Method OIV-MA-BS-19 : R2010

Type IV method

Overall determination of phenolic compounds in spirit drinks of viti-viniculture origin without added caramel

(OIV/OENO 382B/2010)

1. Definition

Folin-Ciocalteu assay measures the total quantities of phenolic compounds originating from wood present in barrel-aged spirits that haven't received any added caramel.

This assay is not specific to phenolic compounds (cf. principle). Caramel also reacts to the Folin-Ciocalteu reagent. However, in the case of wood-aged spirituous beverages, the vast majority of results are related to the presence of phenolic compounds derived from the oak wood (VIDAL and Al, 1991).

Folin-Ciocalteu phenolic compound content corresponds to the response to the result described below. This result is expressed in mg of gallic acid/l by calibration.

2. Principle

All the phenolic compounds are oxidized by the Folin-Ciocalteu reagent. This reagent consists of a mixture of phosphotungstic acid and phosphomolybdic acid which is reduced, during the oxidation of the phenolic substances, into a mixture of blue molybdenum and tungsten oxides.

The blue colouring produced has a maximum absorption of about 750-760 nm. It is proportional to the quantity of oxidized phenolic compounds.

3. Apparatus

3.1. Standard laboratory apparatus, and in particular:

- Temperature-controlled bath (70° C), spectrophotometer.

4. Reagents

4.1. Folin-Ciocalteu reagent

This reagent is available for purchase ready to use. It can be prepared in the following manner:

- 100 g of sodium tungstate (No. CAS : 13472-45-2),
- 25 g of sodium molybdate (No. CAS : 7631-95-0), are dissolved in 700 ml of distilled water (No. CAS : 7732-18-5).

Add:

- 50 ml of 85% phosphoric acid (No. CAS : 7664-38-2) (ρ20=1.71 g/mL),
- 100 ml of concentrated chlorhydric acid (No. CAS : 7647-01-0) (ρ20=1.19 g/mL).

Bring to a boil under reflux for 10 hours, then add:

- 150 G of lithium sulphate (No. CAS : 10377-48-7),
- a few bromine drops (No. CAS : 7726-95-6),
- and bring to a boil for another 15 minutes. Cool and add 1 litre of distilled water.

4.2. Anhydrous sodium carbonate

- Prepare a 4.25% solution (m/v) in distilled water.

4.3. Anhydrous gallic acid (No. CAS : 149-91-7),

5. Procedure

5.1. Calibration in gallic acid

Produce a hydroalcoolic gallic acid stock solution by weighed quantity, then some surrogate solutions by dilution (at least 2). The calibration range also includes a blank (hydroalcoolic solution). As an example, the range can include the following levels: 0.200 and 400 mg/L. Check the linearity of the calibration.

5.2. Preparation of the samples

The sample must be perfectly limpid and free of beeswing.

5.3. Reaction

In a test tube, introduce:

- 0.2 ml of sample (or of calibration solution)
- 1 ml of Folin-Ciocalteu reagent,
- 18.8 ml of sodium carbonate solution.

After stirring, bring to approximately 70°C for 20 minutes in the temperature-controlled bath, then cool under running cold water.

5.4. Absorptance measurement at 760 Nm

Absorbance at 760 nm is measured under a 1 cm optical path.

6. Expression of results

Express the result in mg of gallic acid/L (linear calibration), accounting for the possible dilution of the sample. If the absorptance is greater than 1, a new measurement is carried out after dilution of the sample, if linearity is guaranteed.

7. References

1. BLOUIN J., LLORCA L., MONTREAU F.R., DUFOUR J.H., 1972. Etude des conditions optimales pour la détermination des composés phénoliques totaux par le

réactif de Folin-Ciocalteu. Connaissance de la vigne et du vin, 6, 405-413.

2. VIDAL J.-P., CANTAGREL R., FAURE A., BOULESTEIX J.M., 1991**.** Comparaison de trois méthodes de dosage des composés phénoliques totaux dans les spiritueux. FV OIV n°904.