IMPROVEMENT OF THE METROLOGICAL CHARACTERISTICS AT THE ANALYSIS OF INTERMETALLIC COMPOUNDS

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Introduction

To vary absorption-desorption characteristics of intermetallic compounds (IMC) is possible by means of theirs dope [1]. Therefore doubtless interest represents to increase the precision of definition of small concentration of metals in solutions IMC.

In this case modern high-sensitivity methods of the tool analysis in a combination with preliminary probe concentrate are used. But it worsens metrological characteristics of the analysis, complicates the technology and increases the time of the experiment [2].

The purpose of the present work was development of a technique allowing improving metrological characteristic atomic absorption determination of the small contents of metals in solutions IMC by means of their preliminary concentrate in graphite cavity of the electrothermal analyzer (ETA).

Experimental

The measurements of resonant light-absorption carried out on atomic-absorption spectrophotometer AAS-3 Carl Ceiss Jena in combination with electrothermic atomization device Graphite-2 and automatic batcher with capacities 5, 10, 20, 40, 50 mcl.

In work used the diluted solutions Mn (VII), which was prepared by dissolution of manganese in HNO_3 (1:1).

Conditions of atomic-absorption definition of manganese in researched solutions were following: an analytical line of manganese $\lambda = 279.5$ nm, slot – 0.2 нм, working current of heat of a lamp with hollow cathode $J = 5$mA.

Results and discussion

After series of special experiments an optimal temperature – time regime was selected: evaporation at 90ºC (20 sec) – decomposition at 470ºC (20 sec), atomization at 2300ºC (3 sec), annealing at 2600ºC (12 sec). On a stage of atomization feeding of argon reduced up to ~0.01 l/min.

For concentrate of a researched solution the certain volume (20 mcl) of solution was brought in graphite ETA cavity and evaporated under the specified program, interrupt it after the first stage, brought in the second portion of a solution, dried up a solution and again interrupted the program. In a similar manner the experiments with four portions of a solution were carrying out. The saved dry rest decomposed and atomized according with the accepted program. However the results of concentration were characterized by unsatisfactory reproducibility. For this reason another scheme of concentration was chosen.

In ETA brought in corresponding part of a solution, carried out the first and second stages of the program, interrupted it and after on the received rest deposited the following portion of a solution, carried out two stages of the program and further in the same order acted up to n-1 of a portion of a solution. With last n portion the program realized completely.

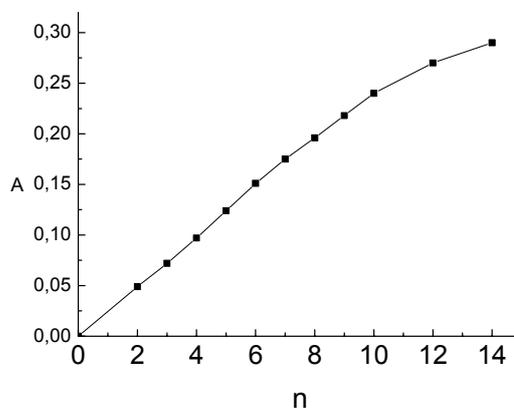Dependence of optical density from quantity of portions of a researched solution a linear up to n = 12 (see fig. 1).

Fig. 1. Dependence of optical density from quantity of portions of a researched solution.

The negative deviation from the Buger-Ber law is was observed at the same contents of manganese, as well as at the work with the one portion of the concentrated solution.

At the replacement of ETA cavity the graduate diagram, as a rule, is not reproduced. The linear dependence between optical density and concentration of a determined component allows using both method of comparison, and method of the additives of a standard solution. The additive of

a standard solution after the necessary concentrate brought in on the rest after the second stage of the program, and then worked under the standard scheme.

The contents of a determined component in a solution in mcg/ml calculated under the formula:

$$X = [A_{x+a}/(A_{x+a} - A_x)] \times 1/nV,$$

where A_{x+a}; A_x – optical density of concentrated sample with the additive and without it; a – the weight of the standard additive, mcg; V – volume of a portion of an analyzed solution, ml; n – number of portions of a solution taken for the concentrate.

At the definition of manganese in a solution with the contents 0,028 mkg/cm^3 (volume of a solution which brought in cavity was 20 mcl) under the traditional scheme it was found 0,027 ± 0,002 mkg/cm^3, and at three-time concentrate - 0,028 ± 0,001 mkg/cm^3 (n = 5, P = 0,95).

Conclusions

The original technique of definition of doped additives in intermetallic compound after their dissolution is developed. For this purpose the concentrate of probe in ETA graphite cavity was carried out.

On an example Mn(VII) of water solutions is shown that correctness and reproducibility of results of definition are satisfactory up to ten-time concentrate of the samples (see fig. 1).

Because the stage of are carried out directly in ETA graphite cavity it is possible to avoid traditional errors of samples concentrated methods which connected with pollution from an environment, chemical reagents, laboratory glassware. In this case the samples are saved and the duration of the analysis is reduced. The offered approach are distinguish by availability and simplicity of realize.

References

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