

# DETERMINATION OF ANTHRANILIC ACID BY POTENTIOMETRIC TITRATION

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Cerium (IV) sulfate solution in sulfuric acid was used for the determination of *o*-aminobenzoic acid in sulfuric acid solution by potentiometric titration. The optima working conditions were set up for the quantitative determination of *o*-aminobenzoic acid. The method is simple, rapid and reliable. The effects of the acidity of the reaction medium and of the titrant concentration have been investigated.

## Introduction

The *o*-aminobenzoic acid is known as anthranilic acid (AA) or vitamin L. The anthranilic acid is a white to pale yellow crystalline powder, freely soluble and stable under ordinary conditions. It is used as an intermediate for production of dyes, pigments and saccharin as well as for perfumes and pharmaceutical products. This acid oxidizes in light and it is used as a corrosion inhibitor for metals. Various methods have been used for determination of *o*-aminobenzoic acid such as: spectrometric [1,2] and chromatographic [3÷6] methods. There are few titrimetric methods that imply direct determination of it [7], the most of them are indirect titration [8,9]. In this work we carried out a simple, rapid and reliable method for assay of *o*-aminobenzoic acid by direct titration. The optima working conditions for the direct quantitative determination of anthranilic acid by redox potentiometric titration with Ce (IV) solution have been established.

## Experimental

### Reagents

All the reagents used were of analytical reagent grade. Anthranilic acid was provided by Sigma,  $1 \times 10^{-1} \text{ mol L}^{-1}$  cerium (IV) sulfate solution in sulfuric acid  $1 \text{ mol L}^{-1}$  and the concentrate sulfuric acid  $d = 1.84 \text{ g cm}^{-3}$  were provided by Merck. Solution  $1 \times 10^{-1} \text{ mol L}^{-1}$  of sulfuric acid has been obtained by dilution of the concentrate sulfuric acid with distilled water.  $5 \times 10^{-2} \text{ mol L}^{-1}$  and  $1 \times 10^{-1} \text{ mol L}^{-1}$  cerium (IV) sulfate solution in  $1 \text{ mol L}^{-1}$  sulfuric acid and respectively anthranilic acid  $1 \times 10^{-2} \text{ mol L}^{-1}$  in sulfuric acid 1M have been prepared. The buffer solutions have been obtained according to standard method [10], (mixtures of  $2 \times 10^{-1} \text{ mol L}^{-1}$  hydrochloric acid solution,  $2 \times 10^{-1} \text{ mol L}^{-1}$  potassium chloride solution and  $2 \times 10^{-1} \text{ mol L}^{-1}$  potassium hydrogen ftalate solution).

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## Apparatus

All the potentiometric measurements were made with a Consort P901 pH/mV-meter. A platinum redox electrode was used as indicative electrode for titration. A saturated calomel electrode was used as reference.

## Procedure

The two electrodes were immersed into Berzelius beaker containing accurately measured volumes of AA solutions diluted to the 20 mL with sulfuric acid solutions of various concentrations and buffer solutions respectively. The electromotive force at the system was recorded after each addition of the titrant solution (cerium (IV) sulfate solution).

## Results and discussion

In order to establish the optima working conditions the influence of the acidity of the reaction medium and of the concentration of Ce(IV) solution have been studied. The data obtained were used to draw the titration curve. From each curve the equivalence volume, the apparent redox standard potential of the AA oxidized form / AA reduced form, the apparent redox standard potential of the couple  $\text{Ce}^{4+} / \text{Ce}^{3+}$ , and the number of electrons exchanged during the redox reaction have been determined. The equivalence volume was determined also by the first and the second derivative of the titration curve. In Fig. 1 a titration curve of AA with Ce(IV) solution is shown.

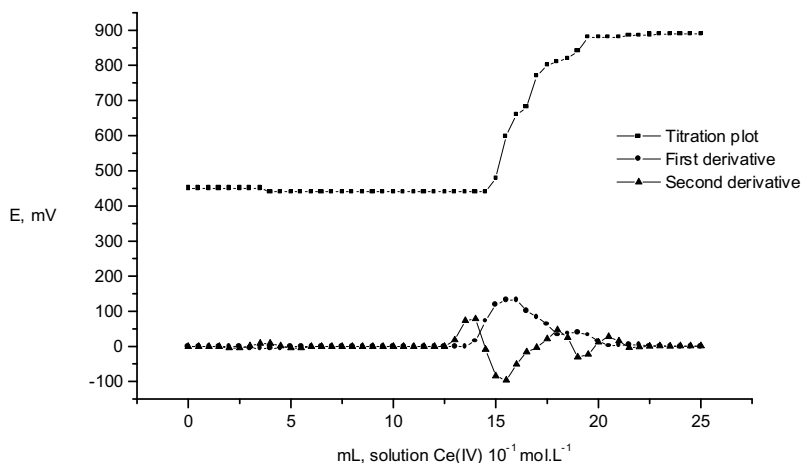


Fig. 1 – Titration curves of AA with  $1 \times 10^{-1} \text{ mol L}^{-1}$  cerium (IV) sulfate solution.

The data reported in Table 1 show that the titrant solution concentration influences the apparent standard potential of the two redox couples and the number of the electrons exchange during the redox reaction. The redox reaction occurs harder when the titrant concentration decreases. In that case the redox standard potential of the  $\text{Ce}^{4+} / \text{Ce}^{3+}$  is lower and the number of electrons exchanged during the redox reaction is modified. For the assay of AA a solution  $1 \times 10^{-1} \text{ mol L}^{-1}$  of cerium (IV) sulfate solution made in sulfuric acid

1mol L<sup>-1</sup> must be used. In these condition the reaction ratio between AA: Ce (IV) is 1:3. The reaction ratio between AA and Ce (IV) is also influences by the acidity of the sample. As it can be seen in the Table 1 the oxidation rate decrease with the increase of the titration medium acidity.

**Table 1. The influence of the cerium (IV) sulfate solution concentration and the sample acidity**

AA sample		Cerium (IV) sulfate solution		$\varepsilon^{\circ}_{OX/RED}$ (mV)	$\varepsilon^{\circ}_{Ce^{4+}/Ce^{3+}}$ (mV)	n
Conc, M	Acidity	Conc, M	*V <sub>e</sub> , mL			
10 <sup>-2</sup>	pH = 0.30	10 <sup>-1</sup>	5.27	746	1263	5
10 <sup>-2</sup>	H <sub>2</sub> SO <sub>4</sub> 1 M	10 <sup>-1</sup>	3.22	806	1160	3
10 <sup>-2</sup>	pH = 2.20	5x10 <sup>-3</sup>	15.71	686	1129	4
10 <sup>-2</sup>	H <sub>2</sub> SO <sub>4</sub> 1 M	5x10 <sup>-3</sup>	18.04	776	1106	4.5

Where:  $\varepsilon^{\circ}_{ox/red}$  = the apparent redox standard potential of the AA oxidised form / AA reduced form; n= the number of electrons exchanged during the redox reaction;  $\varepsilon^{\circ}_{Ce^{4+}/Ce^{3+}}$  = the apparent redox standard potential of the Ce<sup>4+</sup>/Ce<sup>3+</sup>; V<sub>e</sub> = the equivalence volume, mL cerium (IV) sulfate solution.

\* Each value is the mean of five determinations.

### **Analytical application**

The proposed method was applied for the assay of AA in solution containing known amounts of AA. The assay was determined also spectrometrically with reference at a standard solution (Table 2). The results obtained by the two methods proved that the proposed method could be used for the quantitative determination of AA.

**Table 2. Results of AA assay on synthetic samples application of the method**

Taken	Amount (mg)	
	Found by*:	
	Proposed method	Spectrometric method <sup>1</sup>
0.27	0.25±0.02	0.26±0.01
0.55	0.54±0.01	0.56±0.03
1.37	1.36±0.01	1.35±0.01
2.05	2.04±0.03	2.03±0.01
2.45	2.46±0.01	2.43±0.02

\* each value represents the mean of five determination ±s.

### **Conclusions**

The optima conditions for the assay of o-aminobenzoic acid by potentiometric titration with 10<sup>-1</sup> mol.L<sup>-1</sup> cerium (IV) sulfate solution in 1 mol L<sup>-1</sup> sulfuric acid have been determined. From the titration plot the apparent redox standard potential of the o-aminobenzoic acid oxidised form /o-aminobenzoic acid reduced form,  $\varepsilon^{\circ}_{ox/red} = 754 \pm 52$  mV; the number of electrons exchanged during the redox reaction, n = 3; the redox standard apparent potential of the Ce<sup>4+</sup> / Ce<sup>3+</sup>,  $\varepsilon^{\circ}_{Ce^{4+}/Ce^{3+}} = 1165 \pm 70$  mV have been determined. The concentration of

cerium (IV) sulfate solution influences the apparent redox standard potential values and raport ratio between AA: Ce (IV). The optima working conditions for the assay of AA implies the use of a titrant having the concentration of  $1 \times 10^{-1} \text{ mol L}^{-1}$  solution made in sulfuric acid  $1 \text{ mol L}^{-1}$ . In this condition the reaction ratio between AA: Ce (IV) is 1:3. The potential values rise at higher concentration, so the redox process occurs harder. The oxidation rate decreased with the increase of the titration medium acidity. The method shows good results when it was applied on synthetic samples. The results obtained prove that the method proposed can be used for quantitative determination of o-aminobenzoic acid when the optima working conditions are applied.

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