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Polarographic Behaviour and Determination of Selenite and Tellurite in Simple Solutions or in a Binary Mixture

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Summary. The polarographic behaviour of simple solutions of selenite and tellurite in 1 *M* ammonium salts of formate, acetate, tartrate, oxalate, and benzoate solutions in absence and in presence of Triton X-100 as a maximum suppressor and a temperature of 25°C has been investigated. Schemes for the mechanism of reductions occurring at the DME have been deduced. A method for analytical determination of selenite and tellurite in simple solutions as well as in a binary mixture in the presence of 4–14 · 10^{-3}% Triton X-100 is reported.

Keywords. Selenite; Tellurite; Analytical determination; Polarography.

Polarigraphisches Verhalten und die Bestimmung von Selenit und Tellurit in einfachen Lösungen oder in binären Mischungen

Zusammenfassung. Es wurde das polarographische Verhalten von einfachen Lösungen von Selenit und Tellurit in 1 *M* Ammonsalzlösungen von Formiat, Acetat, Tartrat, Oxalat und Benzoat-Lösungen mit und ohne Triton X-100 als Suppressor bei einer Temperatur von 25 °C untersucht. Es wurde ein Reaktionsschema für den Mechanismus der Reduktion an der DME abgeleitet. Eine Methode zur analytischen Bestimmung von Selenit und Tellurit in einfachen Lösungen und in binären Mischungen in Gegenwart von 4–14 · 10^{-3}% Triton X-100 wird berichtet.

Introduction

Selenium and tellurium are important elements because they have several industrial and biological applications. They are much used in xerography and for colouring glass. Metallurgically, the addition of selenium or tellurium to certain stainless steels is said to improve their mechanical properties. Selenium and tellurium have found some application as secondary vulcanizing agents for natural rubber. Therefore, numerous analytical methods have been developed for their determination, e.g. by gravimetry [1, 2], titrimetry [3–6], cyclic and stripping voltametry [7, 8], oscillopolarography [9], and polarography [10–21]. In most supporting electrolytes studied the reduction of tellurite gives rise to complicated behaviours unsuitable for analytical work. This prompted me in the present work to study the effects of Triton X-100 (non-ionic surfactant) on the polarographic behaviour of tellurite ions in ammonium salt solutions to obtain well defined media for analytical determination of tellurite in its simple solutions as well as in a binary mixture with tetravalent selenium.

Experimental

All chemicals used were of BDH grade.

A 0.025 *M* solution of both selenite and tellurite was prepared by dissolving the required amount of sodium selenite or sodium tellurite in bidistilled water. The resulting solutions were then standardized as given in Vogel [22]. Solutions of lower concentrations were prepared by accurate dilution. Ammonium salt solutions and Triton X-100 were prepared by dissolving the required quantities in bidistilled water.

Polarographic measurements were carried out using a Universal OH-105 type polarograph with three electrodes, dropping mercury electrode (DME), auxiliary platinium electrode and a saturated calomel electrode (SCE) serving as the reference electrode. The dropping mercury working electrode had the following characteristics: $m = 1.894\,\mathrm{mg\,s^{-1}}$ and $t = 3.9\,\mathrm{s\ drop^{-1}}$.

Results and Discussion

Polarography of Tetravalent Tellurium

The polarographic behaviour of 0.5 m*M* sodium tellurite in ammonium salt solutions is represented by the polarograms shown in Fig. 1. A most striking feature of the polarograms of Te(IV) is the very large maximum which appears in the diffusion current plateau in all ammonium salt solutions used. An interpretation of this maximum has been discussed previously for universal buffer solutions [21].

In ammonium formate and acetate solutions, the polarograms consist of two waves, the second wave being heigher than the first. A single developed wave was also obtained in ammonium tartrate, oxalate, and benzoate solutions.

The effects produced by Triton X-10 on the polarographic reduction of 0.5 m*M* Te(IV) in ammonium formate solution are illustrated by the polarograms shown in Fig. 2. In the presence of small concentrations of this SAS, the limiting currents

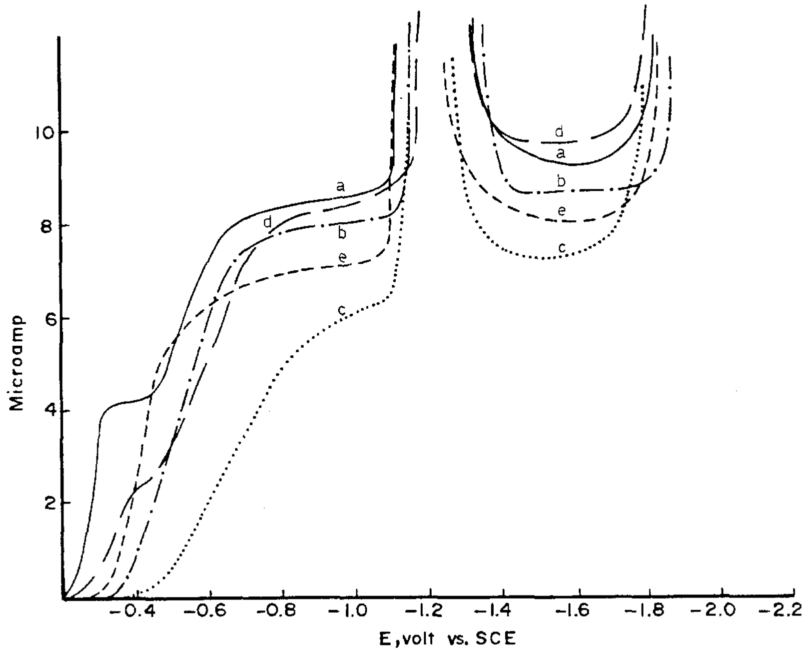

Fig. 1. Polarograms of 0.5 m*M* Te(IV) in 1 *M* ammonium salt solutions: *a* formate; *b* acetate; *c* tartrate; *d* oxalate; *e* benzoate

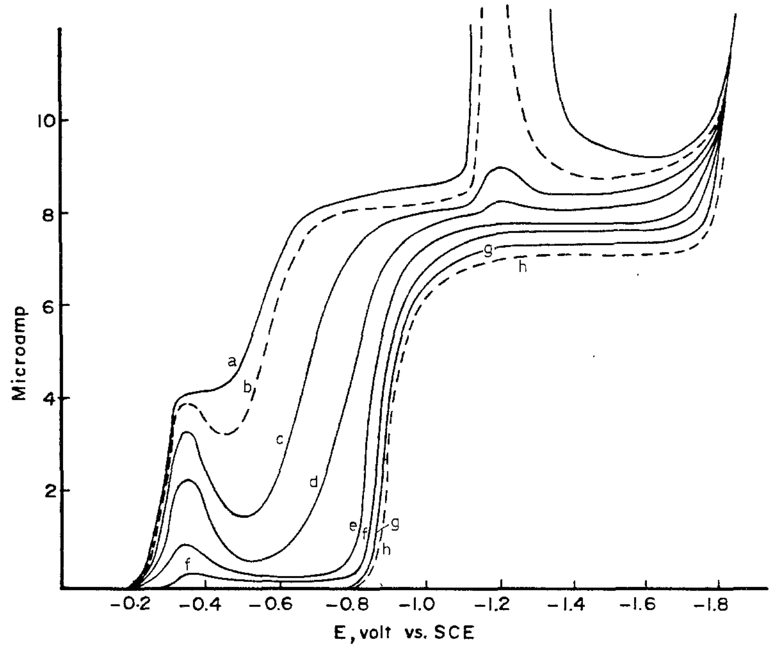

Fig. 2. Polarograms of 0.5 m*M* Te(IV) in 1 *M* ammonium formate solution containing Triton X-100; curve/10^3% SAS: *a* 0.0; *b* 1; *c* 2; *d* 3; *e* 6; *f* 10; *g* 14; *h* 18

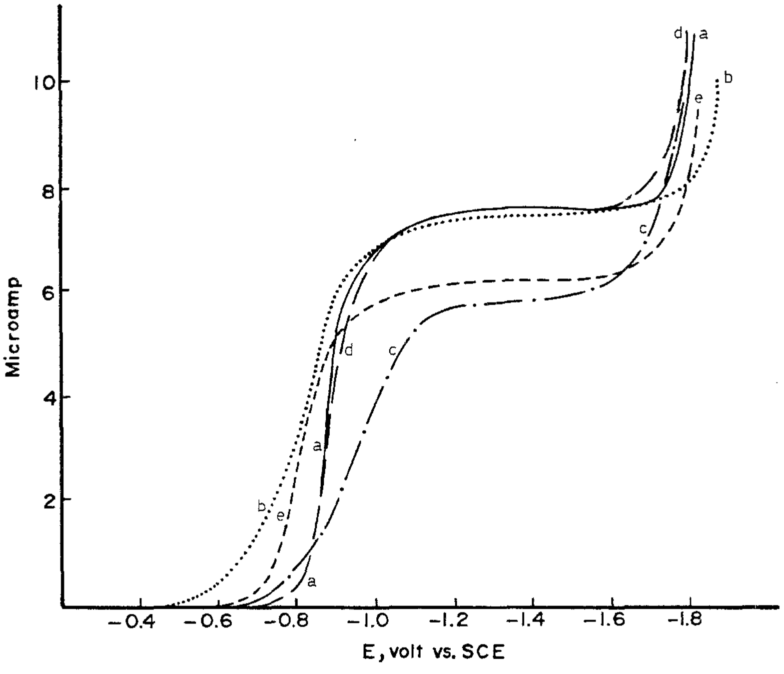

Fig. 3. Polarograms of 0.5 m*M* Te(IV) in 1 *M* ammonium salt solutions in presence of Triton X-100; curve/salt/10^3% SAS: *a* formate/14; *b* acetate/4; *c* tartrate/4; *d* oxalate/6; *e* benzoate/4

of the two waves are decreased and their $E_{1/2}$ shifted to more negative values. On rising [SAS], a minimum develops on the plateau position of the first wave. The depth of this minimum increases with increasing the percentage of Triton X-100. When [SAS] becomes $1.4 \cdot 10^{-2}$%, the hump disappears completely and the polarogram only keeps a strongly shifted wave at more negative potential value ($E_{½} = -0.872$ V vs. SCE). The maximum is suppressed in the presence of $6 \cdot 10^{-3}$% Triton X-100.

The study has been restricted to the reduction waves in ammonium formate, acetate, tartrate, oxalate, and benzoate solutions; the polarograms are shown in Fig. 3, after elimination of the maxima and humps by addition of the smallest amount of Triton X-100.

The results show that reduction of tellurite ions in these ammonium salt solutions occurs along one step. In order to calculate the number of electrons involved in the reduction process, the value of the diffusion coefficient of sodium tellurite in these solutions was calculated using the Stokes-Einstein relation [23]. Substituting for diffusion current (μA) along the wave in the Ilkovič equation, n is found equal to 4, indicating that the reduction of tellurite ions in these media involves libration

Table 1. Kinetic parameters for the reduction wave of 0.5 mM Te(IV) in 1 M ammonium salt solutions in presence of Triton X-100

Ammonium salt 1 M	10^3% SAS	i_d μA	$E_{½}$, V (NHE)	Slope V^{-1}	αn_a	K^0 cm s^{-1}	ΔG^* K. J.	$KD^{-½}$	log i_l/h at −1.3 V (SCE)
Formate	14.0	7.72	−0.630	25.90	1.543	$8.60 \cdot 10^{-20}$	33.41	$2.81 \cdot 10^{-17}$	0.49
Acetate	4.0	7.22	−0.562	6.33	0.374	$4.94 \cdot 10^{-7}$	15.76	$1.61 \cdot 10^{-4}$	0.55
Tartrate	4.0	5.78	−0.705	6.21	0.367	$7.47 \cdot 10^{-8}$	16.90	$2.44 \cdot 10^{-5}$	0.49
Oxalate	6.0	7.22	−0.645	11.46	0.677	$7.24 \cdot 10^{-11}$	21.06	$2.36 \cdot 10^{-8}$	0.49
Benzoate	4.0	6.24	−0.573	10.44	0.617	$1.86 \cdot 10^{-9}$	19.12	$6.07 \cdot 10^{-7}$	0.47

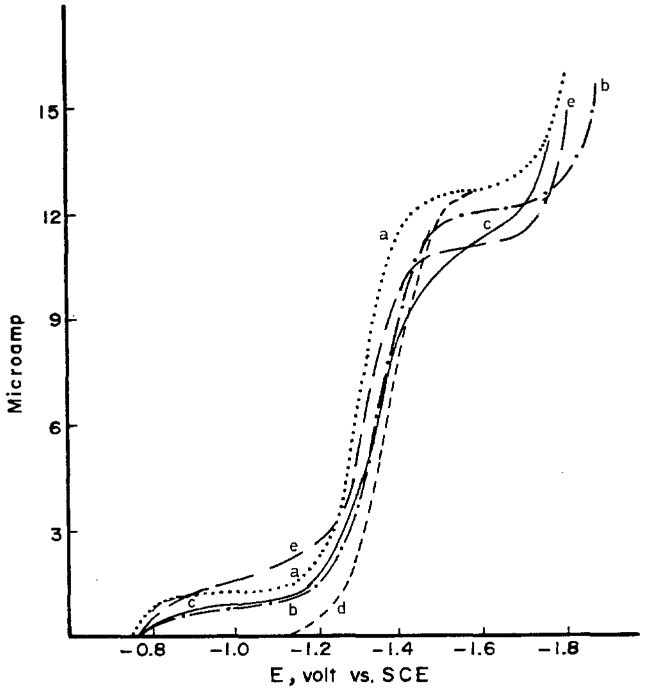

Fig. 4. Polarograms of 0.5 mM Se(IV) in 1 M ammonium salt solutions; curve/salt: a formate; b acetate; c tartrate; d oxalate; e benzoate

of OH$^-$ ions, and passes along a single wave to tellurium by taking four electrons as follows:

$$\mathrm{TeO_3^{2-} + 4\,e^- + 3\,H_2O = Te + 6\,OH^-}\ .$$

These results agree with findings of previous investigations [19–21].

The reduction of Te(IV) in all ammonium salt solutions was irreversible, judging from the log-plot of the wave analysis which was obtained by means of the plots of the values of log $i/i_d - i$ vs. E. These plots were utilized in the evaluation of the corresponding values of the kinetic parameters and wave characteristics including the slope, αn_a (α = transfer coefficient and n_a = number of electrons involved in the rate determining step), K^0 (standard rate constant), ΔG^* (energy of activation) and $KD^{-1/2}$. Table 1 summarizes the kinetic parameters for the reduction of tellurite ions in these ammonium salt solutions. These results coupled with the direct proportionality of the diffusion current to the square root of the effective height of the mercury column, indicated that the reduction of Te(IV) in these solutions is irreversible and entirely diffusion-controlled.

Polarography of Tetravalent Selenium

The polarographic behaviour of 0.5 mM sodium selenite in ammonium salt solutions is represented by the polarograms shown in Fig. 4. The results show that reduction of Se(IV) occurs along a single wave in ammonium oxalate solution and along the two waves in ammonium formate, acetate, tartrate, and benzoate solutions, the first one being a pre-wave.

From the polarograms recorded for different concentrations of selenite ions in these media it was shown that only a single wave was observed at low concentration. A new small pre-wave appeared at $C = 3 \cdot 10^{-4} M$, which exhibits a constant limiting current on further increasing the concentration of the depolarizer. The appearance of a small wave that proceeds the main wave or subsequent to it was described as a pre-wave or a post-wave depending whether the depolarizer or the reduction product itself is adsorbed at the electrode surface [24, 25].

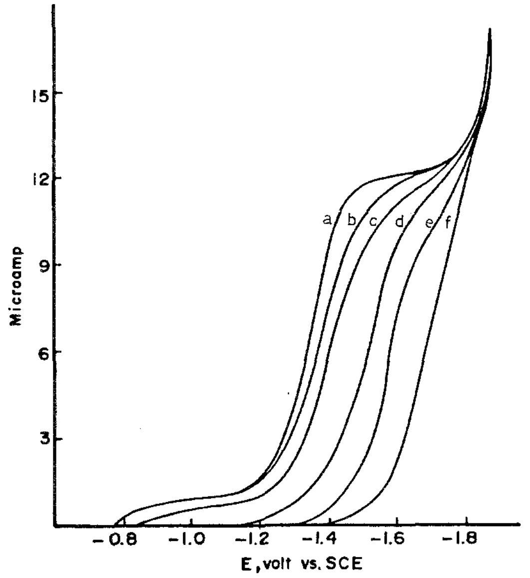

Fig. 5. Polarograms of 0.5 mM Se(IV) in 1 M ammonium acetate containing Triton X-100; curve/10^3% SAS: *a* 0.0; *b* 0.5; *c* 1.5; *d* 4.0; *e* 5.0; *f* 13.0

In order to find out the mechanism of the reduction along the main wave in these solutions, one has to calculate the number of electrons involved in the reduction process. Since the value of the diffusion coefficient of Na_2SeO_3 in these solutions was calculated [23] and by substituting for i_d(μA) along the main wave in the Ilkovič equation, n is found equal to 6, indicating that the reduction process along a single wave to the selenide ion takes six electrons,

$$SeO_3^{--} + 6e^- + 3H_2O = Se^{--} + 6OH^- .$$

These results agree with the findings of several authors [10–17].

The polarograms shown in Figs. 5 and 6 represent the polarographic behaviour of 0.5 mM Se(IV) in ammonium acetate and oxalate solutions in absence and in presence of varying amounts of Triton X-100. In presence of this surfactant, the limiting current of the main reduction wave in ammonium salt solutions is decreased and its $E_{1/2}$ shifted to a more negative value; the pre-wave, which was observed in some ammonium salt solutions, is completely suppressed. No reduction waves were observed when the Triton X-100 concentrations became 14, 13, 6, 6, and $12 \cdot 10^{-3}$% in 1 M ammonium formate, acetate, tartrate, oxalate, and benzoate solutions, respectively.

Table 2 shows wave characteristics and kinetic parameters for the reduction wave of sodium selenite in these ammonium salt solutions in absence and in the presence of varying amounts of Triton X-100. The values of the kinetic parameters show that the reduction process is irreversible and diffusion with an adsorption component and the degree of irreversibility increases with increasing the surfactant concentrations.

Polarographic Determination of Se(IV) *and* Te(IV) *in Simple Solutions*

The plots of i_d vs. concentration for the reduction wave of selenite ions in different ammonium salt solutions at -1.7 Volt vs. SCE are shown in Fig. 7. The plots are straight lines passing through the origin, indicating that the quantitative determination of Se(IV) is perfect and successful in either of these solutions.

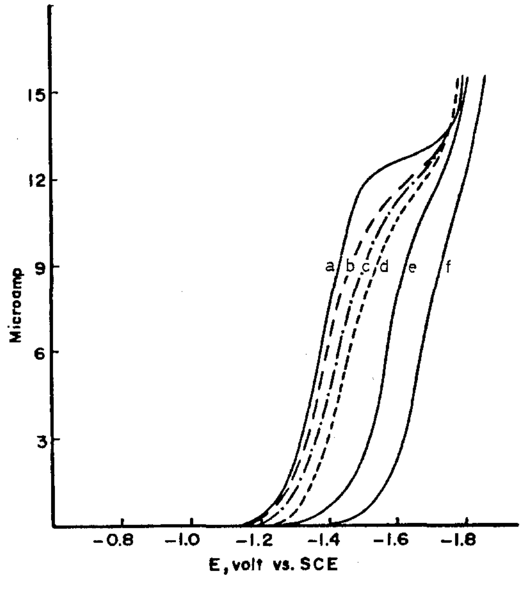

Fig. 6. Polarograms of 0.5 mM Se(IV) in 1 M ammonium oxalate containing Triton X-100; curve/10^3% SAS: *a* 0.0; *b* 0.5; *c* 1.0; *d* 2.0; *e* 4.0; *f* 6.0

Table 2. Effect of Triton X-100 on the polarographic reduction wave of 0.5 m*M* Se(IV) in 1 *M* ammonium salt solutions

% SAS	i_d μA	$E_{½}$, V vs. (NHE)	Slope V^{-1}	αn_a	K^0 cm s^{-1}	ΔG KJ	$KD^{-½}$	log i_l/h at -1.6 V (SCE)
A) Ammonium formate								
0.0	12.75	-1.053	8.71	0.515	$1.26\cdot10^{-12}$	23.50	$3.86\cdot10^{-10}$	0.65
$5.0\cdot10^{-4}$	12.00	-1.090	7.65	0.452	$8.83\cdot10^{-12}$	22.33	$2.70\cdot10^{-9}$	
$1.0\cdot10^{-3}$	11.25	-1.108	7.62	0.450	$6.91\cdot10^{-12}$	22.48	$2.12\cdot10^{-9}$	
$1.5\cdot10^{-3}$	10.80	-1.135	7.67	0.453	$3.78\cdot10^{-12}$	22.84	$1.16\cdot10^{-9}$	
$3.0\cdot10^{-3}$	10.95	-1.245	6.82	0.403	$6.18\cdot10^{-12}$	22.54	$1.89\cdot10^{-9}$	
$5.0\cdot10^{-3}$	10.80	-1.327	8.75	0.517	$4.68\cdot10^{-15}$	26.86	$1.43\cdot10^{-12}$	
B) Ammonium acetate								
0.0	12.00	-1.092	8.29	0.490	$1.09\cdot10^{-12}$	23.96	$5.20\cdot10^{-10}$	0.64
$5.0\cdot10^{-4}$	12.30	-1.130	6.95	0.411	$2.74\cdot10^{-11}$	21.69	$8.22\cdot10^{-9}$	
$1.5\cdot10^{-3}$	11.85	-1.160	6.60	0.390	$4.29\cdot10^{-11}$	21.38	$1.29\cdot10^{-8}$	
$4.0\cdot10^{-3}$	11.40	-1.265	8.31	0.491	$5.91\cdot10^{-14}$	25.33	$1.77\cdot10^{-11}$	
$5.0\cdot10^{-3}$	10.80	-1.330	9.49	0.561	$4.71\cdot10^{-16}$	28.24	$1.41\cdot10^{-13}$	
C) Ammonium tartrate								
0.0	9.40	-1.068	9.25	0.547	$2.24\cdot10^{-13}$	24.54	$7.69\cdot10^{-11}$	0.69
$1.0\cdot10^{-3}$	10.65	-1.100	6.62	0.391	$8.82\cdot10^{-11}$	20.95	$3.03\cdot10^{-8}$	
$2.0\cdot10^{-3}$	10.65	-1.125	6.99	0.413	$2.33\cdot10^{-11}$	21.75	$7.99\cdot10^{-9}$	
$4.0\cdot10^{-3}$	10.05	-1.275	7.19	0.425	$1.17\cdot10^{-12}$	23.54	$4.01\cdot10^{-10}$	
D) Ammonium oxalate								
0.0	13.30	-1.148	6.88	0.406	$2.68\cdot10^{-11}$	21.66	$7.45\cdot10^{-9}$	0.59
$5.0\cdot10^{-4}$	12.30	-1.162	7.26	0.429	$7.65\cdot10^{-12}$	22.41	$2.13\cdot10^{-9}$	
$1.0\cdot10^{-3}$	12.15	-1.190	7.46	0.441	$2.81\cdot10^{-12}$	23.02	$7.82\cdot10^{-10}$	
$2.0\cdot10^{-3}$	12.30	-1.225	7.09	0.419	$4.31\cdot10^{-12}$	22.76	$1.20\cdot10^{-9}$	
E) Ammonium benzoate								
0.0	9.45	-1.083	7.26	0.429	$2.21\cdot10^{-11}$	21.78	$7.99\cdot10^{-9}$	0.68
$1.0\cdot10^{-3}$	8.70	-1.130	7.45	0.440	$6.12\cdot10^{-12}$	22.55	$2.21\cdot10^{-9}$	
$2.0\cdot10^{-3}$	9.00	-1.183	7.03	0.416	$7.69\cdot10^{-12}$	22.41	$2.78\cdot10^{-9}$	
$4.0\cdot10^{-3}$	9.30	-1.277	8.68	0.513	$1.34\cdot10^{-14}$	26.22	$4.85\cdot10^{-12}$	

The current concentration plots for the reduction wave of sodium tellurite in 1 *M* ammonium formate, acetate, tartrate, oxalate, and benzoate solutions containing 14, 4, 4, 6, and $4\cdot10^{-3}$% Triton X-100 are good straight lines passing through the origin as shown in Fig. 7. Accordingly, the reduction current is directly proportional to the concentration of the depolarizer in accordance with the Ilkovič equation. Thus, it is possible to utilize these media for the quantitative determination of Te(IV).

Polarographic Determination of Se(IV) *and* Te(IV) *in a Binary Mixture*

From the foregoing results on the polarographic behaviour of both Se(IV) and Te(IV) in ammonium salt solutions in absence and in presence of Triton X-100, it

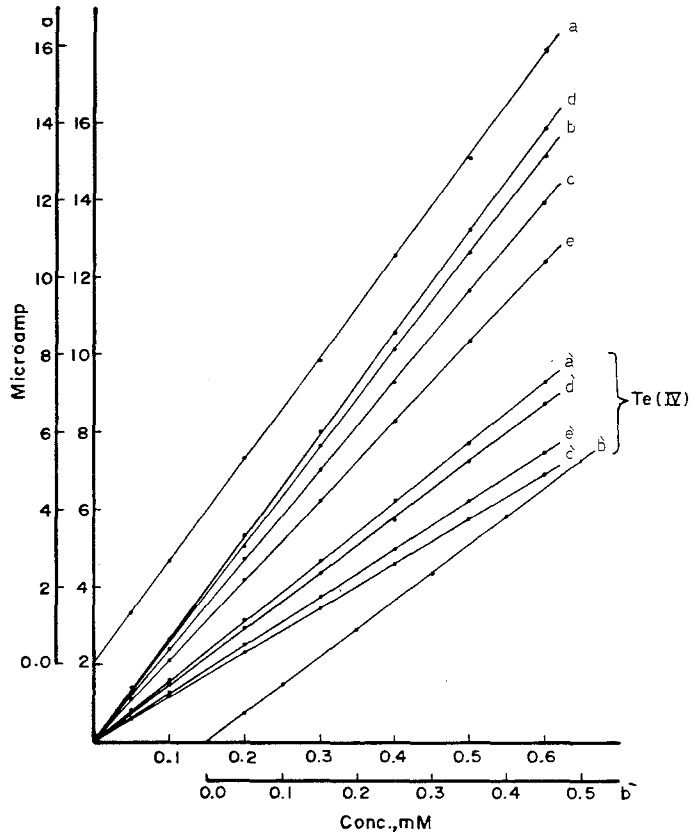

Fig. 7. Effect of both selenite and tellurite concentrations individually in 1 M ammonium salt solutions at -1.3 and -1.6 Volt vs. SCE: a, $\bar{a}$ formate; b, $\bar{b}$ acetate; c, $\bar{c}$ tartrate; d, $\bar{d}$ oxalate; e, $\bar{e}$ benzoate

may be concluded that 1 M ammonium formate or oxalate containing 14 or $6 \cdot 10^{-3}$% SAS, and 1 M ammonium acetate, tartrate, and benzoate containing $4 \cdot 10^{-3}$% SAS are suitable media for successful determination of tellurite ions. Thus Fig. 8 represents the polarograms of a binary mixture of 0.5 mM sodium selenite and 0.5 mM sodium tellurite in these solutions.

In ammonium acetate, tartrate, and benzoate the polarograms consist of two waves correspond to the reduction of tellurite and selenite along the first and second wave, respectively.

The well developed wave obtained in ammonium formate and oxalate corresponds to the reduction of tellurite (no selenite reduction waves were observed in presence of these surfactant concentrations in these media).

From these polarograms it is clear that tetravalent tellurium can be determined in presence of selenite ions in all these media. Measurements of the limiting current are carried out at -1.25 Volt vs. SCE. The $i_d - C$ graph shown in Fig. 9 consists of straight lines passing through the origin, thus confirming the applicability of the Ilkovič equation. Further proof for the validity of this equation comes from the constancy of i_d/C ratios as given in Table 3.

It is also clear from Fig. 8 that selenite ions in presence of Te(IV) can be

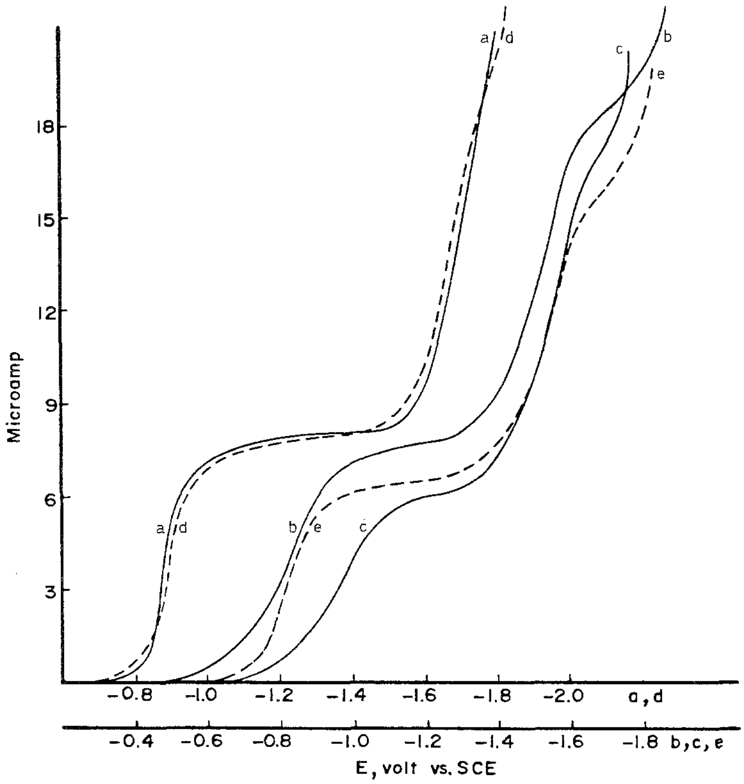

Fig. 8. Polarograms of a binary mixture of 0.5 m*M* Se(IV) and 0.5 m*M* Te(IV) in ammonium salt solutions in presence of Triton X-100; curve/salt/10^3% SAS: *a* formate/14; *b* acetate/4; *c* tartrate/4; *d* oxalate/6; *e* benzoate/4

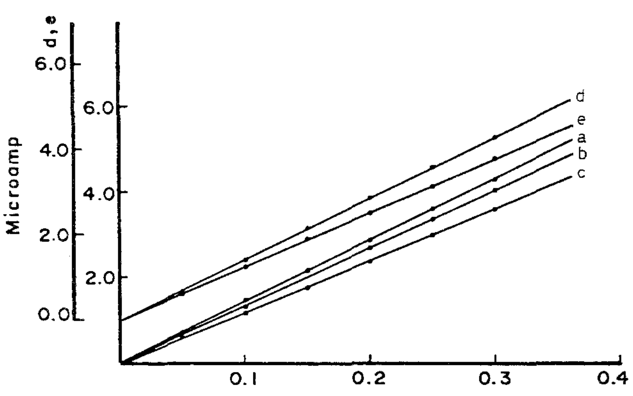

Fig. 9. Effect of [Te(IV)] in 1 *M* ammonium salt solutions containing 0.5 m*M* Se(IV) in presence of Triton X-100 at −1.25 Volt vs. SCE; plot/salt/10^3% SAS: *a* formate/14; *b* acetate/4; *c* tartrate/4; *d* oxalate/6; *e* benzoate/4

determined only in ammonium acetate, tartrate, and benzoate solutions containing $4 \cdot 10^{-3}$% Triton X-100. Measurements of the limiting current are made at −1.7 Volt vs. SCE after subtracting their values from the limiting currents of tellurite. On plotting i_d vs. [Se(IV)], straight lines are obtained passing through the origin as shown in Fig. 10. The constancy of i_d/C ratios listed in Table 4 gives further confirmation that the straight lines obey the Ilkovič equation. It is clear that the

Table 3. Effect of Te(IV) concentration in 1 *M* ammonium salt solutions containing 0.5 m*M* Se(IV) in presence of Triton X-100 at −1.25 Volt vs. SCE

Ammonium salt	10^3% SAS	[Te(IV)] m*M*	i_d μA	i_d/C	Average
Formate	14	0.05	0.722	14.44	14.43
		0.10	1.442	14.42	
		0.15	2.163	14.42	
		0.20	2.886	14.43	
		0.25	3.613	14.45	
		0.30	4.332	14.44	
Acetate	4.0	0.05	0.683	13.66	13.63
		0.10	1.364	13.64	
		0.15	2.042	13.61	
		0.20	2.726	13.63	
		0.25	3.410	13.64	
		0.30	4.084	13.61	
Tartrate	4.0	0.05	0.598	11.96	11.95
		0.10	1.194	11.94	
		0.15	1.793	11.96	
		0.20	2.386	11.93	
		0.25	2.983	11.93	
		0.30	3.588	11.96	
Oxalate	6.0	0.05	0.715	14.30	14.32
		0.10	1.431	14.31	
		0.15	2.148	14.32	
		0.20	2.862	14.31	
		0.25	3.583	14.33	
		0.30	4.296	14.32	
Benzoate	4.0	0.05	0.631	12.62	12.61
		0.10	1.262	12.62	
		0.15	1.890	12.60	
		0.20	2.522	12.61	
		0.25	3.145	12.58	
		0.30	3.780	12.60	

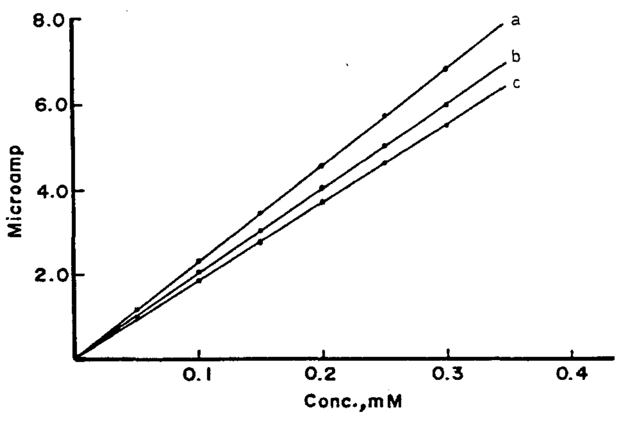

Fig. 10. Effect of [Se(IV)] in 1 *M* ammonium; *a* acetate, *b* tartrate, *c* benzoate solution containing 0.5 m*M* Te(IV) in presence of $4 \cdot 10^{-3}$% Triton X-100 at −1.7 Volt vs. SCE

Table 4. Effect of Se(IV) concentration in 1 *M* ammonium salt solutions containing 0.5 m*M* Te(IV) in presence of $4 \cdot 10^{-3}\%$ Triton X-100 at −1.7 Volt vs. SCE

Ammonium salt	[Se(IV)] m*M*	i_d μA	i_d/C	Average
Acetate	0.05	1.141	22.82	22.81
	0.10	2.281	22.81	
	0.15	3.425	22.83	
	0.20	4.560	22.80	
	0.25	5.700	22.80	
	0.30	6.843	22.81	
Tartrate	0.05	1.005	20.10	20.11
	0.10	2.012	20.12	
	0.15	3.018	20.12	
	0.20	4.020	20.10	
	0.25	5.023	20.09	
	0.30	6.033	20.11	
Benzoate	0.05	0.929	18.58	18.61
	0.10	1.860	18.60	
	0.15	2.793	18.62	
	0.20	3.724	18.62	
	0.25	4.650	18.60	
	0.30	5.592	18.64	

polarographic determination of selenite ions in the presence of 0.5 m*M* Te(IV) in 1 *M* ammonium acetate, tartrate, and benzoate solutions containing $4 \cdot 10^{-3}\%$ Triton X-100 is possible.

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