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M. Litynska
(National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute", Kyiv, Ukraine)
R. Antoniuk
(National Technical University of Ukraine "Igor Sikorsky Kyiv Polytechnic Institute", Kyiv, Ukraine)

INFLUENCE OF TEMPERATURE ON ANALYTICAL SIGNAL IN PHOTOMETRIC DETERMINATION OF ARSENATE-IONS

Nowadays inductively coupled plasma mass spectrometry and atomic fluorescence spectrometry coupled with high performance liquid chromatography are the most popular methods of arsenic determination [1, 2]. But this equipment is not available in the main part of Ukrainian universities and laboratories. Different organizations in developing countries have the same problem.

So, photometric determination is still usable and its optimization is important.

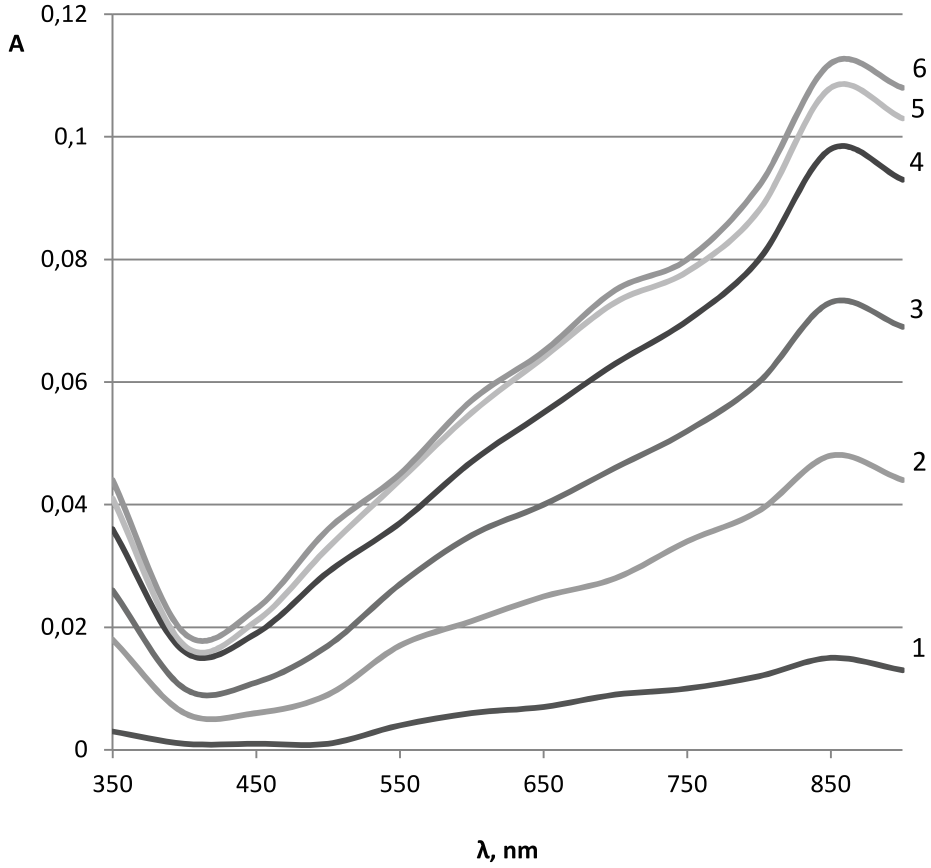
Arsenate-ions can form with molybdate some coloured compounds, which makes possible photometric determination of dissolved arsenates. Presence of reducing agent cause change of colour due to formation blue products of reduction of heteropolyarsenicmolybdate acid.

For determination of arsenic two reagents were used: working reagent and ascorbic acid solution. Working reagent consisted ammonium molybdate solution (40 g/L), sulfuric acid (5 N) and solution of antimony potassium tartrate (3 g/L). The ratio of these components was 3:10:1. Concentration of ascorbic acid was 18 g/L.

For preparation of reagents was used Systea Easy Chem methodology for phosphate determination due to significant similarity of phosphates and arsenates [3]. In volumetric flask (50 mL)10 μg of arsenic (in the form of arsenate solution), 6 mL of working reagent and 6 mL of ascorbic acid solution were pured and this mixture was diluted to 50 mL. Blank solution contained the same components, but without arsenate. Optical density (A) was measured in 2 cm glass cuvette by spectrophotometer Hach DR 2800.

Colour intensity of analytical solution dramatically depended on the duration and temperature regime of preparation (Figures 1-2).

Figure 1 demonstrated significant increasing of solution colour with growing of reaction duration at 15°C. Under these conditions required time for reaching of maximum colour was about 80-90 minutes.

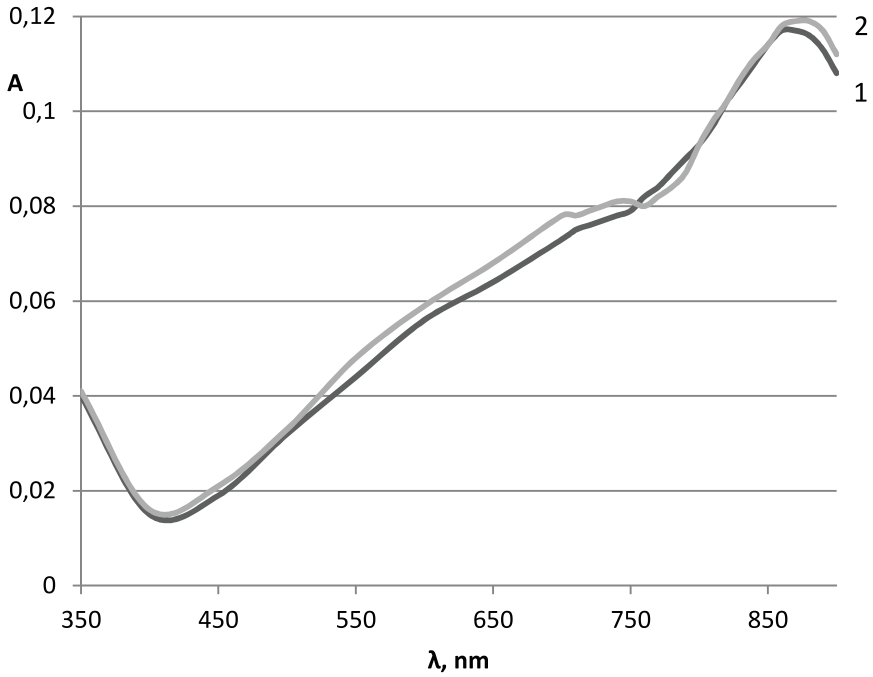


1 – 10 minutes; 2 – 20 minutes; 3 – 30 minutes; 4 – 40 minutes;
5 – 60 minutes; 6 – 80 minutes
Figure 1 – Dependency of colour intensity of photometric solution and reaction duration at 15°C.

During first 10 minutes colour intensity rose slowly than during next 10 minutes (Figure 1, curves 1 and 2). From 10 to 40 minutes of photometric reaction colour intensity increased with the same rate (Figure 1, curves 2-4) and after that rising became more slowly (Figure 1, curves 5-6).

But reaction velocity dramatically rose with increasing of temperature (Figure 2).

At 60°C solution reached maximum colour only after 5 minutes (Figure 2, curve 1), which was significantly less than at 15°C. Heating during 10 minutes led to insignificant increasing of the peak in the optimal for measurements range (840-880 nm) (Figure 2, curve 2).



1 – 5 minutes; 2 – 10 minutes
Figure 2 – Dependency of colour intensity and reaction duration at 60°C.

For temperatures between 15 and 60°C reaction velocities were higher than at 15°C, but lower than at 60°C.

So, heating of samples was very important stage in photometrical determination of arsenate-ions in the form of blue products of reduction of heteropolyarsenicmolybdate acid. Thus, 10 minutes of heating time and 60°C were recommended preparation conditions.

REFERENCES

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