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

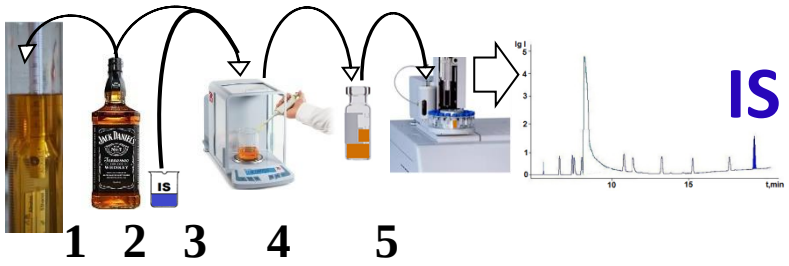
All listed national standards are harmonized with Regulation (EC) 2870/2000 and use the **traditional internal standard method**

AOAC Official Methods 972.10/11, 2005

Norma Mexicana NMX-V-005-NORMEX-2018

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:

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

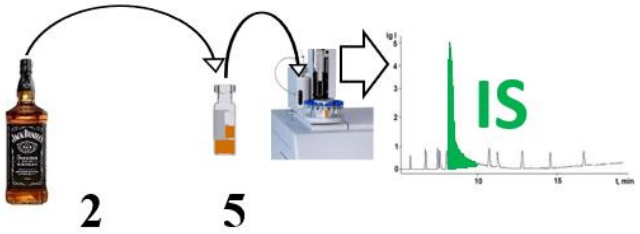
The values of the relative response coefficients of the detector to the investigated volatile component relative to the response to the selected internal standard are calculated using the following formula:

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

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):

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

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

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

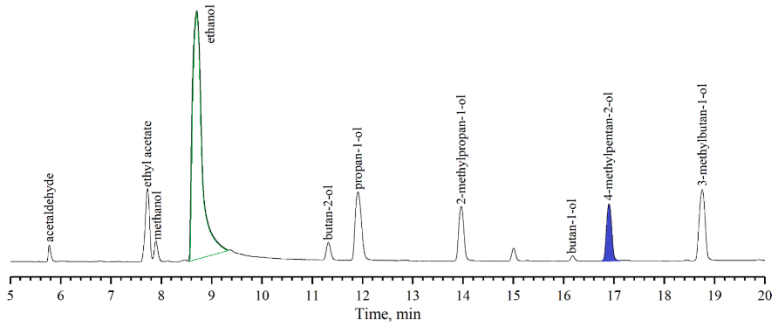
The values of the relative coefficients of the detector response to the investigated volatile component relative to the response to ethanol are calculated using the following formula:

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

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 $\rho_{Eth} = 789270$ 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.



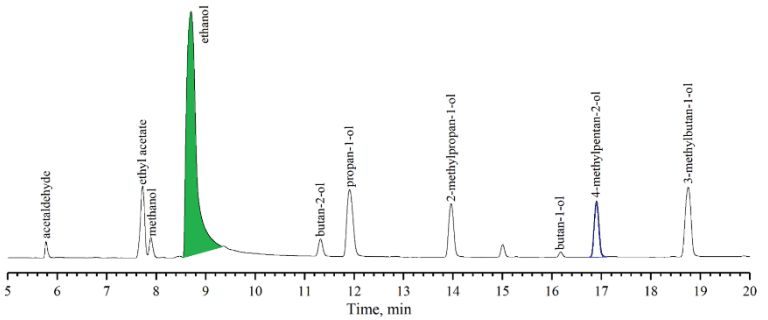
$$C_i(\text{mg/L } AA) = RRF_i^{IS} \cdot \frac{A_i}{A_{is}} \cdot C_{IS}(\text{mg/kg}) \cdot \frac{\rho_{sample}(\text{kg/L}) \cdot 100\ \%}{\text{"Strength" } (\%,\ ABV)}$$

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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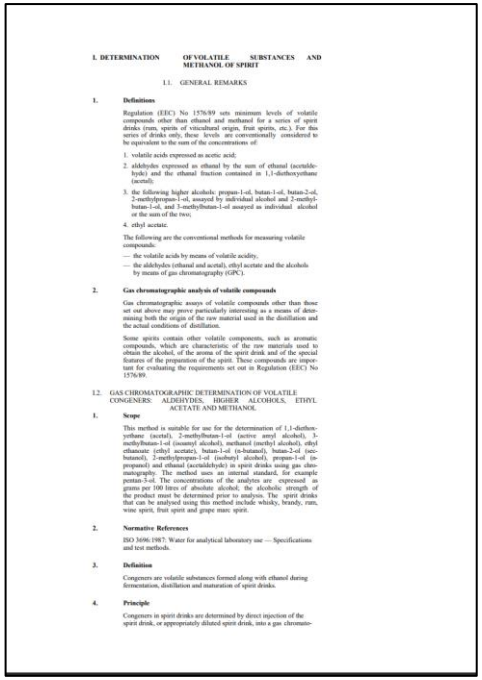
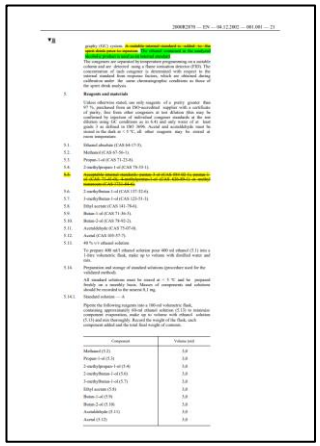
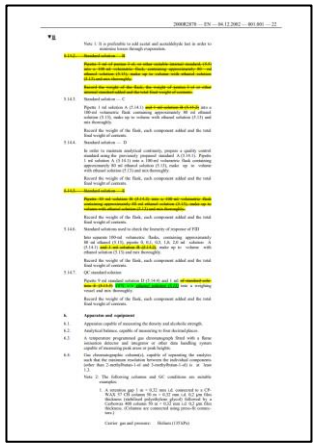
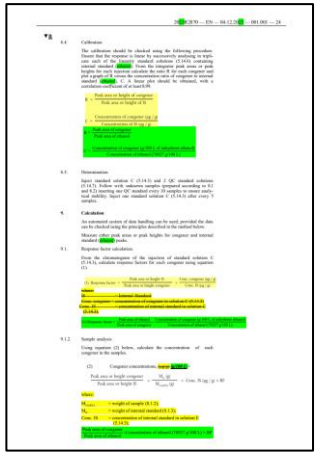
EC 2870/2000 Improvements

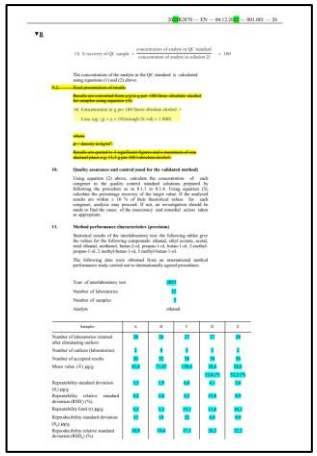
The use of the proposed method ensures high reliability of the data obtained, significantly reduces time, labor, material and financial costs. Analysis of volatile compounds in spirit drinks has never been so easy. Here you can read modified text of official method, which allows to carry out analysis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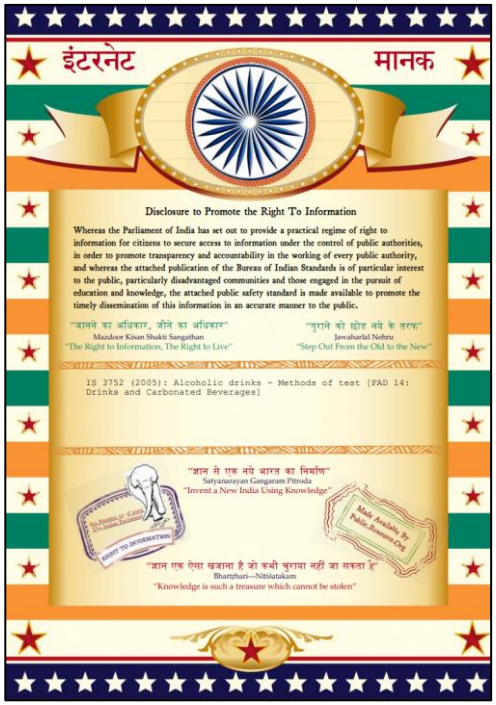

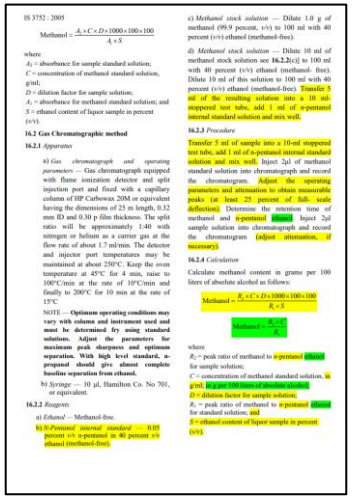

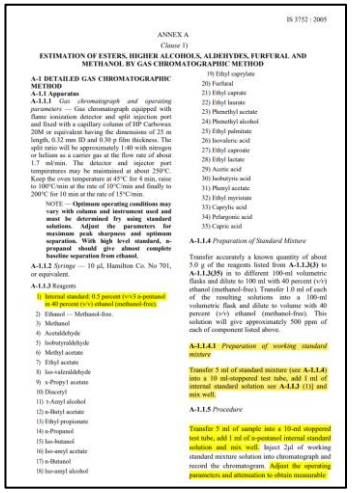

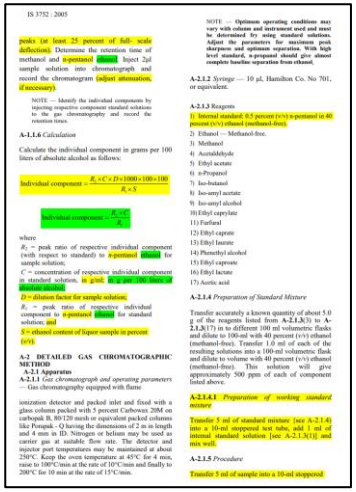

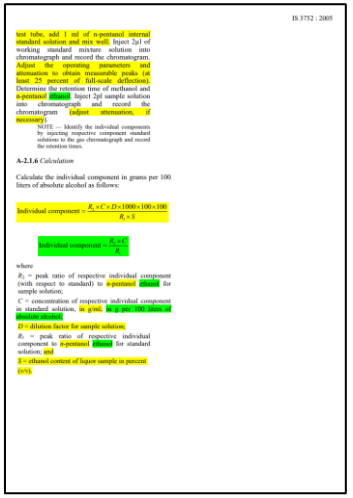

Determination of methanol in alcoholic beverages

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**
	Sambuca	**Egg**	**Herbal**	**Limon**	**Cherry**	**Raspberry**	**Sloe gin**		
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
	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:
siarhei.charapitsa@gmail.com, *lidia.sobolenko@gmail.com*, *yushkevitchdv@gmail.com*