UDC 541.138: 54.061/.062: 543.253: 54-39: 541.459
DOI https://doi.org/10.32689/2663-0672-2025-2-30

Olena MOZGOVA
PhD in Pharmacy, Associate Professor, Institute of Physical Chemistry, Polish Academy of Science, omozgova@ichf.edu.pl
ORCID: 0000-0003-0875-7035

Mykola BLAZHEYEVSKIY
DSc in Chemistry, Professor, Professor at the Department General Chemistry, National University of Pharmacy, blazejowski@ukr.net
ORCID: 0000-0002-8032-347X

Svitlana KARPOVA
PhD in Pharmacy, Associate Professor, Associate Professor at the Department General Chemistry, National University of Pharmacy, za9594506@gmail.com
ORCID: 0000-0001-7274-7750

Natalia BONDARENKO
PhD in Pharmacy, Associate Professor, Associate Professor at the Department General Chemistry, National University of Pharmacy, tropikana2003@ukr.net
ORCID: 0000-0003-0620-2255

Tetiana TOMAROVSKA
PhD in Chemistry, Associate Professor, Associate Professor at the Department Natural Sciences and Health Care, Municipal Establishment «Kharkiv Humanitarian-Pedagogical Academy» of the Kharkiv Regional Counsil, tomarovskaya63@gmail.com
ORCID: 0000-0003-0045-8209

VOLTAMMETRIC DETERMINATION OF HYDROGEN PEROXIDE CONTENT IN PERACETIC ACID DISINFECTANT

Aim of Work. *This study presents the development and validation of a voltammetric method for the quantitative determination of hydrogen peroxide (HP) in the peracetic acid-based disinfectant Delakson.*

Methodology. *The method employs cathodic voltammetry using a carbositall rotating electrode (CRA) as both the working and auxiliary electrode. Measurements were conducted in the potential range of +1.0 to –1.0 V versus a saturated Ag/AgCl/KCl reference electrode.*

Scientific Novelty. *The study offers a novel application of cathodic voltammetry with a carbositall rotating electrode for precise HP determination in a commercial disinfectant matrix. The method demonstrated high linearity, sensitivity, and precision, with low LOD (2.15×10^{-5} mol L^{-1}) and LOQ (7.18×10^{-5} mol L^{-1}) values, making it a robust analytical tool for real-world applications.*

Conclusions. *The proposed voltammetric method is reliable, sensitive, and precise for determining hydrogen peroxide in peracetic acid-based disinfectants. It demonstrated excellent reproducibility (RSD values of 0.028, 0.018, and 0.011) and accuracy (relative errors from –0.77% to +0.92%), with successful application to the Delakson formulation (RSD = 0.012, relative error = +1.69%).*

Key words: *hydrogen peroxide; voltammetry; carbositall electrode; disinfectant.*

Олена Мозгова, Микола Блажеєвський, Світлана Карпова, Наталія Бондаренко, Тетяна Томаровська.
ВОЛЬТАМПЕРОМЕТРИЧНЕ ВИЗНАЧЕННЯ ВМІСТУ ПЕРЕКИСУ ВОДНЮ В ДЕЗІНФІКУЮЧОМУ ЗАСОБІ НА ОСНОВІ ПЕРОЦТОВОЇ КИСЛОТИ

Мета роботи. *У цьому дослідженні представлено розробка та валідація вольтамперометричного методу для кількісного визначення пероксиду водню у дезінфікуючому засобі Делаксон на основі пероцтової кислоти.*

Методологія. *Метод використовує катодну вольтамперометрію з використанням обертального вуглеситалового електроду в якості робочого і допоміжного електрода. Вимірювання проводилися в діапазоні потенціалів від +1,0 до –1,0 В відносно еталонного Ag/AgCl/KCl насиченого електроду.*

Наукова новизна. *Дослідження пропонує нове застосування катодної вольтамперометрії з обертальним вуглеситаловим електродом для точного визначення пероксиду водню у складі комерційного дезінфікуючого засобу. Ме-*

тод продемонстрував високу лінійність, чутливість і точність, з низькими значеннями LOD ($2,15\times10^{-5}$ моль/л) і LOQ ($7,18\times10^{-5}$ моль/л), що робить його надійним аналітичним інструментом для практичного застосування.

Висновки. *Запропонований вольтамперометричний метод є надійним, чутливим і точним для визначення перекису водню в дезінфікуючих засобах на основі пероцтової кислоти. Він продемонстрував чудову відтворюваність (значення RSD 0,028, 0,018 та 0,011) та точність (відносні похибки від –0,77% до +0,92%), з успішним застосуванням до препарату Делаксон (RSD = 0,012, відносна похибка = +1,69%).*

Ключові слова: *пероксид водню; вольтамерометрія; вуглеситаловий електрод; дезінфікуючий засіб.*

Introduction. Hydrogen peroxide (HP, H_2O_2, CAS No. 7722-84-1) is a widely employed antimicrobial agent, commonly used in preservation, disinfection, and sterilization applications across healthcare, pharmaceutical, and industrial settings [3; 17; 22]. Its popularity stems from its potent and broad-spectrum antimicrobial activity, operational versatility, and comparatively favorable safety profile when contrasted with other microbiocidal agents [14]. HP is known to be effective against a wide range of pathogenic organisms, including structurally resistant dormant forms such as bacterial spores and protozoan cysts, as well as atypical infectious agents like prions, contingent on concentration and application conditions [1; 28].

In addition to its microbiocidal effectiveness, HP is valued for its low toxicity and environmentally benign degradation products, primarily water and oxygen, making it suitable for use in sensitive environments and on biological surfaces [2]. Aqueous HP is applied in various formulations, serving as a preservative in pharmaceuticals and cosmetics, a wound disinfectant, and a sanitizer for inanimate surfaces. Recent innovations have focused on enhancing its efficacy at lower concentrations through formulation with synergistic compounds and on the development of vaporized hydrogen peroxide (VHP) systems. These gaseous applications offer several advantages, including low-temperature operation, rapid sterilization, surface material compatibility, and reduced toxicological impact, positioning HP as a leading alternative to conventional chemical or thermal disinfection techniques.

The antimicrobial activity of HP is primarily driven by its oxidative capacity, which enables it to damage essential biomolecules such as proteins, nucleic acids, and lipids [17]. This non-specific mode of action results in irreversible structural and functional disruption of microbial cells, thereby reducing their viability or infectivity. Notably, the broad and nonspecific oxidative mechanism confers a low risk of microbial resistance development, in contrast to many antibiotics and targeted biocidal agents [20]. Although some variability in microbial susceptibility has been observed, particularly due to innate physiological differences, appropriate application strategies can effectively mitigate these challenges and maintain disinfection efficacy.

HP in combination with peracetic acid (PAA) is also used as an active ingredient in certain disinfectants such as "Delakson" («Delana», Kyiv, Ukraine), «Nukdez» («Inter Chemical group», Ukraine), "Dezynfektor", "Septacid", "Steridial W" (Impuls, Gdańsk, Poland), "Sterioks", "Sterisyl" (Baltiachemi, Estonia), etc. The HP and PAA mixture show bactericidal, tuberculocidal, virucidal, sporocidal and fungicidal properties [21; 23; 27] and intended for the final, flow-line and preventive object disinfection in health care institutions and nidus of intestinal and respiratory infections of bacterial and viral etiology, tuberculosis, dermatophytes and Sibirian plague, as well as for the sterilization of medical products (including rigid and flexible endoscopes) and suture material [4; 25].

Various methods have been used for the determination of HP [10]. The most widely used methods for analyzing solutions containing PAA and HP are the method of D'Ans and Frey and its modification of Greenspan and McKellar [13; 26]. It is obvious that the two-step titration method is not suitable for the continuous monitoring of HP because it is very time-consuming. As alternative techniques to the titration methods, electrochemical measurements [5; 7; 8], chromatographic methods [15; 24], and spectroscopic methods [18] have been used. Conductivity measurements are rapid and convenient, but their common disadvantage is their low selectivity. Spectroscopic methods have often been used for the direct determination of a few species in aqueous solutions. Near-infrared (NIR) spectroscopy has recently been of keen interest as a practical technique for a variety of water and aqueous solution analyses. So far, a UV spectroscopic method for the direct determination of HP has not been reported, probably because its UV absorption maximum is located at a very short wavelength (below 180 nm) and the extinction coefficients, obtained by an ordinary UV-visible spectrometer, are very low. But all these methods are not sufficiently sensitive and furthermore require conducting many chemical reactions, complicated cleaning and extraction procedures, and the use of cumbersome equipment, which may interfere with the whole procedure.

Nowadays selective amperometric and potentiometric methods, which allow to perform simultaneous HP and H_2O_2 determination at the compatible presence in water solutions, have been proposed [6; 9]. HP presence in PAA solutions is imminent according to several reasons: so, as the synthesis of PAA is carried out by HP reaction and acetate acid – that is why the HP is a constant technological admixture, and also, due to the course of hydrolysis reaction, in aqueous solutions there is a continuous unilaterally hydrolytic decomposition of PAA into HP and acetate acid.

Given its broad utility and increasing use in disinfectant formulations, reliable and accurate methods for the quantification of HP are essential, particularly in mixed oxidizing systems such as those containing peracetic acid (PAA). One such preparation is Delakson (manufactured by “Delana,” Kyiv, Ukraine), a complex granulated, water-soluble disinfectant. Its composition includes peracetic acid (5–15%), hydrogen peroxide (10–22%), acetic acid (22–26%), and various stabilizing agents. The working solutions are freshly prepared in water immediately prior to use and can be stored for up to five days in sealed containers at room temperature. However, for practical disinfection, the solution is typically used within a single day, necessitating routine monitoring of its active components, particularly HP, due to its oxidative degradation over time.

Currently, the mass fraction of HP in Delakson is commonly determined by redox titration methods [6]. Although titrimetric techniques are established and accessible, they may lack the sensitivity and selectivity required for complex mixtures with overlapping oxidants such as PAA. Previous studies have demonstrated the potential of carbositall electrodes in the electrochemical detection of various peroxide compounds [11; 12; 16].

The present study aims to evaluate the feasibility of using cathodic voltammetry with a carbositall rotating electrode (CRE) as the working electrode for the quantitative determination of HP in the Delakson disinfectant formulation. This approach offers the potential for greater specificity, sensitivity, and operational convenience in the routine analysis of complex oxidizing disinfectants.

Materials and Methods. *Reagents and Solutions.* A stock solution of HP was prepared using a commercially available reagent. The solution was diluted and standardized according to established procedures [19]. Specifically, 10.00 mL of the standardized HP solution was transferred into a 100 mL volumetric flask and diluted to the mark with double-distilled water, yielding a final concentration of 4.70×10^{-3} mol L^{-1}.

The background electrolyte consisted of a mixed solution comprising an acetate buffer (pH 3.6) and sodium sulfate (Na_2SO_4) at a concentration of 0.1 mol L^{-1}. The acetate buffer was prepared by mixing 425 mL of 1.00 mol L^1 acetic acid solution with 50.0 mL of 1.00 mol L^1 sodium hydroxide solution in a 500 mL volumetric flask, then diluting to volume with double-distilled water. A concentrated acetic acid solution (17.5 mol L^{-1}) was prepared by diluting 28.6 mL of glacial acetic acid in a 500 mL volumetric flask with double-distilled water. The 1.00 mol L^{-1} sodium sulfate solution was obtained by dissolving 142.0 g of Na_2SO_4 in a 1000 mL volumetric flask with double-distilled water.

The test sample analyzed for HP content was Delakson disinfectant (produced by “Delana”, Kyiv, Ukraine). A working solution of Delakson, containing approximately 0.1% peracetic acid and 0.9% hydrogen peroxide, was prepared by dissolving 1.0 g of the powdered disinfectant in a 1000 mL volumetric flask with double-distilled water, following the standardization procedure described in [19].

pH Measurement

pH values were measured using an I-160M ion meter equipped with a glass electrode (type ESL-43-07) in combination with a saturated Ag/AgCl/KCl reference electrode.

Electrochemical Measurements

All voltammetric experiments were conducted using an AVS-1.1 electrochemical analyser (Volta) operated in a three-electrode configuration. A carbositall rotating electrode served as both the working and auxiliary electrode, while a saturated Ag/AgCl/KCl electrode (type EVL-1M4) was employed as the reference. Measurements were performed in square-wave mode with a modulation amplitude of 40 mV, a frequency of 65 Hz, and a scan rate corresponding to a rotation speed of 1000 rpm. The potential was scanned in the range of +1.0 V to –1.0 V. Peak potentials were recorded using the “Module EM-04” electrochemical sensor with an accuracy of ±5 mV.

Calibration Procedure

Calibration curves were constructed using working solutions prepared by diluting 1.00–4.00 mL of the HP stock solution, along with 5.00 mL of 1.00 mol L^{-1} Na_2SO_4, in a 50 mL volumetric flask filled to volume with acetate buffer. A 25.0 mL aliquot of each working solution was transferred to the voltammetric cell. Voltammograms were recorded by scanning the potential negatively from +1.0 V to –1.0 V (vs Ag/AgCl/KCl sat). The peak current (I_p, in μA) at –0.65 V was plotted against the corresponding HP concentration (mol L^{-1}) to construct the calibration curve (see Fig. 2). The linear regression equation was derived using the least squares method.

Quantitative Determination of Hydrogen Peroxide in Delakson

For analytical determination, test solutions were prepared by diluting 10.00–15.00 mL of the Delakson solution with 5.00 mL of 1.00 mol L^{-1} Na_2SO_4 in a 50 mL volumetric flask, filled to volume with acetate buffer (pH 3.6). The electrochemical measurements were performed under the same conditions as for the calibration solutions. The concentration of hydrogen peroxide in the test solution, denoted as C_x, was calculated based on the calibration graph using the measured peak current values:

$$C_x = \frac{I_p - b}{a},$$

where I_p represents the peak current (μA) of the working solution, and a and b are the slope and intercept coefficients of the linear regression equation, respectively.

The mass fraction of hydrogen peroxide (%) in the Delakson sample was subsequently calculated using the following expression:

$$X,\% = \frac{C_x \cdot 34.01 \cdot 100 \cdot V_0}{m \cdot 1000 \cdot 10 \cdot V} \cdot 100\%,$$

where C_x is the determined hydrogen peroxide concentration (mol L^{-1}), 34.01 is the molar mass of hydrogen peroxide (g mol^{-1}), V_0 is the volume of the final volumetric flask (mL), V is the aliquot volume of the test solution used for analysis (mL), m is the mass of the disinfectant sample used (g), 10 is the volume (mL) of the stock solution used in dilution, 100 and 100 are the volumetric flask capacities (mL) used during successive dilution steps

Results and discussion. The feasibility of using cathodic voltammetry with a CRE for the quantitative determination of HP in multicomponent disinfectant formulations was thoroughly evaluated. The electrochemical behavior of HP was first investigated across a range of pH values to optimize the supporting electrolyte conditions. Voltammograms recorded for a 9.36×10^{-5} mol L^{-1} HP solution revealed a marked dependence of the reduction peak on the pH of the medium. As the pH increased from 2.15 to 4.78, the peak current (I_p) decreased, and the peak potential (E_p) shifted toward more negative values. Optimal signal intensity and resolution were observed within the pH range of 2.5-3.7, with pH 3.6 selected as the optimum due to its balance between signal strength and stability. Beyond pH 4.5, the analytical signal diminished significantly, indicating reduced electrochemical activity of HP under those conditions.

Figure 1a illustrates the influence of pH on the current peak, while Figure 1b shows the corresponding shift in peak potential. These observations align with the expected proton-coupled electron transfer mechanism of HP, which becomes less favorable at higher pH due to the decreased proton availability in the reaction medium.

A calibration curve was constructed using model solutions of HP with concentrations ranging from $(0.94–3.76)\times10^{-4}$ mol L^{-1}. The resulting linear regression equation was:

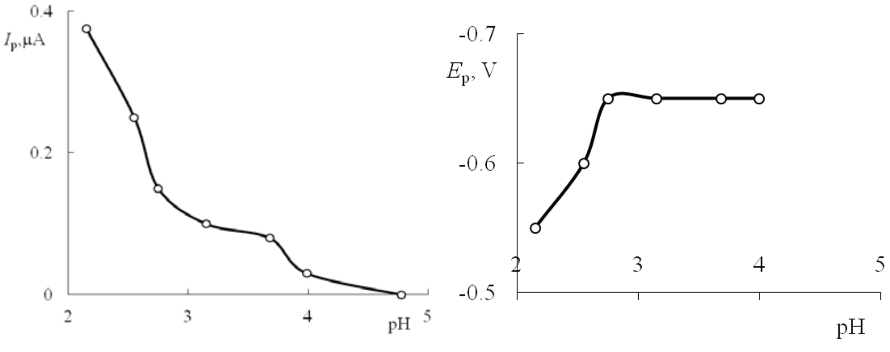
$I_p = (3.57\pm0.26)\times10^3\cdot C + (0.11\pm0.07)$, r = 0.998

where I_p is the peak current (μA) and C is the HP concentration (mol L^{-1}). This strong linear relationship is depicted in (Fig. 2) and further supported by the analytical parameters summarized in Table 1. The limit of detection (LOD) and limit of quantification (LOQ) were determined as 2.15×10^{-5} mol L^{-1} and 7.18×10^{-5} mol L^{-1}, respectively, reflecting the method's high sensitivity.

The method exhibited excellent reproducibility and accuracy. For model solutions, the relative standard deviation (RSD) ranged from 0.011 to 0.028, with relative error (δ) values between –0.77% and +0.92%, as shown in (Tab. 2). For the analysis of Delakson disinfectant, the voltammetric procedure demonstrated a mean recovery of 101.69%, an RSD of 0.019, and a relative error of +1.69% compared to the reference titrimetric method (Tab. 3). These results affirm the method's precision and accuracy in both pure and complex matrices.

The electrochemical method benefits from minimal interference from accompanying substances in the disinfectant formulation, such as peracetic acid and acetic acid, likely due to their distinct redox potentials under the chosen conditions. Furthermore, the use of a chemically inert and mechanically stable carbositall electrode minimizes electrode fouling and contributes to the long-term reproducibility of the measurements.

Conclusions. A novel voltammetric method was developed and validated for the quantitative



a **b**

Fig. 1. The influence of pH on the current peak intensity (*a*) and on the peak potential (*b*) of the HP reduction process at the CRE (vs Ag,AgCl/KCl(sat))

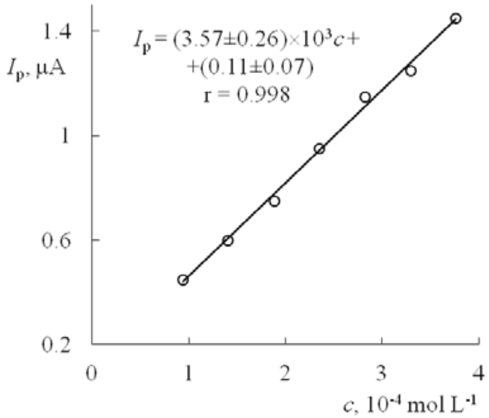


Fig. 2. The calibration graph of the HP reduction current peak vs concentration; $E_p = -0.65$ V

Table 1

Analytical characteristics of the calibration graph of HP voltammetric determination procedure in model solutions ($y = ax+b$)

Parameters	Data
Concentration ranges (mol L^{-1})	$(0.94\text{-}3.76)\times10^{-4}$
Regression equation	$I_p = (3.57\pm0.26)\times10^3 c + (0.11\pm0.07)$
Correlation coefficient (r)	0.998
a	3.57×10^3
b	0.11
S_a	0.26×10^3
S_b	0.07
Δa	0.10×10^3
Δb	0.025
LOD (mol L^{-1})	2.15×10^{-5}
LOQ (mol L^{-1})	7.18×10^{-5}

Table 2

Evaluation of accuracy and precision of H_2O_2 voltammetric determination procedure in HP model solution ($n = 5$; P = 0.95%)

Taken (mol L^{-1})	Found (mol L^{-1})	Reproducibility (%±SD)	RSD	ε (%)	δ* (%)
1.88×10^{-4}	$(1.87\pm0.06)\times10^{-4}$	99.23±3.42	0.28	3.45	-0.77
2.35×10^{-4}	$(2.34\pm0.05)\times10^{-4}$	99.53±2.34	1.89	2.35	-0.47
2.82×10^{-4}	$(2.85\pm0.04)\times10^{-4}$	100.92±1.41	1.12	1.40	+0.92

** Concerning the average reference method [16]*

Table 3

The results of HP voltammetric determination in "Delakson" disinfectant ($n = 5$; $P = 0.95\%$)

Taken (mol L^{-1})	Found (mol L^{-1})	Reproducibility (%±SD)	RSD	ε (%)	δ* (%)
0.900	0.915±0.013	101.69±1.50	0.019	1.47	+1.69

** The calculation has been made according to the average content established through the standard procedure* determination of HP in the peracetic acid–based disinfectant “Delakson.” The method employs cathodic voltammetry using a carbositall rotating electrode as the working and auxiliary electrode. The optimal background electrolyte was found to be an acetate buffer solution at pH 3.6, combined with sodium sulfate, ensuring high signal stability and analytical sensitivity.

The developed approach demonstrated excellent linearity in the HP concentration range of (0.94–3.76) $\times 10^{-4}$ mol·L^{-1}, with a correlation coefficient of 0.998. The method exhibits high precision and accuracy, with relative standard deviations (RSDs) not exceeding 2.8% for model solutions and 1.9% for real samples. The detection and quantification limits were 2.15×10^{-5} mol·L^{-1} and 7.18×10^{-5} mol·L^{-1}, respectively.

The results confirm the suitability of this voltammetric procedure for routine control of HP content in "Delakson" and similar disinfectants, offering a reliable, sensitive, and efficient alternative to traditional titration methods.

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Дата надходження статті: 28.07.2025
Дата прийняття статті: 20.08.2025
Опубліковано: 14.11.2025