ANALYSIS OF NI (II) ION AND DIMETYLGLIOXIME COMPLEX IN USED ADSORBENT

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ABSTRACT
This paper presents the results of the analysis of the complex of nickel (II) ion with dimethylglyoxime from the adsorbent composition used in the purification of MDEA. The spectral characteristics of the complex on the SF 46 spectrophotometer, the determination of the composition of the complex using the method of isomolar series, the determination of the actual molar extinction coefficient of the complex and the equilibrium constant by Tolmachyov's graphical method are presented on the basis of tables.

Key words: used adsorbent, Ni (II) ion, dimethylglyoxime reagent, sandel sensitivity, quenching coefficient, Tolmachyev graph, pH, buffer solution.

Currently, the production has a significant negative impact on the environment due to the toxic content of the adsorbents used as waste in industrial zones. The methods of analysis of the isolated complex of toxic Ni (II) ion formed with dimethylglyoxime in the adsorbent used in this paper are described[4]. Absolute loss of this ion from the adsorbent was achieved, and the spectral characteristics of the complex, the method of isomolar series of the composition of the complex, the equilibrium constant were studied [3].

Spectral description
Light absorption spectra were obtained under selected optimal conditions of the complex formed with dimethylglyoxime reagent and Ni (II).

Process: 2.6 ml of aqueous solution of 0.01% dimethylglyoxime reagent and 5.0 ml of buffer with pH = 8.0 in a 25 ml volumetric flask, 3.5 ml of a solution of Ni (II) of 30.0 μg / ml. from the solution and diluted with distilled water to the mark of the flask. The absorption spectrum of the resulting complex was measured in a
quartz cuvette with a light absorption thickness of $l = 1.0$ cm relative to the reference solution, with a spectrophotometer SF-46. The absorption spectrum of the reagent was obtained with respect to distilled water. The results showed that the maximum absorption area of dimethylglyoxime reagent complex with Ni (II) was located at $\lambda_{\text{comp}} = 550$ nm, the maximum light absorption area of dimethylglyoxime reagent was observed in the region of shorter spectral waves, $\lambda_{\text{reagent}} = 470$ nm.

Using the value of the maximum optical density of the complex compound (in the area $l = 550$ nm), the apparent molar extinction coefficient ($E_{\text{off}}$) [1] was determined by the following formula:

$$E_{\text{off}} = \frac{A}{C \cdot l} \quad (1)$$

The spectral characteristics of the complex and the reagent are given in Table 1. The Sendel sensitivity of the method is $0.001 \text{ mcg} / \text{ cm}^2$. The light absorption is calculated by the following formula [2]:

$$S.b.s = \frac{Q \cdot L \cdot 0.0001}{A \cdot 25} \quad (2)$$

**Table 1.**

Results of the study of the composition of a complex compound using the method of isomolar series

<table>
<thead>
<tr>
<th>Color of complex</th>
<th>pH</th>
<th>$C_{\text{MeR}}$, nm</th>
<th>$C_{\text{HR}}$, nm</th>
<th>$\Delta \lambda$, nm</th>
<th>$C_{\text{Ni}^{2+}}$, mg</th>
<th>$C_{\text{Ni}^{2+}}$, mol/l</th>
<th>$\bar{A}$</th>
<th>Sendel sensitivity Mkg / sm$^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Red-pink</td>
<td>8.0</td>
<td>550</td>
<td>480</td>
<td>70</td>
<td>30</td>
<td>2.0446$\times$10$^{-5}$</td>
<td>0.435</td>
<td>0.0027586 Mkg / sm$^2$</td>
</tr>
</tbody>
</table>

$S.b.s = \frac{Q \cdot L \cdot 0.0001}{A \cdot 25}$

The results show that the reaction has a high contrast (at $\lambda_{\text{comp}} = 550$ nm) and sensitivity ($S.b.s. = 0.0027586$).

Determination of the proportion of constituent moles of the complex formed by nickel (II) with dimethylglyoxime reagent. Determination of complex composition using the method of isomolar series.

The molar ratios of the components in the complex formed by nickel (II) with dimethylglyoxime reagent were determined by the isomolar series method. Equilibrium solutions of nickel (II) and dimethylglyoxime reagents were used to determine the ratio of moles in the complex formed by nickel (II) with dimethylglyoxime reagent by the method of isomolar series:

$$C_{\text{Ni}^{2+}} = C_{\text{HR}} = 2.0446 \times 10^{-5} \text{ mol/l}$$

Procress: A series of solutions were prepared in 25 ml volumetric flasks. To do this, add to each of a variable amount of nickel (II) solution (up to 9.0–1.0 ml) and add a variable amount of dimethylglyoxime reagent solution (up to 1.0 ml-9.0 ml), and 5.0 ml of a universal buffer solution with a pH of 8 were diluted and mixed with distilled water to the mark of the tube.

The optical density of the prepared solutions was measured at KFK-2 at a light filter at 550 nm and relative to the reference solution in cuvettes with a light absorption thickness of $l = 1.0$ cm. The results obtained are given in Table 2.

**Table 2.**

Results of the study of the composition of a complex compound using the method of isomolar series

<table>
<thead>
<tr>
<th>№</th>
<th>Obtained Ni(II), ml</th>
<th>Obtained Vur, ml</th>
<th>Buffer solution</th>
<th>$\bar{A}$</th>
<th>Sendel sensitivity Mkg / sm$^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>9.5</td>
<td>1.5</td>
<td>5.0</td>
<td>0.007</td>
<td></td>
</tr>
<tr>
<td>2.</td>
<td>8.5</td>
<td>2.5</td>
<td>5.0</td>
<td>0.08</td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td>7.5</td>
<td>3.5</td>
<td>5.0</td>
<td>0.165</td>
<td></td>
</tr>
<tr>
<td>4.</td>
<td>6.5</td>
<td>4.5</td>
<td>5.0</td>
<td>0.30</td>
<td></td>
</tr>
<tr>
<td>5.</td>
<td>5.5</td>
<td>5.5</td>
<td>5.0</td>
<td>0.34</td>
<td></td>
</tr>
<tr>
<td>6.</td>
<td>4.5</td>
<td>6.5</td>
<td>5.0</td>
<td>0.39</td>
<td></td>
</tr>
<tr>
<td>7.</td>
<td>3.5</td>
<td>7.5</td>
<td>5.0</td>
<td>0.45</td>
<td></td>
</tr>
<tr>
<td>8.</td>
<td>2.5</td>
<td>7.0</td>
<td>5.0</td>
<td>0.50</td>
<td></td>
</tr>
<tr>
<td>9.</td>
<td>2.0</td>
<td>8.0</td>
<td>5.0</td>
<td>0.43</td>
<td></td>
</tr>
<tr>
<td>10.</td>
<td>1.0</td>
<td>9.0</td>
<td>5.0</td>
<td>0.33</td>
<td></td>
</tr>
</tbody>
</table>
It can be seen from this table that the composition of the complex formed by the dimethylglyoxime reagent with nickel (II) corresponded to the molar ratio of Ni: R = 1: 2.

Determination of the real molar extinction coefficient and equilibrium constant of the complex of nickel (II) with dimethylglyoxime reagent by Tolmachyov's graphical method

For a more detailed study of the complex formation reaction of nickel (II) in the dimethylglyoxime reagent and the adsorbent used, the equilibrium constant of the complex formation was determined by Tolmachyov's graphical method. All the above results (Ni: R = 1: 2) were taken into account when working with this method. The reaction equation for Ni (II) with dimethylglyoxime reagent can be described as follows.

Process: A 25-ml volumetric flask was filled with a stoichiometric reactive reagent and nickel (II) solution and 5.0 ml buffer mixture with a pH of 8.0 and diluted to the mark with distilled water. The optical density was measured at KFK-2 at 6 nf, relative to the specific solution in cuvettes with a light absorption thickness of 1 = 1.0 cm. The results are presented in Table 3.

Table 3.

<table>
<thead>
<tr>
<th>№</th>
<th>V Ni2+</th>
<th>VHR</th>
<th>C Ni2+</th>
<th>A</th>
<th>(1/\sqrt{A})</th>
<th>1/(\sqrt{A})</th>
<th>E</th>
<th>1/(\varepsilon)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.25</td>
<td>0.5</td>
<td>0.075</td>
<td>0.774</td>
<td>3.649</td>
<td>1.022</td>
<td>7338.5</td>
<td>1.36</td>
</tr>
<tr>
<td>2</td>
<td>0.50</td>
<td>1.5</td>
<td>0.100</td>
<td>0.316</td>
<td>3.164</td>
<td>2.044</td>
<td>4892.36</td>
<td>2.04</td>
</tr>
<tr>
<td>3</td>
<td>0.75</td>
<td>2.5</td>
<td>0.128</td>
<td>0.358</td>
<td>2.793</td>
<td>3.066</td>
<td>4174.48</td>
<td>2.40</td>
</tr>
<tr>
<td>4</td>
<td>1.00</td>
<td>3.5</td>
<td>0.157</td>
<td>0.394</td>
<td>2.538</td>
<td>4.088</td>
<td>3791.58</td>
<td>2.64</td>
</tr>
<tr>
<td>5</td>
<td>1.25</td>
<td>4.5</td>
<td>0.160</td>
<td>0.400</td>
<td>2.590</td>
<td>5.11</td>
<td>2622.95</td>
<td>3.13</td>
</tr>
<tr>
<td>6</td>
<td>1.50</td>
<td>5.5</td>
<td>0.180</td>
<td>0.424</td>
<td>2.350</td>
<td>6.13</td>
<td>2517.48</td>
<td>3.40</td>
</tr>
</tbody>
</table>

Tolmachyov’s graphical formula for the real molar extinction coefficient and equilibrium constant of a complex [2]:

\[
\frac{1}{\varepsilon} = f\left(\frac{1}{\sqrt{A}}\right)
\]

(3)

The following formulas were used in the calculations:

\[
\varepsilon_{\text{real}} = \frac{1}{\varepsilon} \times 10^{-\eta} = 1/0.136 \times 10^{-4} = 7.52 \times 10^{-4}
\]

(4)

\(\varepsilon_{\text{real}}\) - real molar extinction coefficient of the complex;

Compared to the calculated numerical values, the developed method showed a much higher sensitivity, and the complex compound had an average stability.

According to the results obtained, the solution of dimethylglyoxime, used as a reagent in the complete complex precipitation of nickel ions in the adsorbent used, gave good results. According to the spectral characteristics of this complex, the absorption spectrum was measured in a quartz cuvette with a light absorption thickness of 1 = 1.0 cm relative to the reference solution, with a spectrophotometer SF-46.

The absorption spectrum of the reagent was obtained with respect to distilled water. The results showed that the maximum absorption area of dimethylglyoxime reagent complex with Ni (II) was located at \(\lambda_{\text{comp}} = 550\) nm, the maximum light absorption area of dimethylglyoxime reagent was observed in the region of shorter spectral waves, \(\lambda_{\text{reagent}} = 470\) nm, real molar extinction coefficient of the complex is 7,52*10\(^{-4}\).

References:


