

Received May 05, 2020; reviewed; accepted June 23, 2020

Temperature influence on the electrode process in the presence of 6-thioguanine and surfactants

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Abstract: The measurements concerning the process of the electroreduction of Bi(III) ions in the presence of 6-thioguanine (6TG) and Triton X-100 as well as Tween 80 in the 2 mol·dm⁻³ chlorates(VII) demonstrate a dependence of the process rate on temperature. The applied electrochemical techniques (DC polarography, cyclic and SWV voltammetry) allowed to determine the kinetic and thermodynamic parameters as well as their correlation with changes in kinetics of the electrode process. The catalytic activity of 6TG was confirmed by the decrease of overall enthalpies of activation. Different values of ΔH^\ddagger and ΔS^\ddagger for Bi(III) electroreduction in the presence of 6-thioguanine as well as Triton X-100 and Tween 80 suggest changes in kinetics of the electrode process towards inhibition.

Keywords: catalytic effect, Bi(III) electroreduction, 6-thioguanine, surfactants, thermodynamic parameters, kinetic parameters

1. Introduction

Investigations of the effect of mixed adsorption layers on the kinetics of Bi(III) ions reduction on the mercury electrode showed a change in the dynamics of the catalytic action of thiopurine derivatives on the electrode process. This is reflected by a decrease in the standard rate constants as a function of surfactant concentration (Kaliszczak, Nosal – Wiercińska, 2018; Kaliszczak, Nosal – Wiercińska, 2019; Kaliszczak, Nosal – Wiercińska, 2020). The obtained effect was associated with the blockade of the electrode surface by surfactants pushing out from the adsorption layer composed of previously formed active complexes, which mediate the electrons transfer.

According to the literature reports, temperature is a factor supporting the catalytic action of amalgamation reactions of many depolarizers (Nosal-Wiercińska, 2010; Nosal-Wiercińska, 2011; Nosal-Wiercińska, 2012; Wiśniewska, 2012; Wiśniewska, 2011; Sharma and Gupta, 2011; Marczak et al., 1999; López-Pérez et al., 2003; Marczevska et al., 1999). These observations are related to the temperature dependence of different kinetic or thermodynamic parameters of electrode processes (Nosal-Wiercińska, 2010; Nosal-Wiercińska, 2011; Nosal-Wiercińska, 2012; Sharma, Gupta, 2011; Marczak et al., 1999; López-Pérez et al., 2003; Marczevska et al., 1999; Fawcett, Jaworski, 1982; Nieszporek and Dagci, 2014). Marczak et al. (Marczak et al., 1999) analyzed the temperature dependence of transfer coefficients for cathodic and anodic electrode reactions of two different types of systems, namely Cr(III)/Cr(II) and Cr(II)/Cr(Hg) in chlorate(VII) solutions.

Moreover, the change of the Tafel slope with temperature was observed for the Ba(II)/Ba(Hg) system in DMF in 0.1 mol·dm⁻³ TEAP (Fawcett, Jaworski, 1982), which was associated with some adsorption of cations of the background electrolyte.

Changes in the rates of electrode reactions are strictly connected with those in the activation energy (ΔH^\ddagger) or standard reaction entropy (ΔS^0) of the process (Kaliszczak, Nosal-Wiercińska, 2018; Nosal-Wiercińska, 2011; Nosal-Wiercińska, 2012; Marczyk et al., 1999; López-Pérez et al., 2003; Marczevska et al., 1999).

The changes of enthalpies of activation ΔH^\ddagger (the values of activation energy decrease significantly) confirm the catalytic activity of methionine (Nosal-Wiercińska, 2010), cysteine (Nosal-Wiercińska, 2011), thiourea (Marczevska et al., 1999), sodium 1 - decanesulfonate (Nieszporek and Nieszporek, 2018) and 6 - mercaptopurine (Kaliszczak and Nosal-Wiercińska, 2018). In turn, in the presence of Tween 80 or Triton X - 100, the values of ΔH^\ddagger increase. This indicates changes in the kinetics of the electrode process towards inhibition.

Whereas, the presence of cysteine in the 1 - 8 mol·dm⁻³ chlorate(VII) solutions decreased the value of $\Delta S_{\text{Bi(III)/Bi(Hg)}}^0$ at all examined chlorate(VII) concentrations and suggested changes in the dynamics of the process of Bi(III) ion electroreduction (Nosal-Wiercińska, 2011). Similar reductions in the value of $\Delta S_{\text{Bi(III)/Bi(Hg)}}^0$ were observed in the presence of 1·10⁻³ mol·dm⁻³ 6-mercaptopurine and the above mentioned surfactants (Kaliszczak and Nosal-Wiercińska, 2018). This is related to the change in the electrode mechanism.

The paper presents the analysis of the temperature dependencies of kinetic parameters of the electrode reactions in the Bi(III)/Bi(Hg)/2 mol·dm⁻³ chlorates(VII) + 6TG and Bi(III)/Bi(Hg)/2 mol·dm⁻³ chlorates(VII) + 6TG + surfactants systems.

The electrochemical methods, such as DC polarography, SWV and cyclic voltammetry CV were used for this purpose. The determined kinetic and thermodynamic parameters indicated changes in the electrode process speed in the presence of 6TG and mixed adsorption layers due the temperature change.

2. Experimental

Polarographic and voltammetric measurements were performed employing a μ Autolab Fra 2/ GPES (Version 4.9) analyser (Eco Chemie, Utrecht, Netherlands). The working electrode was a dropping or hanging controlled - growth mercury drop electrode (CGMDE) and a constant drop surface (0.014740 cm²) made by MTM, Poland. The reference was a silver chloride electrode and the auxiliary electrode was platinum.

Prior to the measurements, the solutions were deaerated using high purity nitrogen. Furthermore, nitrogen was passed over the solutions during the measurements.

All experiments were conducted in the temperature range of 288-303 \pm 0.5 K. The chemicals of analytical grade from Fluka were used.

There were applied the following organic substance concentration ranges: 6TG from 0.1 to 5·10⁻³ mol·dm⁻³, Triton X-100 and Tween 80 from 1·10⁻⁶ to 1·10⁻⁴ mol·dm⁻³.

The values of kinetic and thermodynamic parameters were calculated from the cyclic voltammetry measurements.

For irreversible processes the standard rate constants k_s values were determined from the cathodic peaks potential, whose dependence on the kinetic parameters is described by the following equation (Galus, 1979):

$$E_{pk} = E_f^0 - \frac{RT}{an_\alpha F} [0.78 - \ln k_s + \ln \sqrt{D_{ox} b}] \quad (1)$$

where:

$$b = \frac{an_\alpha F v}{RT} \quad (2)$$

E_f^0 - the formal potential,

an_α - the cathode transfer coefficient

For quasireversible processes the k_s values were determined using the Nicholson (Galus, 1979) method based on the equation:

$$\Psi = \left(\frac{D_{ox}}{D_{red}}\right)^{\alpha/2} \frac{k_s(RT)^{1/2}}{(\pi n F \nu D_{ox})^{1/2}} \quad (3)$$

The dependencies of $n(E_{pa} - E_{pk})$ values as a function of Ψ are presented in the literature (Galus, 1979).

The details of determination of the diffusion coefficient (D_{ox}) of Bi(III) ions, formal potentials (E_f^0) and transfer coefficient (αn_α) in the studied solutions are described elsewhere (Nosal-Wiercińska 2010). The accuracy of the value determination was rated $\pm 5\%$.

The enthalpies of activation ΔH^\ddagger for Bi(III) electroreduction in the studied systems were determined according to equations (1) and (3) (López-Pérez et al., 2003; Mianowski et al., 2018):

$$\Delta H^\ddagger = R \frac{d \ln k_s}{d \frac{1}{T}} \quad (4)$$

whereas the standard reaction entropy ΔS^0 (López-Pérez et al., 2003; Mianowski et al., 2018) from the following formula:

$$\Delta S_{\text{Bi(III)/Bi(Hg)}}^0 = 2F \frac{dE_f^0}{dT} \quad (5)$$

The accuracy of the values of ΔH^\ddagger and ΔS^0 determination were rated $\pm 5\%$

In the DC polarography, SWV and CV voltammetry, the optimal experiment operating conditions were as follows: the step potential 2 mV for DC, the pulse amplitude 20 mV, the frequency 120 Hz and the step potential 2 mV for the SWV, as well as scan rate 5 - 1000 mV s⁻¹ and step potential 5 mV for the CV.

3. Results and discussion

3.1. Polarographic and voltammetric studies

Figures 1a, b, c show the SWV voltammograms of $1 \cdot 10^{-3}$ mol·dm⁻³ Bi(III) in 2 mol·dm⁻³ chlorates(VII) in the presence of $1 \cdot 10^{-3}$ mol·dm⁻³ 6-thioguanine (a) and $1 \cdot 10^{-3}$ mol·dm⁻³ 6-thioguanine with $5 \cdot 10^{-5}$ mol·dm⁻³ Tween 80 (b) and $5 \cdot 10^{-5}$ mol·dm⁻³ Triton X-100 (c). The figures clearly indicate the temperature dependence.

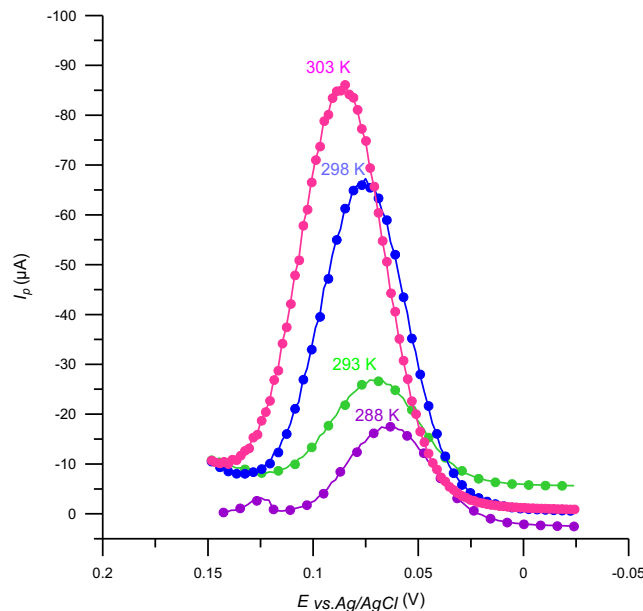


Fig. 1a. The SWV peaks of $1 \cdot 10^{-3}$ mol·dm⁻³ Bi(III) electroreduction in 2 mol·dm⁻³ chlorates(VII) in the presence of $1 \cdot 10^{-3}$ mol·dm⁻³ 6-thioguanine: (●) 288K, (●) 293K, (●) 298K, (●) 303K

With the temperature increase $288\text{K} < 293\text{K} < 298\text{K} < 303\text{K}$ the values of peak currents increase, at the same time, the peak potentials shift towards positive potentials. These relationships are observed for all the studied systems. These observations lead to the conclusion that the temperature increase

affects the growth in kinetics of the electrode process (Nosal-Wiercińska, 2011; Nosal-Wiercińska, 2012; Marczevska et al., 1999; Nieszporek and Nieszporek, 2018).

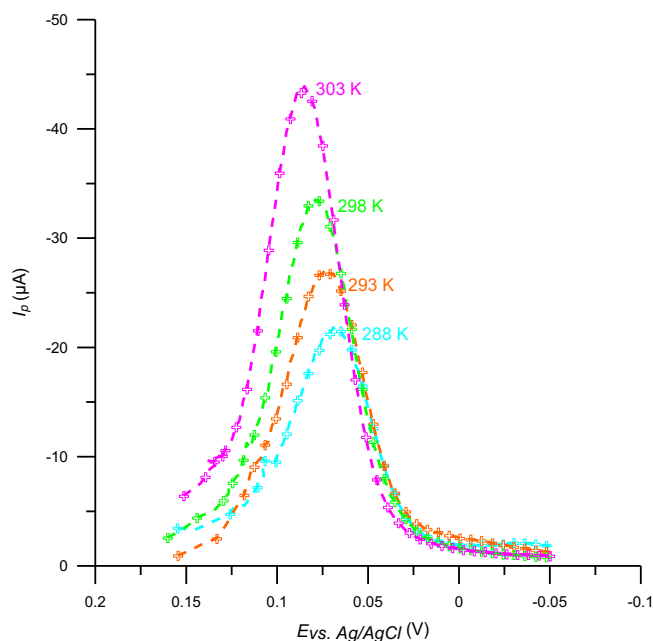


Fig. 1b. The SWV peaks of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ Bi(III) electroreduction in $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) in the presence of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine with $5 \cdot 10^{-5} \text{ mol} \cdot \text{dm}^{-3}$ Triton X-100: (⊕) 288K, (⊕) 293K, (⊕) 298K, (⊕) 303K

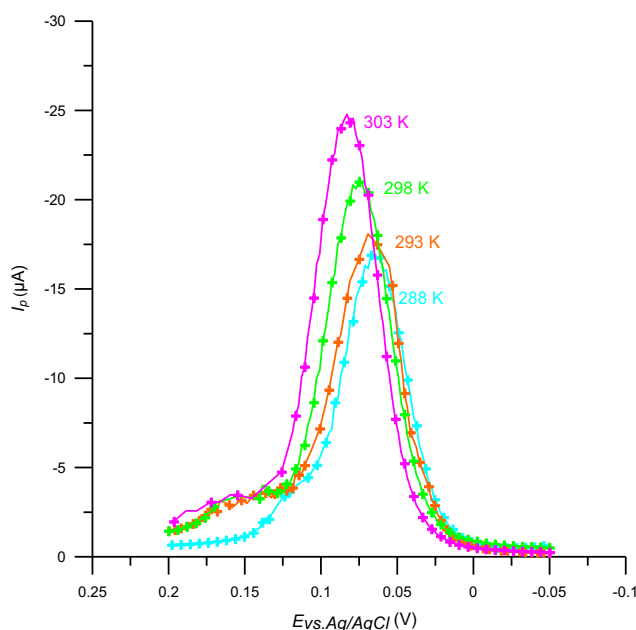


Fig. 1c. The SWV peaks of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ Bi(III) electroreduction in $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) in the presence of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine with $5 \cdot 10^{-5} \text{ mol} \cdot \text{dm}^{-3}$ Tween 80: (⊕) 288K, (⊕) 293K, (⊕) 298K, (⊕) 303K

As follows from the DC polarograms analysis (Fig. 2a, b) of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ Bi(III) electroreduction in $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) in the presence of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine (a) and for example $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine with $5 \cdot 10^{-5} \text{ mol} \cdot \text{dm}^{-3}$ Tween 80 (b) also shows the temperature dependence. With the increase of temperature the DC wave (Fig. 2a) increases and the half - wave potentials ($E_{1/2}$) are shifted towards the positive potentials. However, in the 6-thioguanine - nonionic surfactant system these dependencies are definitely smaller (Fig. 2b). This may be associated with the changes in the viscosity of the basic electrolyte solution (Kaliszczak and Nosal-Wiercińska, 2018) with the increasing temperature. The surfactants presence changes evidently the properties of the studied solution.

Moreover, the previous studies proved that Triton X-100 and Tween 80 change the dynamics of the catalytic activity of thiopurine drugs on Bi(III) ions electroreduction with the tendency of inhibition (Kaliszczak and Nosal-Wiercińska, 2018; Kaliszczak and Nosal-Wiercińska, 2019; Kaliszczak and Nosal-Wiercińska, 2020).

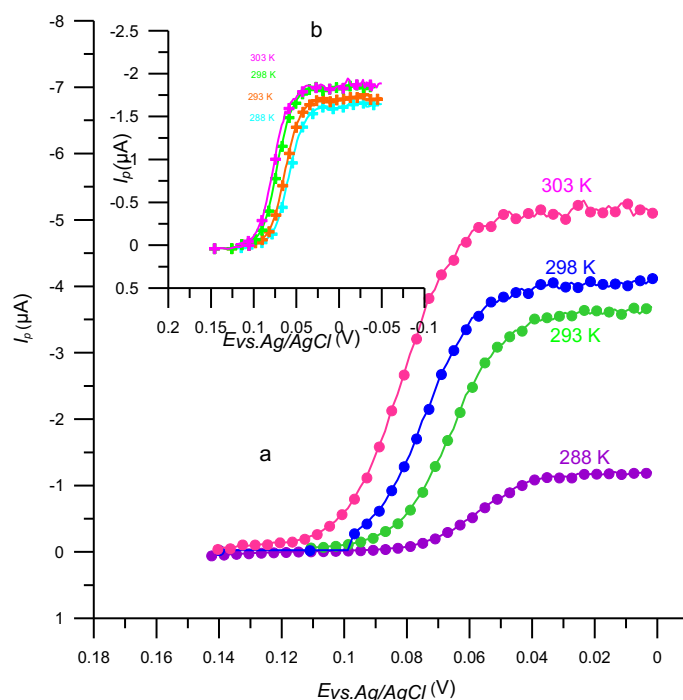


Fig. 2. (a) The DC polarograms of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ Bi(III) in $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) in the presence of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine: (●) 288K, (●) 293K, (●) 298K, (●) 303K; (b) The DC polarograms of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ Bi(III) in $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) in the presence of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine with $5 \cdot 10^{-5} \text{ mol} \cdot \text{dm}^{-3}$ Tween 80: (⊕) 288K, (⊕) 293K, (⊕) 298K, (⊕) 303K

Table 1. The changes in the difference between the anodic and cathodic peak potentials ($\Delta E = E_a - E_c$) for the process of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ Bi(III) ion electroreduction in $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) in the presence of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine and increasing concentrations of the surfactants; polarization rate $v = 50 \text{ mV} \cdot \text{s}^{-1}$

		$\Delta E/V$							
$10^6 c_{\text{surfactant}} / \text{mol} \cdot \text{dm}^{-3}$	Triton X-100				Tween 80				
	288K	293K	298K	303K	288K	293K	298K	303K	
0	0.0281	0.0272	0.0260	0.0253	0.0281	0.0272	0.0260	0.0253	
10	0.0301	0.0299	0.0297	0.0295	0.0347	0.0345	0.0343	0.0340	
20	0.0306	0.0304	0.0300	0.0297	0.0348	0.0347	0.0345	0.0343	
50	0.0314	0.0311	0.0307	0.0302	0.0352	0.0349	0.0347	0.0345	
100	0.0330	0.0328	0.0325	0.0322	0.0366	0.0363	0.0360	0.0356	

In order to perform a more detailed analysis of temperature effect on the electrode process in the examined systems, the values $\Delta E = E_a - E_c$, were included. As can be seen from Table 1, the values ΔE for $v = 50 \text{ mV} \cdot \text{s}^{-1}$ depend evidently on the temperature. A clear decrease in the value is observed with the increasing temperature in all tested systems, which indicates an increase in the reversibility of the electrode process. However, it should be noted that in the systems where the mixed adsorption layers of 6-thioguanine - Tween 80 and 6-thioguanine - Triton X-100 are formed (Nosal-Wiercińska et al. 2019), this effect of temperature on changes in the rate of Bi(III) ion electroreduction are definitely smaller. These observations show the changes in the dynamics of the process acceleration as the temperature increases, which seem to be the biggest in the presence of 6-thioguanine.

3.2. Kinetic and thermodynamic studies

The dependence of the reaction rate constant on the temperature is described by the Arrhenius equation (Mortimer, Taylor, 2002). It should be noted that the determined rate constants satisfy this equation.

As can be seen from Tables 2 and 3, the values of standard rate constants k_s increase with the temperature rise $288\text{K} < 293\text{K} < 298\text{K} < 303\text{K}$. However, it should be noted that the increase in these values in the presence of 6-thioguanine is definitely larger compared to the increase obtained for the mixtures of 6-thioguanine – Triton X-100 as well as 6-thioguanine – Tween 80. The gradual increase in the temperature causes that the acceleration effect loses its dynamics when surfactants are present in the basic electrolyte containing already 6-thioguanine (Nieszporek and Nieszporek, 2018).

Table 2. The values of standard rate constants (k_s) of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ Bi(III) ion electroreduction in $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) and in the presence of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine

$10^3 c_{6\text{-thioguanine}} / \text{mol} \cdot \text{dm}^{-3}$	$10^3 k_s / \text{cm} \cdot \text{s}^{-1}$			
	288K	293K	298K	303K
0	0.167	0.187	0.197	0.243
0.1	2.000	2.343	2.987	3.870
0.5	3.567	5.799	6.998	7.987
1.0	5.657	6.760	9.760	11.56
3.0	7.876	10.21	14.87	17.98
5.0	8.987	11.32	16.76	19.87

Table 3. The values of standard rate constants (k_s) of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ Bi(III) ion electroreduction in $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) and in the presence of $1 \cdot 10^{-3} \text{ mol} \cdot \text{dm}^{-3}$ 6-thioguanine and increasing concentrations of the surfactants

$10^6 c_{\text{surfactant}} / \text{mol} \cdot \text{dm}^{-3}$	$10^3 k_s / \text{cm} \cdot \text{s}^{-1}$							
	TritonX-100				Tween 80			
	288K	293K	298K	303K	288K	293K	298K	303K
0	5.657	6.760	9.760	11.560	5.657	6.760	9.760	11.560
10	5.650	6.000	9.755	10.120	5.000	8.650	8.965	9.890
20	5.560	5.679	9.680	10.001	4.680	4.987	5.678	7.899
50	5.468	5.500	9.540	9.887	4.004	5.120	5.306	6.430
100	4.876	5.000	6.320	9.765	3.000	3.050	4.021	5.021

As it results from Table 4 after the addition of 6 - thioguanine to the supporting electrolyte, the value of activation energy ΔH^\ddagger decreases significantly. This confirms the increase in the reversibility of the Bi(III) ion electroreduction process in the $2 \text{ mol} \cdot \text{dm}^{-3}$ chlorates(VII) (Pérez et al., 1995; Nosal-Wiercińska, 2010; Nosal-Wiercińska, 2011). With the increase in the concentration of 6TG large reduction in the value of the ΔH^\ddagger is observed. On the other hand, in the presence of both surfactants, the values of ΔH^\ddagger (Table 5) increase which confirm the changes in the kinetics of the electrode process towards inhibition (Kaliszczak, Nosal-Wiercińska, 2018). With the increasing concentration of the surfactants the values of ΔH^\ddagger still increase. However, it should be noted that these changes are definitely smaller compared to those for the solutions containing only 6-thioguanine.

An evident decrease in the value of $\Delta S_{\text{Bi(III)}/\text{Bi(Hg)}}^0$ (Table 4 and 5) suggests changes in the dynamics of the of Bi(III) ion electroreduction process in the presence of 6 - thioguanine and both surfactants Tween 80 or TritonX - 100 (Mianowski et al., 2018). Definitely lower values of the standard reaction entropy in the presence of surfactants indicate an evident change in the arrangement of the system, which involves changes in the process dynamics and reversible mechanism of the Bi(III) ion electroreduction. This is probably connected with the change of the film of surfactant and active

complex adsorbed on the electrode surface (Marcus and Loewenschuss, 1984; Rosen and Kunjappu, 2012) Triton X-100 and Tween 80 change the dynamics of the catalytic activity of thiopurine drug towards Bi(III) ions electroreduction with the tendency of inhibition (Kaliszczak and Nosal-Wiercińska, 2018).

Table 4. The values of standard activation energy (ΔH^\ddagger) and standard reaction entropy (ΔS^0) for Bi(III) ion electroreduction in 2 mol·dm⁻³ chlorates(VII) and in the presence of 1·10⁻³ mol·dm⁻³ 6-thioguanine

10 ³ C _{6-thioguanine} /mol·dm ⁻³	ΔH^\ddagger /kJ·mol ⁻¹	$\Delta S^0_{\text{Bi(III)/Bi(Hg)}}$ /J·mol ⁻¹ ·K ⁻¹
0	31.68	337.7
0.1	28.45	326.3
0.5	19.34	299.5
1.0	10.12	267.4
3.0	8.320	254.5
5.0	4.654	202.0

Table 5. The values of standard activation energy (ΔH^\ddagger) and standard reaction entropy (ΔS^0) for Bi(III) ion electroreduction in 2 mol·dm⁻³ chlorates(VII) and in the presence of 1·10⁻³ mol dm⁻³ 6- thioguanine and increasing concentrations of the surfactants

10 ⁶ C _{surfactant} /mol·dm ⁻³	ΔH^\ddagger /kJ·mol ⁻¹		$\Delta S^0_{\text{Bi(III)/Bi(Hg)}}$ /J·mol ⁻¹ ·K ⁻¹	
	TritonX-100	Tween 80	TritonX-100	Tween 80
0	10.12	10.12	267.4	267.4
10	25.23	24.25	172.4	136.3
20	35.43	28.54	93.1	72.2
50	52.34	49.32	57.1	50.3
100	78.32	68.21	39.2	31.3

4. Conclusions

The presented measurements concerning the process of the Bi(III) ions electroreduction in the presence of 6-thioguanine as well as Triton X-100 and Tween 80 in the 2 mol·dm⁻³ chlorates(VII) demonstrate a dependence of the process rate on temperature. However, the standard k_s rate constants determined from the cyclic voltammetry curves, indicate a catalytic effect of 6-thioguanine and changes in its size due to the presence of the 6TG - Triton X-100 and 6MP - Tween 80 mixture as well as a change in temperature at which the experiment was performed.

Changes in the electrode reaction rates are strictly connected with those in the activation energy (ΔH^\ddagger) or standard reaction entropy ($\Delta S^0_{\text{Bi(III)/Bi(Hg)}}$) of the electroreduction process (Kaliszczak and Nosal-Wiercińska, 2018).

The addition of 6-thioguanine to the supporting electrolyte causes a significant decrease of the activation energy value in 2 mol dm⁻³ chlorates(VII) solution, so the electrode process speeds up. In turn, in the presence of both surfactants, the values of ΔH^\ddagger increase which suggests changes in the kinetics of the electrode process towards inhibition. However, definitely smaller changes of ΔH^\ddagger value compared to those obtained for the solutions containing only 6-thioguanine indicate changes in the mechanism of Bi(III) ions electroreduction in the presence of 1·10⁻³ mol dm⁻³ 6-thioguanine and both surfactants.

An evident decrease in the $\Delta S^0_{\text{Bi(III)/Bi(Hg)}}$ value also confirms changes in the dynamics of the Bi(III) ions electroreduction process in the presence of 1·10⁻³ mol dm⁻³ 6-thioguanine and both surfactants.

As results from the foregoing studies (Kaliszczak and Nosal-Wiercińska 2019), the catalytic influence of 6-thioguanine on the electroreduction of Bi(III) ions in chlorates(VII) is connected with the formation of active complexes of Bi - 6-thioguanine. The molecules of surfactants, present additionally in the basic electrolyte solution, start to block the electrode surface pushing out the earlier formed Bi - 6TG active complexes from the adsorption layer. Therefore, the mechanism of the electrode process changes due

to the changes in the arrangement of the system (significant differences in the $\Delta S_{\text{Bi(III)/Bi(Hg)}}^0$ values in the presence of Triton X – 100 and Tween 80).

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