Method OIV-MA-BS-25 : R2017

Type IV method

Determination of ethyl carbamate in spirit drinks of viti-vinicultural origin

OENO 6/94;
OIV-OENO 590-2017

1. Principle

The assay is performed by direct injection of the drink into a chromatograph coupled to a mass spectrometer operating under the principle of electron impact, in "Selected Ion Monitoring (SIM)" acquisition mode.

2. Chromatography

Capillary column of the Carbowax 20 M (50 mx 0.22 mm) type, film thickness 0.2 μm.

- Temperature programming from 60 to 200°C, 3°C per minute.
- Data acquisition method of the mass spectrometer: Selected Ion Monitoring (SIM), MZ = 62, 74, 84.

The chromatograms are re-processed with the single ion M/Z = 62. The other ions are used to confirm peak purity by taking into account the ratio of their respective intensities.

Note: Certain NP or Hall sensors can be used.

3. Sample preparation

3.1. Internal standard

The internal standard is propyl carbamate (reference ICN, K & K Laboratories 217188) at 100 mg/L in a 50% vol. hydroalcoholic solution. (Check that the alcohol used is free

of ethyl carbamate).

3.2. Addition of the internal standard

At 5 ml of the alcoholic beverage, add 50 µL of the solution of propyl carbamate at 100 mg/L which results in 1 mg/1 in the sample.

Note: this final quantity of the internal standard in the sample can be modulated according to the ethyl carbamate content in the medium to be analyzed.

3.3. In the case of sweet alcoholic beverages (over 10 g/L),

after adding the internal standard it is preferable to extract the ethyl carbamate, for example, method (1) can be used, which consists in extracting the ethyl carbamate with ether after saturating the medium with excess sodium sulphate to fix the water, or by method (2) which involves fixing the carbamates (ethyl carbamate or the internal standard) on a porous polymer (of Extrelut type) followed by elution with dichloromethane.

4. Preparation of the reference solution

- According to the alcoholic beverage to be analyzed, prepare a solution of ethyl carbamate at 50 µg/L or 400 µg/L or more if necessary.
- 5 ml of the reference solution are added by 50 µL of the internal standard solution (propyl carbamate at 100 mg/L).
- The solution is injected using the Splitless mode (valve closure for 20 to 30 seconds) by 2 µL of the prepared solution into the chromatograph after being properly adjusted.

5. Expression of results

The ethyl carbamate is expressed in µg/L of the spirit.

6. Bibliography

1. Dosage du Carbamate d'éthyle dans les vins et eaux de vie, 1988, BERTRAND A. et BARROS P.; connaissance Vigne Vin 22 (1) 39-47.

2. Method for the analysis of ethyl carbamate in alcoholic beverages by capillary gas chromatography, 1986, DENNIS M.J. HOWARTH N. MASSEY R.C. PARKER I. SCOTTER M. and STARTIN J.R. J.AOAC, 369 193.<0}