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SENSITIVE SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF SILVER

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A sensitive spectrophotometric method for the determination of silver is described. The method has been applied to the determination of silver in synthetic samples.

Introduction

Nanoparticles, representing the transition region between individual atom/molecule and bulk material have been attracting considerable research input, owing to some unique physical and chemical properties which they exhibit. Nanoparticles of silver are one of the most widely studied and well characterised metal systems due to the high stability of the nanoparticles. The presence of the surface plasmon band gives a convenient handle for the spectroscopic investigations on the silver nanoparticles. The position and shape of silver nanoparticles are strongly dependent on their size, chemical environment of the dielectric media and the nature of the species adsorbed on particle surfaces [1-5].

Many spectrophotometric methods have been reported for the determination of silver. Among the very recent methods, in this paper the spectrophotometric determination of silver as nanoparticles has been reported. In the present investigations, a highly sensitive spectrophotometric method for the determination of silver, that is, a new route involving the reduction of silver(I) by glucose in the presence of gliserin media to produce a yellowish-brown product, is described. Interference studies involving various diverse species have been made. The results of application of the present method for the determination of silver in synthetic samples, compare favorably with the reported method [6-9].

Experimental

Reagents and solution

All the used chemicals were of analytical reagent or higher grade. Distilled demineralized water was used for the preparation of all solutions and for all determinations.

A 0.01 M standard solution of $AgNO_3$ was prepared by dissolving a weighed portion of $AgNO_3$ in 1M HNO_3 and stored in darkness. Standard stock of 0.01M solution of glucose was prepared by dissolving weighed amount in water. The used Glycerin was of higher grade. Solutions of lower concentrations were prepared by diluting the stock solution with distilled water.

Apparatus

The absorbance of the solutions was measured with a Lambda-40 spectrophotometer with a computer interface (Perkin-Elmer, United States) and with a KFK-2 photoelectrocolorimeter (Russia), l = 1 cm.

Recommended procedure

An aliquot of a standard solution containing 2ml 10^{-3}M of silver(I) was transferred to a 25 ml calibrated flask. Then, 5ml 10^{-3}M of glucose solution was added, followed by dilutation to the mark with gliserin and mixed well. Heat the solution to 60-70^{0}C. The absorbance of the yellowish-brown solution was measured at 410 nm after 15 min against a freshly prepared reagent blank (without silver) prepared in the same manner.

Determination of silver in synthetic samples

The recommended procedure was also followed for synthetic samples, by taking known amounts of a silver nitrate solution. To determine the precision and accuracy of the method, recovery experiments were performed using the method of additions, and analyzed within the Beer's law limits.

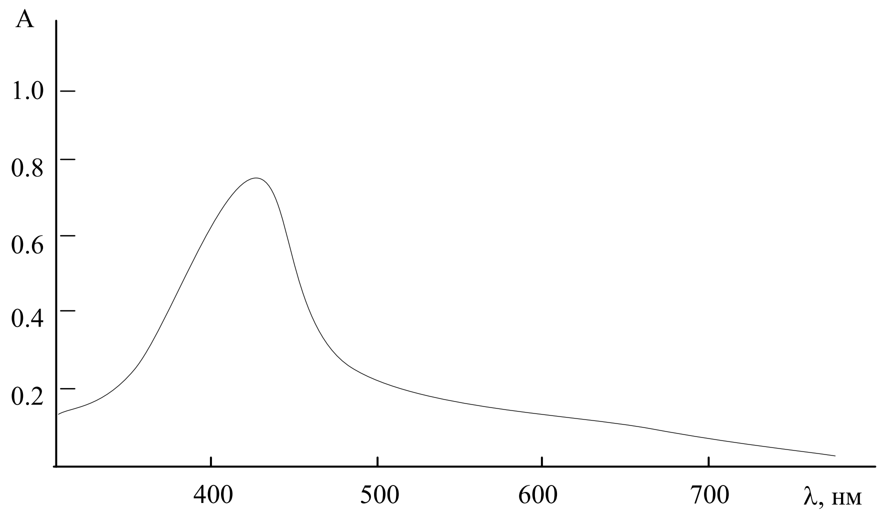

Fig. 1. UV-vis spectra of silver nanoparticle solution.

Results and Discussion

Spectral characteristics of the colored product

The solution of the yellowish-brown silver nanoparticles with maximum absorption at 410 nm was formed when silver (I) ion was reduced by glucose. The absorption spectra of the reduced product is shown in Fig. 1. The yellowish-brown colours observed in solutions are symptomatic of the presence of silver nanoparticles in the solution. Strong absorption bands are observed to occur at ~ 400- 450 nm for silver nanoparticles in the solution. This characteristic resonance corresponds to excitation of surface plasmon vibrations in the silver nanoparticles and is responsible for the striking colours of the different samples. The reaction of reduction silver with glucose was in gliserin medium.The product has maximum absorption at 410 nm. Beer's law is obeyed in the concentration range of 0.04 – 0.61 mkq ml^{-1} of silver, and the molar absorptivity is 3.62×10^{-4} l mol^{-1} cm^{-1} . The optical parameters, optimum reaction conditions and interference studies have been described. The optical characteristics and the precision data are given in Table 1.

Table 1

Optical characteristics and precision data

Optical characteristics and precision	Parameter
Color	yellowish-brown
λ_{opt} (nm)	400-450
λ_{max} (nm)	410
Stability (min)	15
Beer's law range (mkq ml^{-1})	0.04 – 0.61
Molar absorptivity (l mol^{-1} cm^{-1})	3.62×10^4
Sandells' sensitivity (mkq ml^{-1})	0.00024
Detection limit (mkq ml^{-1})	0.009
Regression equation (Y)[a]	
Slope (b)	4.3305
Intercept (a)	–0.0108
Correlation coefficient (r)	0.994.
Relative standard deviationb	0.2386
Range of error[b]	±0.3175

a. Y = bx + a, where x is the con.of silver in mkq ml^{-1}.
b. Five replicates.

Stability of the colored product

The formation of the solution of yellowish-brown silver nanoparticles was observed after heating the solution to temperature range of 60 – 70 C. At temperatures above 70 C, the absorbance was stable. After 15 min, the absorbance of silver nanoparticles solution readings remained constant. However, 30 min was sufficient to complete the analysis.

Precision and accuracy

The precision of the proposed method was ascertained from the absorbance values of the actual determinations of five replicates of fixed amounts of the silver sample. The percentage relative standard deviation was calculated by the proposed method. To determine the accuracy of the proposed method, different amounts of samples containing silver were taken within the Beer's law limits and analyzed by the recommended method.

Application of the recommended method

The proposed method has been applied to the determination of silver(I) in synthetic samples. The results of the analysis by the present method and the reported method (9) agree favorably. The results are given in Table 2.

Table 2

Determination of silver in synthetic samples

Proposed method				Reported method (9)	
Samples	Silver added mkq ml^{-1}	Silver found mkq ml^{-1}	Silver recovery %	Silver found mkq ml^{-1}	Silver recovery %
1	0.02	0.018	99.5	0.0196	98.0
2	0.04	0.039	102.5	0.04	100.0
3	0.12	0.115	99.83	0.1195	99.58
4	0.14	0.142	100.1	0.14	100.0
5	0.20	0.197	98.0	0.198	99.0

Conclusion

The proposed method for the determination of silver(I) is much simpler, and no critical conditions are necessary. The calibration curve can be precisely reproduced; extraction is not necessary. The method has the advantages of being rapid, reproducible, selective and more sensitive than many other reported methods.

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GÜMÜŞÜN HƏSSAS SPEKTROFOTOMETRİK TƏYİNİ METODİKASI

A.M.MƏHƏRRƏMOV, P.R.MƏMMƏDOV

XÜLASƏ

Gümüşün həssas spektrofotometrik təyini metodikasi təklif edilmişdir. Təklif olunan metodika gümüşün suni nümunələrdə təyini üçün tədbiq edilmişdir.

ЧУВСТВИТЕЛЬНЫЙ СПЕКТРОФОТОМЕТРИЧЕСКИЙ МЕТОД ОПРЕДЕЛЕНИЯ СЕРЕБРА

А.М.МАГЕРРАМОВ, П.Р.МАМЕДОВ

РЕЗЮМЕ

В статье описан чувствительный спектрофотометрический метод определения серебра. Метод был применен для определения серебра в синтетических образцах.