Method OIV-MA-BS-32 : R2009

Type IV method

Lead- Determination by atomic absorption in spirit drinks of viti-vinicultural origin

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1. Principle of the method

Lead is determined directly in the alcoholic beverage, using a lead hollow-cathode lamp by flameless atomic absorption spectrometry, using a matrix modifier.

2. Apparatus

All the glassware must be washed prior to use with hot concentrated nitric acid (70-80°C) and rinsed in double-distilled water.

2.1. Atomic absorption spectrophotometer equipped with a graphite oven, a non-selective absorption corrector and a multipotentiometric recorder.

2.2. Lead hollow-cathode lamp.

2.3. 5 l micropipettes with special tips for atomic absorption measurements

3. Reagents

All the reagents must be of analytical purity and, in particular must be lead-free. The water used must be double-distilled in a borosilicate glass apparatus or with water of equivalent purity.

3.1. Phosphoric acid to 85 p. 100 (p20 = 1.71 g/ml)

3.2. Phosphoric acid solution obtained by dilution of 6 ml of phosphoric acid to 100 ml with water.

3.3. Nitric acid (□20 = 1.38 g/ml)

3.4. Lead solution to 1 g per litre.

Use a standard commercial solution. This solution can be obtained by dissolving 1.600 g of lead nitrate II, Pb $(NO_3)_2$ in nitric acid diluted to 1% (v/v) and adjusting the volume to 1 litre.

Keep the solution in a borosilicate glass bottle with a ground glass stopper.

3.5. Nitric acid solution diluted to 1% (v/v).

3.6. The solution is obtained by diluting the phosphoric acid solution at 6% at 1/2 with the nitric acid solution at 1%.

4. Procedure

4.1. Sample preparation

Add to the test sample of the alcoholic beverage an equal volume of the solution (3.6) of phosphoric and nitric acids. Determine its absorbance If it is greater than 0.6, dilute the alcoholic beverage (a dilution of 1/5 is sufficient in most cases).

Prepare the test solution by adding to the test sample of the diluted alcoholic beverage an equal volume of the solution of phosphoric and nitric acids.

4.2. Preparation of the solutions in the calibration range

Using the control solution of lead, prepare dilutions in which 50% of the final volume

is the solution (3.6) of phosphoric and nitric acids The concentration scale of the range depends on the sensitivity of the apparatus. For example, prepare solutions containing 10 - 20 - 30 micrograms of lead per litre.

4.3. Determination

4.3.1. Oven program.

Step	Temperature (°C)	Time (s)	Nitrogen (L/min.)	Reading
1	75	2	3	
2	95	20	3	
3	140	15	3	
4	300	8	3	
5	450	7	3	
6	480	10	3	
7	900	20	3	
8	900	1	0	
9	2 250	0,7	0	L
10	2 250	1	0	L
11	2 250	2	3	

4.3.2. Measurements

Select wavelength 283.3 nm. Set to zero the absorbance scale with double-distilled water Using a micropipette or an automatic sampler, inject into the programmed oven 3 times 5 tl of each solution in the calibration range and of the solution of the sample

to be analysed.

Record the measured absorbances. Calculate the mean absorbance value based on the results for the three injections. The absorbances are measured in height of peaks.

5. Expression of results

5.1. Calculation

Plot the changes in absorbance versus the concentrations of lead in solutions of the calibration range. The relationship is linear. Record the mean value of the absorbance of the sample solution on the calibration curve and determine the concentration C of lead.

The lead concentration in micrograms per litre of alcoholic beverage is equal to:

C x F

- F = dilution factor.

6. Bibliography

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