

### **OIV-MA-VI-15 Measurement of iron content**

#### **Type IV method**

#### **1. Introduction**

As for the other metals, the presence of iron in vinegars mainly has its origin in contaminations from contact materials during their manufacture, and of course the iron of the wine itself from which the vinegar has been made. Excessive content could cause hazes or even serious alterations in the colour.

#### **2. Principle**

Direct measurement of iron content in the vinegar by flame atomic absorption spectrometry, samples being tested with standard acetic solutions.

#### **3. Reagents**

- 3.1. ultra pure demineralized water
  - 3.2. 90% acetic acid
  - 3.3. ready-made standard iron solution at 1.000 g/l
  - 3.4. iron solution at 100 mg/l: put 10 ml of 1 g/l iron solution (3.3.) in a 100 ml flask; make up to the mark with with demineralized water (3.1).
  - 3.5. standard range: 0, 2, 4, 6, 8 mg of iron per litre
- Put successively 0, 1, 2, 3, 4 ml of 100 mg/l iron solution (3.4.) in 4 50 ml flasks (2.1.1.); add 2.5 ml of acetic acid (3.2.); make up to the mark with with demineralized water (3.1).

#### **4. Device and utensils**

Standard laboratory material, including:

- 4.1. 50 and 100 ml volumetric flasks (class A)
- 4.2. 1, 2, 3, 4, 5, 10 ml calibrated pipettes (class A)
- 4.3. atomic absorption spectrophotometer
- 4.4. iron ion hollow cathode lamp

#### **5. Preparation of samples**

Shake the sample to homogenize and filter if necessary. In case of an > 8 mg/l iron content, sample should be diluted with an acetic acid solution at 5% (v/v).

#### **6. Technique**

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### 6.1. test parameters (atomic absorption spectrometry)

6.1.1. oxidizing air-acetylene flame

6.1.2. 248.3 nm wavelength

6.1.3. width of the aperture: 0.2 nm

6.1.4. intensity of the hollow cathode lamp: 5 mA

### 6.2. measurements

Suck successively the standard solutions into the burner of the spectrophotometer. Read the absorbency for 10 s; make 2 measurements. Then suck the samples. Read the absorbencies. Take into account a possible dilution of the vinegar.

### 6.3. quality control

Flame atomic absorption spectrometry tests are usually performed manually. Quality control is achieved by regularly interposing a reference material every 5 measurements, or one after the standard solution, one in the middle and one at the end of the measurements.

## 7. Results

### 7.1. calculation

Draw the absorbance variation curve according to the iron concentration made of the standard solutions. Write down the absorbencies middle value of the vinegar sample and measure the iron content, expressed in milligrams per L of the sample. In most devices the iron concentration in the samples is automatically calculated by the software.

### 7.2. presentation

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

## 8. Bibliography

- OIV, 1990. *Recueil des méthodes Internationales d'analyse des vins et des moûts* (Compilation of the international methods of analysis of wines and musts) OIV Paris, France.