DETERMINATION OF SILVER CONTENT IN BATHS OF PHOTOGRAPHIC FIXATION BY POTENTIOMETRIC TITRATION

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Abstract: Silver ion was determined in baths of photographic fixation by means of potentiometric titration. For this purpose, thiocinamamide (3-phenyl-thiopropenamide) was used as a reagent, in presence of a strong basic medium (*p*H=12÷13). The proposed method is accurate, simple and fast.

Keywords: silver; potentiometric determination; photographic bath.

Introduction

During the process of fixation of photographic materials, by means of different solutions containing as a main component the anion $S_2O_3^{2-}$, a variation of the silver concentration occurs. A proportion of about 70% of silver halides is converting in metallic silver, and the rest of it is will be found in the fixation solutions as a complex with the anion thiosulphate, having the formula: $[Ag(S_2O_3^{2-})_2]^{3-}$. The knowledge of the silver concentration in fixation bath is useful for control of its efficiency. Besides that, in case of the recovery of the silver amount from fixation baths containing residual solutions, the knowledge of silver concentration is also important. For these reasons, there haven been achieved several methods of determining silver content from media containing the ion $S_2O_3^{2-}$ [1-6]. The photographic solutions being darken and containing many suspensions, do not allow the utilization of other methods, like spectrometric methods [7-10]. Unlike the mentioned methods, the potentiometric methods allow the determination of silver content in fixation baths.

In the present work it was achieved a method of determining the silver ion content in fixation baths, using thiocinamamide for the potentiometric titration. In very strong basic solutions this compound is a source of sulfide anion, according to the equilibrium:

$$C_6H_5\text{-}CH{=}CH\text{-}CS\text{-}NH_2 + 2\ OH^- \Leftrightarrow C_6H_5\text{-}CH{=}CH\text{-}COO^- + NH_4^+ + S^{2-}$$

This equilibrium can be used for the determination of Ag(I) due to the formation of Ag_2S. Moreover, thiocinamamide is an etalon substance, which can be weighted and dissolved in methanol in order to obtain a standard solution.

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Experimental

For the measuring the electroworking tension it was used an electrometric cell containing silver electrode as an indicator electrode, and calomel-saturated electrode with a double junction as the reference electrode. The electrochemical cell was linked to a pH-meter/millivoltmeter MV-84 Clahmann-Grahnet. For the titration procedure a solution of 10^{-2} M thiocinamamide was used. Sample to be analyzed that contains about 0.05 – 5 g Ag/l is bring to a pH within the range 12 – 13 with the aid of a 2N NaOH solution, and after that it is titrated with thiocinamamide. The solution is vigorously shaken with a magnetic shaker.

Results and Discussions

For the optimization of the titration procedure, at the beginning a solution containing 5 mg silver, bring to a *p*H=12÷13 is titrated with thiocinamamide. In the Fig. 1 the results of this titration procedure are presented. From this figure a clearly defined potential shift can be observed, which can be used for the calculation of the silver content in the fixation bath.

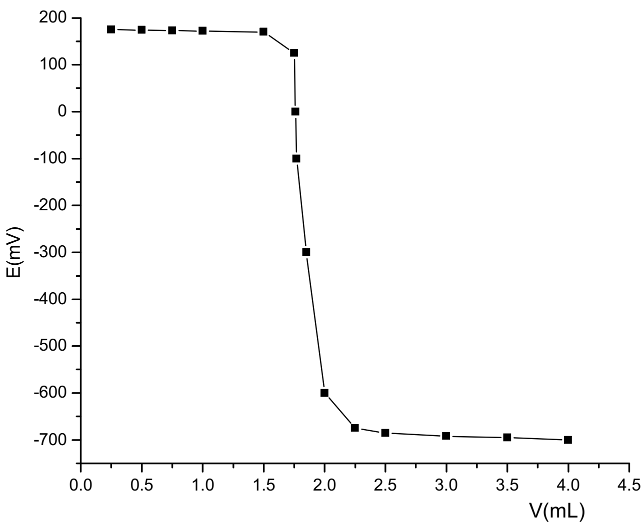

Fig. 1: *Potentiometric titration curve of Silver (I) with thiocinamamide.* (silver amount = 5 mg; $C_{thiocinamamide} = 10^{-2}$ M).

The titration mentioned in Fig. 1 is repeated, but this time in presence of 5 ml solution of 20% $Na_2S_2O_3$. By studying the effect of variation of the $Na_2S_2O_3$ concentration, it was established that it does not influence the results of the determinations. Besides that, variations of the *p*H value within 1÷2 units near the value of *p*H=12 does not influence the determinations.

In Fig. 2 the influence of the temperature upon the titration curve is presented. The experiments were performed in the same conditions like those used for obtaining the curve I, and for temperature value of 25 and 50°C (curve II).

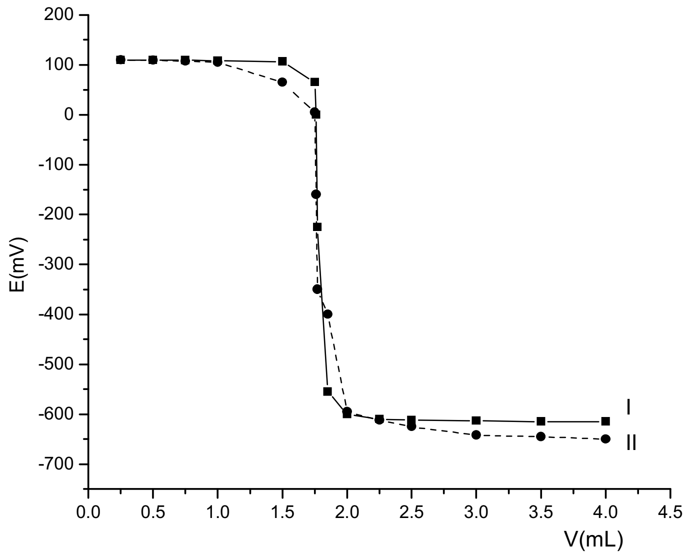

Fig. 2: *Titration curves for the determination of silver with thiocinamamide,* for 25°C (I), and 50°C (II).

From Fig. 2 one can observe that for different temperatures the equivalence volume is the same for a constant quantity of silver in sample analyzed. However, the titration curve is more clearly outlined at temperature of 25°C, and for this reason, this temperature was used in the next determinations.

The determinations of the silver content in the baths of photographic fixation has led to the reproducible analytical results and a good agreement with the gravimetric method for determination o silver, using Na_2S as a precipitation reagent.

Conclusions

The proposed method allows the determination of silver content in the baths of photographic fixation. The method is accurate, simple and fast. In comparison with the other methods for silver determination in the same sample, this method does not require buffer solutions or the maintenance of a certain temperature by using a thermostat.

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