AMPHEROMETRIC TITRATION WITH AIM OF DETERMINATION OF THE CORRELATION COEFFICIENTS

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Abstract. Voltamperometric behavior of thioacetamide and its metallocomplexes on the platinum micro disk anode on different by nature and concentrations buffer solutions and phone electrolytes was investigated. Nature of limited anode currents and number of electron donation oxidation of one molecule of thioacetamide were determined and also the conditions of platinum(IV), palladium(II) and gold(III) determination by its solution were optimized. On the base of obtained data the possibility of using of elaborated electrochemical methods to analysis of some real objects and industrial materials was shown. With aim of elaboration of amperometric methods of platinum(IV), palladium(II) and gold(III) determination by thioacetamide solution and metrological value of obtained experimental data the coefficients of correlation were determined, the results of which are useful for analysis of different by nature standards samples, ores, mineral, tails and natural objects.

Key words: selectivity, amperometry, conductometry, elektroconductivity, rightness, quickness, reproduction, titration; diffusion, kinetic and mixed anodic currents.

Introduction

Analysis of objects of environment containing micro- and traced quantities of platinoids and gold is a very important and actual problem caused by increasing necessity of industry in noble metals and their alloys (Khimicheskaya entsiklopediya, 1992). The most significant demands produced to analysis of different by nature materials are precession, rightness and reproduction equally with expression of determinations. At the same time elaboration of new methods of Pt(IV), Pd(II), Au(III) determination with best metrological characteristics and analytical parameters is very important owing to their low cost and strategically importance (Korenman, 2005, p.232-235).

Method

At determination of micro and trace concentrations of Pt(IV), Pd(II) and Au(III) by solution of thioacetamide different by form and character curves of amperametrical titration were obtained. At this the final point of titration has corresponded to molar ratio of components of reaction Me: Reagent 1:4, 1:2 and 1:3 correspondently. Experiments have shown that from all investigated phones and buffer mixtures the best curves of titration were obtained on the universal buffer system of Britton – Robinson possessing strongly pronounced acid properties (pH=1,81-3,84).

For metrological value of elaborated amperometrical methods and obtained experimental data the correlation analysis of results obtained at determination of different concentrations Pt(IV), Pd(II) and Au(III) by solution of thioacetamide was carried out.

Calculations of the correlation coefficients were carried out by following equations (1:5) (Korita, Dvorjak and Bogachkova, 1977):

\[ r = \frac{n \cdot \sum x_i y_i - \sum x_i \sum y_i}{\left( n \cdot \sum x_i^2 - (\sum x_i)^2 \right) \left( n \cdot \sum y_i^2 - (\sum y_i)^2 \right)^{1/2}} \]  \hspace{1cm} (1);

\[ r = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{\sum (x_i - \bar{x})^2 \sum (y_i - \bar{y})^2} \]  \hspace{1cm} (2);
\[
    r = \frac{1}{n} \sum (x_i - \bar{x})(y_i - \bar{y}) : (S_x S_y) = \left( \frac{1}{n} \sum x_i y_i - \bar{x} \bar{y} \right) : (S_x S_y)
\]  \hspace{1cm} (3);

\[
    S_x^2 = \frac{1}{n} \sum (x_i - \bar{x})^2 \hspace{1cm} (4);
\]

\[
    S_y^2 = \frac{1}{n} \sum (y_i - \bar{y})^2 \hspace{1cm} (5),
\]

where: \( n \) – number of experimental points; \( x_i \) – concentration of determined cation, mkg/ml; \( \bar{x} \) – average determined value of component concentration; \( y_i \) – equivalent point of titrated cation; \( \bar{y} \) – average determined value of highs of voltamperogramm.

It is known (Aleskovskiy, Bardin and Vasilyev, 1964) that strong correlation bond don’t obligatorily designate caused bond between parameters “\( x \)” and “\( y \)”.

Results of determination of the correlation coefficient for Pt(IV), Pd(II) and Au(III) are presented in tables 1-3.

**Table 1**

<table>
<thead>
<tr>
<th>№</th>
<th>( x_i )</th>
<th>( y_i )</th>
<th>( x_i y_i )</th>
<th>( x_i^2 )</th>
<th>( y_i^2 )</th>
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<td>3,3</td>
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<td>225</td>
<td>10,89</td>
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<td>400</td>
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<td>25</td>
<td>5,5</td>
<td>137,5</td>
<td>625</td>
<td>30,25</td>
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<tr>
<td>Σ</td>
<td>75</td>
<td>16,5</td>
<td>302,5</td>
<td>1375</td>
<td>66,55</td>
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**Table 2**

<table>
<thead>
<tr>
<th>№</th>
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<th>( y_i )</th>
<th>( x_i y_i )</th>
<th>( x_i^2 )</th>
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<td>625</td>
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<tr>
<td>Σ</td>
<td>100</td>
<td>42</td>
<td>980</td>
<td>2250</td>
<td>431,2</td>
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</table>

Calculation of the correlation coefficient “\( r \)” at amperometrical titration of Pt(IV) was carried out by:

\[
    r = \frac{5 \cdot 302,5 - 75 \cdot 16,5}{\left( \left[ 5 \cdot 1375 - (75)^2 \right] \left[ 5 \cdot 66,55 - (16,5)^2 \right] \right)^{1/2}} = 1,0
\]

Calculation of the correlation coefficient “\( r \)” at amperometrical titration of Pd(III) was carried out by:

\[
    r = \frac{5 \cdot 980 - 100 \cdot 42}{\left( \left[ 5 \cdot 2250 - (100)^2 \right] \left[ 5 \cdot 431,2 - (42)^2 \right] \right)^{1/2}} = 1,0
\]

Calculation of the correlation coefficient “\( r \)” at amperometrical titration of Au(III) was carried out by:

\[
    r = \frac{5 \cdot 315 - 100 \cdot 14}{\left( \left[ 5 \cdot 2250 - (100)^2 \right] \left[ 5 \cdot 441 - (14)^2 \right] \right)^{1/2}} = 1,0
\]
Table 3

Metrological characteristics using for calculation of the correlation coefficient (r) of determination of Au(III)

<table>
<thead>
<tr>
<th>№</th>
<th>x_i</th>
<th>y_i</th>
<th>x_i·y_i</th>
<th>x_i^2</th>
<th>y_i^2</th>
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<td>625</td>
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<tr>
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<td>126</td>
<td>900</td>
<td>17,64</td>
</tr>
<tr>
<td>Σ</td>
<td>100</td>
<td>14</td>
<td>315</td>
<td>2250</td>
<td>44,1</td>
</tr>
</tbody>
</table>

As shown from obtained data the determinate correlation coefficients “r” in all cases don’t exceed 1,0 what is in accordance with theoretical literature data. Obtained values of “r” have indicated about rightness and reproduction of elaborated amperometrical methods of determination of Pt(IV), Pd(II) and Au(III) by solution of thioacetamide.

Thus these methods are suitable for analysis of different by nature standart samples of ores, minerals, tails and some other materials because the low limits of determinate quantities of investigated noble elements and also their limits of detection (sensibleness) are on the level of PDC and lower.

References