

Adsorption of cystine at mercury/ aqueous solution of chlorate (VII) interface in solutions of different water activity

Research Article

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Abstract: Adsorption of cystine on mercury electrode from 1 to 8 mol dm⁻³ chlorates (VII) was studied. The data obtained from the measurements of differential capacity of the double layer were used, namely the measurements of zero charge potential and surface tension at the zero charge potential. The values of the relative surface excess increase along with the chlorate (VII) and cystine concentrations. The adsorption parameters were obtained from Frumkin and virial isotherms. The changes of these parameters in function of the supporting electrolyte concentration indicate the competitive adsorption of cystine and ClO₄⁻ ions as well as considerable participation of water molecules in adsorption – desorption processes.

Keywords: Adsorption • Amino acids • Chemisorption • Electron transfer • Adsorption isotherms
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1. Introduction

Cystine is a protein amino acid containing sulphur in its molecule. This amino acid is formed as a result of coupling of two cysteine molecules by the disulphur bond. Cystine is a very important compound from a biological and medical point of view. It partakes in the synthesis of insulin and serum proteins, as well as in sulphur supply to the metabolic processes. It is also a component of keratin that is present in hair and nails [1]. Cystine forms a class of atoxic organic compounds which are fully soluble in aqueous media and can be produced with high purity at low cost [2]. These features of cystine and other amino acids justify their use as a corrosion inhibitor of different metals such as copper [3,4], steel [5] or iron [6].

The first publications on cystine by Kolthoff *et al.* [7,8] as well as more recent ones [9-14] where the involvement of electrochemical reactions of cysteine and cystine on mercury electrodes are worth noting. The studies of Stankovich and Bard [15] proved that

in the reaction of cystine with sulphur the cystine adsorption plays the key role. The research shown that the adsorbed monomolecular layer of cystine was involved in the cathodic reaction, and that in the anodic reaction of cysteine a monolayer of the product, a mercury and cysteine compound. This product was made at the electrode. Heyrovsky *et al.* [16] broadened the results mentioned above taking into consideration their own investigations into cystine adsorption and the reactions proceeding at the mercury electrode. Cystine in aqueous solutions come into chemically reaction with mercury, forming the surface-bound cysteine mercuric thiolate Hg(SR)₂. When the potential of mercury is more negative this compound is transformed into cysteine mercurous thiolate Hg₂(SR)₂. Cysteine mercurous thiolate is also formed in a reversible interaction of cystine with mercury in the wide range of electrode negative potentials. In this range of potentials cystine molecule is adsorbed by its central hydrophobic part (-CH₂-S-S-CH₂-). During the moving towards the positive potentials there is a reaction of chemisorptions.

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Where the two S atoms approach one Hg atom the charge transfer occurs [17].

According to Monterroso-Marco and López-Ruiz [17] the adsorption of thiol and disulphide groups at the mercury surface depends on the charge of the molecule, which in turn, is a consequence of the pH. In cystine solutions, the compact film appears only when pH values lie between the pKa 2 and pKa 3. At these pH values cystine carries a zero net charge.

This paper presents the results of studies on the effect of cystine (RSSR) on adsorption parameters of the double layer at the mercury/ chlorate (VII) interface in solutions of different water activity.

The choice of chlorate (VII) solution results from the fact that ClO_4^- ions cause the strongest disruption in water structure [18].

Our previous studies on methionine [19] and cysteine [20] confirmed the considerable influence of these substances and water activity on the adsorption parameters of the double layer at the mercury/ chlorate (VII) interface. In all the systems examined the relative surface excesses of methionine or cysteine compounds were found to increase with the increase of electrode charge and adsorbate concentration; they were also found to depend on chlorates' (VII) concentration. Moreover, the adsorption parameters were found to change together with the change of the supporting electrolyte concentration, which points to the competitive adsorption of methionine or cysteine compounds and ClO_4^- ions.

The results of studies will be helpful in determination of the mechanism of catalytic activity of cystine on the electroreduction of Bi(III) ions in chlorates (VII).

Studies on such reactions give information about the electron transfer in heterogeneous processes and may also be useful in developing some of practical applications, such as the protection of metals and semiconductors against corrosion. These processes may also serve as simple models of ions transport in biological membranes.

2. Experimental procedure

Analytical - grade reagents: cystine (Fluka), NaClO_4 (Fluka) and HClO_4 (Fluka) were used without further purification. The solutions were prepared from freshly double distilled water. Before measurements the solutions were deaerated using high purity nitrogen. Nitrogen was passed over the solution during the measurements.

The supporting electrolytes were $x \text{ mol dm}^{-3} \text{ NaClO}_4 + 1 \text{ mol dm}^{-3} \text{ HClO}_4$ (where $0 \leq x \leq 7$).

As the NaClO_4 concentration increases from 0 to 7 mol dm^{-3} the water activity decreases from 0.962 in $1 \text{ mol dm}^{-3} \text{ HClO}_4$ to 0.543 in $7 \text{ mol dm}^{-3} \text{ NaClO}_4 + 1 \text{ mol dm}^{-3} \text{ HClO}_4$ [21].

The cystine solutions were prepared just before the measurements. The concentrations of cystine were in the range of $0.5\text{--}80 \times 10^{-3} \text{ mol dm}^{-3}$.

Measurements were carried out in thermostated cells at 298 K using an AUTOLAB electrochemical analyzer controlled by GPES software (Version 4.9) (Eco Chemie, Utrecht Netherlands). Experiments were performed in a cell with a hanging controlled-growth mercury drop electrode (CGMDE) (Entech, Cracow, Poland) as the working electrode (electrode area = 0.009487 cm^2 , drop time = 3 s). Ag/AgCl/saturated NaCl and a platinum spiral served as the reference and auxiliary electrodes.

The double layer capacity (C_d) was measured using the AC impedance technique with an Autolab electrochemical analyzer (as described above). The reproducibility of the average capacity measurements was $\pm 0.5\%$. For the whole polarization range, the capacity dispersion was tested at different frequencies between 200 and 1000 Hz. The equilibrium capacities were obtained by extrapolation of the measured capacity versus square root of the frequency to the zero frequency [22]. The potential of zero charge (E_z) was measured for each solution by the method of streaming mercury electrode [23,24], with an accuracy of $\pm 0.1 \text{ mV}$.

The surface tension at potential of zero charge (γ_z) was determined using the method of highest pressure inside the mercury drop presented by Schiffrin [25]. The surface tension values were determined with an accuracy of $\pm 0.2 \text{ m Nm}^{-1}$. The charge density and surface tension were derived by the back - integration of differential capacity – potential dependencies. No correction for the effects of the medium on the activity of the chlorate (VII) electrolyte [26,27] and the activity coefficient of the cystine [28] were made.

3. Results and discussion

3.1. Analysis of experimental data

The introduction of cystine to the supporting electrolyte solution (Fig. 1) results in an increase of differential capacity in the entire potential range examined. With the increase of cystine concentration the differential capacity increases and the “hump” appearing on the curve is shifted towards negative potentials. Moreover, above the cystine concentration of $5 \times 10^{-3} \text{ mol dm}^{-3}$ at the potential approximately - 0.85 V an additional peak is formed, which increases with the increase of the

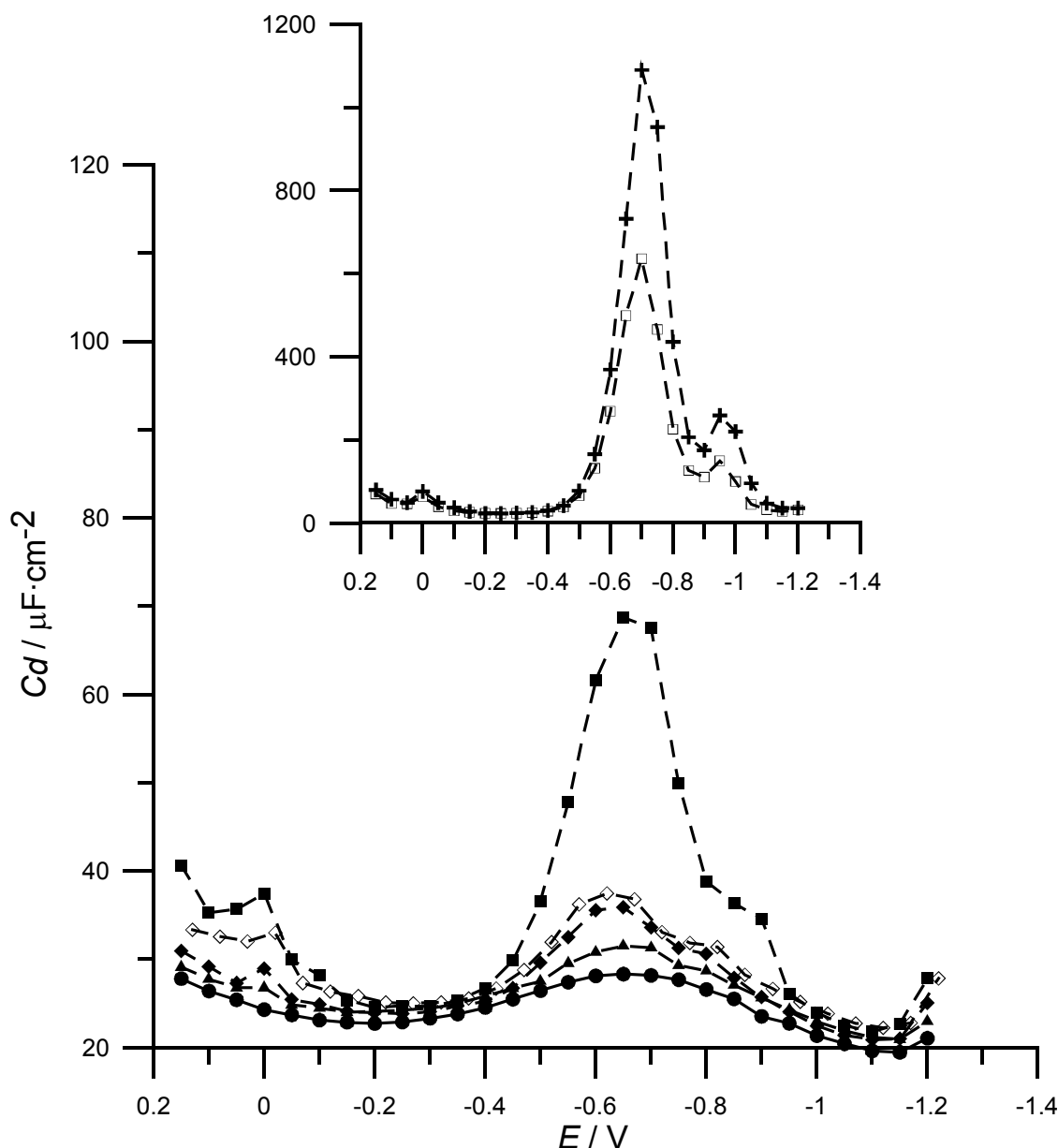
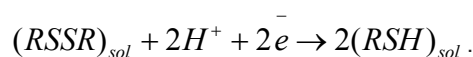


Figure 1. Differential capacity – potential curves of the mercury electrode in 4 mol dm⁻³ chlorate (VII) with various concentrations of cystine (in mol dm⁻³): 0 (●), 8×10⁻⁴ (▲), 5×10⁻³ (◆), 8×10⁻³ (◇), 1×10⁻² (■), 5×10⁻² (□), 8×10⁻² (+).

adsorbate concentration and is shifted towards negative potentials. It should also be observed that above the cystine concentration of 8×10⁻³ mol dm⁻³ at the potential approximately -0.6 V the “hump” increases sharply with the increase of the cystine concentration and is transformed into a peak. At the potential approximately 0 V at $C_d = f(E)$ curves the some small peaks appear, the heights of which increase with the increase of cystine concentration.

This figure of the capacity curves points to complicated effects proceeding at the Hg/ chlorate (VII) solutions interface in the presence of cystine. It

also confirms the results obtained by Heyrovsky *et al.* [16], which suggest that cystine is strongly adsorbed on mercury. According to [16] in acid solutions in the range of positive potentials between the first reduction wave and the anodic wave mercury is positively charged because of chemisorption and that is why the cationic forms of cystine cause a smaller increase of differential capacity in comparison with the neutral or anionic forms. The potential range between -0.5 V and -0.9 V is due to the process:



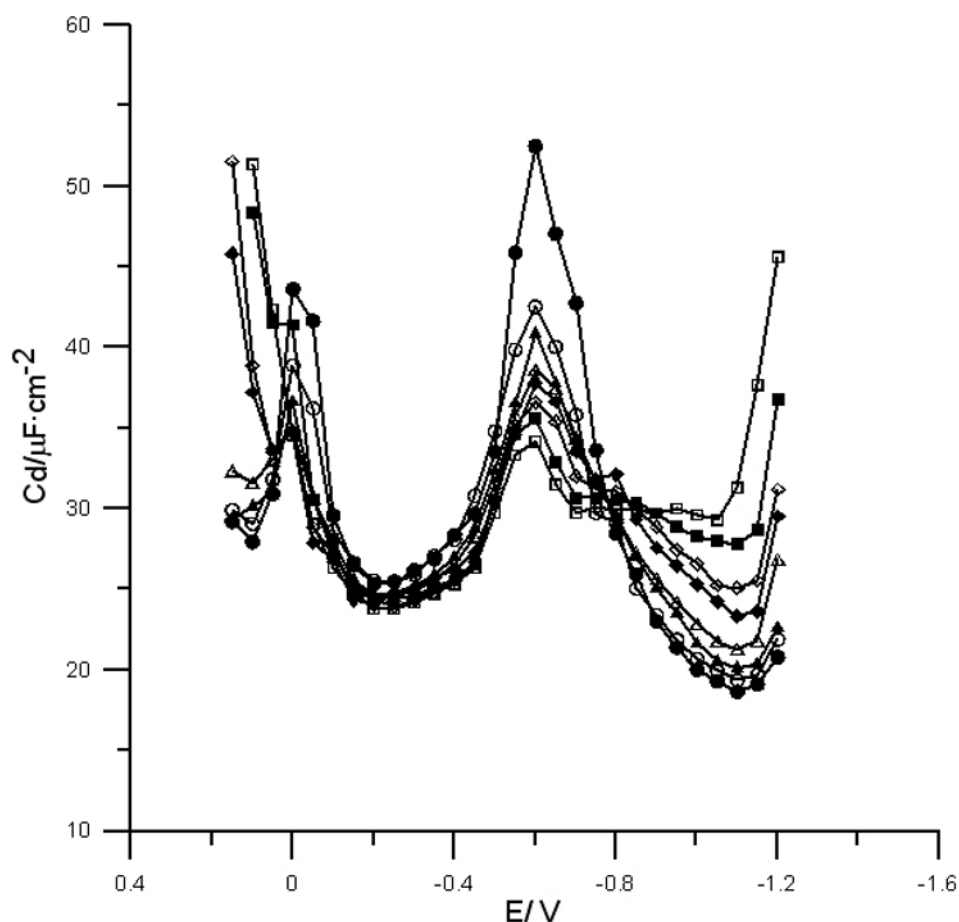
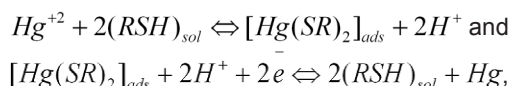


Figure 2. Differential capacity – potential curves of the mercury electrode in 1-8 mol dm⁻³ chlorates (VII): 1 (●), 2 (○), 3 (▲), 4 (Δ), 5 (◆), 6 (◇), 7 (■), 8 (□) with 8 · 10⁻³ mol dm⁻³ cystine.

A part of dissolved RSSR is in reaction with the mercury electrode and these molecules also facilitate the oxidation of mercury, allowing the reactions:



to occur. The overall process can be shown by the following chemical reaction:



(where “ads” and “sol” denote, respectively, the species adsorbed at the electrode or in solution) [17].

These peaks, which are present on the differential capacity curves represent the adsorption of the products of anodic mercury oxidation by cystine. The first peak in differential capacitance measurement is attributed to $\text{Hg}(\text{SR})_2$ and the second peak is attributed to the $\text{Hg}_2(\text{SR})_2$.

At constant cystine concentration with the increase of the supporting electrolyte concentration a decrease

of the peaks at $C_d = f(E)$ curves is observed as well as their slight shift towards more positive potentials (Fig. 2). Such changes indicate a considerable influence of water on the surface properties of the interface. As not all of the obtained curves $C_d = f(E)$ converge at sufficiently negative potentials with the corresponding curve for the base solution, the capacity versus the potential data were numerically integrated from the point of E_z . The integration constants are presented in Fig. 3 and in Table 1. The linear dependences $E_z = f(c_{\text{cystine}})$ (Fig. 3) obtained for all the chlorates (VII) concentrations examined point to the specific adsorption of cystine on mercury [29]. Most probably the adsorbed cystine has not the same behaviour as a passive dielectric, which, when squeezed out of the bulk into the interface, pushes apart the opposing charges of the double layer, but interacts specifically with the metal in a way that consumes the positive charge. It can be understood as the specific interaction occurs through the twin sulphur atoms of cystine donating their free electron pairs to neighbouring mercury atoms [16].

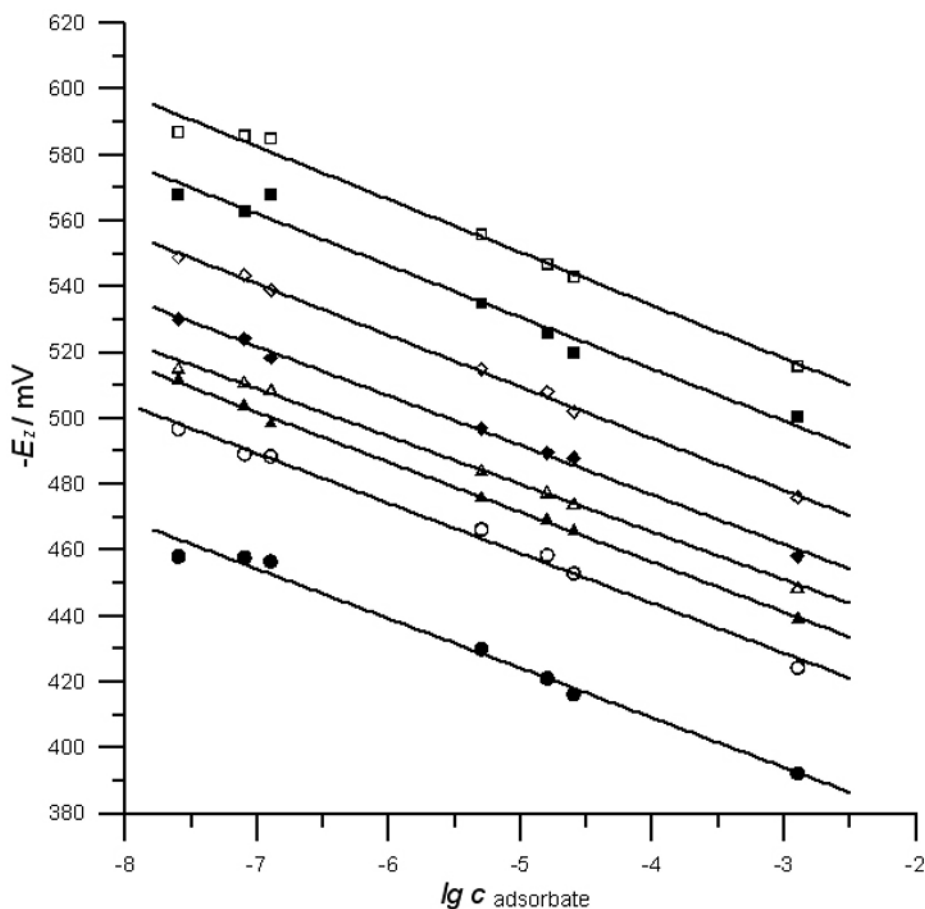


Figure 3. Dependence of the potential of zero charge E_z vs $\lg c_{\text{adsorbate}}$ for cystine-chlorates (VII) systems. Concentrations of chlorate (VII) (in mol dm^{-3}): 1 (●), 2 (○), 3 (▲), 4 (△), 5 (◆), 6 (◇), 7 (■), 8 (□).

Fig. 4 presents the determined electrocapillary curves in 6 mol dm^{-3} chlorates (VII) in the presence of selected cystine concentrations. The increase of cystine concentration causes a decrease of the surface tension at the cathodic part of the electrocapillary curve. Besides, with the increase of cystine concentration the maximum of the curves is shifted towards more positive potentials, which confirms the changes of E_z values measured directly at the streaming electrode. It should also be noticed that for the basic solutions quite the opposite dependences are observed [16].

At constant cystine concentration with a decrease of water activity a decrease of the mercury surface tension is observed as well as a shift of the electrocapillary curve maximum towards negative potentials (Fig. 5).

The data obtained from the integration of differential capacity curves were subsequently used to calculate Parson's auxiliary function ξ , $\xi = \gamma + \sigma_M E$, where σ_M is the electrode charge and E is the electrode potential [30]. Adsorption of cystine was described using the relative surface excess (Γ'), which according to the Gibbs adsorption isotherm is given by:

$$\Gamma'_{\text{cystine}} = \left(\frac{1}{RT} \right) \left(\frac{d\Phi}{d \ln c} \right)_{\sigma_M} \quad (1)$$

where c is the bulk concentration of cystine and Φ is the surface pressure: $\Phi = \Delta\xi = \xi^0 - \xi$ (ξ^0 refers to the supporting electrolyte containing cystine).

The values of Γ' obtained for the studied system depending on the surface charge, cystine concentration and water activity are presented in Figs. 6a and 6b.

The values of Γ' increase with a decrease of surface charge σ_M and an increase of cystine concentration in all the ranges of electrode charges studied (Fig. 6a). Most probably the presence of an additional sulphur atom in the cystine molecule affects its position at the electrode surface. It should be noted that the cystine surface excesses decrease with the increase of electrode charge. Such behaviour is counter to the one observed in the case of the adsorption of cysteine [20] or thiourea [31] and its derivatives [32,33].

With the decrease of water activity the Γ' values increase in the entire range of charges examined (Fig. 6b).

Table 1. Surface tension γ_z / m N m⁻¹ for E_z of 1 - 8 mol dm⁻³ chlorate(VII) + cystine systems.

10 ³ c _{cystine} / mol dm ⁻³	γ_z / m N m ⁻¹							
	1	2	3	4	5	6	7	8
0.0	422.5	420.7	419.8	418.1	417.5	416.5	414.6	414.0
0.5	421.9	420.1	419.2	418.1	417.4	416.1	414.7	413.9
0.8	421.3	419.8	419.0	418.0	416.8	415.5	413.8	413.3
1.0	421.0	419.7	418.8	417.9	416.2	414.9	413.6	412.7
5.0	420.2	418.4	417.3	416.4	415.2	414.1	412.5	411.7
8.0	419.7	417.9	416.8	415.9	414.8	413.4	411.9	411.1
10	419.5	417.6	416.5	415.6	414.6	413.2	411.7	410.9
50	417.7	415.7	414.5	413.6	412.1	410.7	409.4	408.4
80	417.0	415.4	414.0	412.3	410.5	409.6	408.7	407.8

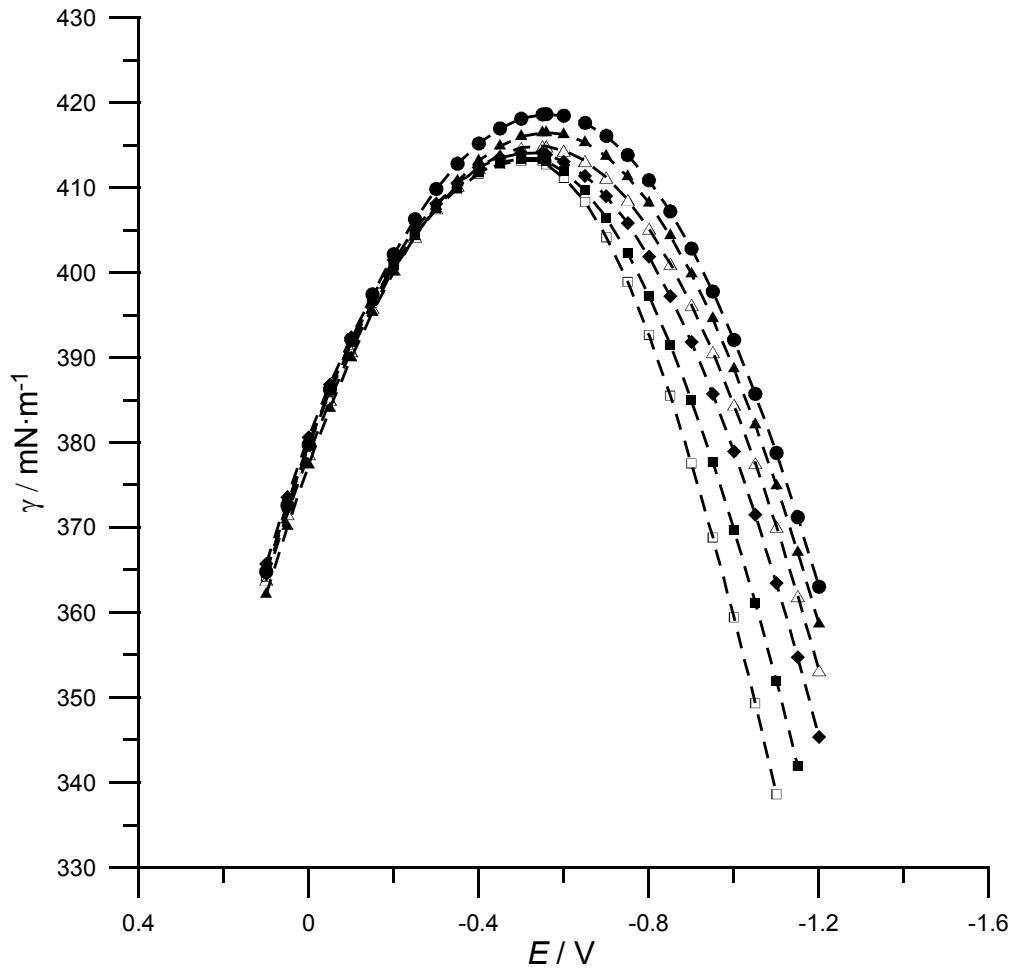


Figure 4. Electrocapillary curves in 4 mol dm⁻³ chlorate (VII) and at various concentrations of cystine (in mol dm⁻³): 0 (●), 8 × 10⁻⁴ (▲), 1 × 10⁻³ (△), 5 × 10⁻³ (◆), 1 × 10⁻² (■), 5 × 10⁻² (□).

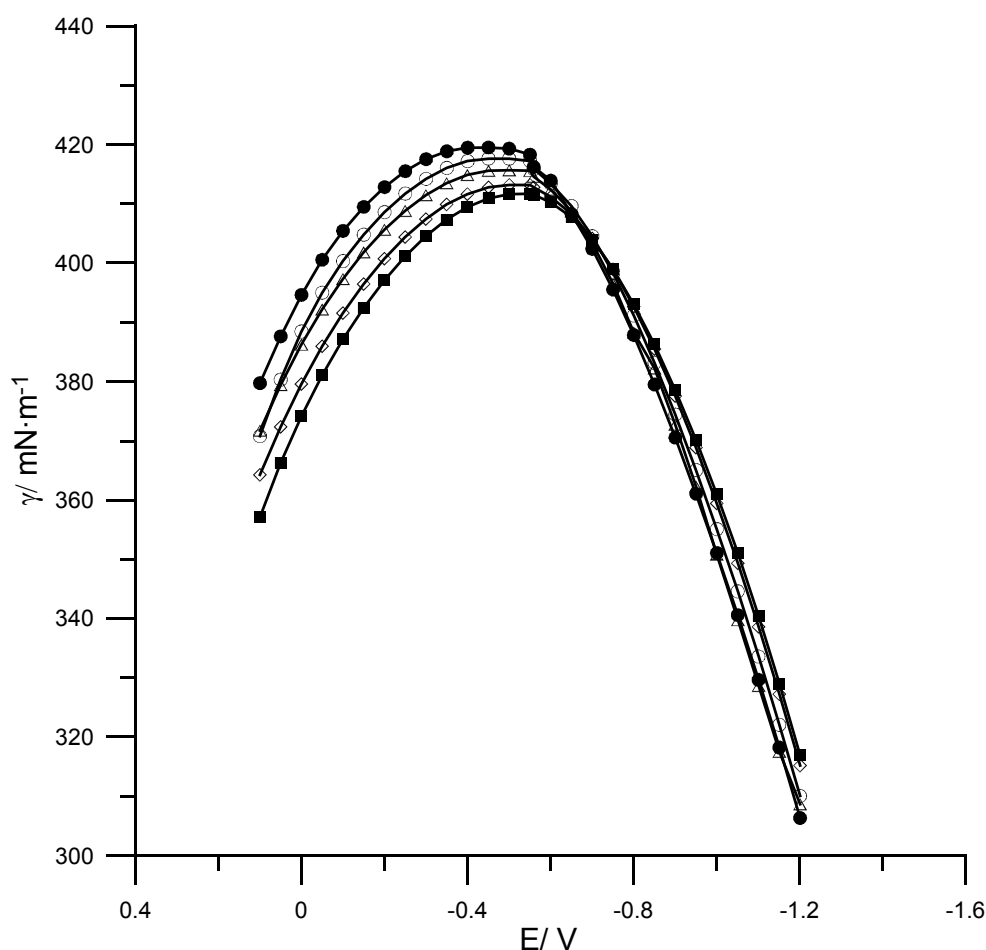


Figure 5. Electrocapillary curves of the $8 \times 10^{-3} \text{ mol dm}^{-3}$ cystine in chlorates (VII) (in mol dm^{-3}): 1 (●), 2 (○), 4 (Δ), 6 (◇), 7 (■).

Table 2. Values of the surface occupied by one adsorbate molecule at a mercury/chlorate (VII) interface (S), dependence on the chlorate (VII) concentration (in mol dm^{-3}).

$S / \text{\AA}^2$	1	2	3	4	5	6	7	8
	70.9	63.3	62.1	59.8	51.0	42.3	38.7	34.6

3.2. Adsorption isotherms

The adsorption of cystine was further analyzed on the basis of the constant obtained from the Frumkin isotherm:

$$\beta x = [\Theta / (1 - \Theta)] \exp(-2A\Theta) \quad (2)$$

where x is the molar fraction of cystine in the solution, β is the adsorption coefficient $\beta = \exp(-\Delta G^0 / RT)$, ΔG^0 is the standard Gibbs energy of adsorption, A is the interaction parameter and Θ is the coverage $\Delta\Theta = \Gamma / \Gamma_s$. The surface excess at saturation (Γ_s) was estimated by extrapolating the $1/\Gamma$ vs. $1/c_{\text{cystine}}$ at different charges to $1/c_{\text{cystine}} = 0$.

The calculated values of the surface occupied by cystine molecule $S = 1/\Gamma_s$ decrease with a decrease of water activity (Table 2), which may indicate that water

present on the electrode surface affects the obtained results of calculations. Stankovich and Bard [15] found that the area occupied by one adsorbed molecule of cystine is about $78 \text{ \AA}^2$. This would correspond to the area of the entire cystine molecule stretched into one plane.

Fig. 7 presents a linear test of the Frumkin isotherm for the 6 mol dm^{-3} chlorates (VII) concentration and selected electrode charges. For all the systems studied a linear dependence $(1 - \Theta)/\Theta = f(\Theta)$ was found.

The values of parameter A were calculated from the slopes of lines of the linear test of the Frumkin isotherm and the corresponding values of ΔG^0 were determined by extrapolation of the line $\ln[(1 - \Theta)x/\Theta]$ versus Θ to the value $\Theta = 0$.

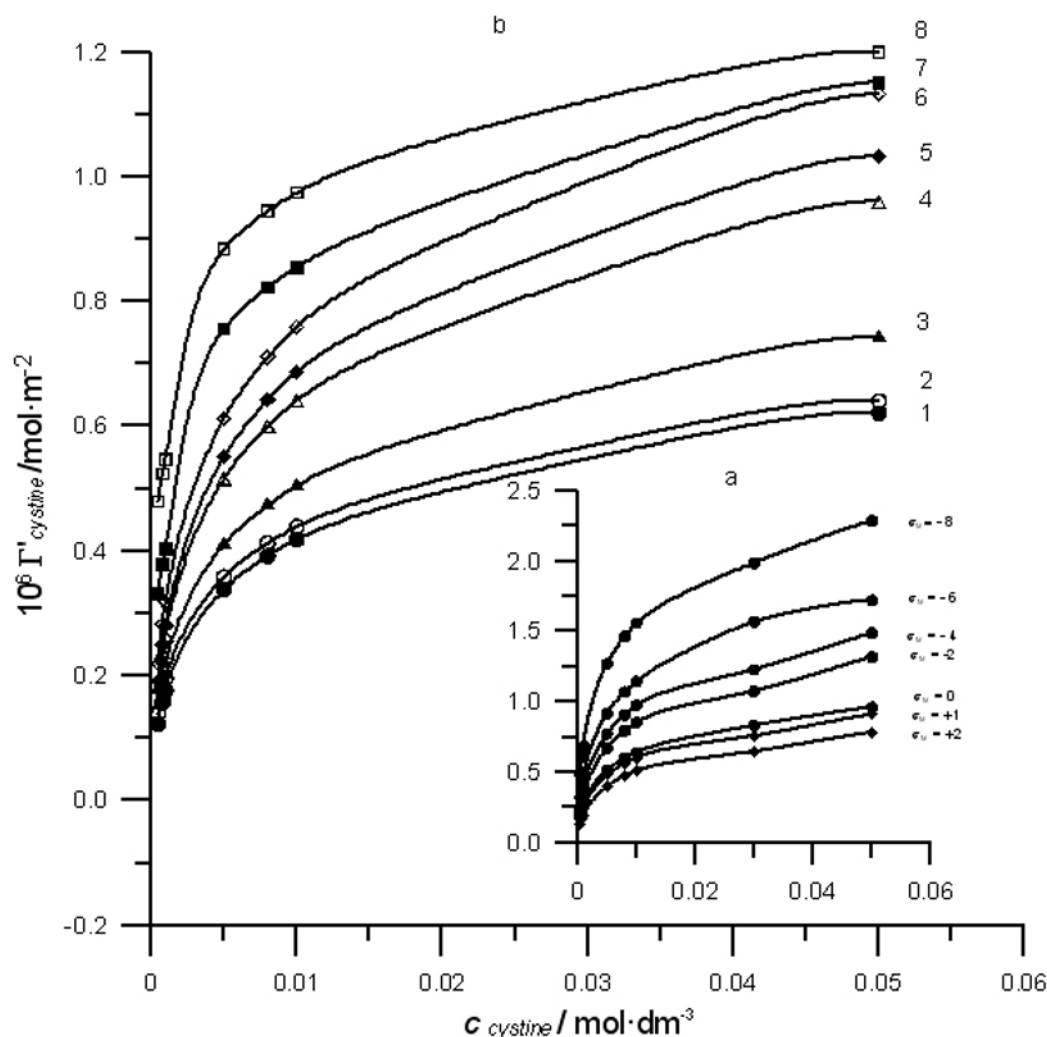


Figure 6. a) Relative surface excess Γ' of cystine against cystine concentration and electrode charge in the bulk for 4 mol dm⁻³ chlorate (VII). Electrode charges σ_M (in 10⁻² C m⁻²) are indicated by each line. b) Relative surface excess Γ' of cystine against cystine concentration in the bulk for various concentrations of chlorates (VII) (in mol dm⁻³) are indicated by each curve. The electrode charge $\sigma_M = 0$ (in 10⁻² C m⁻²).

The changes of adsorption energy ΔG^0 and A values obtained for the Frumkin isotherm as a function of the concentration of chlorate (VII) and electrode charge confirm analogous changes (ΔG^0 and B) obtained for the virial isotherm. The virial isotherm verifies the results obtained from the Frumkin isotherm, because the calculation of the values of surface excess at saturation is not required in this case [34,35]. Fig. 8 presents the linear test of the virial isotherm for the chosen chlorate (VII) concentrations and chosen electrode charges. The values of the 2D second virial coefficient B (Table 3) were calculated from the slope of the lines of the virial isotherm, and the corresponding ΔG^0 values (Table 4) were obtained from the intercepts of those lines with the axis $\log(\Gamma'/c)$.

As results from Table 3, the values of B parameter decrease with a decrease of electrode charge for all the examined chlorates (VII) concentrations. With the increase of chlorates (VII) concentration from 1 mol dm⁻³ to 5 mol dm⁻³ the values of B decrease in the entire range of charges examined. Further increase of the supporting electrolyte concentration from 6 mol dm⁻³ to 8 mol dm⁻³ results in an increase of the values of B parameter. It should be emphasized that with the decrease of σ_M in 1 mol dm⁻³ - 5 mol dm⁻³ chlorates (VII) the repulsive interaction between the adsorbed cystine molecules decreases. The increase of ΔG^0 value with the increase of negative electrode charge for all examined systems might be related to the extraction of water molecules from the electrode surface.

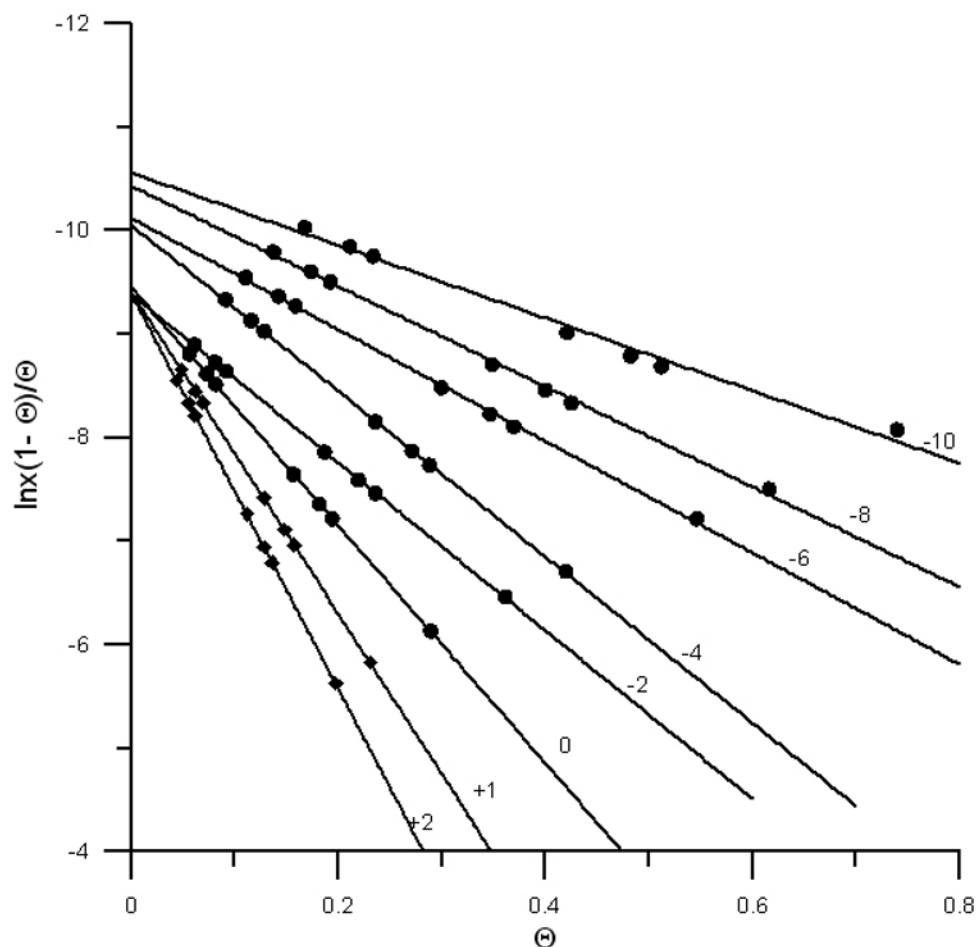


Figure 7. A linear test of the Frumkin isotherm for the system 6 mol dm⁻³ chlorate (VII) + cystine. Electrode charges σ_M (in 10⁻² C m⁻²) are indicated by each line.

Table 3. The values of the second virial coefficient (B) for the chosen charges for 1-8 mol dm⁻³ chlorates (VII) systems containing cystine.

$10^2 \sigma_M / \text{Cm}^{-2}$	$B / \text{nm}^2 \text{ molecule}^{-1}$							
	1	2	3	4	5	6	7	8
-8	1.97	1.14	1.12	1.11	1.10	1.19	1.26	1.29
-6	2.22	1.22	1.50	1.50	1.45	1.27	1.47	1.48
-4	3.13	1.87	1.78	1.66	1.64	1.72	1.88	1.99
-2	4.27	2.33	2.37	1.82	1.80	2.38	2.90	2.94
0	4.32	4.41	3.73	2.72	2.50	2.98	3.63	4.02
1	5.29	4.53	4.51	2.79	2.70	3.33	5.26	5.54
2	7.18	4.79	4.70	3.05	2.93	3.69	6.15	6.93

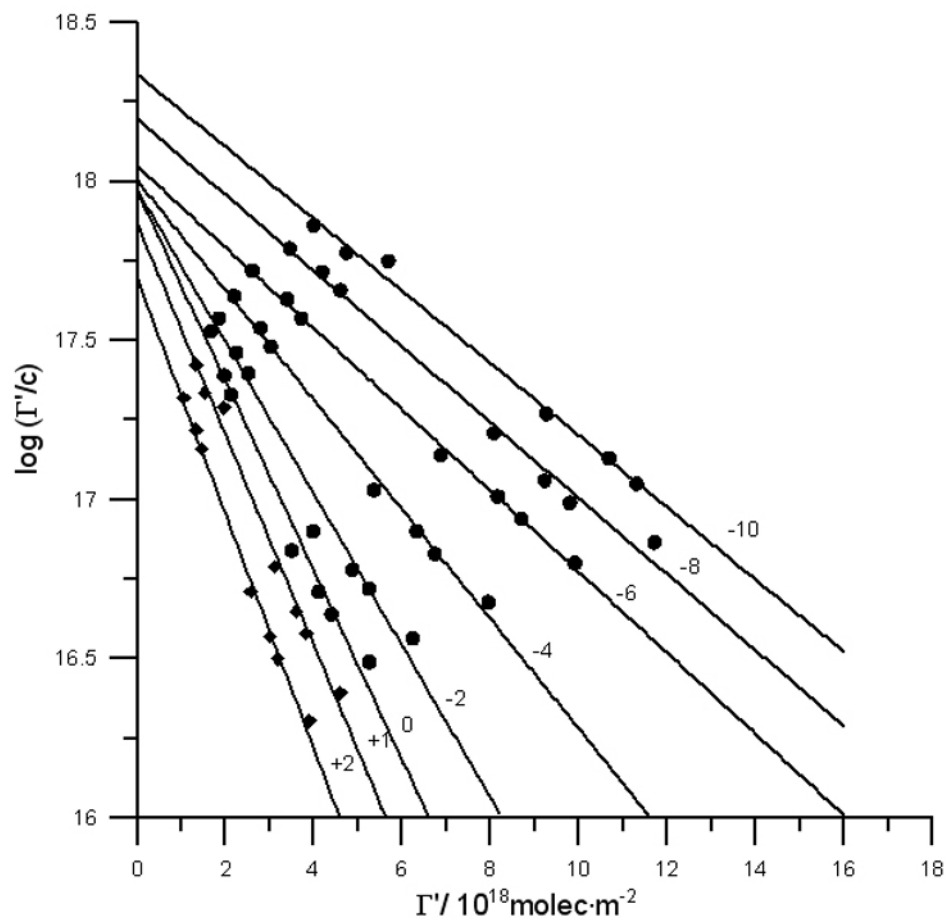


Figure 8. A linear test of the virial isotherm for the system 6 mol dm⁻³ chlorate (VII) + cystine. Electrode charges σ_M (in 10⁻² C m⁻²) are indicated by each line.

Table 4. The values of adsorption energy (ΔG°) obtained from virial isotherm for the chosen charges for 1-8 mol dm⁻³ chlorates (VII) systems containing cystine.

$10^2 \sigma_M / C m^{-2}$	$-\Delta G^\circ / kJ mol^{-1}$							
	1	2	3	4	5	6	7	8
-8	103.76	103.99	104.00	104.36	104.47	104.48	104.79	105.70
-6	102.16	102.60	102.67	102.76	102.77	102.94	103.08	103.53
-4	101.54	101.82	101.87	101.89	101.99	102.68	102.76	102.82
-2	101.42	101.48	101.51	101.59	101.70	102.50	102.51	102.22
0	99.77	100.34	100.45	100.62	100.68	102.45	102.49	102.50
1	99.25	99.43	99.60	100.22	100.39	101.88	101.95	101.96
2	97.88	98.34	99.37	99.38	99.39	100.91	101.00	101.65

4. Conclusions

Cystine is adsorbed specifically at the interface the mercury/ chlorates (VII) solution.

The surface excess of cystine increases with the increase of cystine concentration and the increase of electrode charge as well as of the supporting electrolyte concentration. The adsorption form of cystine is a product of simple fission of – S – S – bond by which the molecule is adsorbed on Hg.

The changes of the determined adsorption parameters against increasing concentration of the

supporting electrolyte point at the increase of cystine adsorption on mercury. The presence of – S – S – bridge in the cystine molecule causes its slightly different behaviour at the electrode surface in comparison with compounds like cysteine, methionine or thiourea and its derivatives. These changes are particularly evident if they are considered in function of electrode charge and activity. It seems that such behaviour of cystine results from the specific distribution of charges in a molecule, which differs from that observed in the molecules of compounds with only one sulphur atom in their structure.

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