RESOLUTION OENO 14/2002

DETERMINATION OF ARSENIC IN WINE

THE GENERAL ASSEMBLY,

HAVING CONSIDERED Article 5 of the October 13, 1954 International Convention on Unification of the Methods of Analysis and Appraisal of Wines,

WITH THE PROPOSAL of the Sub Commission on Methods of Analysis and Appraisal of Wines,

DECIDES to complement Appendix A of the Compendium on International Methods of Analysis of Wines and Musts, with the following method:

DETERMINATION OF ARSENIC IN WINE BY ATOMIC ABSORPTION SPECTROMETRE

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

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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)

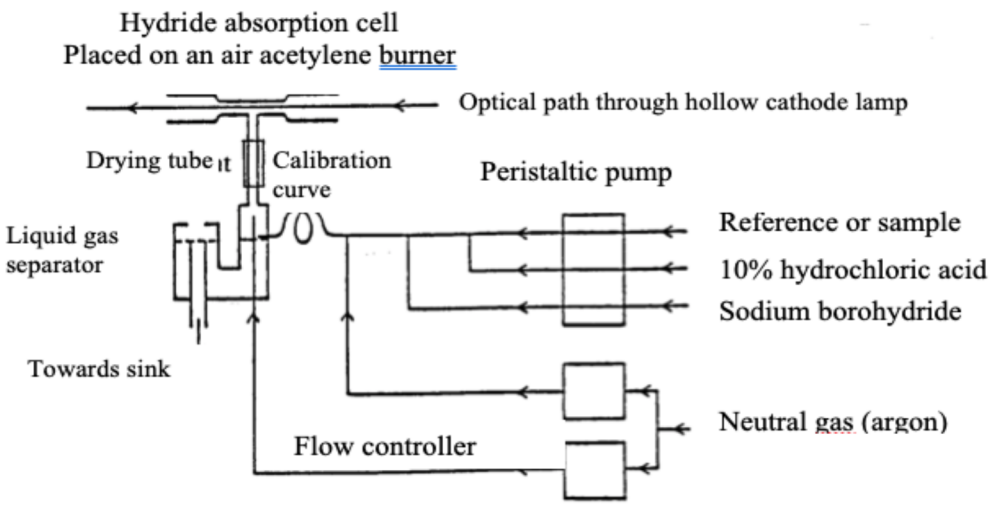

Figure 1. Hydride generator.

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 ($NaBH_4$)
3.8. Sodium hydroxide (NaOH)
3.9. 0.6% Sodium borohydride (containing sodium hydroxide: 0.5% (m/v))
3.10. Calcium Chloride $CaCl_2$ (used as a drying agent)
3.11. 1 g/l Arsenic stock solution prepared in the following manner : dissolve 1.5339 g of

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AS2O5 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.

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7. BIBLIOGRAPHY

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