Method OIV-MA-VI-04 : R2000
Type IV method

Detection and quantification of the presence of synthetic acetic acid in vinegars

(OENO 55/2000)

1. Introduction

The presence of synthetic acetic acid (detection and possibly quantification) in a vinegar is now determined by the content in ^{14}C. Natural vinegars (obtained without the addition of synthetic acetic acid) have ^{14}C contents that are accurately determined (according to the year of production). Values less than the characteristic contents of the assumed year of production represent:

- either a mixture with products from more recent years,
- or, if they are less than the natural radioactive contents ^{14}C of approximately 15 dpmg (disintegration per minute and per gram of carbon), the addition of all or part of the synthetic acetic acid (whose radioactivity ^{14}C is 0 dpmg).

2. Principle

After extracting the acetic acid using sodium hydroxide, complete by liquid scintillation the reactivity ^{14}C of the product converted into benzene.

3. Reagents

3.1. Concentrated solution of sodium hydroxide

3.2. Phenolphthalein, alcoolic solution at 1g for 100 ml

3.3. Synthetic benzene (0 dpmg of ^{14}C)

3.4. 0.01 g of 2-(4-tert-Butylphenyl)-5-(4-biphenylyl)-1.3.4-oxadiazole (Butyl PDB) and of 1.4 Bis (2-methylstyryl)benzene (bis-MSB) for 4 ml of benzene

3.5. International radiocarbon standard: oxalic acid N.B.S:

- 13.56 dpmg.

4. Equipment and utensils

4.1. 1-liter balloon flasks

4.2. Vigreux column combined with coolant

4.3. Balloon flask heater

4.4. Reactor for burning chemical products at 5 bars of oxygen

4.5. Chemical bench for synthesis of benzene from CO_2,

4.6. Liquid scintillation spectrometer

4.7. Glass counting cells (with low content and ^{40}K) and provided with highly sealed plastic covers

5. Preparation of sample

Homogenize the sample by stirring and filter if necessary.

6. Technique

6.1. Extraction of acetic acid

Distill 1 liter of vinegar having a total acid content of 60 g/L (or the corresponding quantity if the vinegar has any other total acidity). Recover the water-acetic acid mixture in the receiving balloon flask.

Recover the distillate, add to it 100 µl of phenolphthalein solution (3.2) and neutralize rapidly with sodium hydroxide concentrated solution (3.1) that has been filtered. Then distill the product obtained again to eliminate the water. The sodium acetate thus formed is recovered in the balloon flask, then dried.

The dry acetate is placed in the combustion reactor, which is raised to 5 bars of oxygen. Combustion is initiated by heating a filament, and the carbon gas formed in this way is trapped in the liquid nitrogen.

On the benzene synthesis bench, the CO_2 is reduced by lithium, then transformed into carbide which, through the addition of H_20, produces acetylene. A vanadium aluminum catalyst enables trimerization into benzene.

6.2. Spectrometric measurements

To determine the natural background noise at the metering flask, add 4 ml of synthetic benzene with the scintillating mixture and count until a background noise statistical margin of less than 0.01 dpmg is obtained. This gives us A_{bdf}

To determine the efficiency of the apparatus, count 4 ml of benzene prepared from international standard ^{14}C using the same concentration of scintillating mixture until a statistical margin of 0.1 dpmg is obtained to provide A_{st}

Then add to the meter, the flask containing the sample to be measured in the form of benzene, again using the same quantity of scintillation mixture. Count the number of times 1,000 minutes needed to obtain a statistical margin of 0.1 dpmg. In this way, we obtain A_{mes}.

7. Results

7.1. Calculation of specific activity

The specific activity ^{14}C of the sample (A_{ech}) compared to standard ^{14}C can be calculated from measured activity A_{ech} from which the background noise of the metering flask is removed.

$$A_{ech} = (A_{mes} - A_{bdf})/A_{st}$$

Where A_{ech} and A_{st} are raised to an efficiency of 100 bar so that they can be compared with the reference values.

7.2. Presentation

This specific activity is expressed in dpm of ^{14}C per gram of carbon and is rounded off to the first decimal.

8. Interpretation of results

Interpretation presupposes that the laboratory has natural vinegar radioactivity data (obtained by acetic fermentation) according to the year of production, starting from the year 1955, the first year when the radiocarbon enrichment due to thermonuclear bombs was detectable.

9. Bibliography

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