

Practical work**Definition of mass concentration of ZINC via the atom absorption spectrometer (AAS)**

General review

The present practical work describes the method for definition of mass zinc concentration in liquid, non-alcohol (or alcohol deprived) samples of grape-born products in the range from 0.5 to 5 mg/L via the atom absorption spectrometer (AAS). Maximum acceptable limit of Zink in wine is 5 mg/L.

Samples are automatically sprinkled in the flame of air-acetylene flame of the atomic absorption spectrometer. Absorption is measured at the wavelength of 213.9 nm. Zinc contains the highest number of absorption lines in the area of the given wavelength.

The sample to be analyzed:

Non-alcohol or (alcohol deprived) samples of grape-born products (grape juice, grape must, wine, brandy, husks of grape)

Equipment-Machinery and Support Materials

Flame atomic absorption spectrometer (AAS) with auto sampler;

Back-compensation Zn cathode lamp;

AAS software;

Air Compressor;

Acetylene – pressed in a can;

100, 250 and 1000 ml volumetric flasks;

Automatic pipettes: 20-1000 μ L;

Glass pipettes; 5, 10 and 25 mL;

Polypropylene test tubes (16x125mm);

Alcohol distillation system;

Source of heating, e.g. oven;

Reagents

Zink standard solution;

Nitric acid;

Distilled and deionized water (DDW);

Triton

Preparation of Solutions:

Zink Calibration Solutions (0.5, 1.0, 2.5 and 1.5 mg/L):

Place 0.5, 1.0, 2.5 and 5 mL of dilute standard Zn-solution (100 mg/mL) respectively into a set of 100 mL volumetric flasks, add to each flask 1 mL nitric acid and make up to 100 mL with DDW.

Calibration solutions contain 0.1, 0.5, 1.0 and 1.5 mg/L zinc respectively. These solutions are valid for use for at least 14 days.



Blind Measurement Solution

1 mL nitric acid is placed in a 100 mL measurement tube and is topped with DDW to the sign.

Zero Solution

DDW is used as a zero solution.

Rinse Solution

In a 1000 mL volumetric flask is placed 250 μ L tritone and 150 μ L nitric acid. The tube is filled with DDW to the sign.

Testing Process

Sample Preparation

Sample must be liquid, homogeneous and alcohol-free. If samples contain alcohol, it is essential that it is deprived through a distillation method. The obtained alcohol-free substance should be filled with DDW (20°C) to the original volume.

Instruments Preparation

- switch on Compressor;
- Open tap of Acetylene can (gas pressure < 5 bars);
- Switch on Vent;
- Switch on AAS equipment and auto sampler;
- Switch on Computer and open SpectrAA program;
- Select the active work shield for Zn;
- Light the Flame by pressing a black button on the equipment;
- Activate sample absorption capillary-wash regime.

Measurement

Place zero solution in the tripod designated for the calibration solutions in accordance with the positions indicated in the program, calibration solutions with increasing concentration and blind measurement solution;

Calibration process shall be activated automatically by pressing the Start button in the program menu.

Specific test sample shall be placed in a separate position of the tripod designated for the samples; the code of each sample shall be noted in the schedule of the operating shield under the applicable number of the tripod position;

To ensure the accuracy of the test, a solution of known zinc content shall be placed after every 10th to 15th sample

Test shall be automatically run by pressing the Start button in the program menu.

Calculation

Calculation is automatic. The obtained results are presented in the following manner:



