

Commission Regulation (EC) No 2870/2000 of 19 December 2000 laying down Community reference methods for the analysis of spirits drinks

- Article 1 The Community reference methods for the analysis of spirits drinks...
- Article 2 Notwithstanding the first indent of Article 1, other analytical methods...
- Article 3 (1.) Where ... analytical reference methods are not laid down...
- Article 4 For the purposes of this Regulation: 'repeatability limit': shall be...
- Article 5 This Regulation shall enter into force on the seventh day...
Signature

ANNEX

I. DETERMINATION OF ALCOHOLIC STRENGTH BY VOLUME OF SPIRIT DRINKS

Introduction

1. Scope
2. Normative References
3. Terms and Definitions
 - 3.1. Reference temperature:
Note 1:
 - 3.2. Density:
 - 3.3. Specific gravity:
Note 2:
 - 3.4. Real alcoholic strength by volume:
Note 3:
4. Principle

APPENDIX I: PREPARATION OF DISTILLATE

1. Scope
2. Principle
3. Reagents and Materials
 - 3.1. Anti-bumping granules.
 - 3.2. Concentrated antifoam emulsion (for crème liqueurs).
4. Apparatus and equipment
Note:
5. Storage of test samples
6. Procedure
 - 6.1. Distillation apparatus verification.
 - 6.2. Spirit drinks with alcoholic strength below 50 % vol.
Note:
 - 6.3. Spirit drinks with alcoholic strength above 50 % vol.
Note:

APPENDIX II: MEASUREMENT OF DENSITY OF DISTILLATE

METHOD A DETERMINATION OF REAL ALCOHOLIC STRENGTH BY VOLUME OF SPIRIT DRINKS...

- A.1. Principle
- A.2. Reagents and materials
 - A.2.1. Sodium chloride solution (2 % w/v)
- A.3. Apparatus and Equipment
 - Note 1:
- A.4. Procedure
 - A.4.1. Calibration of pycnometer
 - A.4.1.1. Calibration using a single-pan balance:
 - A.4.1.1.1. Weigh the clean, dry pycnometer (P).
 - A.4.1.1.2. Fill the pycnometer carefully with distilled water at ambient temperature...
 - A.4.1.1.3. Weigh the tare bottle (T0).
 - A.4.1.1.4. Calculation
 - A.4.1.2. Calibration method using a twin-pan balance:
 - A.4.2. Determination of alcoholic strength of test sample
- A.5. Method performance characteristics (precision)
 - A.5.1. Statistical results of the interlaboratory test

METHOD B DETERMINATION OF REAL ALCOHOLIC STRENGTH BY VOLUME OF SPIRIT DRINKS...

- B.1. Principle
- B.2. Reagents and materials
- B.3. Apparatus and equipment
- B.4. Procedure
- B.5. Method performance characteristics (precision)
 - B.5.1. Statistical results of the interlaboratory test

METHOD C DETERMINATION OF REAL ALCOHOLIC STRENGTH BY VOLUME OF SPIRIT DRINKS...

- C.1. Principle
- C.2. Reagents and materials
 - C.2.1. Float cleaning solution (sodium hydroxide, 30 % w/v)
- C.3. Apparatus and Equipment
 - Note 1:
- C.4. Procedure
- C.5. Method performance characteristics (precision)
 - C.5.1. Statistical results of the interlaboratory test

II. DETERMINATION OF TOTAL DRY EXTRACT OF SPIRIT DRINKS BY GRAVIMETRY...

- 1. Scope
- 2. Normative References
- 3. Definition
- 4. Principle
- 5. Apparatus and Equipment
- 6. Sampling and samples
- 7. Procedure
 - 7.1. Pipette 25 ml of the spirit containing less than 15...
 - 7.2. Complete the drying by placing the evaporating dish in a...
- 8. Calculation
- 9. Method performance characteristics (precision)
 - 9.1. Statistical results of the interlaboratory test

- III. DETERMINATION OF VOLATILE SUBSTANCES AND METHANOL OF SPIRIT DRINKS
 - III.1. GENERAL REMARKS
 - 1. Definitions
 - 2. Gas chromatographic analysis of volatile compounds
 - III.2. GAS CHROMATOGRAPHIC DETERMINATION OF VOLATILE CONGENERS: ALDEHYDES, HIGHER ALCOHOLS, ETHYL...
 - 1. Scope
 - 2. Normative References
 - 3. Definition
 - 4. Principle
 - 5. Reagents and materials
 - 6. Apparatus and equipment
 - 7. Sampling and samples.
 - 8. Procedure (used for the validated method)
 - 9. Calculation
 - 10. Quality assurance and control (used for the validated method)
 - 11. Method performance characteristics (precision)
 - III.3. DETERMINATION OF VOLATILE ACIDITY OF SPIRIT DRINKS
 - 1. Scope
 - 2. Normative references
 - 3. Definitions
 - 3.1. Volatile acidity is calculated by deducting the fixed acidity from...
 - 3.2. Total acidity is the sum of titratable acidities.
 - 3.3. Fixed acidity is the acidity of the residue left after...
 - 4. Principle
 - 5. Reagents and materials
 - 5.1. 0,01 M sodium hydroxide solution (NaOH)
 - 5.2. Mixed indicator solution:
 - 6. Apparatus and equipment
 - 7. Sampling and samples
 - 8. Procedure
 - 8.1. Total acidity
 - 8.1.1. Preparation of sample
 - 8.1.2. Titration
 - 8.1.3. Calculation
 - 8.2. Fixed acidity
 - 8.2.1. Preparation of sample
 - 8.2.2. Titration
 - 8.2.3. Calculation
 - 9. Calculation of volatile acidity
 - 9.1. Expression in milliequivalents per l:
 - 9.2. Expression in mg of acetic acid per l:
 - 9.3. Expression in g of acetic acid per hl of pure...
 - 10. Method performance characteristics (Precision)
 - 10.1. Statistical results of the interlaboratory test
 - (1) 'Protocol for the design, conduct and interpretation of method-performance studies',...
 - (2) Horwitz, W. (1982) Analytical Chemistry , 54, 67A-76A.

- V. ANETHOLE. GAS CHROMATOGRAPHIC DETERMINATION OF TRANS-ANETHOLE IN SPIRIT DRINKS
1. Scope
 2. Normative references
 3. Principle
 4. Reagents and materials
 - 4.1. Ethanol 96 % vol. (CAS 64-17-5)
 - 4.2. 1-methoxy-4-(1-propenyl) benzene; (trans-anethole) (CAS 4180-23-8)
 - 4.3. 4-allylanisole, (estragole) (CAS 140-67-0), suggested internal standard (IS)
 - 4.4. Ethanol 45 % vol.
 - 4.5. Preparation of standard solutions
 - 4.5.1. Standard solution A
 - 4.5.2. Internal standard solution B
 - 4.5.3. Solutions used to check the linearity response of the flame...
 - 4.5.4. Standard solution C
 5. Apparatus and equipment
 - 5.1. A capillary gas chromatograph fitted with a flame ionisation detector...
 - 5.2. Split/splitless injector
 - 5.3. Capillary column, for example:
 - 5.4. Common laboratory equipment: A grade volumetric glassware, analytical balance (precision:...
 6. Chromatography conditions
 7. Samples
 8. Procedure
 - 8.1. Sample screening for estragole
 - 8.2. Preparation of unknown samples
 - 8.3. Blank
 - 8.4. Linearity test
 - 8.5. Determination
 9. Calculation of response factor
 - 9.1. Response factor (RF_i) calculation
 - 9.2. Analysis of the linearity response test solutions
 - 9.3. Analysis of the sample
 10. Calculation of results
 11. Quality assurance and control
 12. Treatment of spirits sample containing large amount of sugar and...
 - 12.1. Principle
 - 12.2. Reagents and materials
 - 12.2.1. Ammonium sulphate, anhydrous, (CAS 7783-20-2).
 - 12.2.2. Sodium phosphate, dibasic, dodecahydrate, (CAS 10039-32-4).
 - 12.3. Apparatus and equipment
 - 12.4. Procedure
 - 12.4.1. Sample screening for estragole
 - 12.4.2. Extraction
 - 12.4.3. Preparation of the extracted sample to be analysed
 - 12.5. Determination
 - 12.6. Calculation of results
 - 12.7. Quality control and assurance
 13. Method performance characteristics (precision)

VI. GLYCYRRHIZIC ACID. DETERMINATION OF GLYCYRRHIZIC ACID USING HIGH PERFORMANCE LIQUID...

1. Scope
2. Normative references
3. Principle
4. Reagents and materials
 - 4.1. Ethanol 96 % vol. (CAS 64-17-5).
 - 4.2. Ammonium glycyrrhizinate, C₄₂ H₆₂ O₁₆ .NH₃...
 - 4.3. Glacial acetic acid, CH₃ COOH, (CAS 64-19-7).
 - 4.4. Methanol, CH₃ OH (CAS 67-56-1).
 - 4.5. Ethanol 50 % vol.
 - 4.6. Preparation of the HPLC elution solutions
 - 4.6.1. Elution solvent A (example)
Note:
 - 4.6.2. Elution solvent B
 - 4.7. Preparation of standard solutions
 - 4.7.1. Reference solution C
 - 4.7.2. Standard solutions used to check the linearity of the response...
5. Apparatus and equipment
 - 5.1. Separation system
 - 5.1.1. High-performance liquid chromatograph.
 - 5.1.2. Pumping system enabling one to achieve and maintain a constant...
 - 5.1.3. UV spectrophotometric detection system: to be set at 254 nm....
 - 5.1.4. Solvent degassing system.
 - 5.2. Computational integrator or recorder, the performance of which is compatible...
 - 5.3. Column (example):
 - 5.4. Laboratory equipment
 - 5.4.1. Analytical balance with a precision of 0,1 mg
 - 5.4.2. A-grade volumetric glassware
 - 5.4.3. Micromembrane filtration arrangement for small volumes.
6. Chromatography conditions
 - 6.1. Elution characteristics: (example)
 - 6.2. Detection:
7. Procedure
 - 7.1. Preparation of the spirit sample
 - 7.2. Determination
8. Method performance characteristics (precision)

VII. CHALCONES. HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY METHOD FOR VERIFYING THE PRESENCE OF...

1. Scope
2. Normative references
3. Principle
4. Reagents and materials
 - 4.1. Ethanol 96 % vol. (CAS 64-17-5)
 - 4.2. Acetonitrile, CH₃ CN, (CAS 75-05-8)
 - 4.3. Reference substance: Glycyrrhiza glabra : liquorice, 'sweet root'
 - 4.4. Sodium acetate, CH₃ COONa, (CAS 127-09-3)
 - 4.5. Glacial acetic acid, CH₃ COOH, (CAS 64-19-7)
 - 4.6. Preparation of solutions

- 4.6.1. Ethanol 50 % volume
- 4.6.2. Solvent A: acetonitrile
- 4.6.3. Solvent B: 0,1 M sodium acetate buffer solution, pH 4,66....
5. Preparation of the reference extract from *Glycyrrhiza glabra* (4.3)
 - 5.1. Weigh 10 g of ground liquorice root (*Glycyrrhiza glabra*...
 - 5.2. Recover the liquorice extract from the filter
 - 5.3. The liquorice root extraction must be performed three times in...
 - 5.4. Combine the three filtrates.
 - 5.5. Evaporate the solvent phase (of 5.4) on a rotary evaporator....
 - 5.6. Take up the residual extract (of 5.5) with 100 ml...
6. Apparatus and equipment
 - 6.1. Separation system.
 - 6.1.1. High-performance liquid chromatograph.
 - 6.1.2. Pumping system capable of achieving and maintaining a constant or...
 - 6.1.3. UV/visible spectrophotometric detection system that can be set at 254...
 - 6.1.4. Solvent degassing system:
 - 6.1.5. Column oven that can be set at a temperature of...
 - 6.2. Computational integrator or recorder, the performance of which is compatible...
 - 6.3. Column
 - 6.4. Common laboratory equipment, including:
 - 6.5. Chromatography conditions (example).
 - 6.5.1. Elution characteristics of solvents A (4.6.2) and B (4.6.3):
 - 6.5.2. Flow rate: 1 ml/minute.
 - 6.5.3. UV detector settings:
7. Procedure
 - 7.1. Preparation of the spirit sample
 - 7.2. Preparation of the residual liquorice extract (5.6)
 - 7.3. Determination
 - 7.3.1. Inject 20 µl of the prepared liquorice extract (7.2). Perform...
 - 7.3.2. Inject 20 µl of the sample (7.1) (aniseed-flavoured spirit sample)....
 - 7.3.3. Compare the two chromatograms. There must be a great similarity...
8. Characteristic chromatogram for a pastis
9. Method performance characteristics (precision)

VIII. TOTAL SUGARS

1. Scope
2. Normative references
3. Principle
4. Reagents and materials
 - 4.1. Glucose (CAS 50-99-7), at least 99 % pure.
 - 4.2. Fructose (CAS 57-48-7), at least 99 % pure.
 - 4.3. Sucrose (CAS 57-50-1), at least 99 % pure.
 - 4.4. Lactose (CAS 5965-66-2), at least 99 % pure.
 - 4.5. Maltose monohydrate (CAS 6363-53-7), at least 99 % pure.
 - 4.6. Pure acetonitrile (CAS 75-05-8) for HPLC analysis.
 - 4.7. Distilled or demineralised water, preferably microfiltered.
 - 4.8. Solvents (example)
 - 4.9. Ethanol absolute (CAS 64-17-5).

- 4.10. Ethanol solution (5 %, v/v).
 - 4.11. Preparation of stock standard solution (20 g/l)
 - 4.12. Preparation of working standard solutions (2,5, 5,0, 7,5, 10,0 and...
 5. Apparatus and Equipment
 - 5.1. HPLC system capable of achieving baseline resolution of all of...
 - 5.1.1. High-performance liquid chromatograph with a six-way injection valve fitted with...
 - 5.1.2. Pumping system enabling one to achieve and maintain a constant...
 - 5.1.3. Differential refractometer.
 - 5.1.4. Computational integrator or recorder, the performance of which is compatible...
 - 5.1.5. Pre-column:
 - 5.1.6. Column (example):
 - 5.1.7. Chromatography conditions (example):
 - 5.2. Analytical balance accurate to 0,1 mg.
 - 5.3. Filtration set-up for small volumes using a 0,45 µm micromembrane....
 6. Sample storage
 7. Procedure
 - 7.1. PART A: Sample preparation
 - 7.1.1. Shake the sample.
 - 7.1.2. Filter the sample through a filter with a pore size...
 - 7.2. PART B: HPLC
 - 7.2.1. Determination
 - 7.2.2. Should any peak of a sample have a greater area...
 8. Calculation
 9. Method performance characteristics (precision)
 - 9.1. Statistical results of the interlaboratory test
 - (1) ' Protocol for the design, conduct and interpretation of method-performance...
 - (2) Horwitz, W. (1982) Analytical Chemistry , 54, 67A-76A.
- IX. EGG YOLK. DETERMINATION OF EGG YOLK CONCENTRATION IN SPIRIT DRINKS...
1. Scope
 2. Normative references
 3. Principle
 4. Reagents and materials
 - 4.1. Double-distilled water
 - 4.2. Diatomaceous earth
 - 4.3. Ethanol 96 % vol. (CAS 64-17-5)
 - 4.4. 15 % magnesium acetate (CAS 16674-78-5) solution
 - 4.5. 10 % sulphuric acid (CAS 7664-93-9)
 - 4.6. 1 N sulphuric acid.
 - 4.7. 0,16 g/l potassium dihydrogen phosphate (CAS 778-77-0), KH₂ PO...
 - 4.8. Reagent for phosphate determination:
 5. Apparatus and equipment
 - 5.1. 100 ml conical flask
 - 5.2. Ultrasonic bath (or magnetic stirrer)
 - 5.3. 100 ml volumetric flask
 - 5.4. 20 o C water bath
 - 5.5. Filter (Whatman No 4 or equivalent)
 - 5.6. Porcelain (or platinum) crucible

- 5.7. Boiling water bath
 - 5.8. Hot plate
 - 5.9. Muffle furnace
 - 5.10. 50 ml volumetric flask
 - 5.11. 20 ml volumetric flask
 - 5.12. Spectrophotometer set at 420 nm
 - 5.13. 1 cm cuvette.
 6. Samples
 7. Procedure
 - 7.1. Sample preparation
 - 7.1.1. Weigh 10 g of the sample into a 100 ml...
 - 7.1.2. Add gradually 70 ml of ethanol (4.3) in small portions,...
 - 7.1.3. Transfer the contents of the flask to a 100 ml...
 - 7.1.4. Add a small amount of diatomaceous earth (4.2) and filter...
 - 7.1.5. Transfer 25 ml of the filtrate to a porcelain (or...
 - 7.1.6. Place the crucibles on a hot plate (5.8) and heat...
 - 7.1.7. Ash the residue by heating to incandescence at 600 o...
 - 7.1.8. Take up the ash with 10 ml of 10 %...
 - 7.2. Photometric phosphate assay
 - 7.2.1. Comparative solution
 - 7.2.1.1. Place 10 ml of 10 % sulphuric acid (4.5) in...
 - 7.2.1.2. Add to a 5 ml aliquot of this solution (7.2.1.1),...
 - 7.2.1.3. Stopper with a loosely inserted stopper, shake, and heat in...
 - 7.2.1.4. Fill a 1 cm cuvette (5.13) with this comparative solution....
 - 7.2.2. Sample solution
 - 7.2.2.1. Add to a 5 ml aliquot of the ash solution...
 - 7.2.2.2. Stopper with a loosely inserted stopper, shake, and heat in...
 - 7.2.2.3. The yellow solution that develops is immediately analysed spectrophotometrically (5.12)...
 - 7.2.3. Calibration curve
 - 7.2.3.1. To construct the calibration curve, add 2 ml aliquots of...
 - 7.2.3.2. Stopper with a loosely inserted stopper, shake, and heat in...
 - 7.2.3.3. Construction of the calibration curve:
 8. Expression of results
 9. Method performance characteristics (precision)
- X. DETERMINATION OF THE FOLLOWING WOOD COMPOUNDS IN SPIRIT DRINKS BY...
1. Scope
 2. Normative reference
 3. Principle
 4. Reagents
 - 4.1. 96 % vol. alcohol.
 - 4.2. HPLC-quality methanol (Solvent B).
 - 4.3. Acetic acid diluted to 0,5 % vol. (Solvent A).
 - 4.4. Mobile phases: (given as an example only).
 - 4.5. Reference standards of 99 % minimum purity: furfural, 5-hydroxymethyl furfural,...

- 4.6. Reference solution: the standard substances are dissolved in a 50...
5. Apparatus
 - 5.1. A high-performance liquid chromatograph capable of functioning in binary gradient...
 - 5.2. Syringes for HPLC.
 - 5.3. Device for membrane-filtration of small volumes.
 - 5.4. Integrator-computer or recorder with performance compatible with the entire apparatus,...
6. Procedure
 - 6.1. Preparation of the solution to be injected
 - 6.2. Chromatographic operating conditions: carry out the analysis at ambient temperature...
 - 6.3. Determination
 - 6.3.1. Inject the reference standards separately, then mixed.
 - 6.3.2. Inject the sample as prepared in 6.1.
 - 6.3.3. Measure the area of the peaks in the reference solution...
7. Expression of results
8. Performance characteristics of the method (precision)
 - 8.1. Furfural
 - 8.2. 5-Hydroxymethylfurfural
 - 8.3. 5-Methylfurfural
 - 8.4. Vanillin
 - 8.5. Syringaldehyde
 - 8.6. Coniferaldehyde
 - 8.7. Sinapaldehyde
 - 8.8. Gallic acid
 - 8.9. Ellagic acid
 - 8.10. Vanillic acid
 - 8.11. Syringic acid
 - 8.12. Scopoletin
 - (1) 'Protocol for the design, conduct and interpretation of method-performance studies',...
 - (2) Horwitz, W. (1982) Analytical Chemistry , 54, 67A-76A.

Changes to legislation: There are currently no known outstanding effects for the
Commission Regulation (EC) No 2870/2000. (See end of Document for details)

- (1) OJ L 160, 12.6.1989, p. 1.
- (2) OJ L 105, 25.4.1990, p. 9.
- (3) OJ L 270, 7.10.1998, p. 9.
- (4) OJ L 372, 31.12.1985, p. 50.

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