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Characteristic of the $Ag(bipy)_2^{2+}/Ag(bipy)_2^{+}$ and $Ag(phen)_2^{2+}/Ag(phen)_2^{+}$ Systems in Acetonitrile

Maksymilian Ignaczak and **Andrzej Grzejdziak**

Department of General and Inorganic Chemistry, University of Łódź,
PL-90-136 Łódź, Poland

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The values of standard potentials of redox systems formed by the complexes of Ag(II) and Ag(I) with 2,2′-bipyridine and 1,10-phenanthroline in acetonitrile have been determined. The properties of the above systems in water and acetonitrile are compared. The possibility of application of these systems for the construction of electrodes with a constant potential in different solvents is discussed.

(Keywords: Acetonitrile; Redox systems; Silver complexes)

Charakteristik der Systeme $Ag(bipy)_2^{2+}/Ag(bipy)_2^{+}$ *und* $Ag(phen)_2^{2+}/Ag(phen)_2^{+}$ *in Acetonitril*

Die Standardpotentialwerte der Redoxsysteme, die durch Komplexbildung von Ag(II)- und Ag(I)-Ionen mit 2,2′-Bipyridin und 1,10-Phenantrolin gebildet werden, wurden in Acetonitril bestimmt. Die Eigenschaften dieser Redoxsysteme in Wasser und Acetonitril wurden verglichen.

Es wurde weiterhin die Möglichkeit der Anwendung dieser Systeme zur Konstruktion einer Elektrode mit unveränderbarem Potential in verschiedenen Lösungsmitteln diskutiert.

Introduction

In the process of oxidation of organic compounds the Ag(II)/Ag(I) system is one of most commonly used. Owing to its high standard potential ($E^\circ = 1.98\,V$) an oxidized compound is often destroyed. A modification of this system by complexing the two oxidation states leads to a decrease of the standard potential and the possibilities of syntheses become wider. Of particular use are ligands stabilizing Ag(II) ions, such as 2,2′-bipyridine, 1,10-phenanthroline, quinoline and picolinic acid [1–7].

Additional possibilities of syntheses also emerge from the use of the above systems in non aqueous solvents. In the last years numerous studies on the complexing and redox systems of the type AgL_2^{2+}/AgL_2^{+} in acetonitrile and propylene carbonate were reported [8–15].

The purpose of our investigation was to characterize the $Ag(bipy)_2^{2+}/Ag(bipy)_2^{+}$ and $Ag(phen)_2^{2+}/Ag(phen)_2^{+}$ systems in acetonitrile. We have also compared the properties of these systems in water and acetonitrile.

Experimental and Results

$Ag(bipy)_2NO_3$ was obtained from 0.1 M solution of $AgNO_3$ by precipitating the complex with an excess of 2,2′-bipyridine.

The complex $Ag(bipy)_2(NO_3)_2$ was obtained according to the method of *Thorpe* and *Kochi* [2]. The purity of the final product was determined by the method described previously [6].

The complex of Ag(I) has been removed by extraction with methylene chloride.

$(C_2H_5)NClO_4$ was obtained according to the method described by *Kolthoff* [21]. The final product was triply crystallized from acetonitrile and dried under vacuum at room temperature.

$Ag(phen)_2NO_3$ and $Ag(phen)_2(NO_3)_2$ were obtained by the method described previously [7].

Acetonitrile produced by VEB Laborchemie Apolda (GDR) was purified by the method of *Coetzee* [16] and distilled over P_2O_5. The fraction collected was the one whose boiling point at $p = 760$ mm Hg was 81.6 °C. Purification was carried out until the cut-off point was reached at $\lambda = 210$ nm, with a conductivity of $2–3 \cdot 10^{-7}$ ohm^{-1} cm^{-1}.

Other analytically pure reagents, produced by POCh Gliwice were applied directly.

The apparatus used in coulometric and chronovoltamperometric measurements was described previously [6].

The Ag/0.01 M $AgNO_3$ (AN) + 0.1 M $(C_2H_5)_4NClO_4$ (AN) electrode was applied as a reference one. Its potential was assumed to be 0.532 V [17]. In chronovoltamperometric measurements the applied rates of potential sweep were in the range of 10–100 mV/s.

Coulometric Measurements

In the coulometric amperostatic titration the weighed samples of $Ag(bipy)_2(NO_3)_2$ and $Ag(phen)_2(NO_3)_2$ complexes in solutions of different ionic strengths from 0.1 to 0 have been reduced by electric current ($i = 5$ mA).

Ionic strength was formed by weighed samples of the complex and $(C_2H_5)_4NClO_4$.

The values of the potentials of redox systems were determined by a measurement of EMF of the following cell:

$$Ag\,|\,0.01\,M\ AgNO_3(AN) + 0.1\,M\ (C_2H_5)_4NClO_4(AN)\ \|\ \mu\,(C_2H_5)_4NClO_4(AN)\ \|$$
$$x\,AgL_2NO_3(AN) + y\,AgL_2(NO_3)_2(AN) + z(C_2H_5)_4NClO_4(AN)\,|\,Pt$$

where L is 2,2′-bipyridine or 1,10-phenanthroline and x, y, z are the concentrations determining the total ionic strength.

The measurements were made after earlier anodic oxidation ($i = 1$ mA) of solutions of Ag(II) complexes to the maximum possible *EMF* values in a given system.

The obtained curves $EMF = \mathrm{f}(t)$ were similar to the ones of the redox titration. The E° values of systems have been obtained by extrapolation of the E_f° values to $\mu = 0$. The results are given in Table 1.

Table 1. *Values of the potentials at equilibrium points for the solutions:* 10^{-3} *M* Ag(*bipy*)$_2$(NO$_3$)$_2$ *and a) saturated* $\sim 10^{-3}$ *M* Ag(*phen*)$_2$(NO$_3$)$_2$; *b)* $2 \cdot 10^{-4}$ *M* Ag(*phen*)$_2$(NO$_3$)$_2$. *Temperature 298 K*

μ	E_f° Ag(II)/Ag(I)—2,2′-Bipyridine [V] vs. *NHE*	E_f° Ag(II)/Ag(I)—1,10-Phenanthroline [V] vs. *NHE*		
		a)		b)
0.1	1.109	1.228	0.851	1.229
0.067	1.114	1.234	0.856	1.236
0.03	1.122	1.242	0.863	1.243
0.01	1.129	1.250	0.871	1.252
0.	1.138	1.260	0.880	1.262

Chronovoltamperometric Measurements

In the case of the Ag(*bipy*)$_2^{2+}$/Ag(*bipy*)$_2^{+}$ system the chronovoltamperometric curves have been made for the following solutions in acetonitrile:

a) c Ag(*bipy*)$_2$(NO$_3$)$_2$ + 0.1 *M* (C$_2$H$_5$)$_4$NClO$_4$;

b) c Ag(*bipy*)$_2$NO$_3$ + 0.1 *M* (C$_2$H$_5$)$_4$NClO$_4$;

in the range of potentials 0.4–1.5 V (vs. *NHE*), $c = 10^{-4}$ *M*, $5 \cdot 10^{-4}$ *M*, and 10^{-3} *M*.

c) $2 \cdot 10^{-3}$ *M bipy* + 0.1 *M* (C$_2$H$_5$)$_4$NClO$_4$;

d) 0.1 *M* (C$_2$H$_5$)$_4$NClO$_4$;

in the range of potentials 1.0–2.5 V vs. *NHE*.

Typical curves are shown on Fig. 1 and 2.

The plot of voltamperograms for 2,2′-bipyridine and (C$_2$H$_5$)$_4$NClO$_4$ solutions indicates that both reactants are not active under the conditions of the process (Fig. 2).

Voltamperograms for Ag(*bipy*)$_2$(NO$_3$)$_2$ and Ag(*bipy*)$_2$NO$_3$ solutions are identical (Fig. 1). They have anodic peaks at about 650 mV and 1 185 mV, and respective cathodic ones at about 570 mV and 1 115 mV (vs. *NHE*).

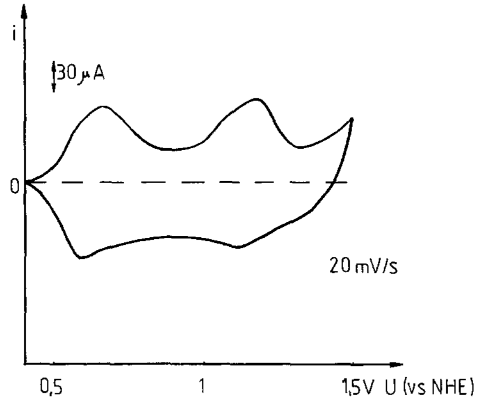

Fig. 1. Voltamperogram of $10^{-3}\,M$ solution of $Ag(bipy)_2(NO_3)_2$ in acetonitrile in the range of potentials φ 0.4–1.5 V; $v = 20\,mV/s$; $\mu = 0.1$; temperature 298 K

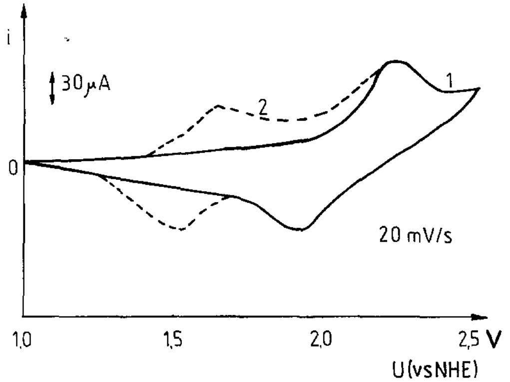

Fig. 2. Voltamperograms of: 1) 0.1 M solution of $(C_2H_5)_4NClO_4$; 2) $2 \cdot 10^{-3}\,M$ solution of 2,2′-bipyridine + 0.1 M $(C_2H_5)_4NClO_4$ in acetonitrile. The range of potentials 1.0–2.5 V; $v = 20\,mV/s$; $\mu = 0.1$; temperature 298 K

The values of ΔE_{pac} and constant values of E_{pc} and E_{pa} potential versus the depolarizer concentration show that both electrodic processes are reversible.

In the case of the $Ag(phen)_2^{2+}/Ag(phen)_2^{+}$ system the chronovoltamperometric curves have been obtained for the following solutions in acetonitrile:

a) $5 \cdot 10^{-4}\,M$ $Ag(phen)_2(NO_3)_2$ + 0.1 M $(C_2H_5)_4NClO_4$;

b) $5 \cdot 10^{-4}\,M$ $Ag(phen)_2NO_3$ + 0.1 M $(C_2H_5)_4NClO_4$;

in the range of potentials 0.4–2.0 V vs. *NHE*.

c) $10^{-3}\,M\,phen$ + 0.1 M $(C_2H_5)_4NClO_4$

in the range of potentials 1.0–2.5 V vs. *NHE*.

The voltamperograms of the phenanthroline solution in 0.1 M $(C_2H_5)_4NClO_4$ in acetonitrile do not exhibit any peaks in the region of potentials used in the investigations of $Ag(phen)_2NO_3$ and $Ag(phen)_2(NO_3)_2$ solutions.

The curves obtained for the solutions of phenanthroline complexes of Ag(I) and Ag(II) are practically identical with an anodic peak at $\varphi = 1.495$ V and the cathodic peak at $\varphi = 1.395$ V. Under the experimental conditions the values of the peak potentials do not depend on the rate of potential sweep (10–100 mV/s).

Discussion

Ag(II)/Ag(I)—2,2′-*Bipyridine System*

The stability of the $Ag(bipy)_2^{+}$ complex and the limited solubility of $Ag(bipy)_2NO_3$ in acetonitrile strictly determine the concentrations of solutions and the forms of Ag(I) complexes, which can be obtained. The value of the stability constant of $Ag(bipy)_2^{+}$ in AN—$\beta_2(I) = 2.16 \cdot 10^5$ shows that beginning with the concentration of the complex $c = 3.4 \cdot 10^{-2}$ M the degree of dissociation is minimal. At this concentration the ion $Ag(bipy)_2^{+}$ is practically the only form of Ag(I). Considering the solubility of the complexes Ag(II) and Ag(I) in acetonitrile such concentration is, however, impossible to obtain.

Within the range of possible concentrations ($\sim 10^{-3}$ M) the complex Ag(I) dissociates completely:

$$Ag(bipy)_2^{+} \rightleftharpoons Ag^{+} + 2\,bipy$$

Accordingly, coulometric and chronovoltamperometric curves must refer to the following redox system:

$$Ag(bipy)_2^{2+} + e \rightleftharpoons Ag^{+} + 2\,bipy$$

[$\beta_2(II) \gg \beta_2(I)$, Ag(II) complex occurs only in the form of $Ag(bipy)_2^{2+}$].

The data from Table 1 give the value of the standard potential of that system $E_l^\circ = 1.138$ V. This value is also confirmed by Fig. 1 with peaks for the potentials $E_a = 1.185$ V and $E_c = 1.115$ V and also $E_f^\circ = 1.150$ V.

Given the value of $\beta_2(I)$ and making use of the value of E_1° one can define the standard potential of the system as:

$$Ag(bipy)_2^{2+} + e \rightleftharpoons Ag(bipy)_2^{+}$$

$$E_2^\circ = E_1^\circ - 0.0591 \lg \beta_2(I) \qquad E_2^\circ = 0.823\ \mathrm{V}$$

The second pair of peaks from Fig. 1 ($E_a = 0.650$ V, $E_c = 0.580$ V, $E_f^\circ = 0.615$ V) corresponds to the reversible reduction of Ag^+ ions in acetonitrile ($E^\circ_{Ag^+/Ag^\circ(AN)} = 0.637$ V [18]).

Ag(II)/Ag(I)—*1,10-Phenanthroline System*

The value of the solubility product of $Ag(phen)_2NO_3$ $K_{so} = 4.02 \cdot 10^{-9}$ [11] and value of the stability constant of $Ag(phen)_2^+$ complex $\beta_2(I) = 7.34 \cdot 10^9$ [11] in acetonitrile show that in saturated solution of the complex ($c \sim 10^{-3}\,M$) in the case of coulometric measurements near the equilibrium point, about 6% of the Ag(I) complex occurs in the form of free Ag^+ ions.

Because of the fact that the value of $\beta_1(I)$ is unknown the concentration of $Ag(phen)^+$ remains unknown too. So, even in saturated solution one will not be dealing exclusively with the $Ag(phen)_2^+$ complex and redox system:

$$Ag(phen)_2^{2+} + e \rightleftharpoons Ag(phen)_2^+$$

The coulometric curves (Table 1) show two potential drops close to each other at $E^\circ = 1.262\,V$ and $E^\circ = 0.880\,V$, while chronovoltamperograms indicate the existence of a redox system with $E_f^\circ = 1.445\,V$. Let us assume that the values of the potential drops in a coulometric titration correspond to the standard potentials of the systems:

$$Ag(phen)_2^{2+} + e \rightleftharpoons Ag(phen)^+ + phen \qquad E_3^\circ$$

and

$$Ag(phen)_2^{2+} + e \rightleftharpoons Ag(phen)_2^+ \qquad E_4^\circ$$

At the conditions of electrolysis in chronovoltamperometric investigations the quantities of $Ag(phen)_2^+$ ion being formed in the case of $Ag(phen)_2^{2+}$ reduction are minimal, so the $Ag(phen)_2^+$ complex undergoes complete dissociation:

$$Ag(phen)_2^+ \rightarrow Ag^+ + 2\,phen$$

Therefore in te range of possible concentrations of reactants the following system will be an object of chronovoltamperometric investigations:

$$Ag(phen)_2^{2+} + e \rightleftharpoons Ag^+ + 2\,phen \text{ with } E_5^\circ = 1.445\,V$$

Those conclusions may be confirmed in a relatively simple way by finding the relationship between the E_3°, E_4° and E_5° values:

$$E_5^\circ = E_4^\circ + 0.0591 \lg \beta_2(I)$$

$$E_3^\circ = E_4^\circ + 0.0591 \lg \beta_1(I)$$

with $\beta_2(I) > \beta_1(I)$: $E_4^\circ < E_3^\circ < E_5^\circ$.

Given the value of $\beta_2(I) = 7.34 \cdot 10^9$ one can find the value of E_5°.

$$E_5^\circ = 1.463\,V.$$

It corresponds approximately to the value E_f° from chronovoltamperometric measurements. However, when the stability constant $\beta_2(I)$ is unknown, it is not possible to confirm the value of E_3°.

Direct experimental evidence could be obtained from a curve of coulometric reduction with one potential drop at $E^\circ = 0.880\,V$. Such a curve could be obtained in a solution of Ag(*phen*)$_2^{2+}$ only with the concentration $c = 6 \cdot 10^{-3}\,M$. However, taking into account the solubility of Ag(*phen*)$_2$(NO$_3$)$_2$ in *AN* such evidence is impossible to be obtained. The coulometric reduction of the Ag(*phen*)$_2^{2+}$ complex with a concentration in the order of $2 \cdot 10^{-4}\,M$ gives a curve which has in practice a one-electron potential drop at $E^\circ = 1.262\,V$.

Comparison of the Properties of the Systems in H_2O *and AN*

Table 2 presents data concerning the stability of Ag(I) complexes [11] and the values of the standard potentials of AgL_2^{2+}/AgL_2^{+} systems in water and acetonitrile.

Table 2. *Comparison of the properties of the* AgL_2^{2+}/AgL_2^{+} *system in water and acetonitrile*

$\lg\beta_2$(I) (H_2O)	$\lg\beta_2$(I) (*AN*)	$\Delta\lg\beta$	Ligand	$E^\circ_{AgL_2^{2+}/AgL_2^{+}}$	
				(H_2O)	(*AN*)
4.35	3.42	0.97	pyridine	—	—
6.67	5.33	1.34	2,2′-bipyridine	0.820 [6]	0.823
11.52 [19]	9.87	1.65	1,10-phenanthroline	1.096 [7]	0.880

The smaller stability of Ag(I) complexes in acetonitrile, compared with water, results from the differences of the interaction energies ion—solvent: $\beta_{AN} = 2.98$, $\beta_{H_2O} = 0.13$.

The complexing of Ag(I) ions in H_2O and *AN* may be shown in the following way:

$$Ag^{+}_{(H_2O)} + 2\,L_{(H_2O)} \rightleftharpoons AgL^{+}_{2(H_2O)} \qquad \beta_{2(H_2O)}$$

$$Ag^{+}_{(AN)_2} + 2\,L_{(AN)} \rightleftharpoons AgL^{+}_{2(AN)} \qquad \beta_{2(AN)}$$

The AgL_2^{+} ions have the same spatial and electronic structure in *AN* and in H_2O and ligand stabilization energy LFSE = 0. Taking into account the very large radii of Ag(*bipy*)$_2^{+}$ and Ag(*phen*)$_2^{+}$ the free enthalpy of complex solvation should be very small and approximately the same for H_2O as for *AN*.

The energy of interaction ligand—solvent (*Debye* ynd *London* type) is also very small in the case of large sizes and relatively small polarizability of the ligand. So, under the above conditions the differences in the stability of the same complex in H_2O and *AN* may arise only from differences of Ag^+ ion—solvent dipol interactions. Hence,

$$\lg\beta_{\mathrm{Ag}L^{+}_{2(\mathrm{H_2O})}} - \lg\beta_{\mathrm{Ag}L^{+}_{2(AN)}} \approx \lg\beta_{\mathrm{Ag}^{+}_{(\mathrm{H_2O})}} - \lg\beta_{\mathrm{Ag}^{+}_{(\mathrm{CH_3CN})_2}} = 1.36$$

The value $\Delta\lg\beta$ for *L*—pyridine shows that an "excess" stability of a complex in *AN* is caused by non-complete screening at central ion charge and partially exposed Ag^+ ion.

In that case, the condition of equality of the free enthalpy of a complex ion in H_2O and *AN* is not satisfied. Probably, we are dealing here with outer-spheric complexes of the type:

$$\mathrm{Ag}(py)_2^+ \cdot n\,\mathrm{H_2O} \text{ and } \mathrm{Ag}(py)_2^+ \cdot n\,\mathrm{CH_3CN}$$

where the stability of $\mathrm{Ag}(py)_2^+ \cdot n\,\mathrm{CH_3CN}$ complexes is greater.

The value of $\Delta\lg\beta$ for $\mathrm{Ag}(bipy)_2^+ = 1.34$ suggests that in this case all considered conditions are satisfied.

However, in the case of $\mathrm{Ag}(phen)_2^+$, $\Delta\lg\beta > 1.36$ indicates that with the full screening of Ag^+ ions we are dealing with relatively large and non-negligible interactions of *London* and *Debye* type: *AN*—1,10-phenanthroline, resulting from the big polarizability of phenanthroline molecule.

In our opinion the above considerations may be applied generally to the problem of electrodes indicating the same potential in different solvents and based on a reversible redox system of the type:

$$MeL_n^{z+}/MeL_n^{+z-1} \text{ [20].}$$

The most important electrodes of this type include such systems as: $\mathrm{Fe}(phen)_3^{3+}/\mathrm{Fe}(phen)_3^{2+}$ [21], Fc^+/Fc [20] and bis-diphenylchromium I/0 [22].

A criticism of these electrodes based on experimental data was published by *Parker* [23].

At present, many intensive studies using cryptates and coronens which should screen the central ion in complexes are continuing. The search is carried out by a "trial and error" method.

Our considerations lead to somewhat different assumptions concerning the construction of the electrodes:

1. The complex ion radius should be large enough to make the screening of central ion complete. As a measure of that screening the quantity $\Delta\lg\beta_{S_1-S_2}$ can be used.
2. The condition of very small charge of central ion is not necessary.
3. The energy of ligand—solvent interactions (*van der Waals* type) must be small and negligible when compared with an energy of solvent—metal ion interaction.

Additional two conditions concerning the stability of complexes forming a redox system may be expressed:

$$\frac{\beta_n[MeL_n]^{z+}}{\beta_n[MeL_n]^{+z-1}} = const. \text{ (for different solvents)}$$

$$\frac{\beta_{Me^{z+}-S}}{\beta_{Me^{+z-1}-S}} = \frac{\beta_{Me^{z+}-H_2O}}{\beta_{Me^{+z-1}-H_2O}} = const. \text{ (for different solvents)}$$

It follows from condition 3 which requires small polarizability and dipol moment of the ligand, that coronens and cryptates, which have big polarizability, do not seem to be useful in solving the problem in question.

On the contrary, the system $Ag(bipy)_2^{2+}/Ag(bipy)_2^+$ satisfying the $\Delta \lg \beta_{S_1-S_2}$ criterion is expected to be useful. Data from Table 2 indicate that this system has in practice the same E° values in H_2O and in *AN*. In addition the studies of *Rahimi* and *Popov* [24] on the interactions solvent—Ag^+ ion show greatest differences of interactions Ag^+—*S* between H_2O and *AN* (anomally high energy of interactions Ag^+—*AN*, soft acid—soft *Pearsons* base).

Therefore, if:

$$E^\circ_{Ag(bipy)_2^{2+}/Ag(bipy)^+_{2(AN)}} = E^\circ_{Ag(bipy)_2^{2+}/Ag(bipy)^+_{2(H_2O)}}$$

than the standard potential of a system should be constant in solvents with a donor number $DN \leqslant 30$ [24].

Unfortunately, it follows from the above investigations that dealing with a limited solubility $Ag(bipy)_2(NO_3)_2$ and $Ag(bipy)_2NO_3$ and a relatively small stability constant $\beta_2(I)$ in *AN*, it is impossible in practice to obtain a $Ag(bipy)_2^{2+}/Ag(bipy)_2^+$ system. This hypothesis can be therefore verified by studies of this system in other solvents.

The value of $\Delta \lg \beta = 1.65$ was indicating that the *London* and *Debye* interaction: *AN*—1,10-phenanthroline was relatively strong and also the $Ag(phen)_2^{2+}/Ag(phen)_2^+$ system appeared to be useless in the construction of an universal electrode.

The values $E^\circ_{AN} \neq E^\circ_{H_2O}$ from Table 2 have also confirmed that assumption.

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