Method OIV-MA-BS-02 : R2009

Type II method

Reference method for the determination of alcoholic strength by volume of spirit drinks of viti-vinicultural origin: Preparation of the distillate

(OIV/OENO 379/2009)

1. Scope

The method is suitable for the preparation of distillates to be used to determine the real alcoholic strength by volume of spirit drinks.

2. Principle

The spirits are distilled to separate the ethyl alcohol and other volatile compounds from the extractive matter (substances which do not distil).

3. Reagents and Materials

3.1. Anti-bumping granules

3.2. Concentrated antifoam emulsion (for crème liqueurs)

4. Apparatus and Equipment

Usual laboratory apparatus and in particular the following.

4.1. Water bath capable of being maintained at 10 °C to 15 °C.

Water bath capable of being maintained at 20 °C (± 0.2 °C)

4.2. Class A volumetric flasks, 100 ml and 200 ml, that have been certified to ± 0.1 % and ± 0.15 % respectively.

4.3. Distillation apparatus:

4.3.1. General requirements

The distillation apparatus must meet the following specifications:

- The number of joints must be no more than the strict minimum needed to ensure the system is leak-tight.
- Inclusion of a device designed to prevent priming (entrainment of the boiling liquid by the vapour) and to regularise the distillation rate of alcohol-rich vapours.
- Rapid and complete condensation of the alcohol vapours.
- Collection of the first distillation fractions in an aqueous medium.

The heat source must be used with a suitable heat-diffuser to prevent any pyrogenic reaction involving the extractive matter.

4.3.2. As an example, a suitable distillation apparatus would include the following parts:

- Round bottomed flask, 1 litre, with a standardised ground-glass joint.
- Rectifying column at least 20 cm high (a Vigreux column, for example).
- Elbow connector with an approximately 10 cm long straight-rimmed condenser (a West-type condenser) fitted vertically.
- Cooling coil, 40 cm long.
- Drawn out tube, taking the distillate to the bottom of a graduated collecting flask containing a small amount of water.

Note: The apparatus described above is intended for a sample of least 200 ml. However, a smaller sample size (100 ml) can be distilled by using a smaller distillation flask, provided a splash-head or some other device to prevent entrainment is used.

5. Storage of test samples

Samples are stored at room temperature prior to analysis.

6. Procedure

6.1. Distillation apparatus verification

The apparatus used must be capable of the following:

The distillation of 200 ml of a water-alcohol solution with known concentration close to 50 % vol. must not cause a loss of alcohol of more than 0.1 % vol.

6.2. Spirit drinks with alcoholic strength below 50 % vol.

Measure out 200 ml of the spirit into a volumetric flask.

Record the temperature of this liquid, or maintain at standard temperature (20 °C).

Pour the sample into the round bottomed flask of the distillation apparatus and rinse the volumetric flask with three aliquots each of approximately 20 ml of distilled water. Add each rinse water aliquot to the contents of the distillation flask.

Note: This 60-ml dilution is sufficient for spirits containing less than 250 g of dry extract per litre. Otherwise, to prevent pyrolysis, the volume of rinse water must be at least 70 ml if the dry extract concentration is 300 g/l, 85 ml for 400 g/l dry extract, and 100 ml for 500 g/l dry extract (some fruit liqueurs or crèmes). Adjust these volumes proportionally for different sample volumes.

Add a few anti-bumping granules (3.1) (and antifoam for crème liqueurs).

Pour 20 ml of distilled water into the original 200 ml volumetric flask that will be used to hold the distillate. This flask must then be placed in a cold water bath (4.1) (10 - 15 °C for aniseed-flavoured spirit drinks).

Distil, avoiding entrainment and charring, occasionally agitating the contents of the flask, until the level of distillate is a few millimetres below the calibration mark of the volumetric flask.

When the temperature of this distillate has been brought down to within 0.5 °C of the liquid's initial temperature, make up to the mark with distilled water and mix thoroughly.

This distillate is used for the determination of alcoholic strength by volume (Annex II)

6.3. Spirit drinks with alcoholic strength above 50 % vol.

Measure out 100 ml of the spirit drink into a 100 ml volumetric flask and pour into the round bottomed flask of the distillation apparatus.

Rinse the volumetric flask several times with distilled water and add the washings to the contents of the round-bottomed distillation flask. Use enough water to bring the flask's contents up to approximately 230 ml.

Pour 20 ml of distilled water into a 200 ml volumetric flask that will be used to hold the distillate. This flask must then be placed in a cold water bath (4.1) (10 °C to 15 °C for aniseed-flavoured spirits).

Distil, agitating the contents occasionally, until the level of distillate is a few millimetres below the calibration mark of the 200 ml volumetric flask.

When the temperature of this distillate has been brought down to within 0.5 °C of the liquid's initial temperature, make up to the mark with distilled water and mix thoroughly.

This distillate is used for the determination of alcoholic strength by volume (Annex II)

Note: The alcoholic strength by volume of the spirit drink is twice the alcoholic strength of the distillate.