

# ELECTROCHEMICAL BEHAVIOUR OF Te(VI) AND ITS QUANTITATIVE DETERMINATION BY SQUARE-WAVE VOLTAMMETRY

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## Abstract

Electrochemical behaviour of Te(VI) is investigated applying both square-wave and cyclic voltammetry. The aim of the work is to develop a method for quantitative determination of Te(VI). The methodology is based on the reduction of Te(VI) to elementary Te at potential of  $-1.80$  V. The electrode reduction product Te(0) is accumulated on the electrode surface and it can be subsequently stripped off by cathodic potential scan due to further reduction of Te(0) to Te<sup>2-</sup>. An optimization of experimental as well as instrumental parameters was attempted. The influence of the interference ions, such as Cu<sup>2+</sup>, Zn<sup>2+</sup>, Co<sup>2+</sup>, Mn<sup>4+</sup>, V<sup>5+</sup>, Cr<sup>3+</sup>, Fe<sup>3+</sup>, Ni<sup>2+</sup> is investigated. The detection limit of  $9 \times 10^{-8}$  mol/L Te(VI) with accumulation time of 10 s was found. The reproducibility of the method varied from 0.2 to 3.0 %.

**Key words:** tellurium, square-wave voltammetry

## Introduction

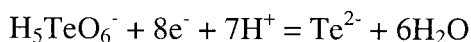
In the last tenth years, the square-wave voltammetry (SWV) has been forced as the most advanced electroanalytical technique. Its performance such as high frequency, high amplitude, its capability to discriminate against the capacitive current as well as the short time required for measurements make it superior compared to other voltammetric techniques. SWV is used for the determination of both organic and inorganic substances as well as for the characterization of the redox processes. Moreover, SWV is a very sensitive technique especially in the cases when the electroactive material shows adsorption properties on the surface area of the working mercury electrode. These substances can be measured by SWV in a trace levels after preconcentration step. The aim of this work is to develop a sensitive square-wave voltammetric method for quantitative determination of Te(VI).

## Experimental

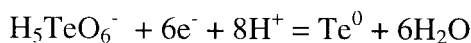
Telluric acid and Na<sub>2</sub>SO<sub>4</sub> were used as received (Merck). The stock solution of Te(VI) was prepared by dissolving of an appropriate amount of telluric acid in twice distilled water. All measurements were performed by Polarographic Analyzer Princeton Applied Research Model 384 B. The working electrode was a hanging mercury drop (HMDE) with a surface area of 0.0149 cm<sup>2</sup>, Ag/AgCl (saturated KCl) was reference and Pt wire was used as a counter. The pure nitrogen was used for deoxygenization of working solutions 8 minutes prior to measurement. All experiments were achieved at room temperature.

## Results and discussion

It is known that in a solution with  $\text{pH} > 8$ ,  $\text{Te(VI)}$  is reduced to  $\text{Te}^{2-}$  at a HMD electrode, at a potential of about  $-1.50 \text{ V}$  vs  $\text{Ag/AgCl}$  (saturated  $\text{KCl}$ ) [1,4]. The obtained peak is a single and the redox process is totally irreversible and the diffusion controlled. This process can be presented with the following equation[2]:

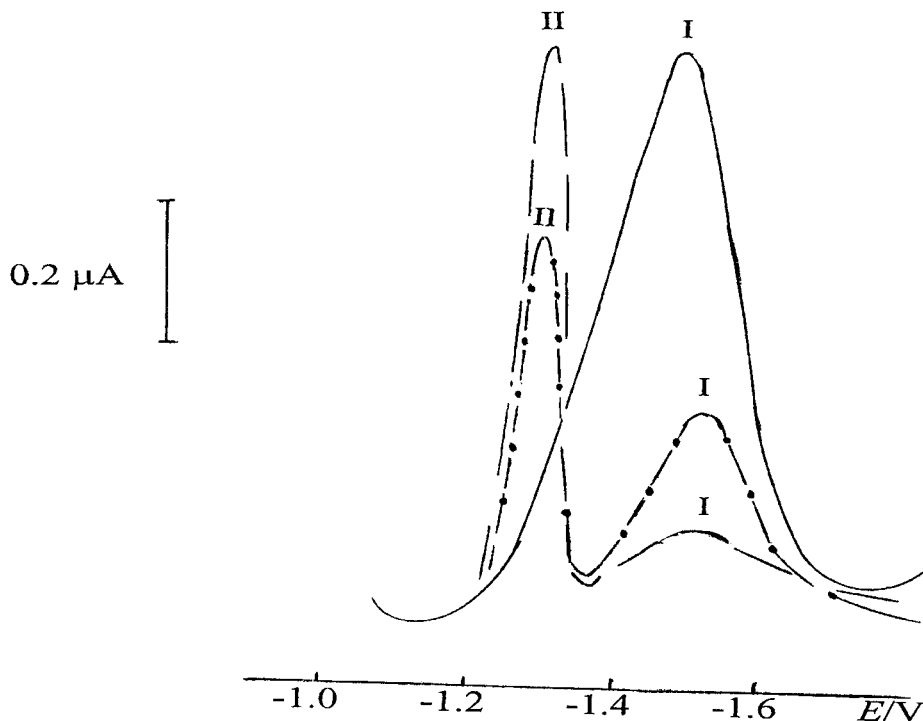


However, in aqueous solutions of  $0.1 \text{ mol/L}$   $\text{Na}_2\text{SO}_4$  and  $\text{NaCl}$ ,  $\text{Te(VI)}$  is reduced to  $\text{Te(0)}$  at a potential of about  $-1.60 \text{ V}$ , according to the equation [3]:



The product of last reaction  $\text{Te(0)}$  is a surface active and it can be adsorbed at the mercury electrode. On the other hand, the reduction of  $\text{Te(0)}$  to  $\text{Te}^{2-}$  was carried out at a potential of about  $-1.25 \text{ V}$  [3,4]. So, to reach better sensitivity, firstly we must produce  $\text{Te(0)}$  species at negative potentials, and than by "jumping" from negative to more positive potentials we can reduce  $\text{Te(0)}$  to  $\text{Te}^{2-}$ . At the deposition potential greater than  $-1.80 \text{ V}$  only the peak at potential of  $-1.25 \text{ V}$  exists. Namely, at accumulation potential of  $-1.80 \text{ V}$  all  $\text{Te(VI)}$  ions are reduced to  $\text{Te(0)}$  and thus by scanning from  $-0.9$  to  $-1.8 \text{ V}$   $\text{Te(0)}$  species can be reduced to  $\text{Te}^{2-}$ . This observation is in agreement with the literature data. A few square-wave voltammograms shown in the Fig. 1, which are recorded at different accumulation potentials confirmed above discussion. The peak I is the reduction of  $\text{Te(VI)}$  to  $\text{Te(0)}$  and the peak II is assigned as the reduction of  $\text{Te(0)}$  to  $\text{Te}^{2-}$ . So, all further measurements were achieved by scanning from  $-1.10$  towards  $-1.80 \text{ V}$  with deposition at a potential of  $-1.80 \text{ V}$ . This approach enables us to develop an adsorptive stripping method for quantitative determination of  $\text{Te(VI)}$ .

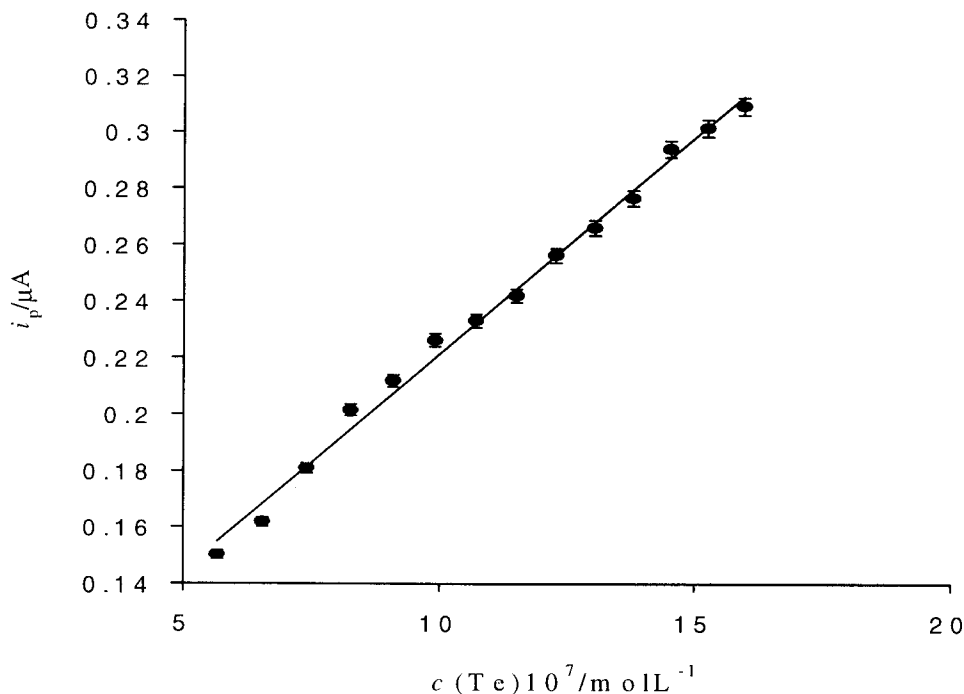
The curve which presents the dependence of the peak current on the accumulation time has a parabolically shape with a maximum located at accumulation time of about  $30 \text{ s}$ . The addition of  $0.5 \text{ mL}$  methanol, whose molecules shown adsorption properties on the mercury electrode surface decrease the peak current of  $\text{Te(VI)}$  because of competitive adsorption. The optimization of the instrumental parameters such as frequency  $f$ , amplitude  $E_{\text{sw}}$  and scan increment  $dE$  was achieved. It was found that the frequency of  $120 \text{ Hz}$ , the amplitude of  $20 \text{ mV}$  and the scan increment of  $4 \text{ mV}$  are suitable for quantitative determination of  $\text{Te(VI)}$ .



**Figure 1.** Square-wave voltammograms of Te(VI) recorded with different accumulation potentials.  $c(\text{Te(VI)}) = 1 \times 10^{-5}$  mol/L, frequency  $f = 120$  Hz, amplitude  $E_{\text{sw}} = 20$  mV, scan increment  $dE = 4$  mV, accumulation time  $t_{\text{acc.}} = 5$  s.  $E_{\text{acc.}} = -1.00$  V (\_\_\_\_),  $-1.50$  V (\_\_\_\_) and  $-1.80$  V(- - - -). Starting potential  $E_s = -0.9$  V and final potential  $E_f = -1.8$  V. Supporting electrolyte was  $0.1$  mol/L  $\text{Na}_2\text{SO}_4$ .

The calibration curve of Te(VI) in the  $10^{-7}$  mol/L concentration range is presented in the Fig. 2. The correlation coefficient  $R^2$  is greater than 0.99. The detection limit is about  $9 \times 10^{-8}$  mol/L, while the reproducibility of the method varied from 0.2 to 3.0 %.

The effect of interferences was studied and it was found that Cr(III) and Co(III) at a concentration level greater than  $1 \times 10^{-7}$  mol/L as well as Zn(II) and Fe(III), at a concentration level greater than  $5 \times 10^{-7}$  mol/L significantly influences the SW voltammetric response of Te(VI).



**Figure 2.** Calibration curve for Te(VI). Accumulation time  $t_{\text{acc.}} = 10$  s, accumulation potential  $E_{\text{acc.}} = -1.8$  V. Other conditions as in the Fig. 1.

## References

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## Апстракт

Извршено е испитување на електрохемиските особини на Te(VI) со циклична и квадратно-бранова волтаметрија. Целта на овој труд е да се разработи метода за негово квантитативно определување. Методата се состои во редукција на Te(VI) до елементарен телур, при потенцијал на акумулација од -1,80 V. Продуктот на редукцијата, Te(0), има особина да се адсорбира на површината од живината капка што овозможува негова предконцентрација. Адсорбираниот Te(0) потоа при катодна поларизација се редуцира до  $\text{Te}^{2-}$  на потенцијали од околу -1,30 V. Извршена е оптимизација на експерименталните и инструменталните параметри за квантитативно определување на Te(VI). Испитано е влијанието на интерферентните јони ( $\text{Cu}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Mn}^{4+}$ ,  $\text{V}^{5+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Ni}^{2+}$ ). Границата на детекција на Te (VI) во 0,1 mol/L раствор на  $\text{Na}_2\text{SO}_4$ , при акумулација од 10 s изнесува  $9 \times 10^{-8}$  mol/L. Репродуцибилноста на методата се движи во границите од 0,2 до 3,0%.