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Research Article

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Electrochemical analysis of reactive orange 16 on glassy carbon electrode

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Abstract

Cyclic voltammetric investigations of reactive orange 16 (RO 16) were performed in aqueous solution using a glassy carbon electrode (GCE). The impact of pH on the electrochemical behavior of reactive orange 16 was studied from pH 1.0 to 13.0 at a scanning rate of 50 mV/s. The highest peak current response was observed at pH 4.0, while others were somewhat lower. Three anodic peaks in the forward scan and two cathodic peaks in the reverse scan were detected at all pHs. The dye solution of RO 16 displays oxidation and reduction peak potentials at approximately -195, 030, 652 mV and -225, -6800 mV, respectively, relative to Ag/AgCl in the voltammogram. A thorough investigation of the experimental conditions influencing the anodic differential pulse stripping voltammetric response was performed. Maximum peak current conditions were achieved. A calibration curve was created under conditions of maximum peak current with varying concentrations of RO 16. The concentration range examined for the measurement is from 300 to 700 ppb. The GCE has a detection lower limit of 200 ppb and an RSD of 3.2%. The appropriateness of this approach for quantifying dye in effluents from the textile industry was also confirmed.

Keywords: CV-Cyclic Voltammetry, RO-12- Reactive Orange 12, SV- Stripping voltammetry, DPV-Differential Pulse Voltametry, GCE- Glassy Carbon Electrode.

Introduction

The textile production sector ranks among the most polluting industries regarding discharge volume and the composition of wastewater.[1] Reactive dyes are commonly utilized in textile manufacturing because their molecular structure contains reactive groups that form covalent bonds with fibers. The primary challenge with reactive dyes is their weak attraction to fabrics, leading to comparatively elevated dye levels in textile wastewater. Reactive dyes display a diverse array of chemical structures, largely centered on substituted aromatic and heterocyclic groups. Due to the high solubility of reactive dyes in water, they are challenging to eliminate from wastewater using traditional treatment methods [2-5]. Our initial study revealed a noticeable variation in the morphology of the graphite anode electrode before and after the electrochemical procedure [6]. In the anode reaction of the electrochemical oxidation process, substrate oxidation entails transferring one or more electrons from the substrate to the anode direct electron transfer (DET). Conversely, substrate oxidation seldom happens without the concurrent oxidation of water, particularly under amperostatic conditions. This suggests that the assault on the substrate begins with reactive intermediates from water oxidation, including hydroxyl radicals, hydrogen peroxide, hydroperoxy radicals, and their fundamental conjugates, which can be either free in solution or attached to the anode surface [7].
Ceria-based composites have been previously created as effective electrolytes for the optimal performance of solid oxide fuel cells, which demand high-performance electrolyte materials capable of delivering excellent ion conductivity for adequate current generation. These composites exhibit a combination of proton and oxygen ion conductivity. We advanced additional composite electrolyte materials incorporating a catalyst like rare earth elements; gadolinium (Gd^{3+}), neodymium (Nd^{3+}), and samarium (Sm^{3+}) doped GCE to explore the electrochemical treatment of C.I Reactive orange 107.

The release of dyes poses a global environmental issue. These dyes are extensively used in various industries such as dyestuff, rubber, paper, leather, textiles, plastics, and cosmetics [8-11]. Reactive azo dyes display high water solubility and are distinct from other dye categories due to their ability to bond with textile fibers such as cotton, wool, and silk, creating covalent bonds. The impact of pH on the adsorption of reactive orange 16 dye using rice husk was examined at room temperature (25 ºC). pH is a crucial factor influencing the dye's adsorption by adsorbent materials, altering both the surface charge of adsorbents and the ionization extent of various adsorbates. Large quantities of synthetic dyes are utilized in various

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sectors, including dyestuffs, leather, tanning, and textiles. In the textile sector, it was estimated that around 10%–15% of all dyes used were wasted during dyeing procedures and discharged into effluent [12-14]. Over 60% of dyes produced globally are azo dyes, which are resistant, non-biodegradable, carcinogenic, mutagenic, and toxic. Wastewaters containing azo dyes need proper treatment to avert potential damage to aquatic organisms, as they can include various pollutants like dyestuff with diverse structures and textile auxiliaries such as surfactants and polyvinyl alcohol used in sizing operations [15-17]. The ferrate (VI) method achieved color removal efficiencies of 85%, while the sono-ferrate (VI) method reached 91%. Kinetic analyses were additionally conducted for the decolorization of RO 16 under optimized conditions at room temperature [18].

pH significantly influences the absorption process and affects absorption capacity, dye solubility and the surface of the absorbent pore. The influence of pH on adsorption capacity was examined from pH 2.0 to 12 at 30°C, with a trembling speed maintained at 150 rpm. The percentage removal of RO16 from 95% to 98.12% as the pH increased from 2 to 8 [19-21]. The research aimed to create electro-analytical methods utilizing specific voltammetric techniques for the precise detection of RO16 in water using a glassy carbon electrode. The direct redox activity of reactive orange 16 on GCE was studied, leading to an examination of its determination using CV and DPV process, with methods tested, developed, and optimized for the selective detection of reactive orange 16.

Methods of Experimentation

All reagents were of analytical grade acquired from commercial sources. The stock solution of reactive orange 16 was created by dissolving the compounds in double distilled water purified through the SG purification system. The pHs solutions of the supporting electrolyte were also made using the same water. The pH levels of the buffer solutions were assessed using a Hanna HI 2211 pH/ORP meter.

The electrochemical investigations were performed in both exploratory and determination modes using software linked to a CH Instruments Electrochemical Workstation (model CH 650C). The cell was constructed from glass, with a capacity of 15 mL, and the top made of Teflon featured three distinct holes for electrode insertion: working electrode (Glassy Carbon), counter electrode (platinum foil), and reference electrode (Saturated calomel). The cell configuration is maintained within a CH Instrument Pico amp Booster and Faraday cage. The cell top includes nitrogen gas purging and eliminate oxygen gas.This arrangement allows for an inert atmosphere to be preserved above the sample solution during the entire experiment.

The GCE's surface is likely to be polluted by surface-active agents and various other substances. Consequently, the effective surface area and various attributes might be altered. Therefore, it is essential to preprocess the electrode prior to usage, if reliable reproducibility is needed. Established and recognized methods for treating glassy carbon electrodes were utilized. Initially, the electrode was rinsed with a mixture of water, ethanol, and ammonia, then with ethanol-ammonia, followed by ethanol-acetic acid, and a blend of ethyl acetate and ethanol. It was subsequently rinsed with distilled water and trichloroethylene. Using this technique, oxides, surfactants, and organic compounds were eliminated from the electrode surface. When the electrode surface is heavily contaminated, the most efficient and simplest method used to renew the surface was to eliminate a layer by rubbing it with 0.05 µM fine powder of Alumina. Following the rubbing, the electrode was cleaned with filter paper and subsequently washed with water. The fine particles of Al_2O_3 on the GCE were eliminated through ultrasonication with water. The electrode was thoroughly cleaned of grease using trichoroethylene and rinsed with deionized water and acetone. The surface of the GCE was effectively prepared for the detection of small quantities of dye. The dye attached to GCE is currently quite useful for the detection of ultra trace substances.

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Results and Discussion

Cyclic Voltammetric Behaviour of Reactive orange 16

CV of RO 16 on GCE in acidic, neutral, and basic conditions at various scan rates ranging from 25 to 500 mV/s was obtained. A notable reaction occurred when the experiments were conducted within the pH range of 1.0 to 13.0. The current was measured for each scan rate and removed in the relevant potential range. The maximum potential Ep (in mV) and maximum current ip (in mA) for different concentrations and scan rates were recorded. The data has been gathered and analyzed.

Influence of pH

Due to a notable effect at pH 1.0, the CV investigations of RO 16 were performed within the pH interval of 1.0 to 13.0. At every pH, 3 anodic peaks and 2 cathodic peaks (Fig.2) were noted during the reverse scan. Among the 3 peaks, anodic peak III demonstrated significance with an increased current in the pH range of 1.0 to 13.0. The anodic peak III and cathodic peak became less distinct as the pH was raised. Therefore, anodic peak III was selected for additional investigations to examine the oxidation mechanism. To examine the effect of pH on the cyclic voltammetric response, pH 4 was selected, and cyclic voltammograms were obtained for 300 ppm RO 16 at a scan rate of 100 mV/s. The peak currents and peak potentials were graphed as functions of pH, and the resulting plots are shown in figure 1. The highest peak current response was observed at pH 4.0(Fig.1). This is because of quicker electron transfer at an acid pH of 4.0, suggesting that the electrochemical oxidation of RO 16 is enhanced in acidic environments. Therefore, it can be regarded as the ideal pH for examining RO 16.

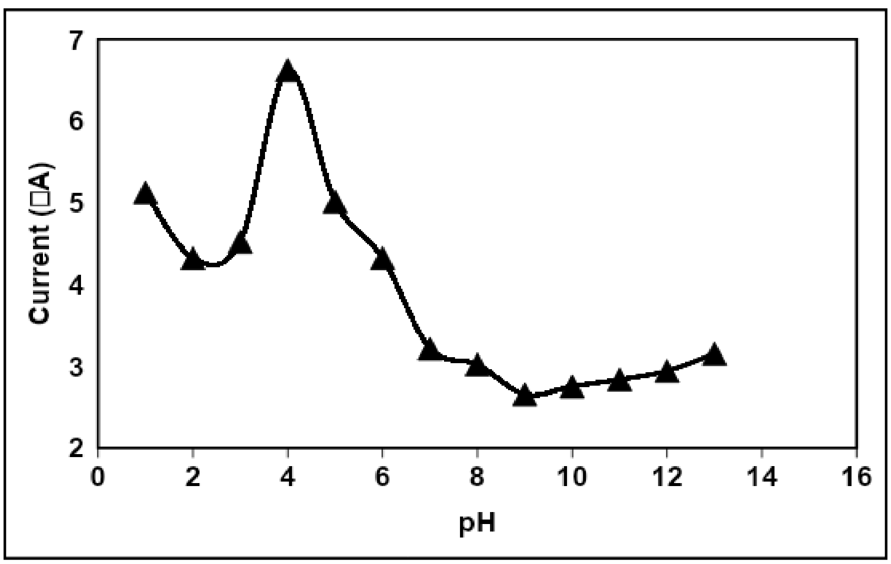

Fig 1.Curve of peak current vs Ph

Effect of scan rate and concentration

The scan rate was altered between 25 and 500 mV/s in the chosen pH medium, and the cyclic voltammetric behavior of 300 ppm RO 16 was analyzed. Peak current was associated with scan rate and the square root of scan rate. A plot of log current against log scan rate was also created. At a fixed scan rate of 100 mV/s, the substrate concentration was altered, and its impact on peak current was examined. A plot of current against the concentration of RO 16 was also created.

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A solution with a pH of 4.0 was selected as the medium for the research. Fig. 1 displays the CV of RO 16 obtained at pH 4.0. 3 anodic peaks were detected at potentials near -195, 030, and 652 mV. In the reverse scan, 2 cathodic peaks were noted at approximately -225 and -6800 mV. The impact of scan rate was examined for the anodic peak I at scan rates ranging from 25 to 500 mV/s. The peak current was graphed versus scan rate, producing a somewhat curved line, while the peak current increased linearly with the square root of the scan rate, yielding a straight line with a strong correlation (R^2 = 0.9902). The plot of log ip against log ν produced a linear graph with a slope of 0.1002. These facts indicated that the electrode reaction is governed by diffusion. The voltage difference between the anodic peak I and the cathodic peak was determined to be approximately 100 mV. Thus, the oxidation of RO 16 at pH 4.0 can be regarded as reversible. The influence of concentration on peak current at a fixed scan rate of 100 mV/s was investigated by varying the concentration from 300 to 700 ppm. An increase in the concentration of RO 16 resulted in a rise in both the peak current and the peak potential. The graph of ip versus concentration produced a straight line. Thus, it can be inferred that at pH 4.0, irreversible diffusion-controlled oxidation and reduction processes occur alongside additional oxidation at elevated anodic potential.

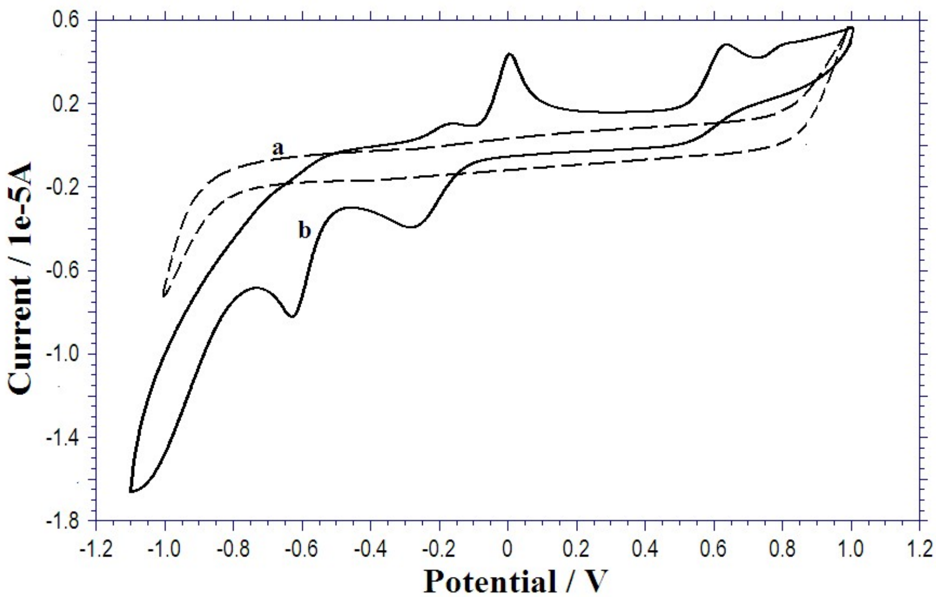

Fig: 2 CV behavior of 300 ppm concentration of RO 16 on GCE in pH 4.0 and at 50 mV/s

Chronocoulometry

The diffusion coefficient is essential for various electrochemical investigations, as the current response at an electrode relies on the speed of mass transport of the electroactive species to its surface. The compound's diffusion coefficient was determined from the slope of Anson's plot, assuming other parameters are known. The geometric area of the utilized GCE is 0.0314 cm². A graph of total charge transmitted, Q versus the square root of time, t½, converts the information into a linear correlation with a slope of $2nFACD^{½}\pi^{-½}$. The graph of Q versus t½ for a concentration of 1.6x10-7 M/cm3 of RO 16 is derived. By applying the forward slope value from the Cottrell equation, the diffusion coefficient 'D' for the compound is determined to be 2.261 x 10-5 $cm2sec^{-1}$ at pH 4.0.

Controlled potential coulometry

Coulometry with controlled potential was conducted at pH 4.0 using the identical cell configuration. The coulometric 'n' value was established following thorough electrolysis of 10 ml of $3.2x10^{-7}$ M dm^{-3} Reactive Orange 16 solution (pH 4.0), resulting in a value of 2.

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This shows the transfer of 2 e^- during the electro oxidation of Reactive Orange 16.

DPSV investigation

After selecting the optimal pH trials, experiments on GCE were conducted to fine-tune different parameters using the solution that contained 300 ppb RO16. The adsorptive stripping voltammetry of RO16 was performed on bare GCE at a positive potential, with the stripping peak potential being near the anodic oxidation potential noted in CV. This implies the adsorptive buildup and anodic stripping of the substrate. The elements influencing the electrode process were optimized and examined as outlined below. Typically, adsorptive stripping voltammetry consists of two primary stages. The analyte molecules were initially gathered through adsorption onto the electrode surface from the bulk, and subsequently, the gathered molecules were removed, producing specific signals. Thus, the optimization of accumulation potential was carried out in the initial phase by keeping other parameters as per default configuration. The impact of accumulation potential was examined by altering it between –800 mV and 800 mV. The highest peak current was recorded at 400 mV, which was established as the potential for maximum peak current accumulation. The dye's anion readily absorbed at a positive potential because of electrostatic attraction. The impact of deposition time on the stripping signal from 15 to 90 seconds was examined, with the peak current attaining its highest value at 60 seconds. The initial scan capability is another crucial factor since it verifies the non-faradic character of the preconcentration phase. It also regulates both the peak voltage and peak current in the stripping voltammogram. The effect of initial potential on the maximum current was examined by changing it from -1300 to -600 mV. An improved response was noted at –1000 mV. The pulse height ranged from 25 mV to 200 mV, with the highest value achieved at 150 mV. The influence of pulse width was examined from 25 msec to 200 msec, revealing that the highest peak current occurred at 75 msec. The impact of scan increment was examined by adjusting it between 2 mV and 20 mV. The peak current showed a linear relationship with the scan increment until it reached 16 mV. The highest peak current was recorded at the scan increment of 16 mV and was therefore selected as the optimal and preferred scan increment. Upon adjusting the aforementioned parameters, the peak current response concerning scan rate was also examined. The scan rate ranged from 20 mV/s to 120 mV/s. A well-defined stripping response featuring the highest peak current was achieved at a scan rate of 50 mV/s. Therefore, it was considered the ideal scan rate for analysis.

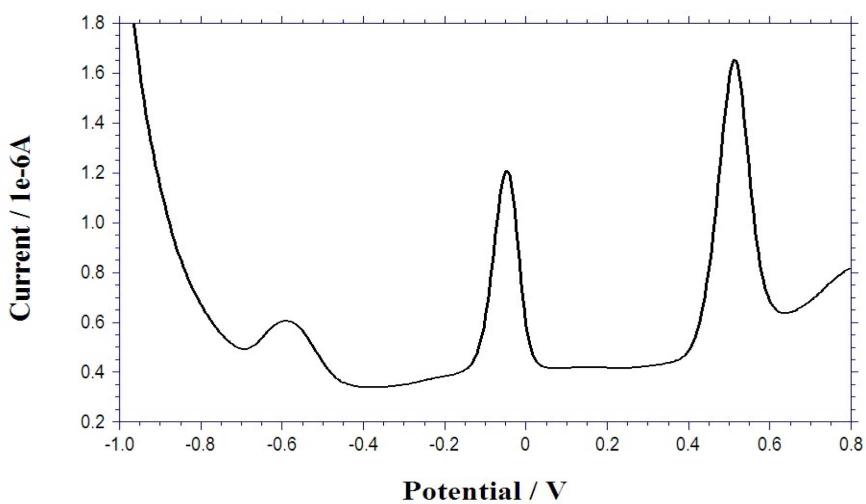

Figure 3 DPV responses of 300 ppb of RO 16 on GCE

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Table 1 Studied range and optimum experimental conditions in DPSV

Parameters	Range studied	Optimum value
Ph	1-13	4.0
Accumulation potential (mV)	-800 to 800	400
Accumulation time (Sec)	10-90	60
Initial scan potential (mV)	-1300 to -600	-1000
Pulse Height (PH) (mV)	25 to 200	150
Pulse width (PW) mSec	25 to 200	75
Scan Increment (SI) mV	2 to 20	16
Scan rate (SR) mV/sec	10 to 100	50
Stirring rate (rpm)	50 to 250	250
Rest period (Sec)	2 to 10	5

Analytical Studies

The relationship between peak current and concentration was examined using the aforementioned fixed optimal parameters. The experiments were conducted on a GCE. Experimental results indicated that the peak current rose with a rise in the concentration of Reactive Orange 16 on the electrode, and the calibration plot was generated as shown in figure 5. The reproducibility of the stripping signal was assessed using relative standard deviation for ten identical measurements performed at a concentration of 300 ppb, resulting in RSD values of 3.0%, while the lower detection limit is 100 ppb.

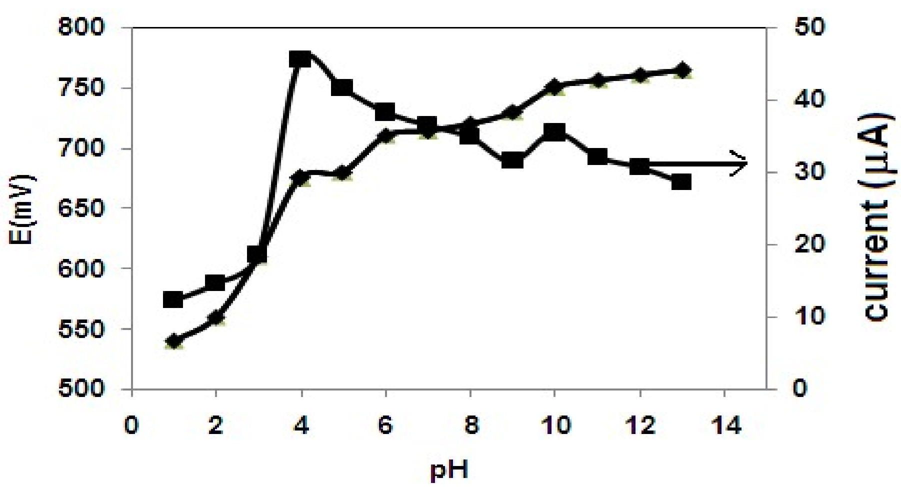

Figure 4 Effect of pH vs. peak current, potential curve on GCE

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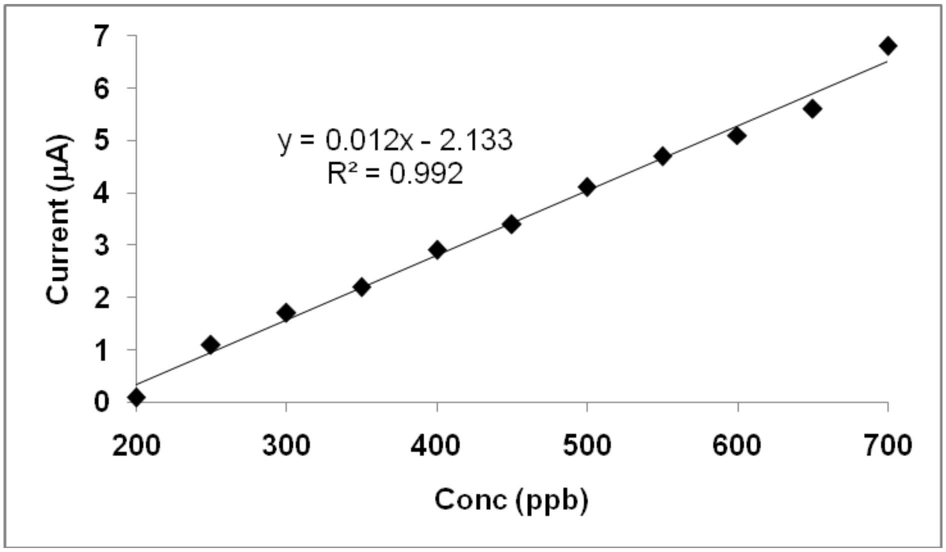

Figure 5 calibration plot of differential pulse stripping voltammetry

Conclusion

Initial exploration of RO 16 dye on a GCE. The electrochemical properties of RO16 were examined in various pH environments. All the pH values exhibit three oxidation peaks and 2 reduction peaks on GCE. Among the 3 pH mediums, the CV of RO16 demonstrates the best response at pH 4.0. The most effective medium utilized in the examination of enhanced electrochemical behavior. Among the 5 redox peaks, the 3 anodic oxidation exhibits a greater peak current and is utilized for additional correlations. The relationship of peak current with scan rate and the square root of scan rate, as well as the log of current against the log of scan rate, indicated that the electrode reaction is irreversible and controlled by diffusion. The Q versus t½ plot for a concentration of $1.6x10^{-7}$ M/cm3 of RO 16 yields a diffusion coefficient 'D of 2.261×10^{-5} cm^2sec^{-1}. The coulometric 'n' value was established after thorough electrolysis of 10 ml of $3.2x10^{-7}$ M dm^{-3} RO 16 solution (pH 4.0), yielding a result of 2. The oxidation of primary amine occurs through a mechanism involving 2 electron and proton reactions, resulting in the formation of hydroxyl amine. The relationship between peak current and concentration was examined using the established optimal parameters. The quantity of RO 16 was measured using DPV technique, with a minimum detection limit of 100 ppb.

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