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DETERMINING TARTRATE IONS IN THE SAMPLES OF MINERAL TABLE WATERS BY THE DECAY OF MOLECULAR LUMINESCENCE OF RUTIN IN COMPLEX WITH YTTRIUM (III)

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Abstract. The yttrium (III)-rutin (Rut) complex in the presence of bovine serum albumin (BSA) is suggested as a luminescent sensor to determine tartrate ions (Tart). It has been experimentally established that tartrate ions reduce the luminescence intensity (I_{lum}) of the Y(III)-Rut complex in the presence of BSA and Tart. The spectral and luminescent properties of the Y(III)-Rut complex in the presence of BSA have been studied. The peak of the luminescence spectrum of the Y(III)-Rut complex in the presence of BSA is at λ=570 nm. In the presence of potassium tartrate, I_{lum} of the Y(III)-Rut complex decreases, and the maximum luminescence shifts to the longwave region of the spectrum (λ=590 nm). It is known that the luminescence decay can be caused by various processes, including reactions in the excited state, energy transfer, formation of complexes, and collisional decay. It can be assumed that the decay effect of the Y(III)-Rut complex is due to the complexation reaction of Y(III) with Tart, that leads to the destruction of the Y(III)-Rut complex. The luminescence decay of the Y(III)-Rut complex in the presence of BSA by means of Tart follows the Stern-Volmer relationship. The Stern-Volmer constant K is 1230 l/mol. The method of luminescent determination of tartrate ions in mineral table waters has been developed. It is based on using the decay of rutin's molecular luminescence in the Y(III)-rutin complex in the presence of BSA. The linear calibration plot for tartrate ions has been obtained over the range of Tart concentrations of 0.02 to 0.20 mg/ml. The limit of determining potassium tartrate is 0.01 mg/ml. The technique has an advantage over the existing ones due to the absence of toxic reagents, and short-time analysis. Besides, it allows rapid screening of samples of mineral table water.

Keywords: tartrate ions, luminescence, ion of yttrium (III), rutin.

ВИЗНАЧЕННЯ ТАРТРАТ-ІОНІВ У ЗРАЗКАХ СТОЛОВОЇ МІНЕРАЛЬНОЇ ВОДИ ЗА ГАСІННЯМ МОЛЕКУЛЯРНОЇ ЛЮМІНЕСЦЕНЦІЇ РУТИНА В КОМПЛЕКСІ З ІТРІЄМ (III)

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Анотація. В якості люмінесцентного сенсора для визначення тартрат-іонів (Tart) запропоновано використання комплекс ітрій (III)-рутин (Rut) у присутності бичачого сироваткового альбуміну (BSA). Експериментально встановлено, що тартрат-іони зменшують інтенсивність люмінесценції ($I_{люм}$) комплексу Y(III)-Rut у присутності BSA. Вивчено спектрально-люмінесцентні характеристики комплексу. Спектр люмінесценції комплексу Y(III)-Rut у присутності BSA має максимум при $\lambda_{люм.}$=570 нм, у присутності Tart $I_{люм.}$ комплексу Y(III)-Rut зменшується та максимум люмінесценції зсувається в довгохвильову область спектру ($\lambda_{люм}$=590 нм). Відомо, що до гасіння люмінесценції може призводити безліч процесів, у тому числі реакції в збудженому стані, перенесення енергії, утворення комплексів і гасіння при зіткненнях. Можна припустити, що виявлений ефект гасіння молекулярної люмінесценції рутина в комплексі Y(III)-Rut, обумовлений утворенням комплексної сполуки ітрію (III) з Tart та руйнуванням комплексу Y(III)-Rut. Гасіння люмінесценції комплексу Y(III)-Rut у присутності BSA за допомогою Tart підпорядковується співвідношенню Штерна-Фольмера. Константа Штерна-Фольмера К становить 1230 л/моль. Розроблено методику люмінесцентного визначення Tart у столових мінеральних водах, яка ґрунтується на використанні гасіння молекулярної люмінесценції рутина в комплексі Y(III)-Rut у присутності BSA. Калібрувальний графік лінійний у діапазоні концентрацій Tart від 0,02 до 0,2 мг/мл. Границя визначення тартрата калія складає 0,01 мг/мл. Методика вигідно відрізняється від тих, що існують, відсутністю токсичних реагентів, нетривалим часом аналізу, дозволяє здійснювати швидкий скринінг зразків столової мінеральної води.

Ключові слова: тартрат-іони, люмінесценція, іон ітрія (III), рутин

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Introduction. Formulation of the problem

In the course of living activity, the human body loses many salts and microelements, and it is mineral waters that can become an adequate source of their replenishment. According to DSTU 878:2006, packaged natural underground mineral waters, characterised by mineralisation from 0.1 g/dm^3 to 1.0 g/dm^3 and the sta-

bility of the physical and chemical composition, are attributed as mineral table waters. Natural mineral waters are used with no restrictions as for the frequency of use. They are used for cooking, so the control of their quality, and development of highly sensitive methods of quantitative determination of impurities in mineral table water samples seem relevant. Information on the mineral composition of the water should be on the label of the bottle. Unfortunately, many manufacturers do not provide all the necessary information, or the information is not always true. The presence of food additives is detrimental to human health, since the influence of any chemical substance on the human body depends on the individual characteristics of the organism, and on the amount of the influencing substance. The food additive E336 (potassium tartrate, potassium tartaric acid) is recognised as safe for human life and health and is therefore widely used in food industry, in particular to improve the quality and taste of mineral table water.

Potassium tartrate belongs to the group of natural antioxidants (Fig. 1), and acts as a regulator of acidity, stabiliser, acidifying agent, emulsifier. It is present in instant soups, candied fruit jelly, jams, juices, and table waters, in amounts from 50 to 200 mg/100 g of product [1,2].

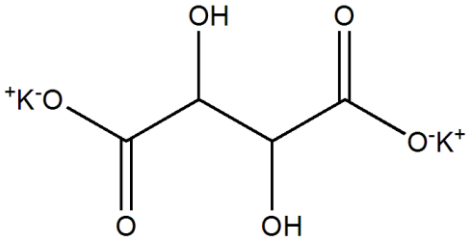

Fig. 1. Structure of potassium tartrate

The additive E336 affects the venous-vascular system of a person, and reduces the flow of venous blood. Besides, potassium tartrate has a mild laxative and diuretic effect, and regulates bile outflow. Tartrates contained in urine are potential inhibitors of the development of stones in the kidneys [3,4], since they form relatively stable complexes with calcium [5,6]. The daily dose of E336 is defined as that of tartaric acid and is 30 mg/kg of body weight.

Analysis of recent research and publications

It is known that amplification or decay of emission of lanthanide luminescent probes by means of different analytes open up new possibilities for highly sensitive determination of substances that cannot sensitise 4f-luminescence on their own. Tartrate potassium is one of these substances. At present, the quantitative determination of tartrate in various objects is carried out mainly by titrimetric, spectrophotometric, electrochemical, and chromatographic methods of analysis [7-10]. One titrimetric and two spectrophotometric methods for the assay of metaprolol tartrate (MPT) in bulk drug and in tablets are described in [7]. The methods employ N-bromosuccinimide (NBS) as the oxidimetric reagent, and two dyes, methyl orange and indigo carmine, as spectrophotometric reagents. In titrimetry, an acidified solution of MPT is treated with a known excess amount of NBS, and after a definite time, the unreacted oxidant is determined by iodometric back titration. Spectrophotometry involves adding a measured excess of NBS to MPT in acid medium followed by determining the residual NBS by reacting with a fixed amount either of methyl orange, with the absorbance measured at 520 nm, or of indigo carmine, with the absorbance measured at 610 nm [7]. The paper [8] presents two validated methods for UV-VIS quantitative assay of metoprolol tartrate in tablets: the UV direct method and the oxidative method with potassium permanganate. The parameters targeted for the validation were: selectivity, quantification range and quantification limits, accuracy, and precision. The authors [9] suggested a simple densitometric method to determine metroprolol tartrate in tablets and ampoules. After separation into silicagel GF254 plates, using acetone-methanol-triethylamine as the mobile phase for the tablets and acetone triethylamine for ampoules, the chromatographic zones corresponding to the spots of metoprolol were scanned. Quantitative evaluation was performed using a computer-controlled Camag TLC scanner and applying five-point calibration with polynomial regression. The calibration function was established in the ranges 1–28 μg for tablets and 1–9 μg for ampoules [9]. The paper [10] deals with the development and validation of an analytical method based on high performance single column ion chromatography and on conductometric detection, developed to measure tartaric acid.

Most methods of determining tartrate are complex, take a lot of time, and, as a rule, require expensive equipment. Consequently, developing cheaper, more selective methods, simple to implement and express, suitable for analysis remains a relevant issue. This paper presents the results of studies on the determination of tartrate ions in the samples of mineral table waters. The determination is based on the decay of molecular luminescence of rutin in complex with yttrium (III).

The aim of this research was to study the possibility of determining tartrate ions in mineral table waters, with the use of the decay of molecular luminescence of rutin in the complex Y(III)-Rut in the presence of bovine serum albumin.

Tasks of the research.

1. To study the spectral-luminescent properties of the Y(III)-Rut complex in the presence of tartrate ions.
2. To study the mechanism of quenching molecular luminescence of Rut in a complex with yttrium (III) in the presence of BSA by tartrate ions.
3. To develop the method of determining tartrate ions in mineral table waters.

Research Materials and Methods

A stock solution of Tart ($1 \cdot 10^{-2}$ mol/l) was prepared by dissolving a weighed quantity of Tart in 100 ml of distilled water. A stock solution of Rut ($1 \cdot 10^{-2}$ mol/l) was prepared by dissolving a weighed quantity of Rut in 100 ml of ethanol. Yttrium (III)

chloride was prepared by dissolving high purity yttrium (III) oxide (99.99%) in hydrochloric acid (1:1), and excess hydrochloric acid was removed by evaporation. The concentration of yttrium (III) was determined by complexometric titration with a standard solution of Complexone III (0.01 mol/l) using arsenazo I as an indicator in a basic buffer solution of urotropine [11].

The steady-state luminescence spectra and the luminescence decay curves were recorded with a Fluorolog FL 3-22 spectrofluorometer (Horiba Jobin Yvon) and an EF-3MA fluorometer. The pH of the solutions was determined using a pH meter OP-211/1 Radelkis (Hungary) with a glass electrode. The desired pH value was created in the solution by means of urotropine.

Results of the research and their discussion

The rare-earth complexes with many organic ligands are coordinatively unsaturated and capable of attaching various electronegative ligands including organic and inorganic anions [12-15]. It is known [12-15] that rutin fluoresces upon excitation, when its ethanolic solution is illuminated with ultraviolet light from a mercury vapour lamp as the excitation source, but rutin's luminescence intensity (I_{lum}) is very weak. However, the intensity of luminescence of the ligand in some cases can increase when rutin is coordinated with a metal ion. For example, the ions of Y(III), La(III), Sc(III), Al(III) form complexes with rutin. It has been found [16] that the highest intensity of luminescence is in the Y(III)-Rut complex. That is why, it is suggested to use the Y(III)-Rut complex as a luminescent sensor to determine tartrate ions. It has been established [17] that the luminescence intensity of this complex increases in the presence of bovine serum albumin (BSA) that belongs to globular proteins. The rutin complex in the local molecular environment of the globular protein solubilises due to electrostatic and hydrophobic interactions. This prevents interaction between the fluorophore and water molecules, thus reducing radiation-free energy losses. In this case, the nature of the excitation and of the luminescence spectra does not change, only their intensity increases, the crests of the bands are not displaced nor split, which can be an evidence that the BSA molecules are not part of the inner sphere of the complex.

It has been experimentally established that tartrate ions reduce I_{lum} of the Y(III)-Rut complex in the presence of BSA. This phenomenon is used to develop a method of determining potassium tartrate. In this regard, it was practical to study the spectroscopic characteristics of the complex and to establish the possibility of using this luminescent marker for the determination of potassium tartrate.

The luminescence spectrum of the Y(III)-Rut complex in the presence of BSA has its maximum at λ=570 nm (Fig. 2). In the presence of potassium tartrate, I_{lum} of the Y(III)-Rut complex decreases, and the maximum luminescence shifts to the longwave region of the spectrum (λ=590 nm).

It is known that the luminescence decay may be caused by various processes, including reactions in the excited state, energy transfer, formation of complexes, and collisional quenching [18]. It can be assumed that the quenching effect of the Y(III)-Rut complex is due to the complexation reaction of yttrium (III) with tartrate ions, which leads to the destruction of the Y(III)-Rut complex. The complexes of yttrium with tartrate $[YH_3Tart]^{2+}$, $[YH_2Tart]^{+}$, $[Y_2H_2Tart]^{4+}$, $[Y(H_2Tart)_2]^{-}$ and HYTart in aqueous solutions were described by the authors [19,20]. Some complexes were isolated and characterised by thermogravimetric analysis, IR-spectroscopy, and X-ray diffraction [19, 20].

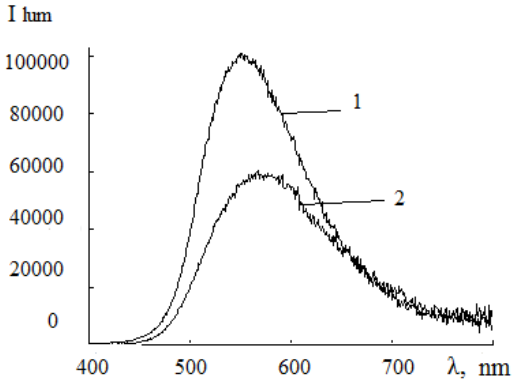

Fig. 2. Luminescence spectra of the Y(III)-Rut complex in the presence of BSA, in the absence of Tart (1), and in the presence of Tart (2)

In the excitation spectrum of the Y(III)-Rut complex in the presence of BSA and in the absence of Tart, there is a large band with the maxima at 300 nm, and in the presence of both Tart and BSA, in the excitation spectrum of the Y(III)-Rut complex, there is a wide, blurred band in the region of 290–380 nm with the maxima at 315 nm and 355 nm (Fig. 3).

The maximum quenching effect I_{lum} of the Y(III)-Rut complex in the presence of BSA is observed at pH 6.0–7.0 created in the solution by means of urotropin. The greatest decay I_{lum} is observed at the concentration Y(III) – $5 \cdot 10^{-3}$ mol/l, rutine – $5 \cdot 10^{-3}$ mol/l.

The luminescence quenching effectiveness is described by the Stern–Volmer equation:

$$\frac{I_0}{I} = 1 + k_q \cdot \tau_0 \cdot C = 1 + K \cdot C \qquad (1),$$

where I_0 and I are the luminescence intensity in the absence and in the presence of the quencher, respectively,

k_q is the biomolecule quenching rate constant, l/mol c

τ_0 is the lifetime of the fluorophore in the absence of the quencher, c,

K is the Stern–Volmer constant, l/mol,

C is the concentration of the quencher, mol/l.

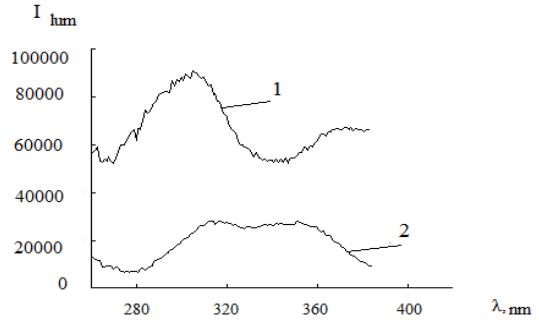

Fig. 3. Excitation spectra of the luminescence of the Y(III)-Rut complex in the presence of BSA, in the absence of Tart (1), and in its presence (2)

Tart's quenching the luminescence of the Y(III)-Rut complex in the presence of bovine serum albumin follows the Stern-Volmer relationship. The Stern-Volmer constant K calculated from the equation (1) was 1230 l/mol.

We used the effect of luminescence quenching of the Y(III)-Rut complex in the presence of BSA to develop the procedure for determining Tart in mineral table waters.

The determination was made by the calibration curve method. When plotting the calibration curve to determine Tart, the following procedure was used: in 10 ml test tubes, we placed 0.1; 0.2; 0.3; 0.4; 0.5; 0.6; 0.7; 0.8; 0.9; 1.0 ml of a standard solution of Tart ($1 \cdot 10^{-2}$ mol/l). To each tube, we added 0.2 ml of yttrium (III) chloride solution ($1 \cdot 10^{-2}$ mol/l), 0.2 ml of rutin solution ($1 \cdot 10^{-2}$ mol/l), 0.2 ml of urotropine solution with a mass fraction of 40%, 0.2 ml of bovine serum albumin (1 mg/ml) and distilled water to 10 ml. The luminescence intensity of the Y(III)-Rut complex was measured at λ_{lum}=570 nm upon excitation at 365 nm.

Based on the obtained data, a calibration curve was plotted. The calibration graph for Tart is linear in the range of $2 \cdot 10^{-4}$ mol/l–$1.0 \cdot 10^{-3}$ mol/l (Fig. 4).

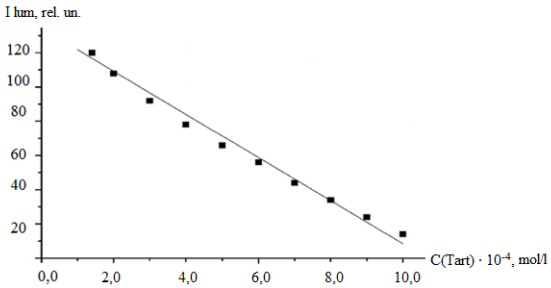

Fig. 4. Calibration curve for determining Tart

Method of determination: In three test tubes, 10 ml each, 1 ml of the water sample was taken. It was analysed, and all the reagents were added, like in the plotting of the calibration curve. The luminescence intensities of these solutions at 570 nm were measured upon excitation at 310 nm.

The results of determining potassium tartrate in 5 samples of mineral table water of various brands are given in the table. The accuracy, reliability, and correctness of the Tart determination is verified by statistical processing of the results of the analysis. When n=5, P=0.95, the relative standard deviation S_r is (3.3–4.5)%. The limit of determination of potassium tartrate is 0.01 mg/ml (Table 1).

Table 1 – Results of determining potassium tartrate in samples of mineral table water (n = 5, P = 0.95)

№	The name of the water sample	Trademark	Content of potassium tartrate (mg/ml)	S_r,%
1	*Zbruchanska*	PE "NSCLD ZBRUCH"	0.93±0.031	3.3
2	*PIRIN*	TM "PIRIN SPRING"	1.10±0.042	3.8
3	*Jermuk*	LLC "FOR FORM"	0.82±0.029	3.5
4	*Oasis green life*	LLC "CEDR LBN TRADE"	0.50±0.022	4.5
5	*Luzhanskaya*	LLC "FOOD TRADE OPTIMA"	0.45±0.017	3.7

As can be seen from the table, in all samples of water, potassium tartrate has been found, from 0.45 mg/ml in the water *Luzhansky* to 1.1 mg/ml in the *PIRIN* water, but manufacturers do not indicate its presence in the product on the label. It was classified as information falsification of a food product.

Conclusions

The method has been developed of luminescence determination of tartrate ions in the samples of mineral table water, which is based on quenching of molecular luminescence of rutin in the complex Y(III)-Rut in the presence of BSA. The technique differs favourably from those that exist by the absence of toxic reagents, expensive equipment, short-time analysis, and allows rapid screening of samples of table mineral water.

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