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## METHOD OF ELEMENTS IDENTIFICATION IN THE MULTICOMPONENT SIGNAL

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*To measure concentrations of toxic elements in analytical systems of ecological monitoring, a generative model of a multicomponent differential signal of stripping chronopotentiometry is used in the coordinates of potentials in time ( $e$ ,  $dt/de$ ) in the form of a model of the capacitive background discharge of electrodes (base curve) and the sum of the models of the stripping components of chemical elements, given by asymmetric normal distribution functions. Identification of elements with slight overlapping of component signals is carried out based on the maximum value and range in which potentials change.*

**Keywords:** *identification, generative model, multicomponent signal, normal distribution, base curve, least squares method.*

### Introduction

When measuring ultra-low concentrations of toxic elements during ecological monitoring of the environment very sensitive electrochemical methods of stripping chronopotentiometry (SCP) are often applied. For this method of analysis, the identification and determination of the stripping time of a chemical element in a multicomponent differential stripping signal (intensity signal) is essential, where time is an analytical characteristic of concentration.

Physically, the intensity signal is the sum of two chemical processes in time – the capacitive background discharge of the electrodes (base curve) and the sum of the stripping components (chemical elements), which may partially overlap. The applica-

tion of the intensity signal model, which takes into account all chemical processes, contributes to a more accurate and stable measurement of the concentrations of elements due to the identification and standardization of the stripping time determination, which will not depend on the human factors of determination of the ranges of each element stripping potentials by analytical chemist.

### Determination of Chemical Elements Concentrations by the SCP Method

According to the method of stripping chronopotentiometry, the process of determining the concentration consists in the electrochemical concentration of chemical elements ions on the measuring

electrode and their subsequent electrolysis in the stripping mode. The main analytical functions of the SCP method are the stripping time of the element, which under standardized conditions of concentration and measurement is directly proportional to the concentration of ions in the solution, as well as to the peaks and range of stripping potentials – a qualitative characteristic of the element [1–5].

The stripping signal is the integral sum of the stripping measurement of individual chemical elements. The process of each element stripping is performed sequentially, in its own range of potentials, according to the increasing value of the standard electrode potential in the activity range of metals. The actual values of peak potentials and element stripping ranges depend on the concentration, chemical composition of the background solution, and the material of the measuring electrode (mercury, gold, platinum) [6].

The concentration of elements is determined by the method of addition. First, a background solution or a blank sample is analyzed, then a sample solution is analyzed, prepared according to the method of measuring an environmental object, then an additive (one or more additives) of a standard solution of element ions is added to the sample [7–8].

During the digital analysis of the stripping signals  $e(t)$ , the following operations are successively performed: high-frequency filtering  $e(t)$ , transformation of the signal  $e(t)$  into  $i(e)$ , differentiation  $dt/de$ , smoothing  $dt/de$ . In the received intensity signals  $T(e) = dt(e)/de$ , which represent a multi-component sum, the chemical element is identified and its stripping time is determined. Based on the obtained background stripping time values, samples, additives the concentration of element ions in the object is determined [9].

Since the identification and concentration of chemical elements from the intensity signal largely depends on the chemist's ability to determine the peaks and ranges of the stripping potentials, let's consider the construction of the intensity signal model, which is performed quite quickly and can help automate the process of concentrations measuring.

## Generative Model of Multicomponent Signal

The analysis of possible methods of modeling multicomponent stripping signals showed that during the processing of real analytical signals it is desirable to present the structure of models in the form of simple, predefined dependencies, in which the determination of model parameters is reduced to the tasks of parametric identification.

Direct construction of a general mathematical model of the intensity signal based on the models of individual components and a base curve with many unknown parameters is a rather long-term and complex problem, so let's build a generative model of a multicomponent signal that will simplify this process.

It is assumed that the baseline and each independent model of the  $k$  component can be represented as a product of the height parameter  $H_k$  and the normalized model  $g_k(e)$ , with other parameters determined by the intensity signal  $T(e)$  unchanged. The base curve model is denoted as  $g_0(e)$ .

Under such assumptions, the generative model of the intensity signal for  $m$  components with a small mutual overlap of signals is as follows:

$$T(e) = \sum_{k=1}^m H_k \cdot g_k(e) + H_0 \cdot g_0(e) \quad (1)$$

The proposed model belongs to the class of one-dimensional regression models as a task of parametric identification, which is solved by determining the optimal values of the parameter vector  $H_k$  by the least squares method.

Let's consider the construction of the model of the base curve of the intensity signal in more detail.

## Base Curve Model

The optimal view of the functional dependence of the model of the base curve  $g_0(e)$  significantly depends on the chemical composition of the background solution and the material of the measuring electrode and is determined by a separate procedure before the start of operation of the concentration measurement system.

According to the performed studies, the base curve model  $g_0(e)$  for a silver measuring electrode

coated with mercury amalgam, the base curve model looks like this

$$g_0(e) = \left( \exp \sum_{k=9}^4 b_p \cdot e^p \right) \quad (2)$$

The unknown coefficients of the vector  $B$  are determined by the method of least squares from a set of selected points that characterize the construction of a piecewise linear base curve in logarithmic coordinates [9].

Consider the method of modeling individual components in the intensity signal.

## Models of Chemical Elements Components

The conducted experiments showed that it is legitimate to model the components of  $g_k(e)$  in the form of normal distribution functions, similar to modeling approaches in chromatography and mass spectrometry. The normal distribution function for half of the component has the form:

$$g_k(e) = H_k \cdot \varphi(e, e_0, \sigma) = H_k \cdot \exp \left[ -\frac{(e - e_0)^2}{\sigma^2} \right], \quad (3)$$

where  $e_0$  is the peak potential of the component signal,  $\sigma$  is the variance.

The model of the individual component  $g_k(e)$  in the form of an asymmetric Gaussian function with left  $\sigma_1$  and right  $\sigma_2$  variances and peak potential  $e_0$  has the form:

$$g_k(e) = \begin{cases} H_k \cdot \phi(e, e_0, \sigma_1), & e \leq e_0, \\ H_k \cdot \phi(e, e_0, \sigma_2), & e > e_0. \end{cases} \quad (4)$$

Thus, to build a component model, it is necessary to determine the values of four parameters:  $H_k$ ,  $e_0$ ,  $\sigma_1$ ,  $\sigma_2$ . The complexity of the problem is determined by the discreteness of the intensity signal with the potential step  $\Delta e$ . In practice, many approaches to its solution are used, let's consider the option proposed in [9].

Take logarithm of the function  $g_k(e)$

$$\ln(g_k(e)) = \left[ \ln(H_k) - \frac{e_0^2}{\sigma^2} \right] + \left[ \frac{e_0}{\sigma^2} \right] \cdot e + \left[ \frac{1}{2\sigma^2} \right] \cdot e^2. \quad (5)$$

Present (5) in the form of a polynomial  $A + B \cdot e + C \cdot e^2$ . Determine the coefficients  $A$ ,  $B$ ,  $C$  by the method of least squares and calculate the parameters of the model  $\sigma$ ,  $H_k$ ,  $e_0$  separately for the left and right halves of the model by the coordinates of points with discrete values of the intensity signal  $(e_i, \ln T_i)$ , where  $i = 1, \dots, N_k$  is the number of considered points of the  $k$  component that are included in the approximation (5–10 points near the peak)

$$\sigma^2 = \frac{1}{2 \cdot |C|}, H_k = \exp \left[ A + \frac{e_0^2}{2 \cdot \sigma^2} \right], e_0 = B \cdot \sigma^2. \quad (6)$$

The values of the model parameters of the component  $H_k$  and  $e_0$  are determined as the average values of the obtained corresponding parameters of the left and right halves of the model, in addition the models height values  $H_k$  will be refined at the stage of building the generative model of the intensity signal  $T(e)$  according to function (1).

## Intensity Signal Model

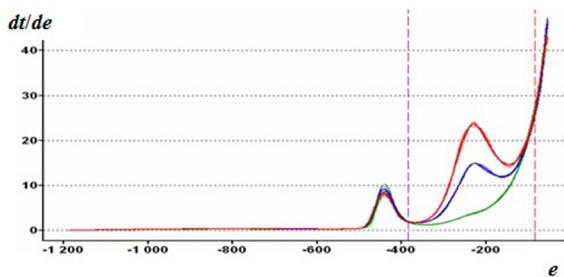
Let's consider the algorithm for building a mathematical model of a multicomponent intensity signal using the example of measuring a standard solution of the element  $Cu$  (copper) with a concentration of  $0,1 \text{ mg/dm}^3$  ( $0,1 \text{ } \mu\text{g/cm}^3$ ) in the presence of  $Pb$  (lead) ions.

Parallel measurements of the background solution stripping ("background"), the sample solution – a standard  $Cu$  solution ("sample"), and the sample solution with the addition of  $Cu$  ions ("additive") were performed. The chemist manually set the limits of the stripping ranges of the  $Cu$  component (dashed vertical lines) (Fig. 1). Based on the values of the background stripping time, sample, and additive, the mass concentration of  $Cu$  in the standard solution with the value of  $C_{Cu} = 0,1189 \text{ } \mu\text{g/cm}^3$  was calculated [9] with a relative error of 18,9%.

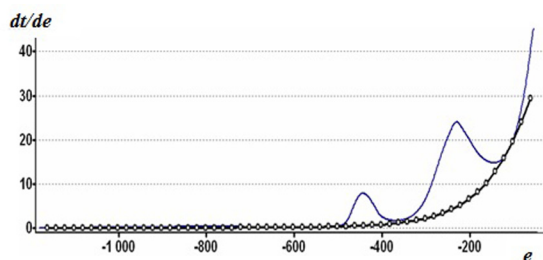
The sequence of building a multicomponent intensity signal model is as follows:

**Step 1.** We define the model of the base curve  $g_0(e)$  according to dependence (2) for a silver measuring electrode covered with mercury amalgam.

**Step 2.** Subtract the amplitudes of the base curve model  $g_0$  from the intensity signal  $T(e)$  and obtain the spectrogram  $G(e) = T(e) - g_0(e)$ .



lower – background, middle – sample, upper – additive  
 Fig. 1. Signals of the background intensity, sample, additives of *Cu* concentration measurement



lower – base curve, upper – additive signal  
 Fig. 2. Additive intensity signal and base curve of *Cu* measurement

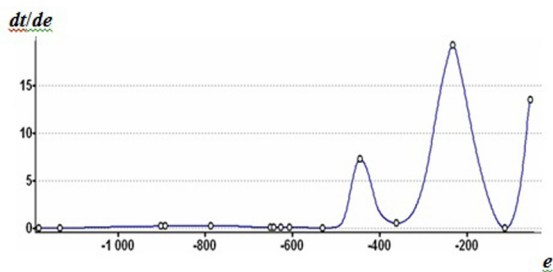


Fig. 3. Spectrogram of the intensity signal

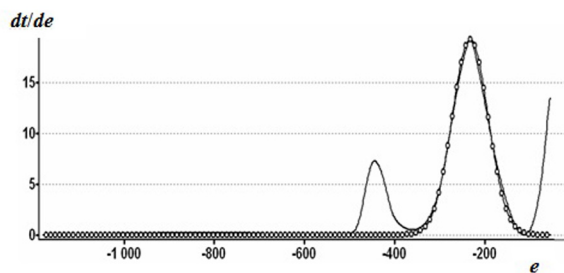


Fig. 4. Additive intensity signal and base curve of *Cu* measurement

Step 3. Analyze the spectrogram  $G(e)$ , find the abscissa of the extremum point with the maximum amplitude  $e_0$  and determine the model of the component  $g_k(e)$  (Fig. 4).

Step 4. From the amplitudes of the spectrogram  $G(e)$ , subtract the calculated amplitudes of the model:  $G(e) = G(e) - g_k(e)$  and repeat steps 3 and 4 of the algorithm, checking the significance of the component amplitudes. The component is considered significant if the stripping time (the area of the component in the spectrogram  $G(e)$ ) or the number of points of amplitudes  $N_k$  is greater than the minimum specified values, otherwise further identification of elements in the intensity signal is stopped.

Step 5. After determining the models of all components, solve the problem of parametric identification (1) by the method of least squares and find the optimal values of the parameter vector  $H_k$  using the amplitudes of the entire intensity signal  $T(e)$ .

When determining the concentrations of a chemical element, the construction of a mathematical model of a multicomponent intensity signal is applied to all parallel stripping signals of the measurement of the background, sample, and additive. For the considered example of *Cu* concentration measurement (Fig. 1), the spectrogram of the model signals of the components looks like this:

The concentration of *Cu* ions in the standard solution, calculated from the values of background stripping time, sample, additive of spectrograms model signals, was  $C_{Cu} = 0,1015 \mu\text{g}/\text{cm}^3$ , with a relative error of 1,5%, which is less than 18,9%. That is, the application of the described method of identification of chemical elements helps to obtain more reliable results of concentrations measurement.

Let's consider another example of building mathematical models of multicomponent intensity signals.

### Analysis of the Results Repeatability of the Chemical Elements Identification

Let's measure the concentration of the chemical element *Zn* in a standard solution with the presence of *Cd*, *Pb*, and *Cu* ions, with a concentration

of  $CZn = 0,1 \mu\text{g}/\text{cm}^3$ . The measured value of  $Zn$  concentration in the standard solution was  $CZn = 0,1027 \mu\text{g}/\text{cm}^3$  with a relative error of 2,7%.

Fix the defined parameters of all components ( $H, e_0, \sigma_1, \sigma_2$ ) and the function of the base curve model  $g_0(e)$  in the background, sample, additive stripping signals during simulation. Calculate the input model signals of the intensity of the background, sample, and additive according to the rounded values of the obtained parameters and perform the identification of the  $Zn$  element again. Perform an analysis of the comparison of the received parameters of the background, sample, additive signal components with the known input model values of these parameters.

Graphical results of comparing the spectrograms of the input model signals of the background intensity, sample, additive of the  $Zn$  component shown in Fig. 6, argue the complete identity of the input and received signals and models.

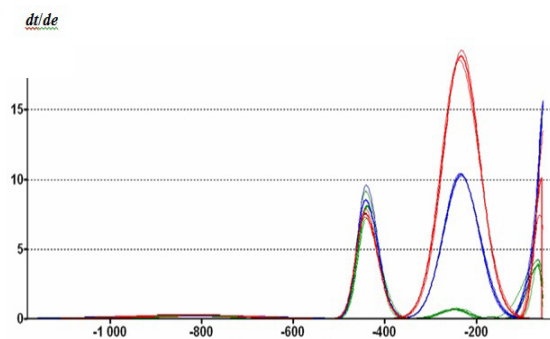
$Zn$  concentration value, calculated from the values of the background stripping time, sample, and spectrogram additive, equated to  $C_{Zn} = 0,1051 \mu\text{g}/\text{cm}^3$  with a relative error of 5,1%, which is greater than the previous concentration value of the input model but less than the normalized value of 20%.

The comparison results of the digital values of the parameters  $H, e_0, \sigma_1, \sigma_2$  of the spectrogram components for the input (“In”) and output (“Out”) signal models of the background intensity, sample, and additive are shown in Table 1. Since the analytical factor for calculating the concentration is the stripping time  $T$  of the  $Zn$  element, the values and the relative error of their comparison is given in percents

$$\delta = 100 \frac{T_{out}}{T_{in}}. \quad (7)$$

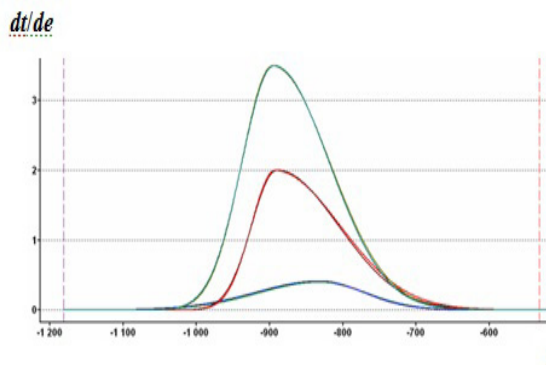
## Conclusions

The paper demonstrates the analysis of the application of chemical elements identification on various test examples when determining the concentrations of chemical elements with the help of stripping chronopotentiometry methods by building a mathematical model of a multicomponent intensity signal, which is a differential stripping signal.



lower – background, middle – sample, upper – additive

Fig. 5. Model signals of the background spectrograms, sample, additive for  $Cu$  concentration measurement



lower – background, middle – sample, upper – additive

Fig. 6. Spectrograms of input and model signals of the background intensity, sample and additive components of  $Zn$  element measurement

It was noted that the application of the intensity signal model does not distort the integral characteristics of the components of chemical elements, which are the analytical characteristics of the concentration measurement. The relative error of determining the integral characteristics did not exceed the value of 5%, which is permissible in engineering calculations.

The use of the proposed approach to the identification of chemical elements with the modeling of multicomponent signals allows you to fully automate the process of measuring concentrations, which will not depend on the analytical chemist's ability to determine the potentials of the stripping ranges of elements, thus increasing the accuracy, repeatability and reliability of analytical research.

Table 1. Spectrogram components parameters of Zn concentration measurement

Signal	Type	H	$e_0, m_V$	$\sigma_1$	$\sigma_2$	T, Ms	$\delta, \%$
Background	in	0,4	831	85	63	74,2	3,64
	out	0,411	831,7	88,3	61	76,9	
Sample	in	2	893	32	89	303,3	1,98
	out	2,002	890,4	34,2	84,3	297,3	
Additive	in	3,5	896	42	78	526,4	0,78
	out	3,499	894,4	43,4	75,7	522,3	

The automation of the identification process made it possible to develop the analytical system "ISP Analyzer", using methods of pulse stripping chronopotentiometry (PSCP) [10], as well as methods of measuring the concentrations of 14 toxic elements (Pb, Cd, Cu, Zn, As, Hg, Ni, Co,

Cr, Se, I, Mn, Sn, Fe) in drinking water, food products, soils and environmental objects, with a sensitivity of up to 0,01  $\mu\text{g}/\text{dm}^3$ .

The further research will be devoted to the development technology for field measurements of toxic elements.

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## МЕТОД ІДЕНТИФІКАЦІЇ ЕЛЕМЕНТІВ У БАГАТОКОМПОНЕНТНОМУ СИГНАЛІ

**Вступ.** При вимірюванні наднизьких концентрацій токсичних елементів при здійсненні екологічного моніторингу доквілля часто застосовують дуже чутливі електрохімічні методи інверсійної хронопотенціометрії (ІХП). Для цього методу аналізу суттєвим є ідентифікація та визначення часу інверсії хімічного елемента в багатоконпонентному диференційному зворотному сигналі інверсії (сигналі інтенсивності), де час є аналітичною характеристикою концентрації.

Фізично сигнал інтенсивності є сумою двох хімічних процесів у часі – ємнісного фонового розряду електродів (базова крива) та суми інверсії компонентів (хімічних елементів), які можуть частково перекривалися. Застосування моделі сигналу інтенсивності, яка враховує всі хімічні процеси, сприяє більш точному та стабільному вимірюванню концентрацій елементів за рахунок ідентифікації та стандартизації визначення часу інверсії, який не буде залежати від людських факторів визначення хіміком-аналітиком діапазонів потенціалів інверсії кожного елемента.

**Мета.** Розробити та застосувати генеративну модель багатокомпонентного диференційного сигналу інверсійної хронопотенціометрії в координатах потенціалів у часі у вигляді моделі ємнісного фонового розряду електродів та суми моделей компонентів інверсії хімічних елементів, заданих несиметричними функціями нормального розподілу.

**Методи.** Визначення концентрації здійснюється методом інверсійної хронопотенціометрії. Параметрична ідентифікація здійснюється методом найменших квадратів.

**Результати.** У роботі проаналізовано застосування ідентифікації хімічних елементів на різних тестових прикладах при визначенні концентрацій хімічних елементів методами ІХП шляхом побудови математичної моделі багатокомпонентного сигналу інтенсивності, який є диференційним сигналом інверсії.

Застосування моделі сигналу інтенсивності не спотворює інтегральні характеристики компонентів хімічних елементів, які є аналітичними характеристиками вимірювання концентрації. Відносна похибка визначення інтегральних характеристик не перевищувала значення 5%, що є допустимим в інженерних розрахунках.

**Висновки.** Використання запропонованого підходу до ідентифікації хімічних елементів з моделюванням багатокомпонентних сигналів дозволяє повністю автоматизувати процес вимірювання концентрацій, який не буде залежати від умінь хіміка-аналітика визначати потенціали діапазонів інверсії елементів, що збільшить точність, повторюваність та надійність аналітичних досліджень.

Автоматизація процесу ідентифікації дозволила розробити аналітичну систему «Аналізатор ІХП», з використанням імпульсних методів ІХП, та методики вимірювання концентрацій 14 токсичних елементів (*Pb, Cd, Cu, Zn, As, Hg, Ni, Co, Cr, Se, I, Mn, Sn, Fe*) у питній воді, харчових продуктах, ґрунтах та в об'єктах довкілля, з чутливістю до 0,01 мкг/дм<sup>3</sup>.

Основними напрямками подальших досліджень буде розроблення технології експрес аналізу для здійснення польових вимірювань токсичних елементів.

**Ключові слова:** параметрична ідентифікація, генеративна модель, багатокомпонентний сигнал, нормальний розподіл, базова крива, пік сигналу, логарифмічні координати, метод найменших квадратів.