The calibration curves were measured in triplicate and evaluated using the linear least squares method. The determination limit in the concentration range (2—10) . 10^{-x} mol l^{-1} was found as ten times the standard deviation for 10 determinations of the analyte with a concentration of 2 . 10^{-x} mol l^{-1} (ref.[14]).

RESULTS AND DISCUSSION

Direct Current Voltammetry of N,N-*Dimethyl-4-aminoazobenzene*

It was found that the test substance in the given medium yields two relatively well developed waves at a glassy carbon RDE; the heights of these waves decrease markedly on repeated recording. Consequently, it is necessary to lightly clean the electrode with emery paper and polish with velvet between measurements in the concentration range 1 . 10^{-3} and 2 . 10^{-4} mol l^{-1}. The most reproducible results can be obtained by first recording the curve of the base electrolyte prior to each determination, after cleaning the electrode. Cleaning with emery paper is not necessary in the concentration range from 1 . 10^{-4} to 2 . 10^{-5} mol l^{-1} and it is sufficient to occasionally polish the electrode with velvet and record the curve of the base electrolyte. The dependence of the heights of both waves on the square root of the angular velocity of electrode rotation is not linear, indicating that this is not a simple, convective diffusion-controlled process. The observed faster decrease in the height of the second wave with decreasing electrode rotation rate is apparently connected with increasing passivation of the electrode by the reaction products, which are not sufficiently rapidly removed from the electrode surface at lower rotation rates. For analytical purposes, the optimum rotation rate is the fastest, i.e. 2 500 rpm. At a constant rotation rate, the wave height increases somewhat with increasing polarization rate, but its shape practically does not change. Simultaneously, however, the slope of the curve for the base electrolyte and the steepness of the curve in the limiting current region also increase. For analytical applications, the optimum polarization rate is 5 mV s^{-1}.

The dependence of the wave height on the concentration of N,N-dimethyl-4--aminoazobenzene under optimum conditions (i.e. polarization rate of 5 mV s^{-1} and rotation rate of 2 500 rpm) is depicted in Fig. 1. Of the methods for the evaluation of the peak height, the best was found to be comparison of the test curve with that of the base electrolyte and measuring the wave height above the base electrolyte line

TABLE I

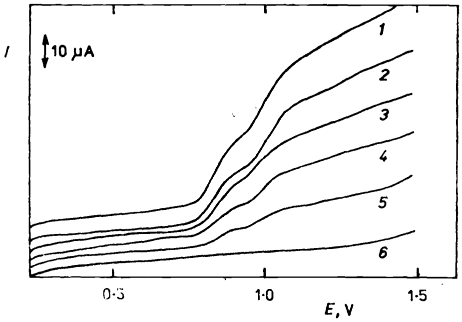
Parameters of the calibration curves and determination limits for N,N-dimethyl-4-aminoazobenzene (*I*), aniline (*II*), N,N-dimethylaniline (*III*) and N,N-dimethyl-*p*-phenylenediamine (*IV*) by various voltammetric methods

Substance	Method	Concentration mol l^{-1}	Slope mA mol^{-1} l	Intercept μA	Correl. coef.	Determination limit mol l^{-1}
I[a]	DC[b]	$(2-10) . 10^{-4}$	15·1	−2	0·9980	—
	DC[b]	$(2-10) . 10^{-5}$	21·7	+0·2	0·9973	$1·3 . 10^{-5}$
	DP[b]	$(2-10) . 10^{-4}$	15·8	−0·7	0·9987	—
	DP[b]	$(2-10) . 10^{-5}$	20·8	+0·2	0·9981	$0·4 . 10^{-5}$
	DP[c]	$(2-10) . 10^{-5}$	8·1	−0·1	0·9976	—
	DP[c]	$(2-10) . 10^{-6}$	10·2	0·0	0·9969	$2 . 10^{-6}$
II	DC[c]	$(2-10) . 10^{-4}$	3·75	0·1	0·9992	—
	DC[c]	$(2-10) . 10^{-5}$	4·25	0·0	0·9975	$1 . 10^{-5}$
	DP[c]	$(2-10) . 10^{-4}$	5·55	−0·2	0·9989	—
	DP[c]	$(2-10) . 10^{-5}$	4·85	0·0	0·9982	$0·5 . 10^{-5}$
III	DC[c]	$(2-10) . 10^{-4}$	2·10	−0·03	0·9972	—
	DC[c]	$(2-10) . 10^{-5}$	2·50	−0·04	0·9810	$3 . 10^{-5}$
	DP[c]	$(2-10) . 10^{-4}$	4·38	0·10	0·9902	—
	DP[c]	$(2-10) . 10^{-5}$	2·25	−0·05	0·9869	$2 . 10^{-5}$
IV	DC[b]	$(2-10) . 10^{-4}$	25·5	0·7	0·9970	—
	DC[b]	$(2-10) . 10^{-5}$	31·0	0·1	0·9968	$4 . 10^{-5}$
	DP[b]	$(2-10) . 10^{-4}$	6·0	0·1	0·9890	—
	DP[b]	$(2-10) . 10^{-5}$	7·5	0·1	0·9875	$3 . 10^{-5}$

[a] Values corresponding to the first wave or peak; [b] rotating electrode (2 500 rpm); [c] stationary electrode.

FIG. 1

DC voltammograms of N,N-dimethyl-4-aminoazobenzene (*I*) at an RDE (2 500 rpm) in acetic acid medium (40% (v/v)) in the presence of sulphuric acid (c = 1 mol . l^{-1}) at a polarization rate of 5 mV s^{-1}. $c(I) . 10^4$ (mol l^{-1}): *1* 10; *2* 8; *3* 6; *4* 4; *5* 2; *6* base electrolyte



at a constant potential of 0·95 V for the first wave and 1·15 V for the second wave. The first wave is more useful for analytical purposes as its height is directly proportional to the concentration in a wide concentration range, permitting the use of the method of standard additions. The dependence of the height of the second wave on the concentration in the range $1 \,.\, 10^{-4}$ to $1 \,.\, 10^{-5}$ mol l^{-1} did not pass through the origin, so that the method of two standard additions had to be used. At concentrations below $1 \,.\, 10^{-5}$ mol l^{-1}, the base electrolyte curve is so steep at the required recorder sensitivity that the wave for the test substance can practically not be evaluated. The parameters of the calibration curve calculated by the method of linear regressions and the determination limits are given in Table I.

Differential Pulse Voltammetry of N,N-*Dimethyl-4-aminoazobenzene*

The differential pulse voltammogram contains two peaks (see Fig. 2) whose height decreases with decreasing electrode rotation rate. This decrease is especially sharp for the more positive peak, which is practically invisible at the stationary electrode. The electrode cleaning procedure described for DCV had to be employed for the pulse technique to ensure reproducible results. As the polarization rate increases, the potential difference between the two peaks decreases somewhat and the depth of the minimum between greatly decreases. The height of the first peak is only slightly dependent on the polarization rate, while that of the second peak increases more rapidly with increasing rate. Consequently, the height of the first peak is greater than that of the second at a polarization rate of 2 mV s^{-1}, while the situation is the opposite at a rate of 10 mV s^{-1}. This phenomenon is apparently connected with the above-mentioned passivation of the electrode by the reaction products formed by oxidation at a potential corresponding to the maximum of the first peak. At higher polarization rates, the period of time during which the electrode is exposed to the effect of these products is shorter (before the second peak potential is attained), so that the decrease in the height of the second peak through passivation is smaller.

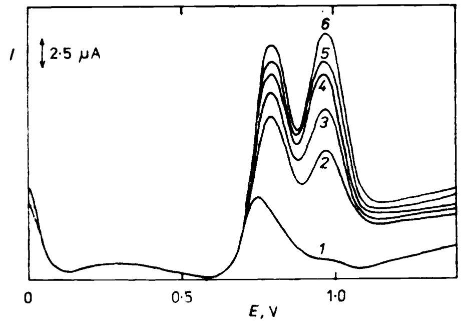


FIG. 2

Effect of the electrode rotation rate on the DP voltammograms of N,N-dimethyl-4-aminoazobenzene (*I*) ($c = 1 \,.\, 10^{-3}$ mol . l^{-1}). Medium the same as in Fig. 1. Polarization rate 5 mV s^{-1}; modulation amplitude 100 mV; rpm: *1* 0, *2* 500; *3* 1 000; *4* 1 500; *5* 2 000; *6* 2 500

However, evaluation of the first peak is preferable in analytical applications and a polarization rate of 2 mV s^{-1} is preferable, especially at lower concentrations, as the base electrolyte curve is less steep. As the size of the modulation amplitude increases, both peaks increase in height; consequently, the largest modulation amplitude, i.e. 100 mV, was employed. However, it is preferable to decrease the modulation amplitude at lower concentrations (of the order of 10^{-5} mol l^{-1}), yielding a less steep base electrolyte curve.

The dependence of the peak height on the concentration of N,N-dimethyl-4-aminoazobenzene is depicted in Fig. 3. Of the methods for evaluation of the curve, that based on measuring the peak height at its maximum value above the straight line connecting the minimum prior to the first and beyond the second peaks was found to yield the best results (see Fig. 3). The dependence of this height on the concentration is linear in the range $2 . 10^{-4}$ to $1 . 10^{-3}$ mol l^{-1} only for the first peak, while marked deviations from linearity occur for the second peak at concentrations above $5 . 10^{-4}$ mol l^{-1}, apparently connected with electrode passivation. When the peak height is measured above the line connecting the minimum between the peaks with the minimum prior to the first peak or beyond the second peak, deviations from linearity were found also for the first peak at concentrations above $8 . 10^{-4}$ mol l^{-1}. The calibration curves in the concentration range $1 . 10^{-4}$ to $2 . 10^{-5}$ mol l^{-1} obtained by measuring the peak height above the line connecting the minimum prior to the first and after the second peak are linear and pass through the origin for both peaks. If the peak height is measured from the line connecting the minima prior to or after the peaks with the minimum between the peaks, then the calibration curve does not pass through the origin even when its deviation from linearity is not great.

The noise level was high at sensitivities required for work with concentrations below $1 . 10^{-5}$ mol l^{-1}, apparently connected with the use of the rotating electrode. Consequently, the determination of the test substances using DPV at the SDE was

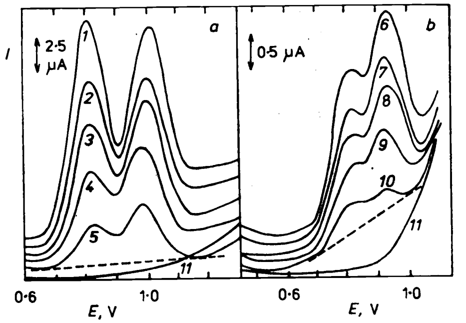


FIG. 3

DP voltammograms of N,N-dimethyl-4-aminoazobenzene (*I*) at an RDE (2 500 rpm). Medium and conditions as in Fig. 2. *c*(*I*) . 10^4 mol l^{-1}: *1* 10; *2* 8; *3* 6; *4* 4; *5* 2; *6* 1; *7* 0·8; *8* 0·6; *9* 0·4; *10* 0·2; *11* base electrolyte. The dashed line corresponds to the baseline from which the peak height was measured studied. The measured voltammograms for the individual concentration ranges are given for illustration in Fig. 4. Difficulties connected with electrode passivation are once again encountered in the concentration range from $1 . 10^{-3}$ to $2 . 10^{-4}$ mol l^{-1}; the effect is even more marked than for work with the rotating electrode. Reproducible results can be obtained only by cleaning the electrode prior to each measurement and then repeatedly recording the curve of the base electrolyte. Under these conditions, only the first peak was observed on the voltammograms; the second peak is suppressed to such a degree on the stationary electrode that only a slight shoulder on the decreasing part of the first peak is apparent. At concentrations above $5 . 10^{-4}$ mol l^{-1}, deviations from linearity were observed. Because of the necessity for energetic cleaning, the results obtained with SDE are somewhat less reproducible than those obtained using the RDE; the latter is clearly preferable for the concentration range $1 . 10^{-3}$ to $2 . 10^{-4}$ mol l^{-1}. At concentrations of $1 . 10^{-4}$ to $2 . 10^{-5}$ mol l^{-1}, this energetic cleaning is no longer necessary after each measurement and the results are more reproducible and the calibration curves are linear. The parameters of these curves are given in Table I. The shape of the voltammograms and their evaluation are depicted in Fig. 4. The lower noise in work with the SDE permitted useful voltammograms to be obtained in the concentration range $1 . 10^{-5}$ to $1 . 10^{-6}$ mol l^{-1}.

Consequently, it can be concluded that the RDE is preferable for determinations at higher concentrations (above $1 . 10^{-4}$ mol l^{-1}) while better results are obtained using the SDE at concentrations from $1 . 10^{-4}$ to $2 . 10^{-6}$ mol l^{-1}. Therefore, the determination of model aromatic amines using DPV at the SDE was studied.

Direct Current and Differential Pulse Voltammetry of Aniline

Work was carried out under conditions found to be optimal for the determination of low concentrations of N,N-dimethyl-4-aminoazobenzene, i.e. at a stationary electrode and polarization rate of 5 mV s^{-1}, modulation amplitude of 100 mV and

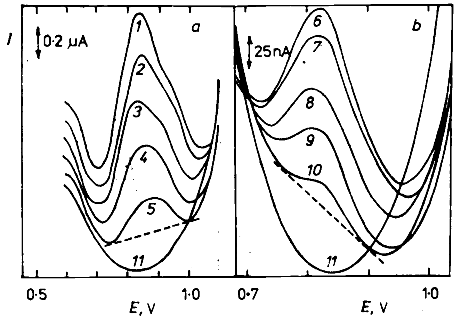


FIG. 4

DP voltammograms of N,N-dimethyl-4-aminoazobenzene (*I*) at an SDE. Conditions as in Fig. 2. $c(I) . 10^5$ mol l^{-1}: *1* 10; *2* 8; *3* 6; *4* 4; *5* 2; *6* 1; *7* 0·8; *8* 0·6; *9* 0·4; *10* 0·2; *11* base electrolyte. The dashed line corresponds to the baseline from which the peak height was measured interval between pulses of 1 s. The electrode cleaning procedure was the same as that for N,N-dimethyl-4-aminoazobenzene, i.e. cleaning with fine emery paper followed by polishing with velvet and recording of the base electrolyte curve prior to recording of the curve for the test substance.

Recording in the DC mode yielded well-developed waves; for aniline concentrations above $5 . 10^{-4} \text{ mol l}^{-1}$ a decrease was observed after attaining the limiting current, apparently again connected with electrode passivation. At higher concentrations a peak is indicated on the voltammograms, while regular, well-developed waves were obtained at lower concentrations (see Fig. 5). It was also found using cyclic voltammetry that the system is irreversible. On repeated measurements without cleaning the electrode, each subsequent curve was several percent lower than the previous curve; this decrease is greater for higher concentrations. The difference between the heights of the first and fifth curves measured after cleaning the electrode at an aniline concentration of $1 . 10^{-3}$, $6 . 10^{-4}$ and $2 . 10^{-4} \text{ mol l}^{-1}$ was 25%, 10% and 3%, respectively. This decrease was practically absent at lower concentrations. The best method of evaluation in the concentration range from $1 . 10^{-3}$ to $1 . 10^{-4} \text{ mol l}^{-1}$ was measurement of the maximum wave height above the straight line obtained by prolonging the linear part of the voltammogram prior to the wave (see Fig. 5*a*). In the concentration range $1 . 10^{-4}$ to $2 . 10^{-5} \text{ mol l}^{-1}$, the peak height was measured as the distance between two straight lines obtained by extending

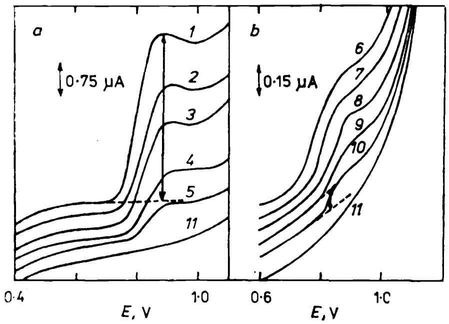


FIG. 5

DC voltammograms of aniline (*II*) at an SDE. Medium and conditions as in Fig. 1. *c*(*II*) . 10^4 (mol l^{-1}): *1* 10; *2* 8; *3* 6; *4* 4; *5* 2; *6* 1; *7* 0·8; *8* 0·6; *9* 0·4; *10* 0·2; *11* base electrolyte. The dashed lines represent the baselines from which the wave height was measured

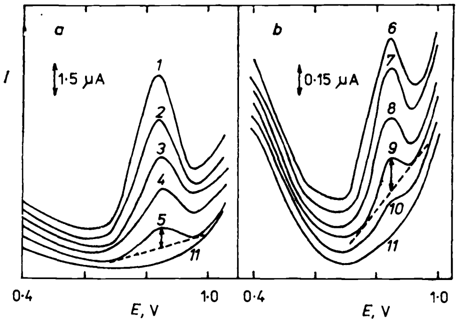


FIG. 6

DP voltammograms of aniline (*II*) at an SDE. Conditions and medium as in Fig. 2. *c*(*II*) . 10^4 (mol l^{-1}): *1* 10; *2* 8; *3* 6; *4* 4; *5* 2; *6* 1; *7* 0·8; *8* 0·6; *9* 0·4; *10* 0·2; *11* base electrolyte. The dashed line corresponds to the baseline from which the peak height was measured the linear part of the voltammogram prior to the wave and that obtained in the limiting current region. This distance was measured at the half-wave potential (Fig. 5*b*). The parameters of the calibration curves and the calculated determination limit are given in Table I. Concentrations below $1 . 10^{-5}$ mol l^{-1} cannot be determined by this method as the base electrolyte curve is too steep.

In DP measurements in the concentration range $1 . 10^{-4}$ to $2 . 10^{-5}$ mol l^{-1}, reproducible results could be obtained by polishing the electrode with velvet after each measurement. At concentrations of $1 . 10^{-3}$ to $2 . 10^{-4}$ mol l^{-1}, cleaning with metallographic paper and polishing with velvet, followed by recording the curve of the base electrolyte was necessary. Under these conditions, the measured voltammograms and evaluation method correspond to those depicted in Fig. 6. The parameters of the measured calibration curves are again given in Table I.

Direct Current and Differential Pulse Voltammetry of N,N-*Dimethylaniline*

The optimal procedures, shapes of the voltammograms obtained (see Figs 7 and 8) and parameters of the calibration curves (see Table I) are analogous with those obtained for aniline. However, at concentrations below $5 . 10^{-5}$ mol l^{-1}, the waves are difficult to evaluate and only a slight indication of a wave is visible on the DC voltammogram at a concentration of $2 . 10^{-5}$ mol l^{-1} (see Fig. 7*b*). It also followed

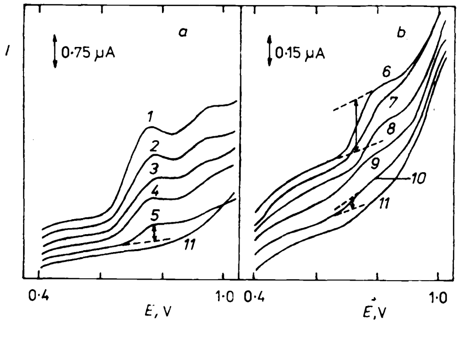


FIG. 7

DC voltammograms of N,N-dimethylaniline (*III*) at an SDE. Medium and conditions as in Fig. 1. c(III) . 10^4 (mol l^{-1}): *1* 10; *2* 8; *3* 6; *4* 4; *5* 2; *6* 1; *7* 0·8; *8* 0·6; *9* 0·4; *10* 0·2; *11* base electrolyte. The dashed line corresponds to the baseline from which the wave height was measured

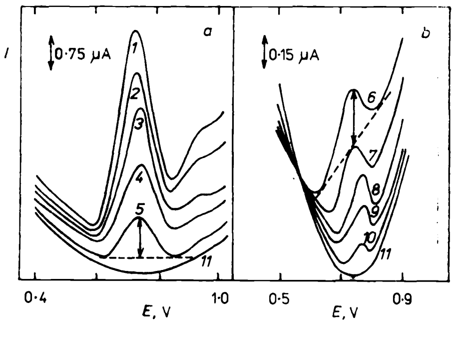


FIG. 8

DP voltammograms of N,N-dimethylaniline (*III*) at an SDE. Medium and conditions as in Fig. 2. c(*III*) . 10^4 (mol l^{-1}): *1* 10; *2* 8; *3* 6; *4* 4; *5* 2; *6* 1; *7* 0·8; *8* 0·6; *9* 0·4; *10* 0·2; *11* base electrolyte. The dashed line corresponds to the baseline from which the peak height was measured from the cyclic voltammetry that this is an irreversible system. Under these conditions, the half-wave potential of N,N-dimethylaniline is about 150 mV more positive than that for aniline alone, as a result of the $+I$ effect of the two methyl groups, which increases the electron density on the nitrogen in the amino group, thus facilitating its electrochemical oxidation.

Direct Current and Differential Pulse Voltammetry of N,N-Dimethyl-p-phenylenediamine

Both the DCV and DPV of N,N-dimethyl-*p*-phenylenediamine yielded very elongated waves or wide and low peaks, corresponding to very irreversible processes. In addition, considerable electrode passivation by the products of the electrode reaction was observed, reflected in a considerable decrease in the height of the wave or peak in the second or third measurement after electrode cleaning. Consequently, it is preferable to employ an RDE at 2 500 rpm, where the products of the electrode reaction are apparently washed away from the electrode surface, decreasing its passivation. It can be seen from Fig. 9 that the oxidation occurs in two steps which are highly irreversible. This irreversibility makes it difficult to evaluate the wave height in DCV or low peaks in DPV, decreasing the sensitivity of the determination of N,N-dimethyl-*p*-phenylenediamine compared to aniline or its N,N-dimethyl derivative. Both DCV and DPV yielded linear calibration curves in the concentration range $1 . 10^{-3}$ to $2 . 10^{-4}$ mol l^{-1}. The wave height in DC voltammetry was measured at a potential of +1·2 V as the distance from the baseline formed by elongating the linear part of the voltammogram prior to the beginning of the first wave of N,N-dimethyl-*p*-phenylenediamine. The height of the DPV peak was measured from the line connecting the minima prior to the first peak and after the second peak (see Fig. 9). The height of the first peak was measured as being more reproducible than that of the second peak, which was affected far more by the above-described electrode passivation. In the concentration range from $1 . 10^{-4}$ to $2 . 10^{-5}$ mol l^{-1}, the splitting of the DC wave is practically indistinguishable and DPV also yields only

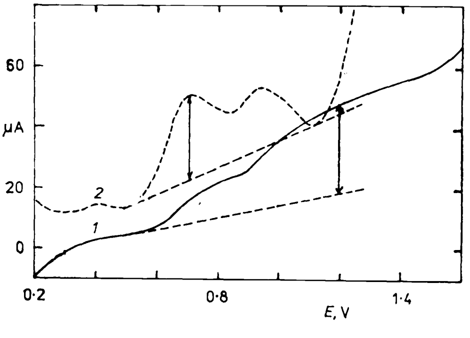


FIG. 9

DC (1) and DP (2) voltammograms of N,N-dimethyl-*p*-phenylenediamine (*IV*) ($c = 1 . 10^{-3}$ mol l^{-1}) at an RDE (2 500 rpm). Medium and conditions as in Figs 1 and 2. The dashed line corresponds to the baseline from which the wave or peak height was measured a single peak. Good quality, reproducible results can be obtained in this concentration range only by carefully cleaning the electrode prior to each measurement, followed by repeated recording of the voltammogram of the base electrolyte alone. The parameters of the calibration lines and the calculated determination limit are again given in Table I.

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