OIV-MA-AS323-01B Arsenic

Type IV method

1. Principle

After mineralization, using sulfuric and nitric acids, arsenic V is reduced to arsenic III by means of potassium iodide in hydrochloric acid and the arsenic is transformed into arsenic III hydride (H3As) using sodium borohydride. The arsenic III hydride formed is carried by nitrogen gas and determined by flameless atomic absorption spectrophotometry at high temperature.

2. Method

2.1. *Apparatus*

2.1.1. Kjeldahl flask (borosilicate glass)

2.1.2. Atomic absorption spectrophotometer equipped with arsenic hollow cathode lamp, hydride generator, background corrector and a chart recorder.

The hydride generator includes a reaction flask (which can eventually be put onto a magnetic stirrer) connected by a tube to a nitrogen gas supply (flow rate: 11 L/min) and by a second tube, to a quartz cell which can be brought to a temperature of 900 oC. The reaction flask also has an opening for the introduction of the reagent (borohydride).

2.2. *Reagents*

All reagents must be of recognized analytically pure quality, and in particular free of arsenic. Double distilled water prepared using a borosilicate glass flask or water of similar purity should be used.

2.2.1. Sulfuric acid (□$_{20}$□= 1.84 g/mL) arsenic free

2.2.2. Nitric acid (□$_{20}$□= 1.38 g/mL) arsenic free

2.2.3. Hydrochloric acid (□$_{20}$□= 1.19 g/mL), arsenic free

2.2.4. 10% (*m/v*) Potassium iodide solution

2.2.5. 2.5% (*m/v*) Sodium borohydride solution obtained by dissolving 2.5 g of sodium borohydride in 100 mL of 4 % (*m/v)* of sodium hydroxide solution. This solution must be prepared at the time of use.

2.2.6. Arsenic reference solution 1 g/L. Use of a commercial standard arsenic solution is preferred.

Alternatively this solution can be prepared in a 1000 mL volumetric flask, by dissolving 1.320 g of arsenic III trioxide AS_2O_3 in a minimal volume of 20 % (*m/v*) sodium

hydroxide. The solution is then acidified with hydrochloric acid, diluted 1/2, and made up to 1 liter with water.

2.3. *Procedure*

2.3.1. Mineralization

Place 20 mL of wine in a Kjeldahl flask, boil and reduce the volume by half to eliminate alcohol. Allow to cool. Add 5 mL sulfuric acid, and slowly add 5 mL nitric acid and heat. As soon as the liquid turns brown, add just enough nitric acid, dropwise, to lighten the liquid while simmering. Continue until the color clears and white sulfur trioxide fumes are formed above the solution.

Allow to cool, add 10 mL distilled water, bring back to the boil and simmer until nitrous oxide and sulfur trioxide fumes are no longer produced. Allow to cool and repeat the operation.

Allow to cool and dilute the sulfuric acid residue with a few milliliters of distilled water.Quantitatively transfer the solution into a 40 mL flask, and rinse the flask with water, combine with the diluted residue and make up to the mark with distilled water.

2.3.2. Determination

2.3.2.1. Preparation of the solution

Place 10 mL of the mineralization solution (2.3.1) into the hydride generator reactor flask. Add 10 mL hydrochloric acid, 1.5 mL potassium iodide solution, then switch on the magnetic stirrer and the nitrogen gas (flow rate: 11 L/minute). After 10 sec, add 5 mL of sodium borohydride solution. The hydride vapor obtained is immediately carried to the measurement cell (at a temperature of 900□C) by nitrogen carrier gas, where dissociation of the compound and arsenic atomization occurs.

2.3.2.2. Preparation of standard solutions

From the arsenic reference solution (2.2.6), prepare dilutions having concentrations of 1, 2, 3, 4 and 5 micrograms of arsenic per liter respectively. Place 10 mL of each of the prepared solutions into the reactor flask of the hydride generator and analyze according to 2.3.2.1.

2.3.2.3. Measurements

Select an absorption wavelength of 193.7 nm. Zero the spectrophotometer using double distilled water and carry out all determinations in duplicate. Record the absorbance of each sample and standard solution. Calculate the average absorbance for each of these solutions.

2.4. *Expression of results*

2.4.1. Calculation

Plot the curve showing the variation in absorbance as a function of the arsenic

concentration in the standard solutions. The relationship is linear. Note the average absorbance of the sample solutions on the graph and read the arsenic concentration C.

The arsenic concentration in wine, expressed in micrograms per liter is given by: 2 C.

Bibliography

- Jaulmes P. et Hamelle G., *Trav. Soc. Pharm. Montpellier,* 1967, 27, no 3, 213-225.
- Jaulmes P., *F.V., O.I.V.,* 1967, no 238
- MEDINA B et SUDRAUD P., *F.V., O.I.V.,* 1983, no 770.

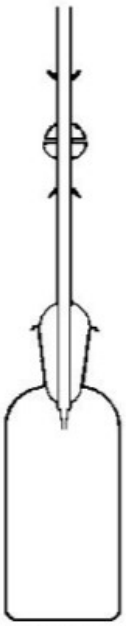

Fig.1: Apparatus used in the limit test of arsenic

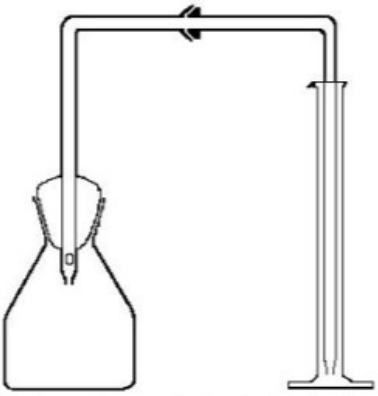

Fig 2: Apparatus used in the determination of arsenic