Commission Implementing Regulation (EU) 2016/635 of 22 April 2016 amending the Annex to Regulation (EC) No 2870/2000 as regards certain reference methods for the analysis of spirit drinks

COMMISSION IMPLEMENTING REGULATION (EU) 2016/635

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amending the Annex to Regulation (EC) No 2870/2000 as regards certain reference methods for the analysis of spirit drinks

THE EUROPEAN COMMISSION,

Having regard to the Treaty on the Functioning of the European Union,

Having regard to Regulation (EC) No 110/2008 of the European Parliament and of the Council of 15 January 2008 on the definition, description, presentation, labelling and the protection of geographical indications of spirit drinks and repealing Council Regulation (EEC) No 1576/89[(1)], and in particular Article 28(2) thereof,

Whereas:

(1) Commission Regulation (EC) No 2870/2000[(2)] lists and describes the reference methods for the analysis of spirit drinks. However, some of the methods listed in the Annex to that Regulation, among which, the methods for the determination of volatile acidity and total sugars in spirit drinks, are not yet described.

(2) The methods for the determination of volatile acidity and total sugars in certain spirit drinks have been subjected to two international validation studies that were conducted in accordance with internationally agreed procedures and their method performance parameters have been found to be acceptable. The studies were carried out as part of a research project under the European Commission (EC) framework IV standards measurements and testing (SMT) programme. The description of those methods should therefore be included in the Annex to Regulation (EC) No 2870/2000.

(3) Regulation (EC) No 110/2008 lays down requirements for some categories of spirit drinks to be aged in wood and provides that others may undergo such ageing. Analysis of the principal compounds coming from wood can be helpful when considering if a sample is consistent with the definition corresponding to the relevant category of spirit drink. The International Organisation of Vine and Wine (OIV) has recognised a method of analysis for the determination of those compounds in its Resolution OIV/OENO 382A/2009. The recognition of the method was based on data obtained from an international method-performance study on different spirit drinks carried out following internationally-agreed procedures. This method and its description should therefore be added to the Union reference methods for the analysis of spirit drinks set out in the Annex to Regulation (EC) No 2870/2000.

(4) Regulation (EC) No 2870/2000 should therefore be amended accordingly.

(5) The measures provided for in this Regulation are in accordance with the opinion of the Committee for Spirit Drinks,

HAS ADOPTED THIS REGULATION:

Article 1

The Annex to Regulation (EC) No 2870/2000 is amended in accordance with the Annex to this Regulation.

Article 2

This Regulation shall enter into force on the third day following that of its publication in the *Official Journal of the European Union*.

This Regulation shall be binding in its entirety and directly applicable in all Member States.

Done at Brussels, 22 April 2016.

For the Commission

The President

Jean-Claude JUNCKER

***Changes to legislation:** There are currently no known outstanding effects for the Commission Implementing Regulation (EU) 2016/635. (See end of Document for details)*

ANNEX

The Annex to Regulation (EC) No 2870/2000 is amended as follows:

(1) The table of contents is amended as follows:

(a) in points III.3 and VIII, the term '(p.m.)' is deleted;

(b) the following point is added:

X. Determination of wood compounds: furfural, 5-hydroxymethylfurfural, 5-methylfurfural, vanillin, syringaldehyde, coniferaldehyde, sinapaldehyde, gallic acid, ellagic acid, vanillic acid, syringic acid and scopoletin.

(2) In Chapter III the following part is added:

III.3. DETERMINATION OF VOLATILE ACIDITY OF SPIRIT DRINKS

1. **Scope**

The method has been validated in an interlaboratory study for rum, brandy, marc and fruit spirits, at levels ranging from 30 mg/l to 641 mg/l.

2. **Normative references**

ISO 3696: 1987 Water for analytical use — Specifications and test methods.

3. **Definitions**

3.1. Volatile acidity is calculated by deducting the fixed acidity from the total acidity.

3.2. Total acidity is the sum of titratable acidities.

3.3. Fixed acidity is the acidity of the residue left after evaporating the spirit to dryness.

4. **Principle**

The total acidity and fixed acidity are determined by titration or by potentiometry.

5. **Reagents and materials**

During the analysis, unless otherwise stated, use only reagents of recognised analytical grade and water of at least grade 3 as defined in ISO 3696:1987.

5.1. 0,01 M sodium hydroxide solution (NaOH)

5.2. Mixed indicator solution:
Weigh 0,1 g of indigo carmine and 0,1 g of phenol red.
Dissolve in 40 ml water and make up to 100 ml with ethanol.

6. **Apparatus and equipment**

Indirect laboratory apparatus, grade A glassware and the following:

6.1. Water pump

6.2. Rotary evaporator or ultrasonic bath

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6.3. Equipment for potentiometric titration (optional).

7. **Sampling and samples**

Samples are stored at room temperature prior to analysis.

8. **Procedure**

8.1. Total acidity

8.1.1. Preparation of sample

The spirit is irradiated with ultrasonic (ultrasonication) or stirred two minutes under a vacuum to rid it of carbon dioxide if required.

8.1.2. Titration

Pipette 25 ml of the spirit into a 500 ml Erlenmeyer flask.

Add about 200 ml of cooled boiled distilled water (prepared fresh daily) and 2-6 drops of the mixed indicator solution (5.2).

Titrate with the 0,01 M sodium hydroxide solution (5.1) until the yellow-green colour changes to violet in the case of colourless spirits, the yellow-brown colour to red-brown in the case of brown-coloured spirits respectively.

The titration may also be carried out by potentiometry, to pH 7,5.

Let n_1 ml be the volume of the 0,01 M sodium hydroxide solution added.

8.1.3. Calculation

The total acidity (TA) expressed in milliequivalents per l of spirit is equal to $0,4 \times n_1$.

The total acidity (TA′) expressed in mg of acetic acid per l of spirit is equal to $24 \times n_1$.

8.2. Fixed acidity

8.2.1. Preparation of sample

Evaporate 25 ml of the spirit to dryness:

Pipette 25 ml of the spirit into a flat-bottomed cylindrical evaporating dish 55 mm in diameter. During the first hour of evaporation the evaporating dish is placed on the lid of a boiling water bath so that the liquid will not boil, as this could lead to losses through splattering.

Complete the drying by placing the evaporating dish in a drying oven at 105 °C for two hours. Allow the evaporating dish to cool in a desiccator.

8.2.2. Titration

Dissolve the residue left after evaporating with cooled boiled distilled water (prepared fresh daily) and make up to a volume to circa 100 ml and add 2-6 drops of the mixed indicator solution (5.2).

Titrate with the 0,01 M sodium hydroxide solution (5.1).

The titration may also be carried out by potentiometry, to pH 7,5.

Let n_2 ml be the volume of the 0,01 M sodium hydroxide solution added.

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8.2.3. Calculation

The fixed acidity (FA) expressed in milliequivalents per l of spirit is equal to $0{,}4 \times n_2$.

The fixed acidity (FA) expressed in mg of acetic acid per l of spirit is equal to $24 \times n_2$.

9. **Calculation of volatile acidity**

9.1. Expression in milliequivalents per l:

Let:

TA = total acidity in milliequivalents per l
FA = fixed acidity in milliequivalents per l

Volatile acidity, VA, in milliequivalents per l is equal to:

TA – FA.

9.2. Expression in mg of acetic acid per l:

Let:

TA′ = total acidity in mg of acetic acid per l
FA′ = fixed acidity in mg of acetic acid per l

Volatile acidity, VA, in mg of acetic acid per l is equal to:

TA′ – FA′.

9.3. Expression in g of acetic acid per hl of pure 100 % vol. alcohol is equal to:

$$\frac{TA' - FA'}{A} \times 10$$

where A is the alcoholic strength by volume of the spirit drink.

10. **Method performance characteristics (Precision)**

10.1. Statistical results of the interlaboratory test

The following data were obtained from an international method performance study carried out to internationally agreed procedures ([1]) ([2]).

Year of interlaboratory test	**2000**
Number of laboratories	18
Number of samples	6

Samples	**A**	**B**	**C**	**D**	**E**	**F**
Number of laboratories retained after eliminating outliers	16	18	18	14	18	18