



IMPROVEMENT OF STATE AND INTERSTATE STANDARDS FOR QUALITY CONTROL AND SAFETY OF ALCOHOLIC PRODUCTS

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Interstate and international standards for the determination of volatile components, including methyl alcohol, in alcoholic products



GB/T 11858-2009
GB/T 15038-2008
GB 5009.266-2016
GB/T 10781-2021



BIS IS 3752:2005(R2009)



Commission Regulation (EC) No. 2870/2000



AOAC Official Methods 972.10/11, 2005

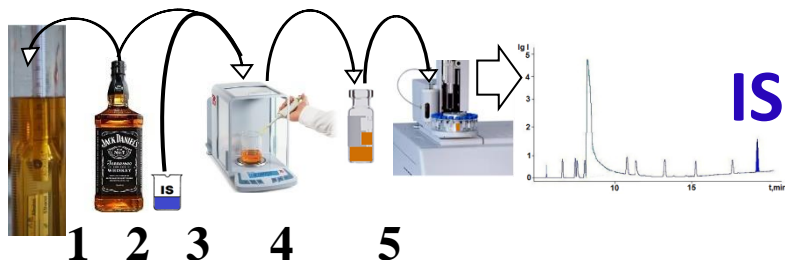


Norma Mexicana NMX-V-005-NORMEX-2018

All listed national standards are harmonized with Regulation (EC) 2870/2000 and use the **traditional internal standard method**

An idea... with long exposure

Today: Traditional internal standard method.
China, India, EU, USA, Mexico, etc.



In accordance with the traditional method of internal standard, the concentration of the i th component in terms of mg/kg is determined by the following formula:

$$C_i(\text{mg/kg}) = RRF_i^{IS} \cdot \frac{A_i}{A_{IS}} \cdot C_{IS}(\text{mg/kg})$$

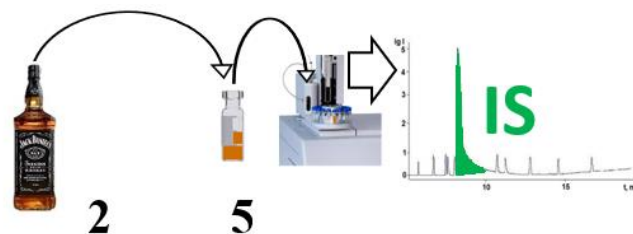
The values of the relative response coefficients of the detector to the investigated volatile component relative to the response to the selected internal standard are calculated using the following formula:

$$RRF_i^{IS} = \frac{C_i^{calibr}(\text{mg/kg})}{C_{IS}^{calibr}(\text{mg/kg})} \cdot \frac{A_{IS}^{calibr}}{A_i^{calibr}}$$

To calculate the concentration of the component, expressed in mg/L AA, it is necessary to measure the density of the sample and determine its strength (volume content of ethanol):

$$C_i(\text{mg/L AA}) = RRF_i^{IS} \cdot \frac{A_i}{A_{IS}} \cdot C_{IS}(\text{mg/kg}) \cdot \frac{\rho_{sample}(\text{kg/L}) \cdot 100\%}{\text{"Strength" } (\%, \text{ ABV})}$$

Tomorrow: Innovative approach
China, India, EU, USA, Mexico, etc.



In accordance with the method “Ethanol as an internal standard”, the concentration of the i th component in the dimension mg/L of anhydrous alcohol (AA) is determined by the following form

$$C_i(\text{mg/L AA}) = RRF_i^{Eth} \cdot \frac{A_i}{A_{Eth}} \cdot \rho_{Eth}(\text{mg/L})$$

The values of the relative coefficients of the detector response to the investigated volatile component relative to the response to ethanol are calculated using the following formula:

$$RRF_i^{Eth} = \frac{C_i^{calibr}(\text{mg/L AA})}{\rho_{Eth}(\text{mg/L})} \cdot \frac{A_{Eth}^{calibr}}{A_i^{calibr}}$$

1. There is no need to add any internal standard to the sample.
2. Ethanol is always present in alcoholic products and its concentration in mg/L AA is always known with a 100% guarantee and is equal to the density of ethanol
 $\rho_{Eth} = 789270 \text{ mg/L}$.

It is possible to make the method easier, cheaper, trust and robust

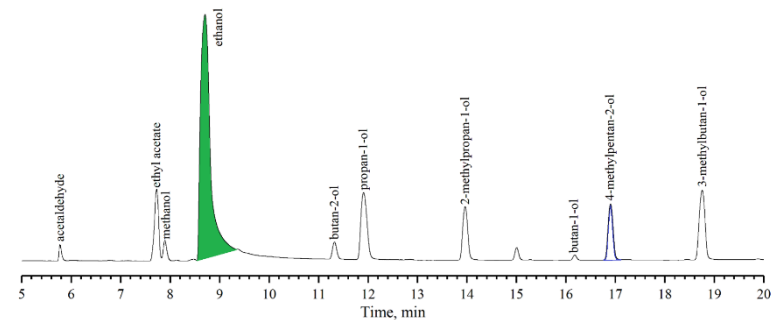
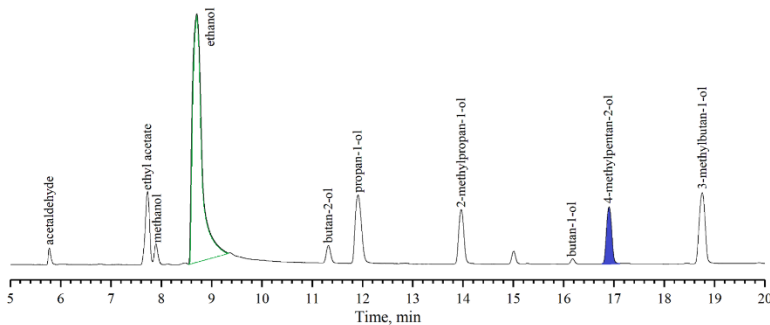
Today: Method of Internal Standard. Traditional way
China, India, EC, USA, Mexico et al.

Tomorrow: Innovative approach
China, India, EU, USA, Mexico, etc.

As an internal standard, ethanol is used directly in the test sample. So, there is no need for a manual procedure for the quantitative addition of the internal standard substance into the test sample.

The coefficients RRF_i^{Eth} are highly reproducible and for modern gas chromatographs they can be tabulated.

Refinement of values RRF_i^{Eth} can be performed no more than once a year.



$$C_i(\text{mg/L AA}) = RRF_i^{IS} \cdot \frac{A_i}{A_{IS}} \cdot C_{IS}(\text{mg/kg}) \cdot \frac{\rho_{\text{sample}}(\text{kg/L}) \cdot 100\%}{\text{"Strength"} (\%, \text{ABV})}$$

$$C_i(\text{mg/L AA}) = RRF_i^{Eth} \cdot \frac{A_i}{A_{Eth}} \cdot 789300 (\text{mg/L})$$

Done

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Chinese GB/T 11858-2008 Improvements

The use of the proposed method ensures high reliability of the data obtained, significantly reduces time, labor, material and financial costs. Analysis of volatile compounds in spirit drinks has never been so easy. Here you can read modified text of official method, which allows to carry out analysis of alcoholic beverages using the developed method.

The places in the text document to be deleted are **highlighted in yellow**. Embedded parts of the test are **highlighted in green**.

GBT 11858-2008 Vodka



National Standards of People's Republic of China

GB/T 11858-2008

National Food Safety Standards

Vodka

Issued on: 2008-10-19

Implemented on: 2009-06-01

Issued by the General Administration of Supervision, Inspections and Quarantine of the People's Republic of China and National Standardization Management Committee

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5.3.6 Precision
Discrepancies between the results of two independent tests conducted under iterative conditions and the average value of the test results should not exceed the 2% range.

5.4 Total Aldehyde

5.4.1 Gas Chromatography Method

5.4.1.1 Principle
Channel vaporized sample along with the carrier gas into the chromatography column and then perform separation of individual components that are meant to be measured by the process of ionizing on the difference of partition coefficients between components while transferring between the two phases (gas-liquid) and the consequent differences between the migration speeds of each component within the column. Separated components will flow out of the chromatography column in a specific order into the hydrogen flame ionization detector. Conduct qualitative analysis by comparing sample standard values with the retention values of the peaks of individual components based on the resultant chromatogram (only by internal standard method with the use of peak area (or peak height)).

5.4.1.2 Apparatus

5.4.1.2.1 Gas Chromatography: With hydrogen flame ionization detector (FID).

5.4.1.2.2 Chromatography Column: PEG20M cross-linked quartz capillary chromatography column, column length 25m-30m, inner diameter 0.25mm. Or any other capillary chromatography column with equal effect of analysis.

5.4.1.2.3 Micro Injector: 10 µL.

5.4.1.3 Reagents and Solutions

5.4.1.3.1 40% Ethanol Solution: Mix ethanol (chromatographically pure) with water.

5.4.1.3.2 Acetaldehyde Solution (2%) Use as standard sample. Extract 2 mL acetal (chromatographically pure) and then dilute it with 40% ethanol solution till it reaches 100 mL.

5.4.1.3.3 Isobutanol Solution (2%) Use as internal standard. Extract 2 mL isobutanol (chromatographically pure) and then dilute it with 40% ethanol solution till it reaches 100 mL.

5.4.1.4 Chromatographic Conditions

Carrier Gas (Nitrogen Gas of High Purity): Flow rate at 0.5 mL/min-1.0 mL/min; diversion ratio >37:1, make gas flow rate at about 30 mL/min-50 mL/min.

Hydrogen Gas: Flow rate at 33 mL/min.

Air: Flow at 400 mL/min.

Temperature of Detector (T_d): 230°C.

Temperature of Sample Inlet (T_i): 230°C.

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In the formula:
X_i - Total acetaldehyde content, unit is milligram per liter (mg/L);
V₁ - Volume of iodine standard reagent used on the sample, unit is milliliter (mL);
V₂ - Volume of iodine standard reagent used on the control experiment, unit is milliliter (mL);
c - Concentration of the iodine standard (titration reagent), unit is mol per liter (mol/L);
22 - Molar mass value of iodine, unit is mol per gram (mmol) (M=22);
V - Volume of sample absorbed, unit is milliliter (mL);
X₀ - Total acetaldehyde content in a liter of 100% ethanol of the sample, unit is milligram per liter (mg/L);
I - Actual alcohol content of sample determined.

Result should be presented in one decimal place format.

5.4.2 Precision
Discrepancies between the results of two independent tests conducted under iterative conditions and the average value of the test results should not exceed the 10% range.

5.5 Total Ester

5.5.1 Gas Chromatography Method

5.5.1.1 Principle
Same as 5.4.1.1.

5.5.1.2 Apparatus
Same as 5.4.1.2.

5.5.1.3 Reagents and Solutions

5.5.1.3.1 40% Ethanol Solution: Mix ethanol (chromatographically pure) with water.

5.5.1.3.2 Ethyl Acetate Solution (2%) Use as standard sample. Extract 2 mL ethyl acetate (chromatographically pure), then dilute it with 40% ethanol solution till it reaches 100 mL volume.

5.5.1.3.3 M Methanol Solution (2%) Use as internal standard. Extract 2 mL isobutanol (chromatographically pure), then dilute it with 40% ethanol solution till it reaches 100 mL volume.

5.5.1.4 Chromatographic Conditions
Same as 5.4.1.4.

5.5.1.5 Analysis Procedure
Entropy of the analysis operation procedure is the same as what is described in section 5.4.1.5, with the

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Column Temperature (T_c): Initial temperature at 70°C. Maintain temperature for 3 mins and then systematically increase the temperature of GC oven to 100°C. Maintain temperature for another 10 mins.

The flow rate of carrier gas, hydrogen and air may differ according to different chromatographic conditions between apparatus used. Experiments should be conducted to determine the best operating conditions, with the end goal of complete separation of internal standard peak and individual peaks of each component present in the alcohol sample achieved as the basis.

5.4.1.5 Analysis Procedure

5.4.1.5.1 Determination of Calibration Factor (F value)
Extract 100 mL of standard sample directly into a 100 mL volumetric flask and then add 0.50 mL of Isobutanol solution (prepared as in 5.4.1.3.3) as internal standard. Shake the flask and then dilute the mixture with 40% ethanol solution to full. The concentration of acetaldehyde should both be 0.02%, 0.04% and 0.06% respectively. Then transfer 10 mL of the mixture into a micro injector, where the amount of sample injected will be dependent on the sensitivity of the apparatus. Make records of the retention time of acetaldehyde in the sample respectively, with acetaldehyde as well as their individual peak area (or peak height). Use these values to calculate the relative calibration factor (F value) of acetaldehyde.

The relative calibration factor (F value) of acetaldehyde to isobutanol is according to experience value, at about 0.02.

5.4.1.5.2 Determination of Sample Solution
Extract 100 mL of alcohol sample directly into a 100 mL volumetric flask and then add 0.50 mL of Isobutanol solution (prepared as in 5.4.1.3.3) as internal standard. Shake the flask and then dilute the mixture with 40% ethanol solution to full. The concentration of acetaldehyde should both be 0.02%, 0.04% and 0.06% respectively. Then transfer 10 mL of the mixture into a micro injector, where the amount of sample injected will be dependent on the sensitivity of the apparatus. Make records of the retention time of acetaldehyde in the sample respectively, with acetaldehyde as the basis of measurement.

5.4.1.6 Result Calculation

a) Calibration Factor (F value) can be calculated with the following formula (6):

$$F = \frac{A_i \cdot A_s}{A_s \cdot A_i} \quad (6)$$

b) Acetaldehyde (or Acetal) content in the sample can be calculated with the following formula (7):

$$X_i = F \cdot \frac{A_i}{A_s} \cdot X_0 \quad (7)$$

5.4.1.7 Total Aldehyde (Acetaldehyde) Content in a liter of 100% ethanol can be calculated with the following formula (8):

$$X_0 = \frac{X_i \cdot V_1}{V} \quad (8)$$

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5.5.2.3.7 Ethyl Acetate Series Standard Reagent: Use a micro burette to extract volumes of 0.0 mL, 0.75 mL, 1.5 mL, 2.25 mL, 3.0 mL, 4.5 mL ethyl acetate standard storage reagent (prepared as in 5.5.2.3.3) into an individual 100 mL, corral flasks respectively. Dilute each solution with 40% ethanol solution till each flask is full and mix evenly. These ready formulated standard reagents should contain ethyl acetate at 0.0 mg/L, 2.50 mg/L, 5.00 mg/L, 7.50 mg/L, 10.00 mg/L, and 15.00 mg/L.

5.5.2.4 Analysis Procedure

5.5.2.4.1 Preparation of Sample Solution
If alcohol sample does not contain any external substances, take sample directly during bottling. Otherwise, distill the sample before any further tests.

5.5.2.4.2 Standard Curve Illustration
Extract 2.0 mL of each of the ethyl acetate series of standard reagents and place them individually in a 25 mL volumetric tube with stopper. Add 2.0 mL hydrochloric hydrochloric solution (prepared as in 5.5.2.3.1) and 2.0 mL sodium hydroxide solution (prepared as in 5.5.2.3.2), mix evenly and let it settle for the used 10 mins. Then add 2.0 mL hydrochloric acid solution (prepared as in 5.5.2.3.3), mix evenly. Then add 2.0 mL ferric chloride solution (prepared as in 5.5.2.3.4), mix evenly again. Use a 1 cm cuvette, readable to zero with a control tube and then determine the light absorbance of each under a wavelength of 525 nm. Plot the standard curve.

5.5.2.4.3 Determination of Sample Solution
Extract 2.0 mL sample solution (prepared as in 5.5.2.4.1) into a 25 mL volumetric tube with stopper and then operate in the same manner as in section 5.5.2.4.2. Determine the ethyl acetate content on the standard curve and that will be the total ester content. Alternatively, use linear regression to calculate the total ester content.

5.5.2.5 Precision
Discrepancies between the results of two independent tests conducted under iterative conditions and the average value of the test results should not exceed the 10% range.

5.6 Methanol

5.6.1 Principle
Same as 5.4.1.1.

5.6.2 Apparatus
Same as 5.4.1.2.

5.6.3 Reagents and Solutions

5.6.3.1 40% Ethanol Solution: Mix ethanol (chromatographically pure) with water.

5.6.3.2 Methanol Solution (2%) Use as standard sample. Extract 2 mL methanol (chromatographically pure), then dilute it with 40% ethanol solution till it reaches 100 mL volume.

5.6.3.3 M Isobutanol Solution (2%) Use as internal standard. Extract 2 mL isobutanol (chromatographically pure), then dilute it with 40% ethanol solution till it reaches 100 mL volume.

5.6.3.4 Chromatographic Conditions
Same as 5.4.1.4.

5.6.3.5 Analysis Procedure
Entropy of the analysis operation procedure is the same as what is described in section 5.4.1.5, with the

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5.6.3.5.1 Total aldehyde (acetaldehyde) content in a liter of 100% ethanol can be calculated with the following formula (9):

$$X_0 = X_i \cdot X_0 \cdot 0.37 \quad (9)$$

In the formula:
F - Relative calibration factor of acetaldehyde (or acetal);
A_i - Peak area (or peak height) of the internal standard; (or peak height) during the determination of standard sample value;
A_s - Peak area (or peak height) of acetal during the determination of standard sample value;
X₀ - Relative calibration factor of acetal; (or peak height) of acetaldehyde (or acetal) in the standard sample;
X_i - Internal standard (or peak height) of acetaldehyde (or acetal) content, unit is milligram per liter (mg/L);
V₁ - Volume of iodine standard reagent used on the sample, unit is milliliter (mL);
V₂ - Volume of iodine standard reagent used on the control experiment, unit is milliliter (mL);
V - Volume of sample absorbed, unit is milliliter (mL);
X₀ - Acetaldehyde (or Acetal) content in sample, unit is milligram per liter (mg/L);
A_i - Peak area (or peak height) of acetaldehyde (or acetal) in sample;
A_s - Peak area (or peak height) of internal standard added in the alcohol sample;
X₀ - Internal standard (or peak height) of acetaldehyde (or acetal) content, unit is milligram per liter (mg/L);
X_i - Acetaldehyde (or Acetal) in a liter of 100% ethanol in the sample, unit is milligram per liter (mg/L);
0.37 - Actual alcohol content of the sample;
X₀ - Total aldehyde (acetaldehyde) content in a liter of 100% ethanol in the sample, unit is milligram per liter (mg/L);
X_i - Acetaldehyde content in a liter of 100% ethanol in the sample, unit is milligram per liter (mg/L);
X₀ - Acetal content in a liter of 100% ethanol in the sample, unit is milligram per liter (mg/L);
0.37 - Conversion coefficient of acetal to acetaldehyde.

5.4.1.7 Precision
Discrepancies between the results of two independent tests conducted under iterative conditions and the average value of the test results should not exceed the 10% range.

5.4.2 Isobutanol

5.4.2.1 Principle
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5.6.4 Chromatographic Conditions
Same as 5.4.1.4.

5.6.5 Analysis Procedure
Entropy of the analysis operation procedure is the same as what is described in section 5.4.1.5, with the specific exception that the standard sample used will be replaced by isobutanol solution (prepared as in 5.6.3.2) instead.

5.6.6 Result Calculation
Same as 5.4.1.6.

5.6.7 Precision
Same as 5.4.1.7.

5.7 High Quality Alcohols

5.7.1 Principle
Same as 5.4.1.1.

5.7.2 Apparatus
Same as 5.4.1.2.

5.7.3 Reagents and Solutions

5.7.3.1 40% Ethanol Solution: Mix ethanol (chromatographically pure) with water.

5.7.3.2 Isobutanol Solution (2%) Use as standard sample. Extract 2 mL isobutanol (chromatographically pure), then dilute it with 40% ethanol solution till it reaches 100 mL volume.

5.7.3.3 Isobutanol Solution (2%) Use as internal standard. Extract 2 mL isobutanol (chromatographically pure), then dilute it with 40% ethanol solution till it reaches 100 mL volume.

5.7.4 Chromatographic Conditions
Same as 5.4.1.4.

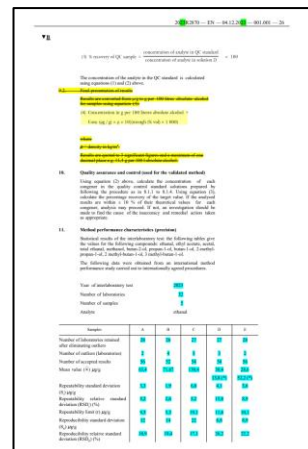
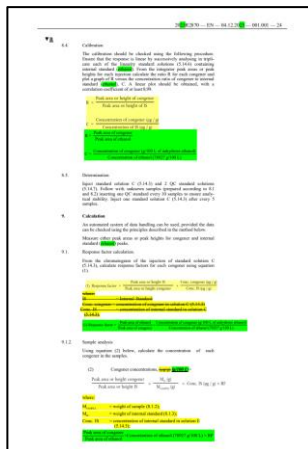
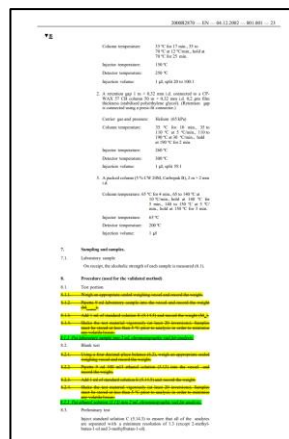
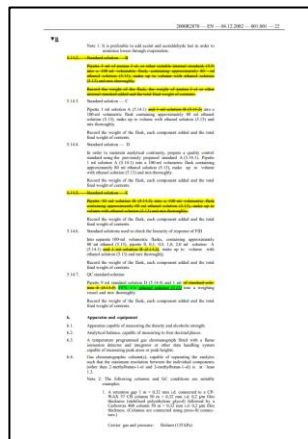
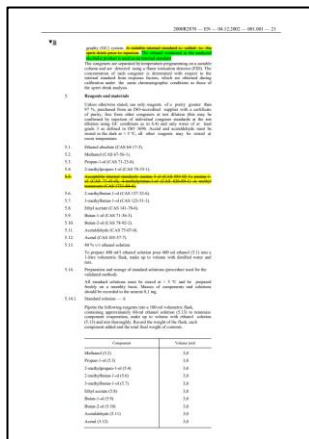
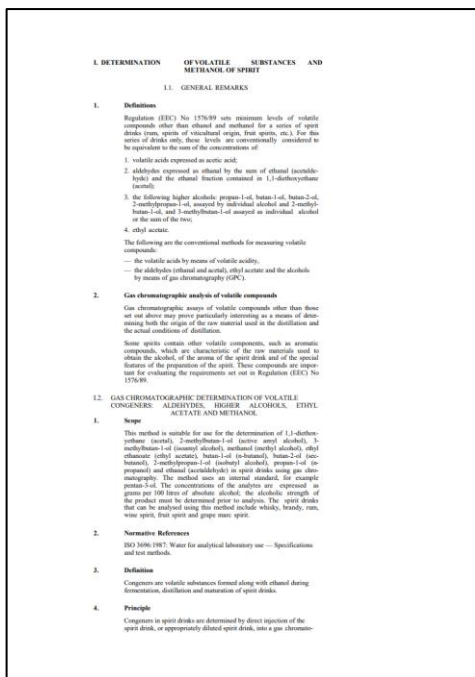
5.7.5 Analysis Procedure
Entropy of the analysis operation procedure is the same as what is described in section 5.4.1.5, with the specific exception that the standard sample used will be replaced by isobutanol solution (prepared as in 5.7.3.2) and internal standard used will be replaced by isobutanol solution (prepared as in 5.7.3.3) instead.

5.7.6 Result Calculation
Same as 5.4.1.6, determine total content of isobutanol and isocetyl ethanol.

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EC 2870/2000 Improvements

The use of the proposed method ensures high reliability of the data obtained, significantly reduces time, labor, material and financial costs. Analysis of volatile compounds in spirit drinks has never been so easy. Here you can read modified text of official method, which allows to carry out analysis of alcoholic beverages using the developed method. The places in the text document to be deleted are **highlighted in yellow**. Embedded parts of the test are **highlighted in green**.



BIS IS 3752:2005(R2009) Improvements

The use of the proposed method ensures high reliability of the data obtained, significantly reduces time, labor, material and financial costs. Analysis of volatile compounds in spirit drinks has never been so easy. Here you can read modified text of official method, which allows to carry out analysis of alcoholic beverages using the developed method. The places in the text document to be deleted are highlighted in yellow. Embedded parts of the test are highlighted in green.



IS 3752: 2005
 Methanol = $\frac{A \times C \times D \times 2000}{A \times S} \times 100 - 100 \times 100$

where:
 A = absorbance for sample standard solution;
 C = concentration of methanol standard solution, g/ml;
 D = dilution factor for sample solution;
 A = absorbance for methanol standard solution; and
 S = ethanol content of liquor sample in percent (v/v).

16.2 Gas Chromatographic method

16.2.1 Apparatus

a) Gas chromatograph and operating parameters — Gas chromatograph equipped with flame ionization detector and split injector port and fitted with a capillary column of HP-5 or equivalent having the dimensions of 25 m in length, 0.32 mm ID and 0.30 µm film thickness. The split ratio will be approximately 1:40 with fittings or balloons as a carrier gas at the flow rate of about 1.7 ml/min. The detector and injector port temperatures may be maintained at about 250°C. Keep the oven temperature at 45°C for 4 min, raise to 100°C/min at the rate of 10°C/min and finally to 200°C for 10 min at the rate of 15°C.

NOTE — Optimum operating conditions may vary with ethanol and instrument used and must be determined by using standard solutions. Adjust the parameters for maximum peak sharpness and optimum separation from ethanol.

b) Syringe — 10 µl Hamilton Co. No. 701, or equivalent.

16.2.2 Reagents

a) Ethanol — Methanol free.

16.2.3 Procedure

Transfer 5 ml of sample into a 10-ml stoppered test tube, add 1 ml of prepared internal standard solution and mix well. Inject 2 µl of working standard mixture into chromatograph and record the chromatogram. Adjust the operating parameters and retention time of methanol and ethanol to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol. Inject 2 µl sample solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol.

16.2.4 Calculation

Calculate the individual component in grams per 100 liters of absolute alcohol as follows:

$$\text{Methanol} = \frac{R \times C \times D \times 2000}{A \times S} \times 100 - 100 \times 100$$

where:
 R = peak ratio of methanol to ethanol for sample solution;
 C = concentration of methanol standard solution, in g/ml; **100 ml of standard solution**; **2000** — **conversion factor** for standard solution; **D** — dilution factor for sample solution;
 A = peak ratio of ethanol to ethanol in standard solution; **in g/ml**; **100 ml of standard solution**; and
 S = ethanol content of liquor sample in percent (v/v).

16.2.5 Reagents

a) Ethanol — Methanol free.

16.2.6 Procedure

Transfer 5 ml of sample into a 10-ml stoppered test tube, add 1 ml of prepared internal standard solution and mix well. Inject 2 µl of working standard mixture into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol. Inject 2 µl sample solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol.

IS 3752: 2005

ANNEX A
 Clause 1
ESTIMATION OF ESTERS, HIGHER ALCOHOLS, ALDEHYDES, FURFURAL AND METHANOL BY GAS CHROMATOGRAPHIC METHOD

A-1 DETAILED GAS CHROMATOGRAPHIC METHOD

A-1.1 Apparatus

A-1.1.1 Gas chromatograph and operating parameters — Gas chromatograph equipped with flame ionization detector and split injector port and fitted with a capillary column of HP-5 or equivalent having the dimensions of 25 m in length, 0.32 mm ID and 0.30 µm film thickness. The split ratio will be approximately 1:40 with fittings or balloons as a carrier gas at the flow rate of about 1.7 ml/min. The detector and injector port temperatures may be maintained at about 250°C. Keep the oven temperature at 45°C for 4 min, raise to 100°C/min at the rate of 10°C/min and finally to 200°C for 10 min at the rate of 15°C/min.

NOTE — Optimum operating conditions may vary with ethanol and instrument used and must be determined by using standard solutions. Adjust the parameters for maximum peak sharpness and optimum separation. Use high level standard, a prepared standard gas, alcohol component separation from ethanol.

A-1.1.2 Reagents

A-1.1.2.1 Ethanol — Methanol free.

A-1.1.2.2 Reagents

- Methanol
- Ethanol — Methanol free.
- Isobutylalcohol
- Acetylaldehyde
- Methyl acetate
- Ethyl acetate
- Isobutylalcohol
- n-Propyl acetate
- n-Butyl acetate
- n-Pentyl acetate
- n-Hexyl acetate
- n-Heptyl acetate
- n-Octyl acetate
- n-Nonyl acetate
- n-Decyl acetate
- n-Dodecyl acetate

A-1.1.4 Preparation of working standard mixture

Transfer 5 ml of standard mixture (see A-1.1.2) into a 10-ml stoppered test tube, add 1 ml of prepared internal standard solution and mix well.

A-1.1.5 Procedure

Transfer 5 ml of sample into a 10-ml stoppered test tube, add 1 ml of prepared internal standard solution and mix well. Inject 2 µl of working standard mixture into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol. Inject 2 µl sample solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol.

IS 3752: 2005

NOTE — Optimum operating conditions may vary with ethanol and instrument used and must be determined by using standard solutions. Adjust the parameters for maximum peak sharpness and optimum separation. Use high level standard, a prepared standard gas, alcohol component separation from ethanol.

A-2.1.2 Reagents

A-2.1.2.1 Ethanol — Methanol free.

A-2.1.2.2 Reagents

- Methanol
- Ethanol — Methanol free.
- Isobutylalcohol
- Acetylaldehyde
- Methyl acetate
- Ethyl acetate
- Isobutylalcohol
- n-Propyl acetate
- n-Butyl acetate
- n-Pentyl acetate
- n-Hexyl acetate
- n-Heptyl acetate
- n-Octyl acetate
- n-Nonyl acetate
- n-Decyl acetate
- n-Dodecyl acetate

A-2.1.4 Preparation of Standard Mixture

Transfer accurately a known quantity of about 5.0 g of the reagents listed from A-2.1.2.1 to A-2.1.2.11 in to different 100 ml volumetric flasks and dilute to 100 ml with 40 percent (v/v) ethanol (methanol-free). Transfer 1.0 ml of each of the resulting solutions into a 100-ml volumetric flask and dilute to volume with 40 percent (v/v) ethanol (methanol-free). This solution will give approximately 500 ppm of each of component listed above.

A-2.1.5 Procedure

Transfer 5 ml of sample into a 10-ml stoppered test tube, add 1 ml of prepared internal standard solution and mix well. Inject 2 µl of working standard mixture into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol. Inject 2 µl sample solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol.

IS 3752: 2005

NOTE — Optimum operating conditions may vary with ethanol and instrument used and must be determined by using standard solutions. Adjust the parameters for maximum peak sharpness and optimum separation. Use high level standard, a prepared standard gas, alcohol component separation from ethanol.

A-2.1.2 Reagents

A-2.1.2.1 Ethanol — Methanol free.

A-2.1.2.2 Reagents

- Methanol
- Ethanol — Methanol free.
- Isobutylalcohol
- Acetylaldehyde
- Methyl acetate
- Ethyl acetate
- Isobutylalcohol
- n-Propyl acetate
- n-Butyl acetate
- n-Pentyl acetate
- n-Hexyl acetate
- n-Heptyl acetate
- n-Octyl acetate
- n-Nonyl acetate
- n-Decyl acetate
- n-Dodecyl acetate

A-2.1.4 Preparation of Standard Mixture


Transfer accurately a known quantity of about 5.0 g of the reagents listed from A-2.1.2.1 to A-2.1.2.11 in to different 100 ml volumetric flasks and dilute to 100 ml with 40 percent (v/v) ethanol (methanol-free). Transfer 1.0 ml of each of the resulting solutions into a 100-ml volumetric flask and dilute to volume with 40 percent (v/v) ethanol (methanol-free). This solution will give approximately 500 ppm of each of component listed above.

A-2.1.5 Procedure

Transfer 5 ml of sample into a 10-ml stoppered test tube, add 1 ml of prepared internal standard solution and mix well. Inject 2 µl of working standard mixture into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol. Inject 2 µl sample solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time to obtain measurable peaks (at least 2% percent of full scale deflection). Determine the retention time of methanol and ethanol.

Norma Mexicana NMX-V-005-NORMEX-2018 Improvements

The use of the proposed method ensures high reliability of the data obtained, significantly reduces time, labor, material and financial costs. Analysis of volatile compounds in spirit drinks has never been so easy. Here you can read modified text of official method, which allows to carry out analysis of alcoholic beverages using the developed method. The places in the text document to be deleted are highlighted in yellow. Embedded parts of the test are highlighted in green.


NMX-V-005-NORMEX-2013
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5.0 DETERMINACION DE ALDEHIDOS, ESTERES, METANOL Y ALCOHOLES SUPERIORES. METODO POR CROMATOGRAFIA DE GASES

5.1 Fundamento
 Este método se basa en los principios de la cromatografía de gases y consiste en la inyección de una pequeña cantidad de la muestra (que contiene una mezcla de sustancias volátiles) en el inyector de un cromatógrafo de gases en el que son vaporizadas y transportadas por un gas inerte a través de una columna empacada o capilar con un líquido de partición que presenta solubilidad selectiva con los componentes de la muestra, ocasionando su separación.

Los componentes que eluyen de la columna pasan uno a uno por el "detector", el cual genera una señal eléctrica proporcional a su concentración, la que es transformada por el registrador, integrador o sistema de manejo de datos en una gráfica llamada cromatograma.

La identificación de cada componente registrado como un pico en el cromatograma, se realiza por inyección del o de los componentes en forma pura y con las mismas características y entidades que se sospecha contiene la muestra, midiendo el tiempo de retención en esas condiciones. También se puede comprobar por adición del componente a la muestra e inyectándola nuevamente para apreciar el incremento de altura o área del pico correspondiente.

La cuantificación se puede efectuar por cualquiera de estos tres métodos: normalización, estandarización externa y estandarización interna, siendo este último el único que se describe a continuación:


La cuantificación por estandarización interna consiste en obtener el cromatograma de la muestra estandarizada, adicionada de una Sustancia llamada estándar interno que debe aparecer en un sitio del cromatograma, libre de interferencias y desde luego no debe ser componente de la muestra, aunque es recomendable que sea de la misma naturaleza química y del mismo intervalo de concentración que el componente de la muestra por cuantificar. Deben obtenerse cromatogramas paralelos con soluciones de concentración conocida de cada componente por cuantificar y del estándar interno que sea adecuada muestra y trazar una curva de calibración que tenga por ordenada la relación de concentraciones correspondientes al componente por cuantificar y al estándar interno y en las abscisas la relación de áreas correspondientes al compuesto por cuantificar y a las áreas del estándar interno.

Esta curva sirve para situar en sus ordenadas la relación de áreas correspondientes al componente por cuantificar y el estándar interno del cromatograma de la muestra estandarizada y así ubicar la relación correspondiente de concentraciones.

5.2 Alcance
 Este método determina la concentración de aldehídos, ésteres, alcoholes superiores y metanol en bebidas alcohólicas por cromatografía de gases.

5.3 Equipos e instrumentos
 Todos los equipos e instrumentos de medición deberán ser calibrados y/o verificados.

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5.4.1 ampollada (por los materiales volátiles y la toxicidad de este compuesto se recomienda usar una ampollada sellada).

5.4.2 Acetil.

5.4.3 Metanol.

5.4.4 Sesi-butanol (2-butanol).

5.4.5 n-propanol (1-propanol).

5.4.6 n-butanol (1-butanol).

5.4.7 iso-butanol (2-metil-1-propanol).

5.4.8 isomillico (3-metil-1-butanol).

5.4.9 Analizador Activo(2-metil-1-butanol) (aplicable en caso de que el volumen haga la separación de este metanol). Ver 5.11.

5.4.10 acetona (1-propanol).

5.4.11 Acetato de etilo.

5.4.12 Lente de etilo.


5.4.13 **Ensamblaje de sonda Hidratado de sodio.**

5.4.14 Alcohol etílico grado cromatográfico y/o libre de los componentes a cuantificar verificado por cromatografía de gases antes de usarlo.

5.4.16 Solución de alcohol etílico al 40% v/v.

Mod: 400 ml de etanol en una probeta y llevar al volumen de 1000 ml con agua, agitar el pH de 8.2 a 8.5 con bicarbonato de sodio o hidróxido de sodio para evitar la evaporación de agua de los componentes en un análisis ácido.

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La adición de la cantidad necesaria de acetilbaldedo se puede realizar de las siguientes maneras:

a) Medir con una pipeta o micropipeta preferencia goteo; o
 b) Medir con una pipeta o micropipeta previamente refrigerada; o
 c) Transferir el contenido de un vial o ampollita sellada, en todos los casos el material debe utilizarse como mínimo a 273 K (0°C).

Tapar el matraz y determinar su masa nuevamente; anotar el valor de la masa, aguar solución de etanol al 40 % v/v cercana a la línea de sifón; mantener el matraz volumen seco en ambiente controlado (por el menor tiempo 30 minutos); llevar al baño homogenizador. Si la solución se va a utilizar posteriormente se almacena en refrigeración.

Nota: Todos los reactivos deberán almacenarse de acuerdo a las indicaciones del fabricante.

5.4.3 - Preparación de soluciones de estándares internos
 Efectuar como se expone en el procedimiento del 2.1.

En un matraz volumétrico de 100 ml adicionar aproximadamente 50 ml de etanol al 40 % v/v, tapar el matraz y determinar su masa nuevamente; la cantidad requerida de estándar interno; agitar y determinar la masa nuevamente; agregar solución de etanol al 40 % v/v cercana a la línea de sifón; y homogenizar. Calentar el volumétrico en un ambiente controlado hasta llevarlo a 273 K (0°C) y 5 minutos y homogenizar.


La concentración de las soluciones se valida a la siguiente manera:

Concentración del analito en g/100ml (P)
 Estando
C = concentración del matraz con etanol al 40% v/v y cantidad interna
P = masa del matraz con etanol al 40% v/v
C tiene su propia solución la densidad de base en la balanza analítica. Ver Tabla 1 y Tabla 2.

5.4.4 Preparación de las diluciones de calibración
 Para preparar las soluciones de calibración se transferirá a matraces volumétricos de 100 ml las cantidades necesarias de la solución concentrada a temperatura controlada del laboratorio para obtener las concentraciones en $\mu\text{g}/100 \text{ ml}$ AA recomendadas en la Tabla No. 2, adicionando el volumen necesario de solución estándar interna. Posteriormente llevar al volumen con la solución de etanol.

Estas soluciones deben guardarse bien tapadas en refrigeración.

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Con el objeto de obtener cromatogramas confiables debe tomarse en cuenta las siguientes precauciones:

- Acondicionamiento de la columna.
- Limpieza del inyector, detector y columna.
- Dirección de flujo del sistema.

Inyectar al cromatógrafo la cantidad de muestra apropiada. La cantidad sugerida de inyección es de 1 a 2 μl .

5.6 Preparación de la muestra
 A las muestras que requieren reportarse en $\text{mg}/100 \text{ ml}$ de alcohol anhidro (AA) se les debe determinar el contenido de alcohol en % AA. Ver a. 2.6.3.2.2.2.2 de acuerdo a la NMX-V-013-NORMEX vigente.

Para tener estándares confiables en un experimento prepare los como una solución volumétrica con un volumen volumétrico y a temperatura de 20 ± 0.2°C. Use pipetas volumétricas o micropipetas calibradas a temperatura ambiente y asegure de que el volumen del estándar interno que se agrega a las diluciones de calibración sea el mismo que el del componente a purificar. Mantenga la solución de calibración en un ambiente controlado a 273 K (0°C) y 5 minutos y homogenizar.

5.6.7 Curva de calibración
 Se requiere mínimo cinco niveles en la elaboración de la curva de calibración y se requiere mínimo por duplicado cada nivel para obtener los cromatogramas respectivos y con estos realizar la curva de calibración en el equipo.

5.6.8 Análisis de la muestra
 Inyectar al cromatógrafo la cantidad adecuada a muestra para obtener el cromatograma correspondiente.

5.7 Cálculos y resultado

5.7.1 Expresión de resultado
 Los resultados se deben expresar en mg de aldehídos, ésteres, alcoholes superiores y metanol referidos a 100 ml de alcohol anhidro ($\text{mg}/100 \text{ ml}$ AA) utilizando al menos una cifra decimal. En caso de ser necesario se podrá expresar en otra unidad reflejando la conversión correspondiente.

Los aldehídos isomillico y acetil activo pueden expresarse por separado o como la suma de estos.


5.7.2 Cálculo de relación de concentraciones y de áreas, en la curva de calibración y de la muestra.
 Cuando el equipo cuenta con software, este realiza los cálculos en forma automática, basándose en el modelo matemático de regresión lineal:

$$y = mX + b$$

En donde:

Relación de área del compuesto a cuantificar entre el área del estándar interno $\left(\frac{A}{A_s}\right)$

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X= relación de la concentración del analito entre la concentración del estándar interno en $\text{mg}/100 \text{ ml}$ AA por su coeficiente de respuesta relativo
 m= pendiente (factor de respuesta relativo)
 b= intercepto en el origen de la unidad, "y".

Sustituyendo variables:

$$\left(\frac{A}{A_s}\right) = m \left(\frac{C}{C_s}\right) + b$$

Despejando para obtener la concentración del compuesto C_x en 100 ml:

Comparar concentración C_x

Concediendo el factor de dilución el contenido de alcohol de la muestra se puede determinar del compuesto expresado en $\text{mg}/100 \text{ ml}$ AA se tiene como el siguiente: Fórmula

$$\text{Concentración en } \frac{\text{mg}}{100 \text{ ml}} \text{ AA} = C_x = \left(\frac{A}{A_s}\right) \cdot \left(\frac{C_s}{m}\right) - \frac{b}{m}$$

En donde:

100 = Punto de dilución en la preparación de la muestra con el estándar interno (Volumen total del matraz volumétrico). Volumen de muestra empleado en la preparación.




























100 = Volumen de alcohol de la muestra en la unidad que volumen = 200 K (0°C)

Nota: Para realizar registros en cada uno de los puntos de la curva, se puede preparar los áreas para validar los factores de respuesta.
 Los datos de calibración se convierten a valores del coeficiente de correlación (R) en mayor o igual a 0.99 los aldehídos superiores pueden hacerse utilizando otros de pureza de grado.

5.7 Repetibilidad y reproducibilidad
 5.8.1 Repetibilidad
 5.8.1.1 La repetibilidad de los resultados de las mediciones con este método.




























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Determination of **methanol** in alcoholic beverages

Result for	 40 % ABV	 40 % ABV	 43 % ABV	 40 % ABV	 40 % ABV	 40 % ABV	 40 % ABV	 47 % ABV	 45 % ABV
	Rum	Whiskey	Bourbon	Grain spirit	Brandy	Grappa	Calvados	Gin	Slivovice
Official method, mg/L AA	22.2±0.5	132±2	88.4±1.2	110±1.6	297±2	414±5	910±5	4.16±0.09	10546±97
Developed method, mg/L AA	22.3±0.6	130±1	88.9±0.5	111±0.7	297±1	412±2	913±2	4.19±0.16	10603±18
Δ , %	0.7	-0.9	0.6	0.9	-0.2	-0.6	0.3	0.8	0.5
Result for	 38 % ABV	 14.5 % ABV	 38 % ABV	 15 % ABV	 18 % ABV	 8.5 % ABV	 70 % ABV	 27.5 % ABV	 40 % ABV
	Tsikoudia	Sake	Tequila	Vermouth	Nalewka	Mulled wine	Rectified spirit	Cocktail	Vodka
Official method, mg/L AA	755±50	18.2±1.3	1456±35	17.5±0.1	168±5	25.3±3.0	6.05±0.39	77.3±0.7	21.8±0.2
Developed method, mg/L AA	761±20	18.1±1.4	1460±10	17.6±0.2	169±4	25.1±2.7	6.03±0.40	76.3±1.5	21.7±0.2
Δ , %	0.8	-1.0	0.3	0.6	0.9	-0.6	-0.4	-1.2	-0.7
Result for	 38 % ABV	 17 % ABV	 35 % ABV	 25 % ABV	 16 % ABV	 16.5 % ABV	 35 % ABV	 40 % ABV	 56 % ABV
	Liqueurs							Rakia	Baijiu
Official method, mg/L AA	2.32±0.04	9.75±0.28	19.5±0.1	29.1±0.9	9.77±1.34	127±5	20.5±0.7	118623	115±5
Developed method, mg/L AA	2.34±0.05	9.81±0.14	19.6±0.1	29.4±1.0	9.82±1.27	126±4	20.7±0.4	11791	116±4
Δ , %	0.8	0.7	0.4	0.8	0.5	-1.1	0.5	0.6	0.6

The relative difference between obtained values of concentrations (Δ , %) measured in accordance with the EC 2870/2000 according to the official internal standard method and in accordance with the proposed modified internal standard method does not exceed **1.5 %**.

Determination sums of aldehydes, esters and high alcohols in alcoholic beverages

Result for	 40 % ABV	 40 % ABV	 43 % ABV	 40 % ABV	 40 % ABV	 40 % ABV	 40 % ABV	 47 % ABV	 45 % ABV
	Rum	Whiskey	Bourbon	Grain spirit	Brandy	Grappa	Calvados	Gin	Slivovice
Official method, mg/L AA	48.1 / 145 / 1043	162 / 589 / 6693	150 / 645 / 5546	44.0 / 84.7 / 4662	143 / 396 / 4801	191 / 289 / 2113	182 / 583 / 3690	1.70 / 0 / 1.54	210 / 907 / 6255
Developed method, mg/L AA	48.4 / 146 / 1051	160 / 584 / 6635	151 / 649 / 5580	44.4 / 85.4 / 4703	142 / 396 / 4794	190 / 288 / 2100	182 / 585 / 3702	1.72 / 0 / 1.55	211 / 912 / 6288
Δ, %	0.7 / 0.7 / 0.7	-0.9 / -0.9 / -0.9	0.6 / 0.6 / 0.6	0.9 / 0.9 / 0.9	-0.2 / -0.2 / -0.2	-0.6 / -0.6 / -0.6	0.3 / 0.3 / 0.3	0.8 / - / 0.9	0.5 / 0.5 / 0.5
Result for	 38 % ABV	 14.5 % ABV	 38 % ABV	 15 % ABV	 18 % ABV	 8.5 % ABV	 70 % ABV	 27.5 % ABV	 40 % ABV
	Tsikoudia	Sake	Tequila	Vermouth	Nalewka	Mulled wine	Rectified spirit	Cocktail	Vodka
Official method, mg/L AA	356 / 266 / 2297	37.6 / 47.0 / 1367	34.8 / 126 / 2895	30.5 / 0 / 5.94	47.4 / 74.4 / 10.3	22.7 / 55.9 / 871	4.83 / 25.2 / 0	61.9 / 84.0 / 728	0.504 / 0 / 0
Developed method, mg/L AA	359 / 268 / 2316	37.2 / 46.5 / 1352	34.9 / 127 / 2904	30.6 / 0 / 5.98	47.8 / 75.1 / 10.4	22.5 / 55.6 / 866	4.81 / 25.1 / 0	61.1 / 83.0 / 719	0.50 / 0 / 0
Δ, %	0.9 / 0.8 / 0.9	-1.1 / -1.1 / -1.1	0.4 / 0.3 / 0.3	0.6 / - / 0.6	0.9 / 0.9 / 0.9	-0.6 / -0.5 / -0.6	-0.4 / -0.4 / -	-1.3 / -1.2 / -1.2	-0.7 / - / -
Result for	 38 % ABV	 17 % ABV	 35 % ABV	 25 % ABV	 16 % ABV	 16.5 % ABV	 35 % ABV	 40 % ABV	 56 % ABV
	Liqueurs							Rakia	Baijiu
	Sambuca	Egg	Herbal	Limon	Cherry	Raspberry	Sloe gin		
Official method, mg/L AA	4.20 / 0 / 2.44	6.89 / 0 / 125	38.1 / 13.5 / 9.39	25.1 / 0 / 0	18.4 / 266 / 0	36.6 / 31.8 / 0	1.12 / 0 / 0	92.2/1334/6165	63.9 / 1072 / 2114
Developed method, mg/L AA	4.24 / 0 / 2.46	6.94 / 0 / 125	38.2 / 13.5 / 9.43	25.3 / 0 / 0	18.5 / 267 / 0	36.2 / 31.5 / 0	1.13 / 0 / 0	91.6/1325/6217	64.3 / 1079 / 2128
Δ, %	0.8 / - / 0.8	0.8 / - / 0.7	0.4 / 0.4 / 0.4	0.8 / - / -	0.5 / 0.6 / -	-1.0 / -1.1 / -	0.6 / - / -	0.6/0.7/0.6	0.6 / 0.6 / 0.6

Aldehydes = acetaldehyde + acetal / Esters = ethyl acetate / Highs alcohols = butan-2-ol + propan-1-ol + 2-methylpropan-1-ol + butan-1-ol + 3-methylbutan-1-ol

The relative difference between obtained values of concentrations (Δ , %) measured in accordance with the EC 2870/2000 according to the official internal standard method and in accordance with the proposed modified internal standard method does not exceed **1.5 %**.

You can ask any questions and for collaboration you are interested in at these email addresses:
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