

OIV-MA-VI-14 Measurement of zinc content

Type II method

1. Introduction

As for copper, the presence of zinc in vinegars mainly has its origin in contaminations from contact materials during their manufacture, and, likewise, excessive content could cause hazes or even serious alterations in the colour.

2. Principle

Direct measurement by atomic absorption spectrophotometry.

3. Reagents

- 3.1. zinc metal
- 3.2. concentrated nitric acid 65% ($\rho_{20} = 1.38$ g/ml).
- 3.3. diluted nitric acid at 1:2 (v/v).
- 3.4. diluted nitric acid at 1% (v/v).
- 3.5. acetic acid solution at 5% (v/v).
- 3.6. standard solution of zinc 1.000 g per L.

Weigh 1.000 g of zinc metal, and transfer it entirely into a 1000 ml volumetric flask. Add diluted nitric acid (3.3) in strictly sufficient quantity to dissolve the metal and make up to the mark with diluted nitric acid (3.4).

This solution can be purchased ready-made.

- 3.7. standard solutions of zinc, from 0.05 to 2.00 mg/L.

Prepare standard solutions of 0.05 to 2.00 mg/L, by diluting the solution (3.6) with the acetic acid solution (3.5).

4. Devices and utensils

Standard laboratory material, plus:

- 4.1. atomic absorption spectrophotometer
- 4.2. zinc ion hollow cathode lamp

5. PREPARATION OF THE SAMPLE

Shake the sample to homogenize and filter if necessary.

6. Technique

Select a 213.9 nm wavelength. Adjust the absorbance scale to zero with the acetic acid

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solution (3.5). Suck the vinegar sample directly into the burner of the spectrophotometer, then successively the standard solutions (3.7). Read the absorbencies. The measurements should be done at least twice.

7. Results

7.1. calculation

Draw the absorbence variation curve according to the zinc concentration of the standard solutions. Write down the absorbencies middle value of the vinegar sample and measure the zinc content, expressed in milligrams per L of the sample.

7.2. presentation

Round the results expressed in milligrams of zinc per L, to the first decimal place.

8. Inter-laboratory validation (Hitos et al., 00)

Units: mg/L

| Sample | R | S _r | RSD _r | R | S _R | RSD _R | RSD _R (Horwitz) | HORRAT Index |
|------------------|--------|----------------|------------------|--------|----------------|------------------|-------------------------------|-----------------|
| 1 - 0.39 mg/L | 0.0390 | 0.014 | 3.55 | 0.2531 | 0.090 | 23.02 | 18.44 | 1.25 |
| 2 - 0.06 mg/L | 0.0454 | 0.016 | 25.41 | 0.0932 | 0.033 | 52.22 | 24.44 | 2.14 |
| 3 - 0.16 mg/L | 0.0618 | 0.022 | 14.13 | 0.1800 | 0.064 | 41.15 | 21.08 | 1.95 |
| 4 - 0.25 mg/L | 0.0503 | 0.018 | 7.28 | 0.2183 | 0.078 | 31.61 | 19.71 | 1.60 |
| 5 - 0.63 mg/L | 0.0689 | 0.025 | 3.92 | 0.4157 | 0.148 | 23.64 | 17.15 | 1.38 |

9. Bibliography

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