ETAAS DETERMINATION OF SOME TRACE ELEMENTS IN WINE

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The analyze of trace metals in wines is also of great importance from several points of view – quality control of wine, authenticity control of wine, wine metals toxicity and bioavailability.

Electrothermal atomic absorption spectrometry is one of the most frequently used techniques, which corresponds to the searchings for trace elements analyzes in wine. However, very often, some additional operations in the preparation of wine samples are necessary because of matrix interferences. In order to avoid these interferences and because of the very low concentration of trace elements in the samples investigated, it is necessary to separate and concentrate these elements. To reach this goal, different methods could be employed: extraction, ion exchange, precipitation, etc.

In this work investigations on the atomization behaviour of the microelements in wine, on the reasons for strong wine matrix interferences, on the accuracy of the calibration procedures are presented. Appropriate methods for preliminary separation and preconcentration of trace elements from wine matrix are compared and discussed.

Optimal analytical procedures for direct ETAAS determination of As, Cd, Cr, Ni, Pb and extraction ETAAS for Se, Tl are developed and validated.

Results for the content of trace elements in Macedonian and Bulgarian wines are presented.

THE FIRE-ASSAY-ATOMIC-ABSORPTION DEFINITION WITH USE OF ULTRASOUND OF THE GOLD CONTENTS IN ORES

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Key words: fire assay, ultrasound, sample preparation, atomic absorption, gold, silver, palladium

The techniques, which are based on the ultrasonic fire assay of the preconcentration for sample preparation, have been developed for determination of gold concentration in geological ores by the atomic-absorption method. Is shown, that the ultrasonic fluctuations accelerate process in 6 times in comparison with a classical method, and also raise a degree of extraction of gold from the ores.
In the given operation researched the possibility of the fire - assay minerals raw material of geological prospecting under effect of low frequency ultrasound the subsequent definition of noble metals in a lead alloy. The ultrasonic emitter represented a spool with the steel core. From the point of view energy losses, most favourably to introduce an emitter is immediate in a melt. At melting have utilised classical composition of fusion mixture. Instead of eitherage applied lead acetate.

There are utilised the ultrasonic generator UZDN -1 with frequency of ultrasonic oscillations 22 kHz. Crucible with a test and components of fusion mixture omitted in a crucible furnace, had preheated up to 1000°C and put in fusion mixture an emitter of the ultrasonic generator. After reaching by mixture of temperature red heating order processing by ultrasound at intensity 8-9 Wt/sm² before termination of rough response. A melt poured out in steel cup and, after cooling, separated lead beads that of slag.

For definition of gold and palladium obtained lead bead dissolved in dilute (1:3) to nitric acid, boiled out up to wet salts, boiled in mixture by hydrochloric and nitric acids (3:1), triply boiled out, adding a hydrochloric acid. Then flooded 1M by hydrochloric acid, led up to boil and, after cooling a solution, filtered in a measuring flask. The ground mass of the lead thus is deleted as chloride. For definition of silver and palladium lead bead dissolved in dilute (1:3) to nitric acid, filtered in a measuring flask.

In the obtained solutions determined gold, palladium and silver by atomic absorption method on the atomic absorption spectrophotometer «S115-PKS» in a flame acetylene - air with deuterium by the proffer of the background. Definition of gold carried out on a resonance line 242,8 nm, palladium and silver - 247,6 nm and 328,1 nm accordingly, in a range of concentrations appropriate to linearity a calibration graphics. The excess lead does not hinder definition of gold. In a parallel way carried out fire-assay (FA) atomic-absorption definition without application of ultrasound (Us).

The obtained results are testified about ultrasound usage efficiency with test intensification purpose and determination accuracy increasing in analytical control of geological tests; the obtained stags do not require padding remelting.

AN ACCELERATED ATOMIC ABSORPTION DETERMINATION OF METALS IN SOILS WITH USAGE OF ULTRASONIC AT SAMPLE PREPARATION STAGE

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Key words: soils, ultrasonic, sample preparation, atomic absorption, bulk content Cd, Co, Cr, Cu, Ni, Pb u Zn

An increase of technogenic loading on soil in modern cities puts before the analysts a problem of development of express and available techniques permitting to determine different contents of microelements such as Cd, Co, Cu, Cr, Ni, Pb, Zn possessed high toxicity for living organisms at relatively low concentrations and also participated actively in biological processes having an ability to bioaccumulation.

Full acid soil decomposing of soil carried out with usage acid mixture standard agrochemical receptions: HF+H₂SO₄, HCl+HNO₃, HNO₃+H₂O₂+H₂SO₄ providing simultaneous treatment by these mixtures or by individual acid. Element determination in obtained solutions