Gravimetric Determination of Saccharin in Some Sweetners Using Mercurous Nitrate as Reagent

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Propõe-se neste trabalho um método gravimétrico rápido, preciso e de baixo custo para determinação de sacarina em adoçantes, usando nitrato mercuroso. Os melhores resultados encontrados foram em pH igual a 2 com um desvio padrão relativo de 4,3%. Ciclamato, ácido benzoico, lactose, frutose, glicose e sacarose não interferem quando presentes, mesmo em concentrações elevadas.

A rapid, precise and low cost gravimetric method for saccharin determination in some sweetners using mercurous nitrate is proposed. The best results were at pH 2 with a relative standard deviation of 4.3%. Cyclamate, benzoic acid, lactose, frutose, glucose and sucrose do not interfere even in significant amounts.

Key Words: *saccharin ; gravimetry; mercurous ion; sweetners.*

Introduction

Saccharin (o-benzoic sulfimide, $C_6H_4COSO_2NH$) and its salts are white crystalline powders, odorless and in diluted solution are about 400-500 times[1] sweeter than sucrose. Due to their characteristics, they are widely used in medicine, toilete articles, dietary products and also in galvanic process as brightener. There are several analytical procedures cited in literatute for determining saccharin such as chromatography[2-8], potentiometry[9-17], polarography[18-22], ultraviolet spectrphotometry[23-28] fluorimetry[29], volumetry[30] and gravimetry[18,31]

The gravimetric method[18,31] for the determination of this sweetner is based in the bromation in alkaline medium (Hoffman reaction) following the precipitation of $BaSO_4$(s) with $BaCl_2$ solution. Nevertheless, this method is time-consuming and laborious and low sensitivity.

Recently[32] we have developed a potentiometric method for saccharin determination in several diet products based on the low solubility of mercurous saccharinate, $Hg_2[NSO_2COC_6H_4]_2$ (s).

In this work, a new gravimetric method for determination of this sweetner is proposed. Saccharin in aqueous solutions was quantitatively precipitated with mercurous nitrate and weigthed directly after drying. The proposed method is faster than that gravimetric method reported[18,31] precise, accurate, inexpensive and does not need previous preparation to remove interfering substances.

Experimental

Apparatus. pH measurements were carried out using a Orion potentiometer, model EA 940 with a precision of 0.01 units of pH. The indicator electrode used was Analion glass electrode, model V 620 and the reference was a silver-silver chloride electrode in 0.1 M KCl solution with a 3.0 M $NaNO_3$ brigde. All weigthing were carried out on a Mettler analytical balance, model H10 with 0.05 mg precision.

Reagents. All reagents used were of analytical grade. The saccharin stock solution was prepared by dissolving 4.580 g of saccharin (Aldrich, 99%) in 0.1 M NaOH solution and diluted to 250 ml in a volumetric flask.

The reference solutions were prepared from the stock solution. The 10^{-2} M mercurous nitrate solution was prepared by dissolving 2.625 g of $Hg_2(NO_3)_2$ (Aldrich, 99%) in 10^{-2}M HNO_3, diluted with the same solution to 500 ml and then standardized with 0.01 M KCl solution by potentiometric titration as described elsewhere[32].

Assugrin liquid sweetener and Doce Menor powder (Vepê Indústria Alimentícia Ltda., São Bernardo do Campo, S.P., Brasil), Dietil liquid sweetner (Nutrícia S.A., Rio de Janeiro, R.J., Brasil) and Sucaryl liquid sweetener (Abbott Laboratório do Brasil, S.P., Brasil) were purchased from a local food store.

General Experimental Procedures

a)Powder Sweetners. An accurately weigthed amount of 2-5 g of the solid sweetner was transfered to 50.0 ml volumetric flask and the sample was dissolved and diluted to volume with 0.1 M HNO_3 to pH 1.5- 2.0. An aliquot containing 3 - 30 mg of saccharin was transfered to the Erlenmeyer flask and saccharin was precipitate with 10 ml of 10^{-2}M mercurous nitrate solution.

b)Liquid Sweetners. A volume of 2.0 ml of each liquid sweetener sample was diluted to 50.0 ml in a volumetric flask with 0.1 M HNO_3 to pH 1.5-2.0. An aliquot of 5.0 ml of this solution was treated as describes to powder sweetner (a). After precipitation of $Hg_2[NSO_2COC_6H_4]_2$(s), this substance was collected on a tared gooch crucible, dry during 2 h in a furnace, cooled in a desiccator and weigthed.

Results And Discussion

Effect of pH. The effect of pH, ranging from 1.0 to 3.0, on the determination of 10^{-2} M saccharin by 10^{-2} M of mercurous nitrate was studied. The best results was obtained in the pH range 1.5-2.0. There is a decrease of the yield at pH less than 1.5 due the protonation of the saccharinate anion (pKa = 1.6)[33] also at pH above 3.0 due the hydrolysis of mercurous cation to Hg_2O. A 10^{-2}M HNO_3 solution at (pH 2) was then chosen for further work. Analysis of ten analytical solutions containing 37.9 mg of sodium saccharinate in pH 2, gave a mean weight of 36.5±1.6 mg and relative standard deviation of 4.3%.

Table 1. *Determination of saccharin in some sweetner using the gravimetric method.*.

	Saccharin (mg/ml)			
sweetner	spectrophotometric	found*	error(%)	Δ**
Assugrin	58.7±2.1	59.3±2.3	+1.0	3.9
Dietil	49.5±1.0	46.8±0.84	-5.4	1.7
Sucaryl	59.7±1.3	61.2±0.3	+2.5	0.5
Doce Menor*	26.2±1.9	24.5±0.2	-6.5	0.7

**mg/g; ** coefficient of variation.*

+ Assay values represent the average of five (n = 5) determinations per sample with a confidence level of 95%.

Effect of Foreign Substances. Several potential interferentes which would be expected to exist in sweetner such as cyclamate, benzoic acid, lactose, frutose, glucose and sucrose were investigated in amount of at least twenty times higher than that of saccharin and no one cause any interference. Only anions (e.g. chloride, phosphate) that precipitate Hg(I) cation cause interferences. In these cases, saccharin should be extracted with ethyl acetate before analysis.

Determination of Saccharin in Sweetners. Table 1 presents a comparison between a UV spectrophotometric method, reported as one of the most used methods for saccharin determination[23] and the gravimetric method proposed in this paper. The results are in close agreement with those reported and within an acceptable range of error.

The gravimetric method for the determination of saccharin in some sweetner reported in this paper is faster, more precise and of lower cost than that reported earlier[18,31]. It is also suitable for routine analysis of saccharin in various sweetners.

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