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ЖУРНАЛ ИССЛЕДОВАНИЯ И ИННОВАЦИИ | JOURNAL OF RESEARCH AND INNOVATIONS

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PHOTOMETRIC DETERMINATION OF IRON (III) WITH SULFOSALICYLIC ACID

ANNOTATION

The most prevalent element in nature is iron, which is also one of the essential, active macro elements. Today, photometric techniques are frequently employed to identify harmful and extremely poisonous heavy metals. Due to its sensitivity, simplicity, and short analysis time, this approach is extremely important. The technique of photometrically determining Fe (III) was devised, and the ideal conditions for the formation of a complex of Fe (III) with sulfosalicylic acid reagent were explored. The following steps were taken during the photometric determination of Fe (III) using sulfosalicylic acid: selection of a light filter, dependence of complex formation on environmental acidity, dependence on the composition of the buffer solution, dependence on the composition of the reagent, field of obedience to Beer's law, the ratio of the component moles of the complex, the Sendal sensitivity, the lowest detection limit of Fe (III), the molar extinction coefficient, and the technique of photometrically determining Fe (III).

Keywords: KFK-3, sulfosalicylic acid, beer's law, buffer solution, sensitivity according to molar extinction coefficient, photometric determination, complex formation.

ФОТОМЕТРИЧЕСКОЕ ОПРЕДЕЛЕНИЕ ЖЕЛЕЗА (III) С СУЛЬФОСАЛИЦИЛОВОЙ КИСЛОТОЙ

АННОТАЦИЯ

Наиболее распространенным элементом в природе является железо, которое также является одним из важнейших активных макроэлементов. Сегодня для выявления вредных и чрезвычайно ядовитых тяжелых металлов часто используются фотометрические методы. Из-за своей чувствительности, простоты и короткого времени анализа этот подход чрезвычайно важен. Была разработана методика фотометрического определения Fe (III) и исследованы идеальные условия для образования комплекса Fe (III) с реагентом сульфосалициловой кислоты. При фотометрическом определении Fe (III) с использованием сульфосалициловой кислоты были предприняты следующие шаги: выбор светофильтра, зависимость образования комплекса от кислотности окружающей среды, зависимость от состава буферного раствора, зависимость от состава реагента, область подчинения закону Бера, соотношение молярные доли компонентов комплекса, чувствительность датчика, нижний предел обнаружения Fe (III), коэффициент молярной экстинкции и методика фотометрического определения Fe (III).

Ключевые слова: КФК-3, сульфосалициловая кислота, закон Бера, буферный раствор, чувствительность по молярному коэффициенту экстинкции, фотометрическое определение, комплексообразование.

TEMIRNING FOTOMETRIK TA'RIFI (III) SULFOSALITSIL KISLOTASI BILAN

ANNOTATSIYA

Tabiatdagi eng keng tarqalgan element temir bo'lib, u ham eng muhim faol makroelementlardan biridir. Bugungi kunda zararli moddalarni aniqlash uchun va juda zaharli og'ir metallar ko'pincha fotometrik usullardan foydalanadi. O'zining sezgirligi, soddaligi va qisqa tahlil vaqti tufayli ushbu yondashuv juda muhimdir. Fe (III) ni fotometrik aniqlash usuli ishlab chiqildi va sulfosalitsil kislotasi reagenti bilan Fe (III) kompleksini hosil qilish uchun ideal sharoitlar o'rganildi. Sulfosalitsil kislotasi yordamida Fe (III) ni fotometrik aniqlashda quyidagi qadamlar qo'yildi: yorug'lik filtrini tanlash, kompleks hosil bo'lishining atrof-muhitning kislotaliligiga bog'liqligi, bufer eritmasining tarkibiga bog'liqligi, reaktiv tarkibiga bog'liqligi, ber qonuniga bo'ysunish maydoni, kompleks tarkibiy qismlarining molyar fraktsiyalari nisbati, sensorning sezgirligi, Fe (III) ni aniqlashning pastki chegarasi, molyar yo'q bo'lib ketish koeffitsienti va Fe (III) fotometrik aniqlash usuli.

Kalit so'zlar: CPK-3, sulfosalitsil kislotasi, Beer qonuni, bufer eritmasi, molyar yo'q bo'lib ketish koeffitsientiga sezgirlik, fotometrik aniqlash, komplekslanish.

The control of the composition of natural and wastewater for the content of harmful elements is complicated by the low content of the analyzed substances and matrix effects. To solve this problem, sorption concentration is widely used in analytical practice, which makes it possible to isolate micro-components from large volumes of solutions of complex composition, thus reducing the limits of their detection and the influence of macro-components. In combination with various instrumental methods of analysis [1, 2, 3, 5] sorption concentration makes it possible to determine directly from the surface of a solid concentrate. There are quite a lot of sorbents used for these purposes. Recently, more and more preference has been given to the use of complexing sorbents [1, 2, 6, 7].

The final product of most industrial enterprises (pharmacology, processing industry, etc.) cannot be produced without maximally purified water. Enterprises should use a high-quality purified liquid when processing products during production. Anyway, good and deep-water purification directly affects the continuous cyclic production of products, and also increases the service life of equipment, protecting it from breakdowns. Iron is present in all the waters of the farm. In natural waters, it is in a dissolved state in the form of complex compounds, mainly with various organic substances. Along with dissolved iron, natural waters contain particles of iron oxides. Significant sources of iron enrichment of feed water of steam boilers are condensates of industrial steam consumers. They contain iron exclusively in the form of oxides of varying degrees of dispersion.

Preparation of dishes: in order to avoid distortion of the results of iron determination due to the leaching of iron from glass or the adsorption of iron from the sample by glass, all chemical dishes used in analyses should be treated as follows: rinse with a chromium mixture, rinse with water, pour a warm solution of hydrochloric acid (1:1) and stand in this solution 3-4 h. Then rinse the dishes with water and finally rinse with de-ironed water.

Preparation of de-ironed water: turbine condensate should be used, in which almost all iron is contained in the form of oxides of varying degrees of dispersion. The release of condensate from these particles is achieved by filtering it through a double paper filter. In a glass funnel with a diameter of 100-150 mm, place a double filter folded along the funnel from sheets of ordinary filter paper and filter the condensate through it, discarding the first 200-250 cm³ of filtrate. When water passes through the filter over time, the water quality improves as the filter is compacted. When the filter resistance increases greatly and the filtration rate decreases significantly, the filter should be replaced. The filter also needs to be replaced after a break in operation.

Store the de-ironed water in a clean bottle with a stopper. Long-term storage of de-ironed water for more than 2-3 days is not recommended.

Preparation of reagents and all work related to analyses in the future should be carried out only on de-ironed or purified water.

- Reagents and solutions:

- Sulfosalicylic acid solution 30%

- Concentrated hydrochloric acid;

- Ammonia solution, 10%

- EDTA-Na 0.1 normal;

- Sodium sulfosalicylate "ch.", 10% solution, in the absence of sodium salicylate, 10 g of sulfosalicylic acid "ch.d.a" is dissolved in

60 cm³ of water and add 35 cm³ of normal caustic soda. Mix the solutions, the mixture should have an acid reaction according to methylorange;

Detection method: A standard solution of iron "A" containing 100 mg/dm³ Fe³⁺, for which dissolve 0.8634 g of iron-ammonium alum

Fe (NH₄) (SO₄)₂ 12 H₂O "H.H." in a measuring flask at 1 dm³ in 50 cm³ approximately 1 normal hydrochloric acid.

After dissolution, bring the volume of liquid to the mark with de-bleached or purified water and mix thoroughly. The resulting solution contains 100 mg/dm³ Fe³⁺. According to hydrochloric acid, it is approximately 0.05 normal.

For the preparation of a working solution "B" containing

10 mg / dm³ Fe³⁺, dilute solution "A" 10 times 0.05 with normal hydrochloric acid.

To prepare a working solution "B" containing

1 mg / dm³ Fe³⁺, dilute the solution "B" by 10 times 0.05 with normal hydrochloric acid.

Checking the titer of solution "A" is mandatory (in accordance with Appendix H).

When determining small amounts of iron from 10 to 100 µg / dm³, the analysis should be carried out by concentrating the sample, 100 cm³ samples should be taken for analysis. When analyzing waters with a high iron content, the analysis should be performed without concentration, in this case, 50 cm³ samples should be taken.

Transfer the sample in an amount of 50-100 cm³ to a beaker, acidify 2 cm³ of concentrated hydrochloric acid and evaporate to a volume of 4-5 cm³. If there are organic substances in the analyzed water that give it color, then instead of hydrochloric acid, introduce 2-3 cm³ of concentrated sulfuric acid and evaporate the liquid until thick white vapors appear. Without stopping heating, put several crystals of sodium nitrate or 2-3 drops of concentrated nitric acid into the glass, after which the dark-colored liquid should become colorless and transparent.

Cool the sample and transfer it to a measuring flask with a capacity of 50 cm³, rinse the walls of the glass twice with de-ironed water and transfer everything to the flask, then inject 2 cm³ of 30% sulfosalicylic acid and 10 cm³ of 10% ammonia solution to neutralize. It is necessary to make sure that ammonia is added in excess, as indicated by the smell of ammonia and the yellowing of the solution in the flask.

Bring the contents of the flask to the mark with de-ironed water, mix thoroughly and colorimetrically, using as a comparison solution a blank sample prepared as follows: pour about 30 cm³ of de-ironed water into a measuring flask for 50 cm³, add 2 cm³ of concentrated hydrochloric acid, 2 cm³ of 30% sulfosalicylic acid and 10 cm³ of ammonia for neutralization. Add ammonia in a small excess. Bring the volume of the solution to the mark with de-ironed water and mix well.

The analysis of raw waters, which remain turbid after filtration, should be carried out in the same way, while taking a non-ironed sample as a blank sample. and the source water in the same amount that was taken for analysis, add 2 cm³ of concentrated hydrochloric acid and evaporate to a volume of 4-5 cm³, transfer to a flask with a capacity of 50 cm³ and bring to the mark with de-ironed water, do not add the remaining reagents.

Colorimetry is carried out on a photoelectrolorimeter KFK-2, KFK-3, etc. with a wavelength of 440 nm, sensitivity-2 in cuvettes of 50 mm. When colorimetry of small quantities, it is necessary to pre-select the cuvettes. The calculation of the iron content (Fe) in micrograms per liter ($\mu\text{g}/\text{dm}^3$) is performed according to the formula:(1) where: a is the iron content in the sample, found according to the graph μg ; V is the volume of the sample taken for evaporation, cm^3 .

Construction of a calibration curve for large concentrations of iron from 100-1000 $\mu\text{g}/\text{dm}^3$: in a series of measuring flasks with a capacity of 50 cm^3 , enter 0; 1; 2; 5; 7; 10 cm^3 of the standard solution "B" containing 10 $\text{mg} / \text{dm}^3 \text{Fe}^{3+}$, add 2 cm^3 of 30% sulfosalicylic acid to each flask, according to

10 cm^3 of a 10% ammonia solution (to excess), top up to the mark with de-ironed water, mix and colorimetricate, using as a blank sample, into which a standard iron solution was not injected.

Colorimetry should be carried out in cuvettes at 50 mm with a wavelength of 440 nm, sensitivity-2. According to the data obtained, plot a graph, postponing the photoelectrocolorimeter readings on the ordinate axis, and the iron content in the sample on the abscissa axis.

Construction of a calibration curve for small concentrations of iron from 10 to 100 $\mu\text{g}/\text{dm}^3$: in a series of measuring flasks with a capacity of 50 cm^3 , enter 0; 1; 2; 5; 7 and 10 cm^3 of a standard solution "B" containing 1 $\text{mg} / \text{dm}^3 \text{Fe}$, add the same reagents and in the same sequence as and for high concentrations, top up to the mark with de-ironed water and colorimetricate on a photocolorimeter in cuvettes at 50 cm^3 with a wavelength of 440 nm, sensitivity-2. Build a graph similar to the previous one. The graph should look like a straight line.

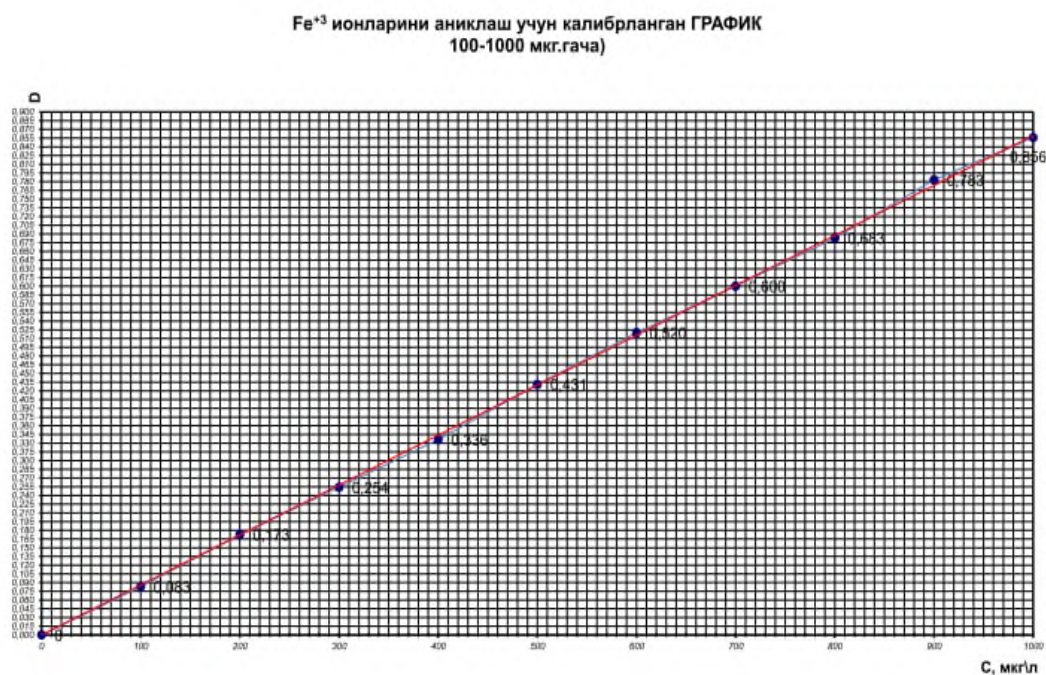


Figure 1. The graph of optical density depending on the amount of added Iron (III)

As can be seen from the obtained results, the field of obedience to Bouguer-Lambert-Beer law was observed in the range of solution. At higher concentrations, there was a deviation from the linear relationship, that is, the range from allows the quantitative determination of Fe (III).

*Reaction results and discussion of results**Spectral description of iron (III) complex with sulfosalicylic acid reagent.*

Absorption spectra of the complex formed with sulfosalicylic acid reagent and Iron (III) were obtained under selected optimal conditions.

Blank sample - pour approximately 20 cm³ of water into a 50 cm³ flask, add 2 cm³ of concentrated hydrochloric acid and all reagents in the same sequence (without evaporation).

Conclusions This article proves that organic chemistry and analytical chemistry are inextricably linked. Studies on the photometric determination of a very small amount of Fe (III) ion with one of the new organic synthesis products were carried out. It was proved that A can be used in the determination of Fe (III) ion. We are continuing our research to identify other d-metal ions similar to the Fe (III) ion by this method.

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