COEI-2-ARSENI Measuring arsenic by hydride generation and atomic absorption spectrometry

1. Field of application

This method applies to the analysis of arsenic in the concentration range of 0 to 200 µg/l with prior mineralisation for oenological products.

2. Description of the technique

2.1. Principle of the method

After reducing arsenic (V) into arsenic (III), arsenic is determined by hydride generation and atomic absorption spectrometry.

2.2. **Principle of the analysis** (figure n°1)

The peristaltic pump draws up the borohydride solution, hydrochloric acid solution and calibration or sample.

The hydride formed in the gas-liquid separator is entrained by a neutral gas (argon).

The gaseous current passes in a dessicator made up of calcium chloride.

The arsenic hydride is analysed in an quartz absorption cell in the flame of a air-acetylene burner.

The optical path of the hollow-cathode lamp of the atomic absorption spectrometer passes in the quartz cell.

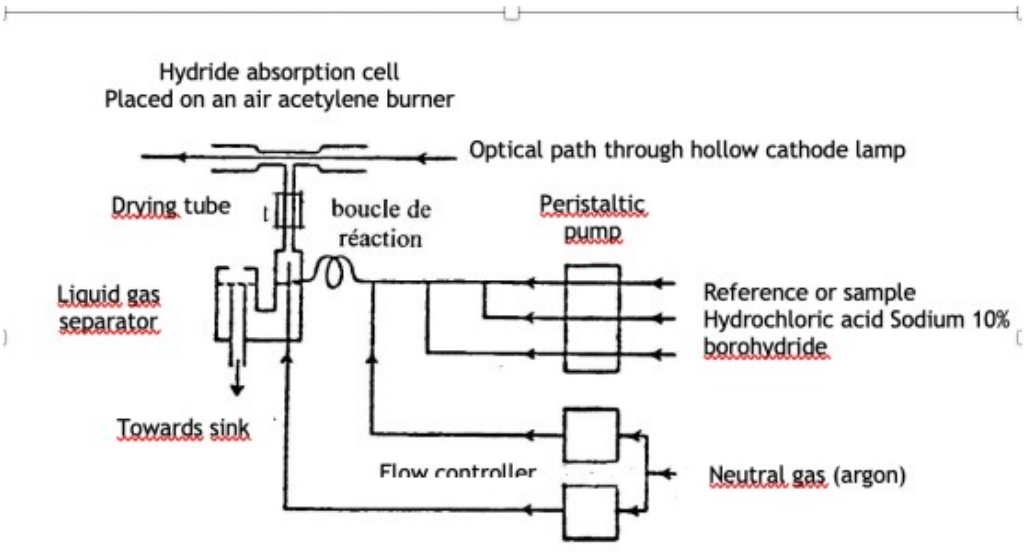


Figure n°1. Hydride generator

3. Reagents and preparation of reagent solutions

3.1. Ultra-pure demineralised water
3.2. Ultra-pure nitric acid at 65%
3.3. Potassium iodide KI
3.4. Potassium iodide at 10% (m/v)
3.5. Concentrated hydrochloric acid
3.6. Hydrochloric acid at 10% (m/v)
3.7. Sodium borohydride $NaBH_4$
3.8. Sodium hydroxide NaOH in patches
3.9. Sodium borohydride solution at 0.6% (containing 0.5% of NaOH)
3.10. Calcium chloride $CaCl_2$ (used as a dessicator)
3.11. Silicone antifoam
3.12. Arsenic calibration solution at 1 g/l containing 2% of nitric acid and prepared from the following acid: H_3AsO_4½ H_20
3.13. Arsenic solution at 10 mg/l: place 1 ml of the calibration solution (3.12.) in a 100 ml flask; add 1% of nitric acid (3.2.); complete to volume with demineralised water (3.1.).
3.14. Arsenic solution at 100 µg/l: place 1 ml of the arsenic solution at 10 mg/l (3.13.) in a 100 ml flask; add 1% of nitric acid (3.2.); complete to volume with demineralised water (3.1.).

4. Apparatus

4.1. Glassware:
4.1.1. graduated flasks 50 and 100 ml (class A)
4.1.2. graduated pipettes 1, 5, 10 and 25 ml (class A)
4.1.3. cylindrical vases 100 ml
4.2. Hot plate with thermostat
4.3. Ashless filter paper
4.4. Atomic absorption spectrophotometer:
4.4.1. air-acetylene burner
4.4.2. hollow-cathode lamp (arsenic)
4.4.3. deuterium lamp

4.5. Accessories:

4.5.1. vapour generator (or gas-liquid separator)

4.5.2. quartz absorption cell placed on the air-acetylene burner

4.5.3. bottle of neutral gas (argon)

5. Preparation of the set of calibration solutions and samples

5.1. Set of calibration solutions 0, 5, 10, 25 µg/l

Place successively 0, 5, 10, 25 ml of the arsenic solution at 100 µg/l (3.14.) in 4, 100 ml flasks; add to each flask 10 ml potassium iodide at 10% (3.4.) and 10 ml of concentrated hydrochloric acid (3.5.); complete to volume with demineralised water (3.1.); allow to stand at room temperature for one hour.

5.2. Samples of oenological products

The sample is mineralised by wet process (cf. mineralisation methods of samples before determination by atomic absorption spectrometry) then filtered. Transfer 10 ml of filtered mineralisate to a 50 ml flask; add 5 ml of potassium iodide at 10% (3.4.) and 5 ml of concentrated hydrochloric acid (3.5.); add a drop of anti-foam (3.11.); adjust to volume with demineralised water (3.1.). Allow to stand at room temperature for one hour. Filter on an ashless filter paper.

6. Procedure

6.1. Instrumental parameters of the atomic absorption spectrophotometer (given as an example)

6.1.1. oxidant air-acetylene flame

6.1.2. wave length: 193.7 nm

6.1.3. width of the monochromator's slit: 1.0 nm

6.1.4. intensity of the hollow-cathode lamp: 7 mA

6.1.5. correction of the non specific absorption with a deuterium lamp

6.2. Analytical determination

The peristaltic pump draws up the reagent solutions (3.6.) and (3.9.) and the calibrations or samples (5.1.) or (5.2).

Present successively the calibration solutions (5.1.); wait long enough so that the hydride formed in the gas-liquid separator, passes in the absorption cell; perform an absorbance reading for 10 seconds; perform two measurements; the spectrometer's computer software sets up the calibration curve (absorbance depending on the concentration of arsenic in µg/l).

Then present the samples (5.2.). Perform two measurements.

6.3. Self-check

Every five determinations, an analytical blank solution and a calibration are analysed in order to correct a possible deviation of the spectrometer.

7. Expression of results

The results are directly printed by the printer connected to the computer.

The concentration of arsenic in oenological products is expressed in µg/kg while taking into account the test sample.

8. Control pf results

The quality control is performed by placing, after the set of calibration solutions and every five samples, a reference material whose content in arsenic is known with certainty.

A control card is set up for each reference material used. The control limits were set at: +/- $2S_R$ intra (S_R intra: standard deviation of reproductibility).

9. Bibliograhy

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