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## Electrochemical method of measuring the antioxidant activity of compounds of phenol nature.

### Cover Page Footnote

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### Erratum

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## ELECTROCHEMICAL METHOD OF MEASURING THE ANTIOXIDANT ACTIVITY OF COMPOUNDS OF PHENOL NATURE

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**Abstract:** The article describes one of the types of electrochemical methods for determining the antioxidant activity of compounds of phenolic nature - the method of voltammetry, which is one of the most common today. Experimental installations for the determination of antioxidant activity using voltammetry modified with various kinds of electrodes are given; in addition, the features of the chosen technique are given.

**Keywords:** antioxidant, antioxidant activity, electrochemical analysis methods Voltammetry, electrode, electrochemical cell

In recent years, various methods have been proposed for the determination of antioxidants (AO) and antioxidant activity (AOA) of materials, substances and products of natural origin; synthesized new reagents, model systems and instrumentation; published many literary and analytical reviews in this subject area [1-3].

Among the multitude of methods, electrochemical methods of analysis (EMA), based on the study of processes occurring at the electrode surface or in the near-electrode space, have become particularly popular. The analytical signal is an electrical parameter that is functionally related to the concentration of the

detected component of the solution and is amenable to correct measurement [3-7].

Considerable attention is paid to electrochemical cells and sensors of the analytical signal, since it is these primary electrochemical converters that determine the analytical capabilities of any method. EMAs are divided into methods in which the excited electric signal is constant or equal to zero and to methods in which the excited signal changes in time. These methods are classified as follows: voltamperometric ( $I \neq 0$ ;  $E = f(t)$ ); potentiometric ( $I = 0$ ); amperometric; chronopotentiometric,  $E - f(t)$ ;  $I = \text{const}$ ; impedance, or conductometric - measurements that use the application of alternating voltage of small amplitude; others combined (for example, spectroelectrochemical). The most common method today is the voltameter method (VA).

There are various schemes for determining the AOA. For example, in [1], studies were carried out using VA with chemically modified electrodes (CHLOE) with a catalytic response. The experimental setup is shown in fig. 1 [3].

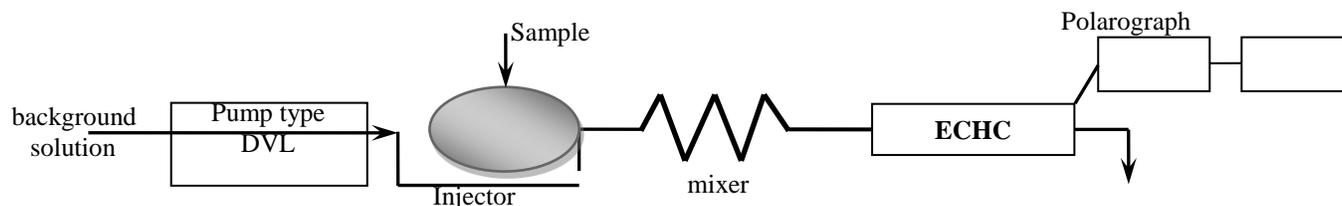


Fig. 1. The scheme of the experimental setup for determining the AOA using voltammetry modified by electrodes: ECHC - Electrochemical Cell.

The objects of study were amino acids (glycine, cysteine, cystine, carbohydrates (sorbitol, mannitol, glucose and sucrose), vitamins of group B (thiamine, riboflavin, pyridoxine) and medicinal compounds (insulin and gentamine)

The study of the AOA of the studied biologically active substances (BAS) was carried out on electrodes modified with hexacyanometal. The disadvantage of this scheme is that it is possible to study only these types of connections. In the study of an extensive class of phenol derivatives (flavonoids), water- and fat-soluble vitamins, as well as a number of enzymes, this scheme cannot be used due to a number of specific properties of these compounds.

Also one of the current, but still underdeveloped directions of AA is the creation of sensors that allow you to quickly and selectively determine organic substances. The new generation of these sensors is based on chemically modified electrodes.

So, in work [5] such a technique was considered. Particular attention is paid to coal-paste electrodes as an indicator analyzer. The component from the aqueous (organic) solution is concentrated on the electrode, and the magnitude of the electrochemical response depends not only on the concentration of the analyte in the reactor, but also on its specific interaction with the modifier. The aim of the work was to study the processes of selected concentration and voltammetric determination of phenol and its derivatives on carbon-paste electrodes (EPE) modified with silica gels, polymeric and diatolite sorbents, determining the factors affecting the process of selective concentration and VA of determining these compounds with the joint presence.

Chlorinated phenols, ortho-isomers of nitrophenol and aminophenol are sorbed on IPE modified with silica gel grafted with alkyl and nitrile groups and polymeric sorbents, and phenol, para-isomer of nitrophenol and aminophenol are adsorbed on IPE modified by silica gel and diatolite.

The disadvantage of this method is that antioxidant activity can only be determined for aqueous solutions, which makes it impossible to use it in the study of the food and pharmaceutical industry, where there is no possibility of obtaining an aqueous solution.

As an optimal setup in this work, we proposed to conduct experimental studies on the PU-1 serial polarograph and the TA-2 computerized voltammetric analyzer [6].

Polarograph PU-1 has a constant-current, alternating-current with a sinusoidal shape and a rectangular form of polarizing voltage, differential-pulse types of polarography. It can work in simple and differential modes of polarography. In the mode of alternating current polarography with a sinusoidal polarizing voltage, the polarograph PU-1 provides measurement of the active and capacitive components of the current. The set of polarograph includes: built-in block of time intervals; discrete set of speeds, providing a change in the potential sweep speed in the range of 0.5 h - 100 mV / s; electronic voltmeter with a range of polarizing voltages of 0 - 4 V and an accuracy of 0.5 mV. The voltammogram is recorded on a laboratory compensation two-coordinate recorder such as LCD-4, included in the polarograph kit. The device allows you to work in two - and three-electrode modes.

TA-2 analyzer is intended for automated analysis by voltammetry of samples of food and agricultural products, beverages, ecological and biological objects, cosmetics, pharmaceuticals, high-purity materials, ores, minerals, etc. It has four forms of polarization voltage sweeps: constant-current, stepped, square-wave and differential-pulse. It can work in simple and differential modes. Analyzer features:

- simultaneous analysis of 3 different samples;
- mixing the solution by stabilizing the vibration of the indicator electrode;
- efficient way of washing the electrochemical cell;
- the possibility of processing the analyzed solutions with inert gas and ozone;

programmatic way to implement the analysis;  
reliable and convenient mounting of the electrodes, excluding the contact of the operator with the working surface of the electrode;

built-in UV lamp to eliminate the interfering influence and destruction of organic substances;

the ability to work in two - and three-electrode modes.

The analyzer works in conjunction with a personal computer. The most important technical

characteristics of the analyzer: sensitivity  $5 \times 10^{-11}$  A; reproducibility of analytical signals 1–5%; the duration of the simultaneous analysis of three prepared samples is 5–30 min.

For carrying out experimental investigations at PU-1, a conventional electrochemical cell with openings for the working and auxiliary electrodes, a reference electrode and a glass tube for supplying nitrogen gas can be used.

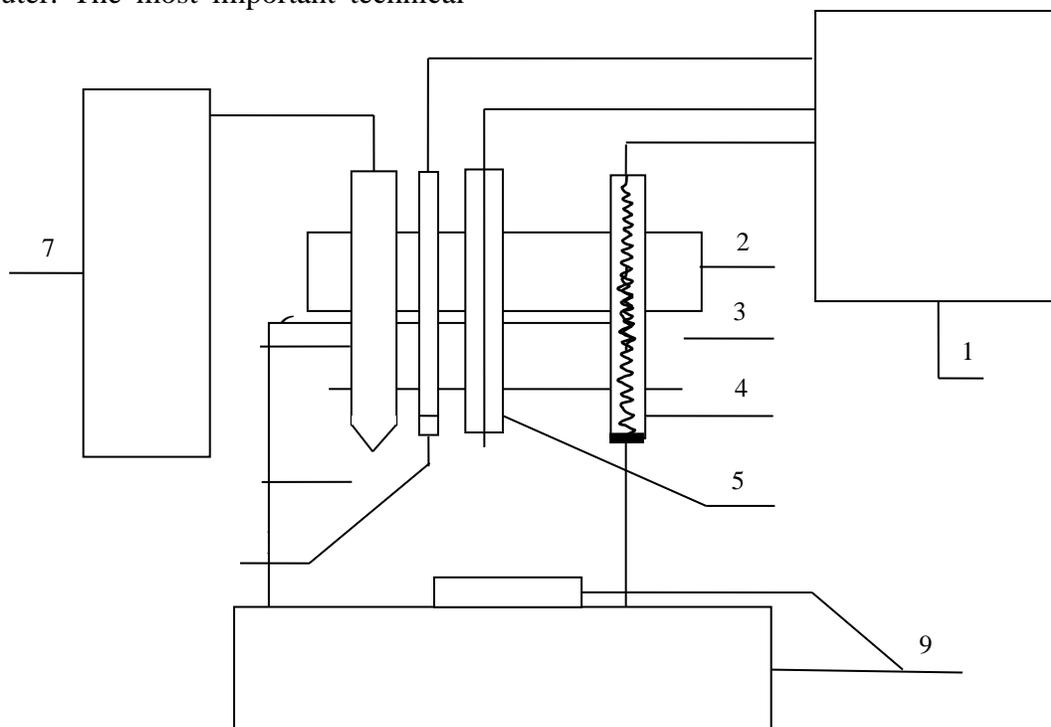


Fig. 2. Schematic representation of the experimental setup:

1. PU-1 or TA-2; 2. PTFE cover; 3. electrochemical cell; 4. HSE; 5. RPE or TME; 6. glass tube; 7. gas cylinder (nitrogen); 8. electrolyte; 9. magnetic stirrer; 10. TMN.

The surface of the electrode before work was polished on the filter and kept for 2 - 5 min in ethyl alcohol to remove surface-active substances (surfactants).

Chloride-silver electrode (HSE) was used as a reference electrode and auxiliary electrode.

The method of obtaining current-

For greater efficiency in the work can be used the following indicator electrodes:

1. Mercury film electrode (RPE) with silver contact. It was a pure silver wire soldered to a copper conductor. The area of the working surface of the electrode is  $S = 0.275$  cm. A thin film of mercury was applied to its working surface by

electrolysis from a saturated solution of nitrous acidic mercury  $Hg_2(NO_3)_2$  or mechanically by ordinary dipping.

2. Glass carbon electrode (CEM). It was a plate of glass graphite, 2 mm in diameter, sealed with a copper contact conductor into a glass tube. The area of the working surface of the electrode is  $S = 0.275$  cm<sup>2</sup>. voltage dependencies can be the same as that used in constant-current voltammetry.

A schematic depiction of an experimental setup on which research can be conducted is shown in Fig. 2.

The method of determining the antioxidant activity is as follows. An electrolyte solution was

placed in an electrochemical cell; for example, NaClO<sub>2</sub> dissolved in dimethylformamide or acetonitrile can be used for non-aqueous media. A three-electrode cell consisting of a working RPE, a reference HSE electrode and an auxiliary electrode is assembled. Electrodes are lowered into the background electrolyte solution and connected to the PU-1 polarograph or TA-2 analyzer. Use should be constant-current mode cathode voltammetry with a working range of potentials from 0 to -1V. Next, the first wave of cathode reduction of oxygen dissolved in the background solvent is removed. It is recommended to repeat the voltammogram at least five times. An addition of the prepared solution of the test compound is made and each time the voltammograms of the first wave of cathodic oxygen reduction are taken. According to the obtained results, a graph of the dependence of the relative current of the electroreduction of oxygen on the concentration of the substance in solution is plotted.

According to the tangent of the angle of inclination of the tangent to the rectilinear portion of this curve, the concentration ( $k_{end}$  (ml / g)) and kinetic ( $k_{kinet}$  ( $\mu\text{mol} / \text{l}\cdot\text{min}$ )) antioxidant activity criteria are calculated.

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