



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

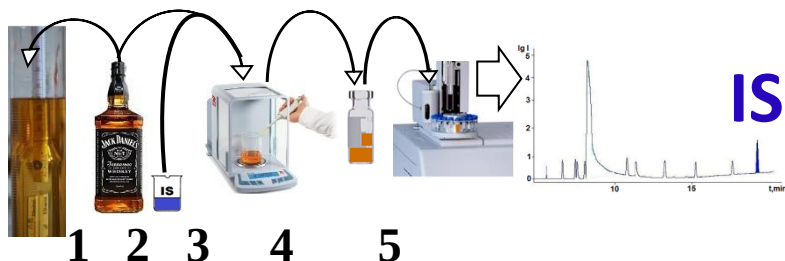


Norma Mexicana NMX-V-005-NORMEX-2018

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:

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

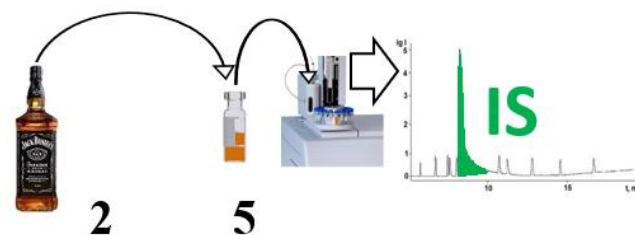
The values of the relative response coefficients of the detector to the investigated volatile component relative to the response to the selected internal standard are calculated using the following formula:

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

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):

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

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

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

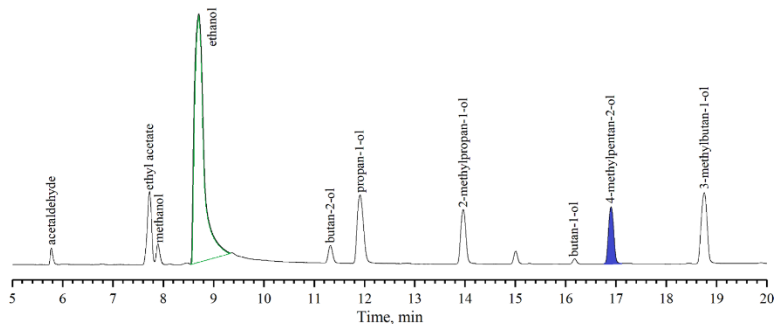
The values of the relative coefficients of the detector response to the investigated volatile component relative to the response to ethanol are calculated using the following formula:

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

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
 $\rho_{\text{Eth}} = 789270 \text{ mg/L}.$

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.



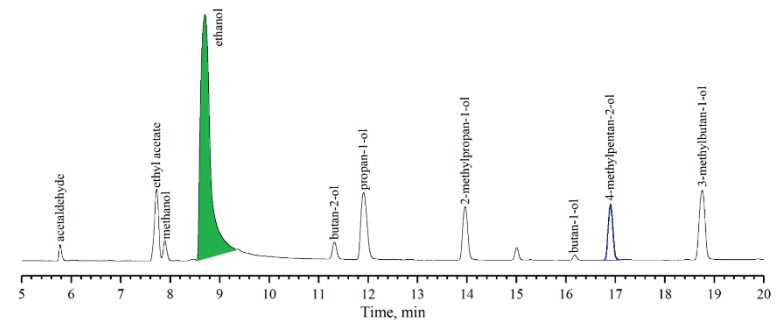
$$C_i(\text{mg/L AA}) = RRF_i^{IS} \cdot \frac{A_i}{A_{IS}} \cdot C_{IS}(\text{mg/kg}) \cdot \frac{\rho_{\text{sample}}(\text{kg/L}) \cdot 100 \%}{\text{"Strength" } (\%, \text{ ABV})}$$

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.



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

Done

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26. Improved document National standards of People's Republic of China GB/T 11858 <https://elab.bsu.by/download.php?id=307>
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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.

1. DETERMINATION OF VOLATILE SUBSTANCES AND METHANOL OF SPIRIT

1.1. GENERAL REMARKS

Definitions

1. **Regulation** (EEC No 1759/89 sets minimum levels of volatile components other than ethanol and methanol for a series of spirit drinks (from spirits of vitaceae origin, fruit spirits, etc.) For this series of drinks only, these levels are conventionally considered to be equivalent to the sum of the concentrations of:

- 1. volatile acids expressed as acetic acid,
- 2. aldehydes expressed as ethanol by the sum of ethanol (acetaldehyde) and the ethanol fraction contained in 1,3-dithiolanepentane (acetol),
- 3. the following higher alcohols: propyl 1-ol, butan-2-ol, butan-2-ol, 2-methylbutan-1-ol, isopentyl 1-ol, 2-methylpentan-1-ol, 2-methylbutan-1-ol, and 3-methylbutan-1-ol expressed as individual alcohol or the sum of the five,
- 4. ethyl acetate.

The following are the conventional methods for measuring volatile components:

- the volatile acids by means of volatile acidity;
- the aldehydes (ethanol and acetol), ethyl acetate and the alcohols by means of gas chromatography (GPC).

2. **Gas chromatographic analysis of volatile components**

Gas chromatographic analysis of volatile components other than those set out above may be particularly interesting in a series of distillates, following both the regular use of the material used in the distillation and the actual conditions of distillation.

Some spirits contain other volatile components, such as aromatic compounds, which are characteristic of the raw materials used to obtain the alcohol, of the aroma of the spirit drink and of the special features of the preparation of the spirit drink. These components are important for evaluating the requirements set out in Regulation (EEC) No 1759/89.

3. **GAS CHROMATOGRAPHIC DETERMINATION OF VOLATILE CONGENERS: ALDEHYDES, HIGHER ALCOHOLS, ETHYL ACETATE AND METHANE**

1. **Scope**

This method is suitable for use for the determination of 1,3-dithiolanepentane, 2-methylbutan-1-ol (active amyl), 3-methylbutan-1-ol (isomer) alcohol (active amyl), ethyl acetate, ethanone [ethyl acetone], butan-1-ol (n-butanol), butan-2-ol (iso-butanol), 2-methylpentan-1-ol (active amyl), 2-methylbutan-1-ol (isopentyl) and ethanol (acetaldehyde) in spirit drinks using gas chromatography. The method uses an internal standard, for example penta-3-ol. The concentrations of the analytes are expressed as grams per 100 litres of absolute alcohol (g/100 l abs). The strength of the product may be determined by means of the spirit drinks that can be analysed using this method include whisky, brandy, rum, wine, rum, fruit spirit and grape marc spirit.

2. **Normative References**

ISO 1831 (1977): Water for analytical laboratory use—Specifications and test methods.

3. **Definitions**

Congeners are volatile substances formed along with ethanol during fermentation, distillation and maturation of spirit drinks.

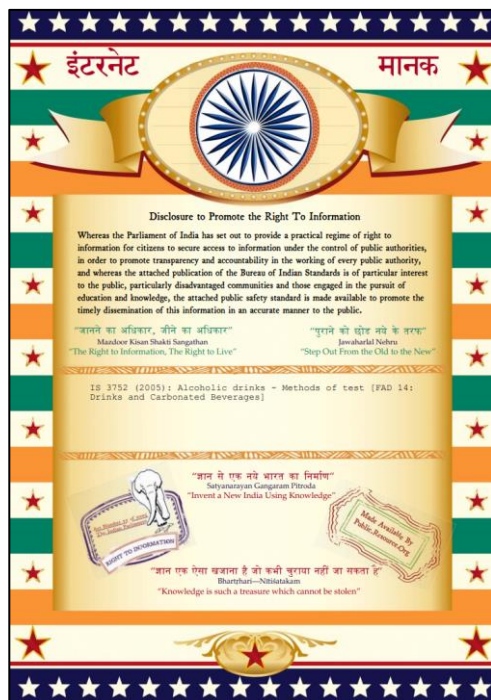
4. **Principle**

Congeners in spirit drinks are determined by direct injection of the spirit drink, or appropriately diluted spirit drink, into a gas chromatograph.

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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.



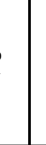
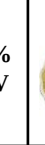
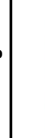
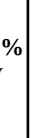
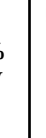
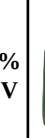
IS 3752: 2005	
Methanol : $\frac{A \times C \times D \times 1000}{A \times S}$	
where: A = absorbance for sample standard solution; C = concentration of methanol standard solution, g/ml; D = dilution factor for sample solution; A = absorbance for methanol standard solution; and S = ethanol content of 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 length, 0.32 mm ID and 0.30 µm film thickness. The split ratio will be approximately 1:40 with nitrogen or helium 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.	
b) Methanol stock solution — Dilute 1.0 g of methanol (99.9 percent, v/v) to 100 ml with 40 percent (v/v) ethanol (methanol-free).	
c) Methanol standard solution — Dilute 10 ml of methanol stock solution to 100 ml with 40 percent (v/v) ethanol (methanol-free). Dilute 10 ml of this solution to 100 ml with 40 percent (v/v) ethanol (methanol-free). Transfer 10 ml of the resulting solution into a 10 ml stoppered test tube, add 1 ml of potassium periodate standard solution and mix well.	
16.2.2 Procedure	
Transfer 5 ml of sample into a 10 ml stoppered test tube, add 1 ml of potassium periodate standard solution and mix well. Inject 2 µl of methanol standard solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time of methanol peak (at least 25 percent of full scale deflection). Determine the retention time of methanol and inject 2 µl of sample solution into chromatograph and record the chromatogram. Adjust retention time of methanol.	
16.2.3 Calculation	
Calculate the individual component in grams per 100 liters of absolute alcohol as follows: $\text{Methanol} = \frac{R_s \times C \times D \times 1000}{A \times S}$	
where: R _s = peak ratio of methanol to approximate 100 percent for sample solution; C = concentration of methanol standard solution, g/ml; Inject 2 µl of sample solution into chromatograph and record the chromatogram. Adjust retention time of methanol. D = dilution factor for sample solution; A = peak ratio of methanol to approximate 100 percent for standard solution; S = ethanol content of liquor sample in percent (v/v).	
16.2.4 Reagents	
a) Ethanol — Methanol free. b) Potassium periodate standard solution — 0.10 percent v/v prepared in 40 percent v/v ethanol (methanol-free).	

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 length, 0.32 mm ID and 0.30 µm film thickness. The split ratio will be approximately 1:40 with nitrogen or helium 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.	
A-1.1.2 Reagents	
a) Ethanol — Methanol free. b) Potassium periodate standard solution — 0.10 percent v/v prepared in 40 percent v/v ethanol (methanol-free). c) Ethanol — Methanol free. d) Methanol. e) Acetylaldehyde. f) Isobutylaldehyde. g) Methyl acetate. h) Ethyl acetate. i) Iso-valeraldehyde. j) n-Propyl acetate. k) Butyryl acetate. l) n-Butyl alcohol. m) n-Hexyl acetate. n) Ethyl capitate. o) Iso-butanol. p) Iso-valeryl acetate. q) n-Butanol. r) Iso-valeryl alcohol. s) n-Propyl alcohol.	
A-1.1.3 Preparation of Standard Mixture	
Transfer accurately a known quantity of about 5.0 g of the reagents listed from A-1.1.2(a) to A-1.1.2(r) in to different 100 ml volumetric flasks and dilute to 100 ml with 40 percent (v/v) ethanol (methanol-free). Transfer 10 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 each of component listed above.	
A-1.1.4 Preparation of working standard mixture	
Transfer 5 ml of standard mixture (see A-1.1.3) into a 10 ml stoppered test tube, add 1 ml of potassium periodate 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 potassium periodate standard solution and mix well. Inject 2 µl of working standard mixture solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time of methanol.	

IS 3752: 2005	
Notes — Optimum operating conditions may vary with column and instrument used and must be determined by using standard solutions. Adjust the parameters for maximum peak shape and optimum separation. With high level standard, a prepared should give almost complete baseline separation from ethanol.	
A-2.1.2 Reagents	
a) Ethanol — Methanol free. b) Potassium periodate standard solution — 0.10 percent v/v prepared in 40 percent v/v ethanol (methanol-free). c) Ethanol — Methanol free. d) Methanol. e) Acetylaldehyde. f) Isobutylaldehyde. g) Methyl acetate. h) Ethyl acetate. i) Iso-valerylaldehyde. j) n-Propyl acetate. k) Butyryl acetate. l) n-Butyl alcohol. m) n-Hexyl acetate. n) Ethyl capitate. o) Iso-butanol. p) Iso-valeryl acetate. q) n-Butanol. r) Iso-valeryl alcohol. s) n-Propyl alcohol.	
A-2.1.3 Preparation of Standard Mixture	
Transfer accurately a known quantity of about 5.0 g of the reagents listed from A-2.1.2(a) to A-2.1.2(r) in to different 100 ml volumetric flasks and dilute to 100 ml with 40 percent (v/v) ethanol (methanol-free). Transfer 10 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 each of component listed above.	
A-2.1.4 Preparation of working standard mixture	
Transfer 5 ml of standard mixture (see A-2.1.3) into a 10 ml stoppered test tube, add 1 ml of potassium periodate standard solution and mix well.	
A-2.1.5 Procedure	
Transfer 5 ml of sample into a 10 ml stoppered test tube, add 1 ml of potassium periodate standard solution and mix well. Inject 2 µl of working standard mixture solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time of methanol.	

IS 3752: 2005	
Notes — Optimum operating conditions may vary with column and instrument used and must be determined by using standard solutions. Adjust the parameters for maximum peak shape and optimum separation. With high level standard, a prepared should give almost complete baseline separation from ethanol.	
A-2.1.2 Reagents	
a) Ethanol — Methanol free. b) Potassium periodate standard solution — 0.10 percent v/v prepared in 40 percent v/v ethanol (methanol-free). c) Ethanol — Methanol free. d) Methanol. e) Acetylaldehyde. f) Isobutylaldehyde. g) Methyl acetate. h) Ethyl acetate. i) Iso-valerylaldehyde. j) n-Propyl acetate. k) Butyryl acetate. l) n-Butyl alcohol. m) n-Hexyl acetate. n) Ethyl capitate. o) Iso-butanol. p) Iso-valeryl acetate. q) n-Butanol. r) Iso-valeryl alcohol. s) n-Propyl alcohol.	
A-2.1.3 Preparation of Standard Mixture	
Transfer accurately a known quantity of about 5.0 g of the reagents listed from A-2.1.2(a) to A-2.1.2(r) in to different 100 ml volumetric flasks and dilute to 100 ml with 40 percent (v/v) ethanol (methanol-free). Transfer 10 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 each of component listed above.	
A-2.1.4 Preparation of working standard mixture	
Transfer 5 ml of standard mixture (see A-2.1.3) into a 10 ml stoppered test tube, add 1 ml of potassium periodate standard solution and mix well.	
A-2.1.5 Procedure	
Transfer 5 ml of sample into a 10 ml stoppered test tube, add 1 ml of potassium periodate standard solution and mix well. Inject 2 µl of working standard mixture solution into chromatograph and record the chromatogram. Adjust the operating parameters and retention time of methanol.	

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 **highs 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
	Sambuca	Egg	Herbal	Limon	Cherry	Raspberry	Sloe gin	Rakia	Baijiu
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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