



УЛУЧШЕНИЕ ГОСУДАРСТВЕННЫХ И МЕЖГОСУДАРСТВЕННЫХ СТАНДАРТОВ КОНТРОЛЯ КАЧЕСТВА И БЕЗОПАСНОСТИ АЛКОГОЛЬНОЙ ПРОДУКЦИИ

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Государственные и межгосударственные стандарты для определения летучих компонентов, в том числе метилового спирта, в алкогольной продукции



GB/T 11858-2009
GB/T 15038-2008
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Commission Regulation (EC) No. 2870/2000



AOAC Official Methods 972.10/11, 2005



Norma Mexicana NMX-V-005-NORMEX-2018



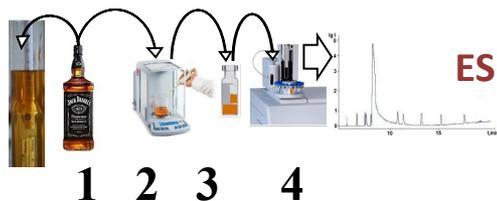
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ГОСТ 10749.6
ГОСТ 10749.13
ГОСТ 10749.14
ГОСТ Р 57893
GOST P 52363
GOST P 51999
GOST P 55878
GOST P 57893
СТБ ГОСТ Р 51698

Все перечисленные национальные стандарты гармонизированы с Регламентом (ЕС) 2870/2000 и используют традиционный метод внутреннего стандарта.

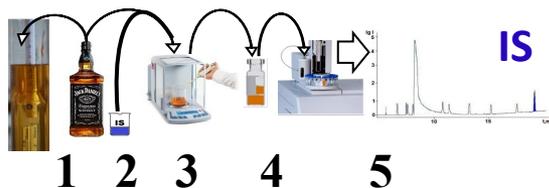
В государствах ЕАЭС одновременно действует более 20 стандартов по методу внешнего стандарта.

Отличия методов

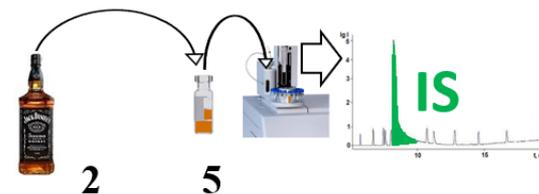
Сегодня: Метод внешнего стандарта.
Только страны ЕАЭС.



Сегодня: Метод внутреннего стандарта
Китай, Индия, ЕС, США, Мексика, etc.



Завтра: Инновационный метод
Китай, Индия, ЕС, США, Мексика, etc.



Определение массовой концентрации i -го летучего компонента $C(i)$ в l -ом исследуемом образце в размерности мг/л по методу внешнего стандарта проводится по формуле

$$\bar{C}_i(l) = RF_i \cdot \frac{1}{M} \sum_{j=1}^M A_{ij}(l),$$

где $A_{ij}(l)$ – величина отклика детектора на i -ый летучий компонент, полученная в результате j -го измерения l -го исследуемого образца, о. е.; M – число измерений l -го исследуемого образца, $M \geq 2$.

Значения относительных коэффициентов отклика детектора на исследуемый летучий компонент рассчитывают по следующей формуле:

$$RF_i = \frac{\sum_{k,j}^{M,N} C_i^{st}(k,j) \cdot A_i^{st}(k,j)}{\sum_{k,j}^{M,N} A_i^{st}(k,j)^2}$$

Для расчета концентрации компонента, выраженной в мг/л б.с., необходимо крепость (объемное содержание этанола) образца и разделить на её процентное содержание по объёму.

В соответствии с традиционным методом внутреннего стандарта концентрацию i -го компонента в пересчете на мг/кг определяют по следующей формуле:

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

Значения относительных коэффициентов отклика детектора на исследуемый летучий компонент по отношению к отклику на выбранный внутренний стандарт рассчитывают по следующей формуле:

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

Для расчета концентрации компонента, выраженной в мг/л б.с., необходимо измерить плотность образца и определить его крепость (объемное содержание этанола):

$$C_i \left(\frac{\text{мг}}{\text{л б.с.}} \right) = RRF_i^{IS} \cdot \frac{A_i}{A_{IS}} \cdot C_{IS}(\text{мг/кг}) \cdot \frac{\rho_{\text{образца}}(\text{кг/л}) \cdot 100\%}{\text{"Крепость" } (\%, \text{ABV})}$$

В соответствии с методом «Этанол как внутренний стандарт» концентрация i -го компонента в размерности мг/л безводного спирта (б.с.) определяют по следующей формуле:

$$C_i(\text{мг/л б.с.}) = RRF_i^{Eth} \cdot \frac{A_i}{A_{Eth}} \cdot \rho_{Eth}(\text{мг/л})$$

Значения относительных коэффициентов отклика детектора на исследуемый летучий компонент по отношению к отклику на этанол рассчитывают по следующей формуле:

$$RRF_i^{Eth} = \frac{C_i^{calibr}(\text{мг/л б.с.}) \cdot A_{Eth}^{calibr}}{\rho_{Eth}(\text{мг/л}) \cdot A_i^{calibr}}$$

1. Нет необходимости добавлять в образец какой-либо внутренний стандарт.

2. Этанол всегда присутствует в алкогольной продукции и его концентрация в мг/л б.с. всегда известна со 100% гарантией и равна плотности этанола

$$\rho_{Eth} = 789270 \text{ мг/л.}$$

Пропечатано

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Улучшения китайского стандарта GB/T 11858-2008

Использование предлагаемого метода обеспечивает высокую достоверность полученных данных, значительно сокращает временные, трудовые, материальные и финансовые затраты. Анализ летучих соединений в спиртных напитках еще никогда не был таким простым. Здесь вы можете ознакомиться с измененным текстом официальной методики, позволяющей проводить анализ алкогольных напитков по разработанной методике.

Места в текстовом документе, которые нужно удалить, выделены желтым цветом. Добавленные части текста выделены зеленым цветом.

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 averaging on the differences 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 chromatography quantity by internal standard method with the use of peak areas (or peak height). **Remove this sentence**

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-40m, 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 up gas flow rate at about 20 mL/min-30 mL/min.
Hydrogen Gas: Flow rate at 33 mL/min.
Air: Flow at 400 mL/min.
Temperature of Detector (T_d): 220°C.
Temperature of Sample Inlet (T_i): 220°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 (mol) [M(2) = 22];
V – Volume of sample absorbed, unit is milliliter (mL);
X_i – Total acetaldehyde content in a liter of 100% ethanol of the sample, unit is milligram per liter (mg/L);
E – 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 n-Butanol Solution (2%) Use as internal standard. Extract 2 mL n-butanol (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 at 0°C/min 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 acetaldehyde solution (prepared as in 5.4.1.3.2) and transfer into a 100 mL volumetric flask. Add 1 mL isobutanol solution (prepared as in 5.4.1.3.3) and transfer into the flask and then dilute the mixture with 40% ethanol solution to full. The concentration of acetaldehyde should both be 0.02%. **Remove the following sentence: The concentration of isobutanol should both be 0.02%. Make records of the retention time of acetaldehyde and the internal standard peak (isobutanol) 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 is according to experiment value (F value), at about **0.37**.

5.4.1.5.2 Determination of Sample Solution
Extract 10 mL of alcohol sample directly with a 10 mL volumetric flask and then add 0.5 mL n-butanol solution (prepared as in 5.4.1.3.3) as internal standard. Test samples in order the same conditions as the iodine test and then determine the positions of acetaldehyde and isobutanol according to the retention time. Determine the peak (or peak height) of the acetaldehyde and isobutanol according to the retention time. **Remove this sentence: Compute the difference between peak areas (or peak height) and calculate the proportion of acetaldehyde (or isobutanol) 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):
$$F = \frac{A_s \cdot A_i}{A_i \cdot A_s} \quad (6)$$

b) Acetaldehyde (or Acetal) content in the sample can be calculated with the following formula (7):
$$X_s = F \cdot \frac{A_s}{A_i} \cdot X_i \quad (7)$$

Acetaldehyde (or Acetal) content in a liter of 100% ethanol can be calculated with the following formula (8):
$$X_s = \frac{X_s \cdot E}{100} \quad (8)$$

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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 reagent (prepared as in 5.5.2.3.3) into an individual 100 mL, central flask respectively. Dilute each solution with 40% ethanol solution till each flask is full and mix evenly. These seven formaldehyde 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 tests. 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 colorimetric tube with stopper. Add 2.0 mL hydrochloric hydrobromic 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 next 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 colorimetric 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 n-Butanol Solution (2%) Use as internal standard. Extract 2 mL n-butanol (chromatographically pure), then dilute it with 40% ethanol solution till it reaches 100 mL volume.

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Total aldehyde (acetaldehyde) content in a liter of 100% ethanol can be calculated with the following formula (9):
$$X_s = X_i + X_i \cdot 0.37 \quad (9)$$

In the formula:
F – Relative calibration factor of acetaldehyde (or acetal);
A_i – Peak area (or peak height) of the internal standard (isobutanol) during the determination of standard sample value;
A_s – Peak area (or peak height) of acetal during the determination of standard sample value;
X_i – Concentration of acetaldehyde (or acetal) of standard sample solution (mg/L);
X_s – Concentration of acetaldehyde (or acetal) of sample solution (mg/L);
E – Actual alcohol content of the sample;
X_i – Acetaldehyde (or Acetal) content in sample (chromatographically pure) (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;
X_i – Internal standard (isobutanol) content, unit is milligram per liter (mg/L);
X_s – Acetaldehyde (or Acetal) content in a liter of 100% ethanol in the sample, unit is milligram per liter (mg/L);
E – Actual alcohol content of the sample;
X_s – 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);
X_s – 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 Ethanol 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 ethanol solution (prepared as in 5.7.3.3) instead.

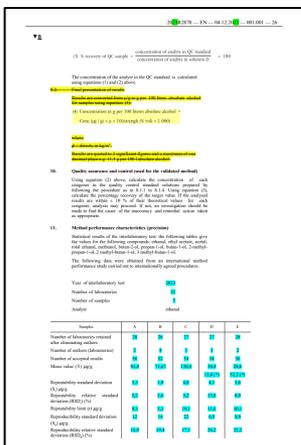
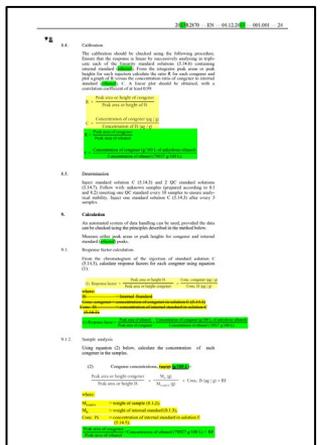
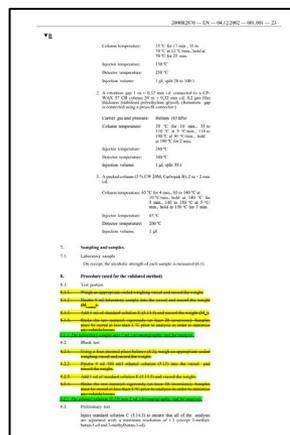
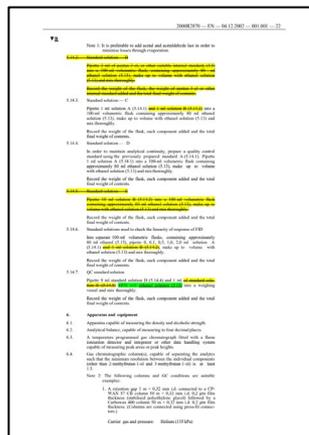
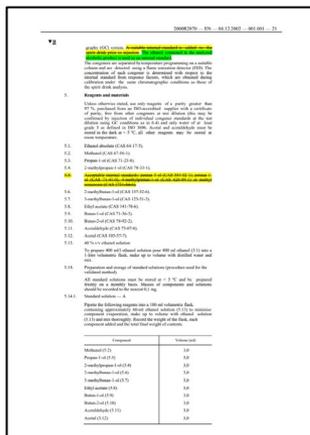
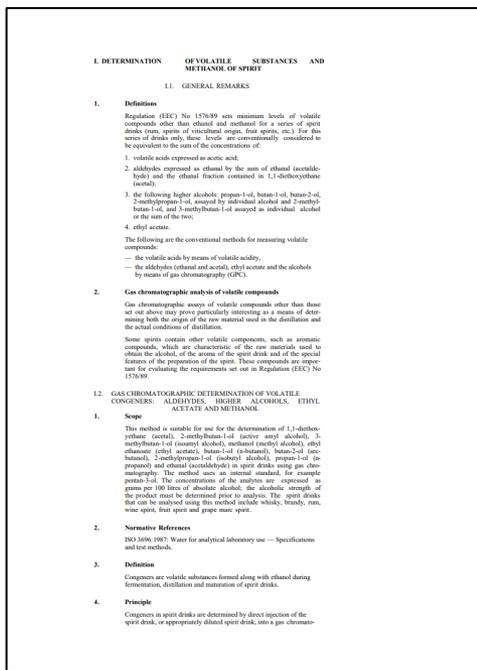
5.7.6 Result Calculation
Same as 5.4.1.6, determine total content of isobutanol and isobutyl ethanol.

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Улучшения европейского стандарта ЕС 2870/2000

Использование предлагаемого метода обеспечивает высокую достоверность полученных данных, значительно сокращает временные, трудовые, материальные и финансовые затраты. Анализ летучих соединений в спиртных напитках еще никогда не был таким простым. Здесь вы можете ознакомиться с измененным текстом официальной методики, позволяющей проводить анализ алкогольных напитков по разработанной методике.

Места в текстовом документе, которые нужно удалить, выделены **желтым цветом**. Добавленные части текста выделены **зеленым цветом**.



Определение метанола в спиртных напитках

Результаты для	 40 % ABV	 40 % ABV	 43 % ABV	 40 % ABV	 40 % ABV	 40 % ABV	 40 % ABV	 47 % ABV	 45 % ABV
	Ром	Виски	Бурбон	Зерн. нап.	Бренди	Граппа	Кальвадос	Джин	Сливовица
Внеш. станд, мг/л б.с.	46.6	130	81.9	108.9	275	328	702	2.7	10884
Инн. метод, мг/л б.с.	22.3	132	88.9	110.7	297	412	913	4.2	10603
Внутр. станд, мг/л б.с.	22.2	132	88.4	111.6	297	414	910	4.16	10546
Результаты для	 38 % ABV	 14.5 % ABV	 38 % ABV	 15 % ABV	 18 % ABV	 8.5 % ABV	 70 % ABV	 27.5 % ABV	 40 % ABV
	Цикудия	Саке	Текила	Вермут	Наливка	Глинтвейн	Нап. сп. кр.	Коктейль	Водка
Внеш. станд, мг/л б.с.	705	10.7	1259	10.8	57	8.87	9.6	41.0	25.4
Инн. метод, мг/л б.с.	761	18.1	1460	17.6	169.5	25.1	6.0	76.3	21.7
Внутр. станд, мг/л б.с.	755	18.2	1456	17.5	168	25.3	6.05	77.3	21.8
Результаты для	 38 % ABV	 17 % ABV	 35 % ABV	 25 % ABV	 16 % ABV	 16.5 % ABV	 35 % ABV		
	Ликёры								
	Самбука	Яичный	Травяной	Лимонный	Вишнёвый	Малиновый	Терновый		
Внеш. станд, мг/л б.с.	0.89	6.78	12.5	10.4	3.08	41.12	14.49		
Инн. метод, мг/л б.с.	2.3	9.81	19.6	29.4	9.82	126	20.7		
Внутр. станд, мг/л б.с.	2.32	9.75	19.5	29.1	9.77	127	20.5		



1 400

МИЛЛИОНОВ



1 400

МИЛЛИОНОВ



450

МИЛЛИОНОВ



333

МИЛЛИОНА

Республика Беларусь не должна отказаться от того, чтобы контроль качества и безопасности десятков миллионов литров спиртных напитков, производимых ежедневно по всему миру, выполнялся по белорусскому методу.

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