OIV-MA-AS323-01A Determination of arsenic in wine by atomic absorption spectrometer

Type IV method

1. Principle

After evaporating ethyl alcohol and reducing the arsenic V in arsenic III, wine arsenic is measured by hydride generation and by atomic absorption spectrometry.

2. Equipment

2.1. Glass ware:

2.1.1. Graduated flask 50, 100 ml (class A)

2.1.2. Graduated pipettes 1, 5, 10, 25 ml (class A)

2.2. Water bath at 100°C

2.3. Filters without ashes

2.4. Spectrophotometer :

2.4.1. Atomic absorption spectrophotometer

2.4.2. Instrumental parameters

2.4.2.1. Air-acetylene oxidising flame

2.4.2.2. Hollow cathode lamp (arsenic)

2.4.2.3. Wave length: 193.7 nm

2.4.2.4. Split width: 1.0 nm

2.4.2.5. Intensity of hollow cathode lamp: 7 mA

2.4.2.6. Correction of non-specified absorption with a deuterium lamp

2.5. Accessories:

2.5.1. Hydride absorption cell, placed on an air-acetylene burner.

2.5.2. Vapour generator (liquid gas separator)

2.5.3. Neutral gas (argon)

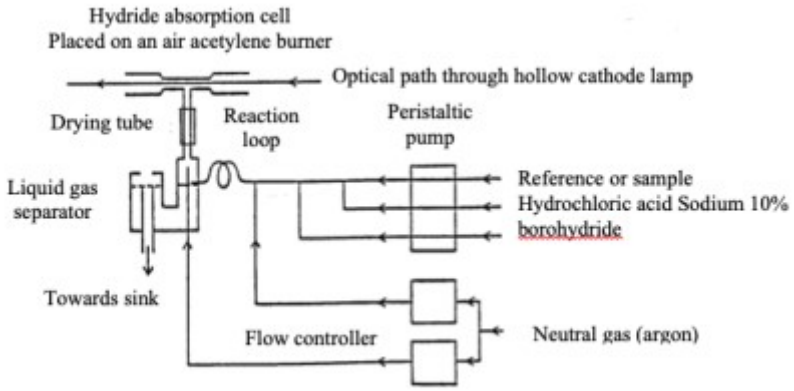


Figure 1. Hydride generator.

3. Reagents

3.1. Ultra-pure demineralised water

3.2. Ultra-pure 65% nitric acid

3.3. Potassium iodide (KI)

3.4. 10% .Potassium iodide (m/v)

3.5. Concentrated hydrochloric acid (R)

3.6. 10% Hydrochloric acid (R)

3.7. Sodium borohydride (NaBH4)

3.8. Sodium hydroxide (NaOH)

3.9. 0.6% Sodium borohydride (containing sodium hydroxide: 0.5% (m/v))

3.10. Calcium Chloride CaCl2 (used as a drying agent)

3.11. 1 g/l Arsenic stock solution prepared in the following manner : dissolve 1.5339 g of AS_2O_5 in demineralised water, adjust to 1 l.

3.12. 10 mg/l Arsenic solution: place 1 ml of stock solution (3.11.) in a 100 ml flask (2.1.1.) ;add 1 % nitric acid (3.2.) ; fill up to volume with demineralised water (3.1.).

3.13. 100 µg/l Arsenic solution: place 1 ml of 10 mg/l arsenic solution (3.12.) in a 100 ml flask (2.1.1.) ; fill up to volume with demineralised water (3.1.).

3.14. Set of callibration standards: 0, 5, 10, 25 µg/l

Successively place 0, 5, 10, 25 ml of 100 µg/l arsenic solution (3.13.) in 4 100 ml flasks (2.1.1.) ; add 10 ml of 10% potassium iodide to each flask (3.4.) and 10 ml of concentrated hydrochloric acid (3.5.) ; leave for 1 hour, fill up to 100 ml with demineralised water.

4. Sample preparation

25 ml of water is evaporated over a 100 °C water bath. This is then brought to 50 ml in the presence of 5 ml of 10% potassium iodide and 5 ml of concentrated hydrochloric acid; leave for 1 hour; filter on an ashless filter.

Make a blank reference sample.

5. Determination

The peristaltic pump sucks in the borohydride solution, the 10% hydrochloric acid solution and the sample solution.

Present the calibration standards in succession (3.14.); take an absorbency reading for 10 seconds; take two readings; the operating software establishes a calibration curve (absorbency according to concentration of arsenic in µg/l).

Then present the samples (4) ; the software establishes the sample's arsenic concentration in µg/l; deduct the arsenic concentration in the wine in µg/l taking into account that the solution be diluted by 1 / 2 .

6. Quality control

Quality control is assured by placing a control sample of internal quality (*) in a regular manner in 5 samples, or after the set of calibration solutions, or in the middle of a series or at the end the measurement.

Two deviation types are accepted compared to known value.

(*) Samples from the Bureau Communautaire de Référence (Community Bureau of reference): red wine, dry white wine and sweet white wine.

7. Bibliography

- Varian Techtron, 1972. Analytical methods for flame spectroscopy.
- Hobbins B., 1982. Arsenic Determination by Hydride Generation. Varian Instruments at Work.
- Le Houillier R., 1986. Use of Drierite Trap to Extend the Lifetime of Vapor Generation Absorption Cell. Varian Instruments at Work.
- Varian, 1994. Vapor Generation Accessory VGA-77.