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

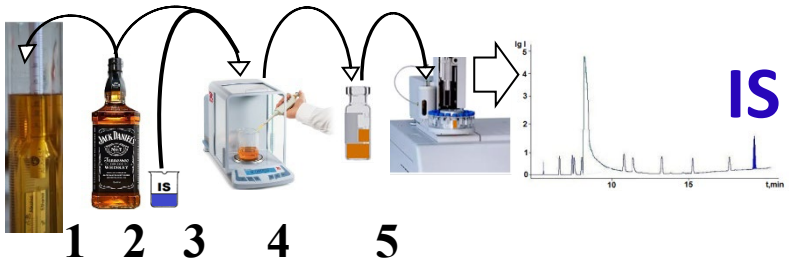
All listed national standards are harmonized with Regulation (EC) 2870/2000 and use the **traditional internal standard method**

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:

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

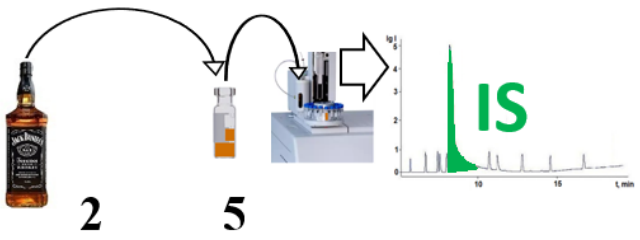
The values of the relative response coefficients of the detector to the investigated volatile component relative to the response to the selected internal standard are calculated using the following formula:

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

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.

Done

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4. *Journal of AOAC International***, 2019**, 102(2), 669-672. https://doi.org/10.5740/jaoacint.18-0258
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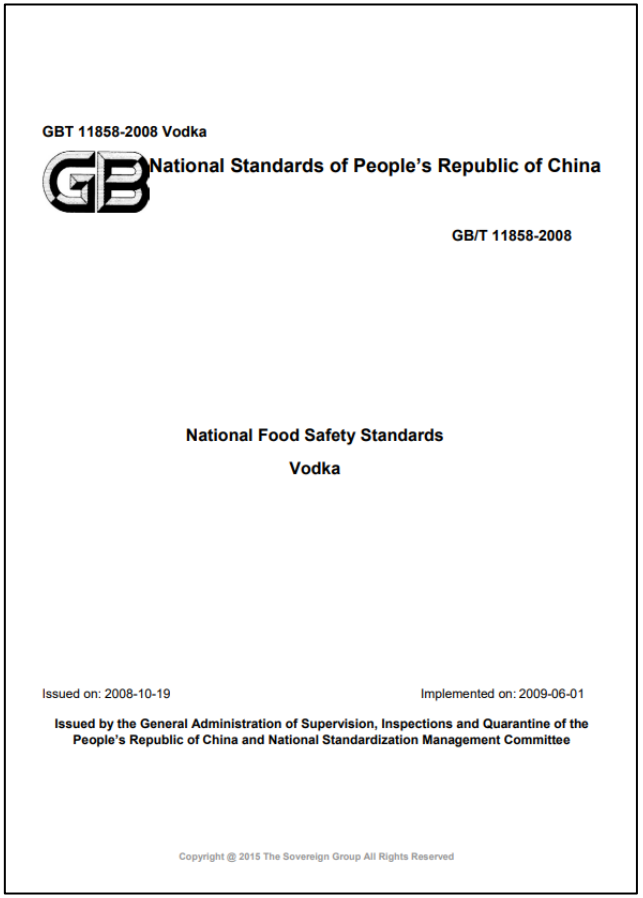
16. *Контроль качества продукции*, **2021**, 11, 34-38. https://ria-stk.ru/mos/adetail.php?ID=204383
17. *Заводская лаборатория*, **2022**, 88(5), 13-21. https://doi.org/10.26896/1028-6861-2022-88-5-13-21
18. Способ определения в этанолсодержащей жидкости газохроматографическим методом концентрации летучих примесей / *Eurasian Patent № 036994,* 2021, https://www.eapo.org/ru/patents/reestr/patent.php?id=36994
19. Improved document COMMISSION REGULATION EC2870/2000 https://elab.bsu.by/download.php?id=308
20. Improved document OIV-MA-BS-14, https://elab.bsu.by/download.php?id=312
21. Improved document OIV-MA-AS312-03A https://elab.bsu.by/download.php?id=317
22. Improved document OIV-MA-AS315-27 https://elab.bsu.by/download.php?id=316
23. Improved document Indian Standard 3572-2005, https://elab.bsu.by/download.php?id=315
24. Improved document Norma Mexicana NMX-V-005-NORMEX-2013 https://elab.bsu.by/download.php?id=311
25. Improved document National standards of People's Republic of China GB/T 15038 https://elab.bsu.by/download.php?id=309
26. Improved document National standards of People's Republic of China GB/T 11858 https://elab.bsu.by/download.php?id=307
27. Improved document AOAC Official Method 972.10 (USA), https://elab.bsu.by/download.php?id=306
28. Improved document AOAC Official Method 972.11 (USA), https://elab.bsu.by/download.php?id=305
29. Improved document ГОСТ 30536-2013 https://elab.bsu.by/download.php?id=314
30. Improved document СТБ ГОСТ Р 51698-2001 https://elab.bsu.by/download.php?id=313

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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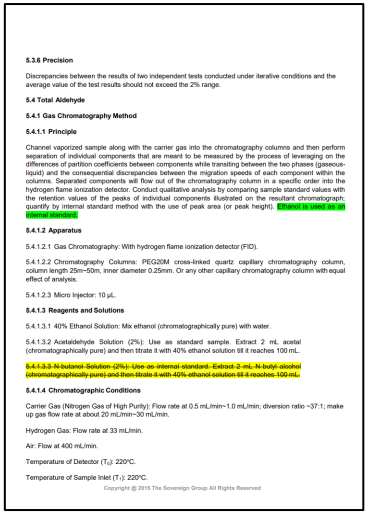

5.3.6 Precision

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 columns and then perform separation of individual components that are meant to be measured by the process of leveraging on the differences of partition coefficients between components while transiting between the two phases (gaseous-liquid) and the consequential discrepancies between the migration speeds of each component within the columns. 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 illustrated on the resultant chromatograph; quantify by internal standard method with the use of peak area (or peak height). Ethanol is used as an internal standard.

5.4.1.2 Apparatus

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

5.4.1.2.2 Chromatography Columns: PEG20M cross-linked quartz capillary chromatography column, column length 25m~50m, 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 titrate it with 40% ethanol solution till it reaches 100 mL.

5.4.1.3.3 N-butanol Solution (2%): Use as internal standard. Extract 2 mL N-butyl alcohol (chromatographically pure) and then titrate 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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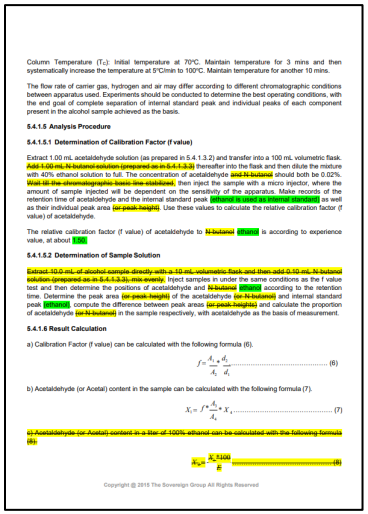

Column Temperature (T_C): Initial temperature at 70°C. Maintain temperature for 3 mins and then systematically increase the temperature at 5°C/min to 100°C. Maintain temperature for another 10 mins.

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 1.00 mL acetaldehyde solution (as prepared in 5.4.1.3.2) and transfer into a 100 mL volumetric flask. Add 1.00 mL N-butanol solution (prepared as in 5.4.1.3.3) thereafter into the flask and then dilute the mixture with 40% ethanol solution to full. The concentration of acetaldehyde and N-butanol should both be 0.02%. Wait till the chromatographic basic line stabilizes, then inject the sample with 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 and the internal standard peak (ethanol is used as internal standard) 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 N-butanol (ethanol) is according to experience value, at about 1.50.

5.4.1.5.2 Determination of Sample Solution

Extract 10.0 mL of alcohol sample directly with a 10 mL volumetric flask and then add 0.10 mL N-butanol solution (prepared as in 5.4.1.3.3), mix evenly. Inject samples in under the same conditions as the f value test and then determine the positions of acetaldehyde and N-butanol (ethanol) according to the retention time. Determine the peak area (or peak height) of the acetaldehyde (or N-butanol) and internal standard peak (ethanol), compute the difference between peak areas (or peak heights) and calculate the proportion of acetaldehyde (or N-butanol) 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).

$$f = \frac{A_{s,4} \cdot d_s}{A_s \cdot d_4} \quad \text{.....(6)}$$

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

$$X_1 = f * \frac{A_1}{A_s} * X_4 \quad \text{.....(7)}$$

c) Acetaldehyde (or Acetal) content in a liter of 100% ethanol can be calculated with the following formula (8):

$$X_{1,0} = \frac{X_1 \cdot 1000}{E} \quad \text{.....(8)}$$

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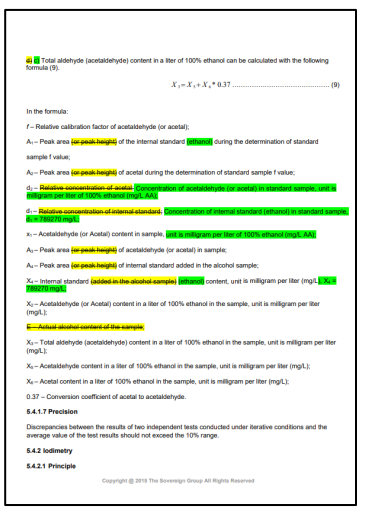

d) Total aldehyde (acetaldehyde) content in a liter of 100% ethanol can be calculated with the following formula (9).

$$X_3 = X_1 + X_4 * 0.37 \quad \text{.....(9)}$$

In the formula:

f – Relative calibration factor of acetaldehyde (or acetal);

A_1 – Peak area (or peak height) of the internal standard (ethanol) during the determination of standard sample f value;

A_s – Peak area (or peak height) of acetal during the determination of standard sample f value;

d_1 – Relative concentration of acetal, Concentration of acetaldehyde (or acetal) in standard sample, unit is milligram per liter of 100% ethanol (mg/L AAL)

d_4 – Relative concentration of internal standard, Concentration of internal standard (ethanol) in standard sample, d_4 = 1ml/100 ml=1%

x_1 – Acetaldehyde (or Acetal) content in sample, unit is milligram per liter of 100% ethanol (mg/L AAL)

A_4 – Peak area (or peak height) of acetaldehyde (or acetal) in sample;

$A_{s,4}$ – Peak area (or peak height) of internal standard added in the alcohol sample;

$X_{4,0}$ – Internal standard (added in the alcohol sample) (ethanol) content, unit is milligram per liter (mg/L, $X_{4,0}$ = 1ml/100 ml=1%);

$X_{1,0}$ – 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_3 – Total aldehyde (acetaldehyde) content in a liter of 100% ethanol in the sample, unit is milligram per liter (mg/L);

X_4 – Acetaldehyde content in a liter of 100% ethanol in the sample, unit is milligram per liter (mg/L);

X_1 – 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 Iodimetry

5.4.2.1 Principle

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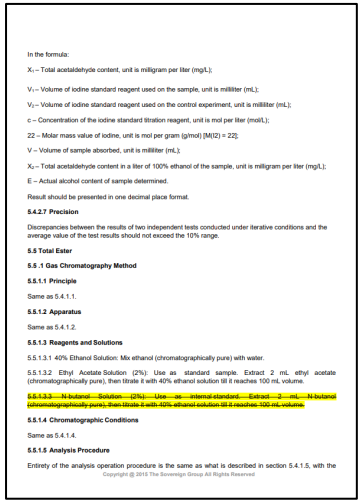

In the formula:

X_1 – Total acetaldehyde content, unit is milligram per liter (mg/L);

V_1 – Volume of iodine standard reagent used on the sample, unit is milliliter (mL);

V_2 – 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 (g/mol) [M(I2) = 22];

V – Volume of sample absorbed, unit is milliliter (mL);

X_0 – 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.

5.4.2.7 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.

5.5 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 titrate 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 titrate 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

Entirety of the analysis operation procedure is the same as what is described in section 5.4.1.5, with the

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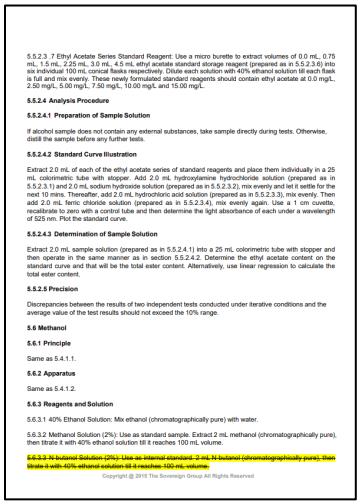

5.5.2.3 .7 Ethyl Acetate Series Standard Reagent: Use a micro burette to extract volumes of 0.0 mL, 0.75 mL, 1.5 mL, 2.25 mL, 3.0 mL, 4.5 mL ethyl acetate standard storage reagent (prepared as in 5.5.2.3.6) into six individual 100 mL conical flasks respectively. Dilute each solution with 40% ethanol solution till each flask is full and mix evenly. These newly 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 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 hydroxylamine hydrochloride 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. Thereafter, 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, recalibrate 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 Solution

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 titrate 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 titrate it with 40% ethanol solution till it reaches 100 mL volume.

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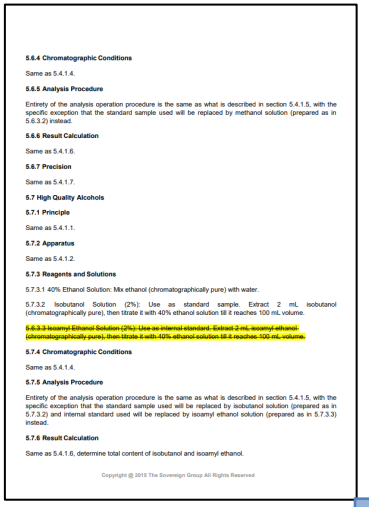

5.6.4 Chromatographic Conditions

Same as 5.4.1.4.

5.6.5 Analysis Procedure

Entirety 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 methanol 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 titrate it with 40% ethanol solution till it reaches 100 mL volume.

5.7.3.3 Isoamyl Ethanol Solution (2%): Use as internal standard. Extract 2 mL isoamyl ethanol (chromatographically pure), then titrate 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

Entirety 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 isoamyl 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 isoamyl ethanol.

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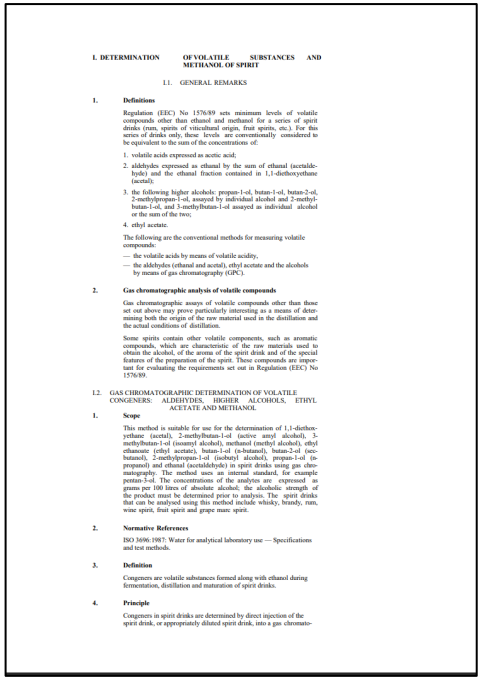
EC 2870/2000 Improvements

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.



I. DETERMINATION OF VOLATILE SUBSTANCES AND METHANOL OF SPIRIT

I.1. GENERAL REMARKS

1. **Definitions**

Regulation (EEC) No 1576/89 sets minimum levels of volatile compounds other than ethanol and methanol for a series of spirit drinks (rum, spirits of viticultural 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 ethanal by the sum of ethanal (acetaldehyde) and the ethanal fraction contained in 1,1-diethoxyethane (acetal);
3. the following higher alcohols: propan-1-ol, butan-1-ol, butan-2-ol, 2-methylpropan-1-ol, assayed by individual alcohol and 2-methylbutan-1-ol, and 3-methylbutan-1-ol assayed as individual alcohol or the sum of the two;
4. ethyl acetate.

The following are the conventional methods for measuring volatile compounds:

— the volatile acids by means of volatile acidity,
— the aldehydes (ethanal and acetal), ethyl acetate and the alcohols by means of gas chromatography (GPC).

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

Gas chromatographic assays of volatile compounds other than those set out above may prove particularly interesting as a means of determining both the origin of the raw 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. These compounds are important for evaluating the requirements set out in Regulation (EEC) No 1576/89.

I.2. GAS CHROMATOGRAPHIC DETERMINATION OF VOLATILE CONGENERS: ALDEHYDES, HIGHER ALCOHOLS, ETHYL ACETATE AND METHANOL

1. **Scope**

This method is suitable for use for the determination of 1,1-diethoxyethane (acetal), 2-methylbutan-1-ol (active amyl alcohol), 3-methylbutan-1-ol (isoamyl alcohol), methanol (methyl alcohol), ethyl ethanoate [ethyl acetate), butan-1-ol (n-butanol), butan-2-ol (sec-butanol), 2-methylpropan-1-ol (isobutyl alcohol), propan-1-ol (n-propanol) and ethanal (acetaldehyde) in spirit drinks using gas chromatography. The method uses an internal standard, for example pentan-3-ol. The concentrations of the analytes are expressed as grams per 100 litres of absolute alcohol; the alcoholic strength of the product must be determined prior to analysis. The spirit drinks that can be analysed using this method include whisky, brandy, rum, wine spirit, fruit spirit and grape marc spirit.

2. **Normative References**

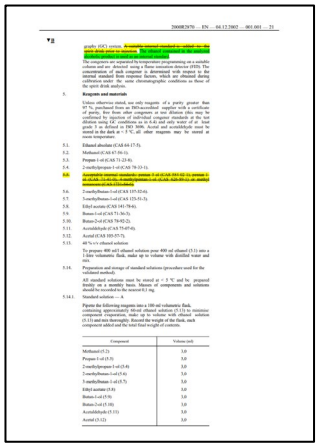
ISO 3696:1987: Water for analytical laboratory use — Specifications and test methods.

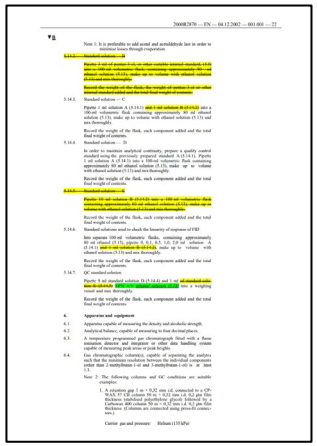
3. **Definition**

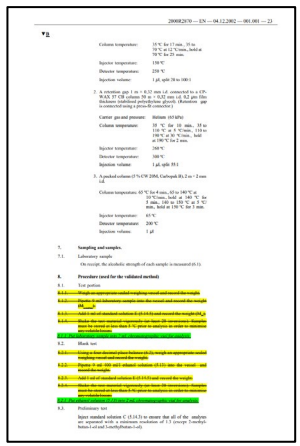
Congeners are volatile substances formed along with ethanol during fermentation, distillation and maturation of spirit drinks.

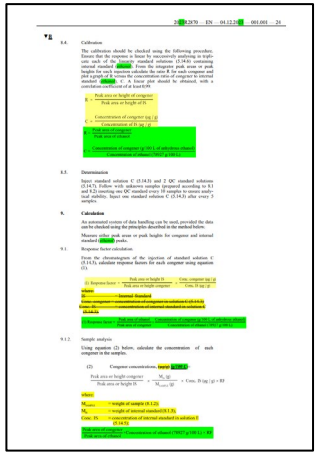
4. **Principle**

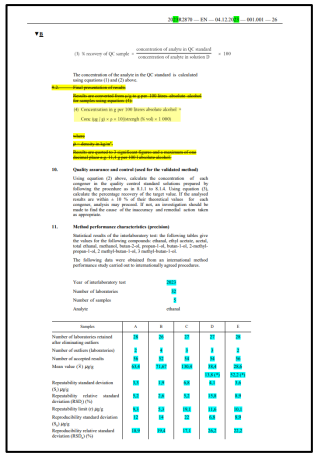
Congeners in spirit drinks are determined by direct injection of the spirit drink, or appropriately diluted spirit drink, into a gas chromato-





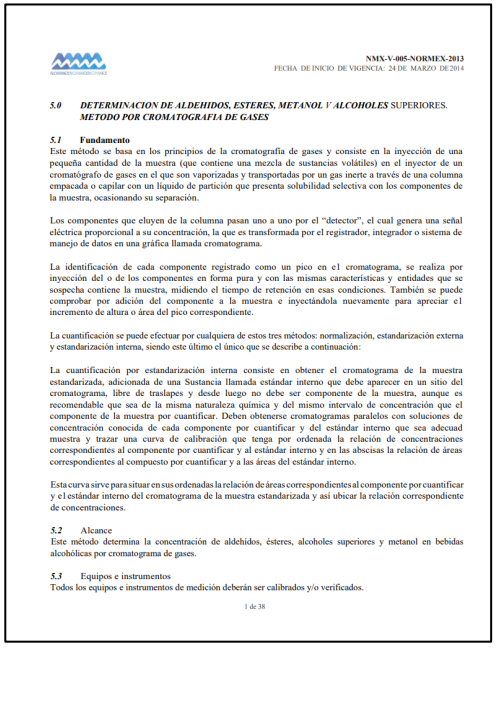






Norma Mexicana NMX-V-005-NORMEX-2018 Improvements

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
FECHA DE INICIO DE VIGENCIA: 24 DE MARZO DE 2014

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 traslapes 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 adecuad 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.

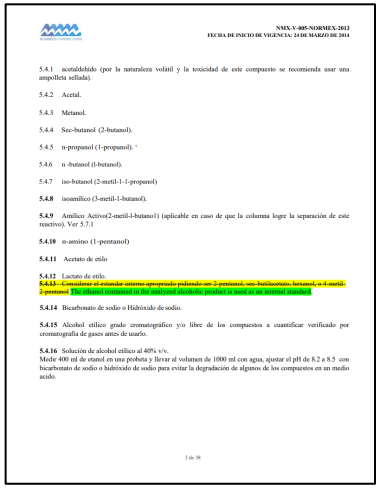
5.2 Alcance

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

5.3 Equipos e instrumentos

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

1 de 38



NMX-V-005-NORMEX-2013
FECHA DE INICIO DE VIGENCIA: 24 DE MARZO DE 2014

5.4.1 acetaldehído (por la naturaleza volátil y la toxicidad de este compuesto se recomienda usar una ampolleta sellada).

5.4.2 Acetal.

5.4.3 Metanol.

5.4.4 Sec-butanol (2-butanol).

5.4.5 n-propanol (1-propanol).

5.4.6 n -butanol (l-butanol).

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

5.4.8 isoamílico (3-metil-1-butanol).

5.4.9 Amílico Activo(2-metil-l-butanol) (aplicable en caso de que la columna logre la separación de este reactivo). Ver 5.7.1

5.4.10 n-amino (1-pentanol)

5.4.11 Acetato de etilo

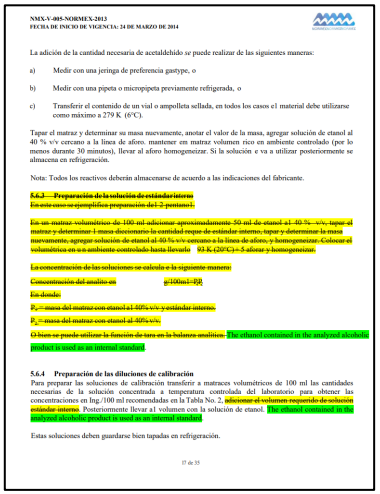
5.4.12 Lactato de etilo.
~~**5.4.13 Considere el estándar interno apropiado pidiendo ser 2-pentanol, sec-butilacetato, hexanol, o 4-metil-2-pentanol.**~~ The ethanol contained in the analyzed alcoholic product is used as an internal standard.

5.4.14 Bicarbonato de sodio o Hidróxido de sodio.

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

5.4.16 Solución de alcohol etílico al 40% v/v.
Medir 400 ml de etanol en una probeta y llevar al volumen de 1000 ml con agua, ajustar el pH de 8.2 a 8.5 con bicarbonato de sodio o hidróxido de sodio para evitar la degradación de algunos de los compuestos en un medio acido.

3 de 38



NMX-V-005-NORMEX-2013
FECHA DE INICIO DE VIGENCIA: 24 DE MARZO DE 2014

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

a) Medir con una jeringa de preferencia gastype, o

b) Medir con una pipeta o micropipeta previamente refrigerada, o

c) Transferir el contenido de un vial o ampolleta sellada, en todos los casos el material debe utilizarse como máximo a 279 K (6°C).

Tapar el matraz y determinar su masa nuevamente, anotar el valor de la masa, agregar solución de etanol al 40 % v/v cercano a la línea de aforo. mantener en matraz volumen rico en ambiente controlado (por lo menos durante 30 minutos), llevar al aforo homogeneizar. Si la solución e va a utilizar posteriormente se almacena en refrigeración.

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

~~**5.6.3 Preparación de la solución de estándar interno**~~
~~**En este caso se ejemplifica preparación del 2 pentanol.**~~

~~En un matraz volumétrico de 100 ml adicionar aproximadamente 50 ml de etanol al 40 % v/v, tapar el matraz y determinar l masa diexinarlo la cantidad reque de estándar interno, tapar y determinar la masa nuevamente, agregar solución de etanol al 40 % v/v cercano a la línea de aforo, y homogeneizar. Colocar el volumétrico en o a ambiente controlado hasta llevarlo 93 K (20°C) ± 5 aforar y homogeneizar.~~

~~La concentración de las soluciones se calcula e la siguiente manera:~~

~~Concentración del analito en g/100ml= ρ/100ml= ρ/l~~

~~En donde:~~

~~P_a = masa del matraz con etanol a l 40% v/v, y estándar interno.~~

~~P_b = masa del matraz con etanol al 40% v/v.~~

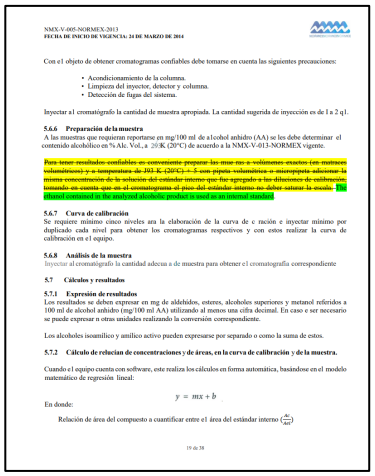
~~O bien se puede utilizar la función de tara en la balanza analítica.~~ The ethanol contained in the analyzed alcoholic product is used as an internal standard.

5.6.4 Preparación de las diluciones de calibración

Para preparar las soluciones de calibración transferir a matraces volumétricos de 100 ml las cantidades necesarias de la solución concentrada a temperatura controlada del laboratorio para obtener las concentraciones en mg /100 ml recomendadas en la Tabla No. 2, ~~aforar al volumen requerido de solución estándar interno.~~ Posteriormente llevar a l volumen con la solución de etanol. The ethanol contained in the analyzed alcoholic product is used as an internal standard.

Estas soluciones deben guardarse bien tapadas en refrigeración.

17 de 35



NMX-V-005-NORMEX-2013
FECHA DE INICIO DE VIGENCIA: 24 DE MARZO DE 2014

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

- Acondicionamiento de la columna.
- Limpieza del inyector, detector y columna.
- Detección de fugas del sistema.

Inyectar a l cromatógrafo la cantidad de muestra apropiada. La cantidad sugerida de inyección es de 1 a 2 q l.

5.6.6 Preparación de la muestra
A las muestras que requieran reportarse en mg/100 ml de alcohol anhidro (AA) se les debe determinar el contenido alcohólico en % Alc. Vol., a 293K (20°C) de acuerdo a la NMX-V-013-NORMEX vigente.

~~Para tener resultados confiables es conveniente preparar las mue nn a volúmenes exactos (en matraces volumétricos) y a temperatura de 293 K (20°C) + 5 con pipeta volumétrica o micropipeta aforar la misma concentración de la solución del estándar interno que fue agregado a las diluciones de calibración; tomando en cuenta que en el cromatograma el pico del estándar interno no deber saturar la escala.~~ The ethanol contained in the analyzed alcoholic product is used as an internal standard.

5.6.7 Curva de calibración
Se requiere mínimo cinco niveles ara la elaboración de la curva de c nción e inyectar 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 adecua a de muestra para obtener el cromatografía correspondiente

5.7 Cálculos y resultados

5.7.1 Expresión de resultados
Los resultados se deben expresar en mg de aldehídos, esteres, alcoholes superiores y metanol referidos a 100 ml de alcohol anhidro (mg/100 ml AA) utilizando al monos una cifra decimal. En caso e ser necesario se puede expresar o otras unidades realizando la conversión correspondiente.

Los alcoholes isoamílico y amílico activo pueden expresarse por separado o como la suma de estos.

5.7.2 Cálculo de relacian de concentraciones y de áreas, en la curva de calibración y de la muestra.

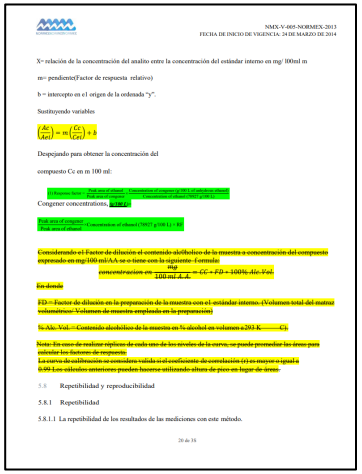
Cuando el equipo cuenta con software, este realiza los cálculos en forma automática, basándose en el modelo matemático de regresión lineal:

$$y = mx + b$$

En donde:

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

19 de 38



NMX-V-005-NORMEX-2013
FECHA DE INICIO DE VIGENCIA: 24 DE MARZO DE 2014

X= relación de la concentración del analito entre la concentración del estándar interno en mg/ 100ml m

m= pendiente(Factor de respuesta relativo)

b = intercepto en el origen de la ordenada "y".

Sustituyendo variables

$$\left(\frac{Ac}{Aei}\right) = m\left(\frac{Cc}{Cei}\right) + b$$

Despejando para obtener la concentración del

compuesto Cc en m 100 ml:

$$Cc = \left[\left(\frac{Ac}{Aei}\right) - b\right]\left(\frac{Cei}{m}\right)$$

Congener concentraties, ~~g/ml~~ g/l

~~$$\text{Peak area of congener} = \text{Concentration of congener (g/100 ml of absolute ethanol)} \times \text{Peak area of ethanol}$$~~

~~$$\text{Peak area of congener} = \text{Concentration of congener (g/100 ml of absolute ethanol)} \times \text{Peak area of ethanol}$$~~

~~$$\text{Peak area of congener} = \text{Concentration of ethanol (%ABV)} \times \text{Peak area of ethanol}$$~~

~~Considerando el Factor de dilución el contenido alcohólico de la muestra a concentración del compuesto expresado en mg/100 ml/AA se o tiene con la siguiente formula:~~

~~$$\frac{\text{concentracion en } \frac{mg}{100\ ml\ A.A.}}{} = CC * FD * 100\% \ Alc.\ Vol.$$~~

~~En donde~~

~~FD = Factor de dilución en la preparación de la muestra con el estándar interno. (Volumen total del matraz volumétrico/ Volumen de muestra empleada en la preparación)~~

~~% Alc. Vol. = Contenido alcohólico de la muestra en % alcohol en volumen a 293 K (20 °C).~~

~~Nota: En caso de realizar réplicas de cada uno de los niveles de la curva, se puede promediar las áreas para calcular los factores de respuesta~~
~~La curva de calibración se considera valida si el coeficiente de correlación (r) es mayor o igual a 0.99. Los cálculos anteriores pueden hacerse utilizando altura de pico en lugar de área.~~

5.8 Repetibilidad y reproducibilidad

5.8.1 Repetibilidad

5.8.1.1 La repetibilidad de los resultados de las mediciones con este método.

20 de 35

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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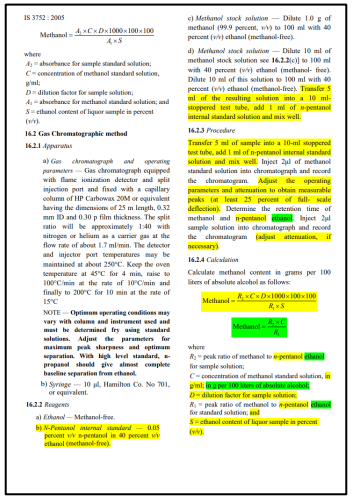

IS 3752 : 2005

$$\text{Methanol} = \frac{A_2 \times C \times D \times 1000 \times 100 \times 100}{A_1 \times S}$$

where
A_2 = absorbance for sample standard solution;
C = concentration of methanol standard solution, g/ml;
D = dilution factor for sample solution;
A_1 = 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 injection port and fixed with a capillary column of HP Carbowax 20M or equivalent having the dimensions of 25 m length, 0.32 mm ID and 0.30 µ 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

NOTE — **Optimum operating conditions may vary with column and instrument used and must be determined fry using standard solutions. Adjust the parameters for maximum peak sharpness and optimum separation. With high level standard, n-propanol should give almost complete baseline separation from ethanol.**

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

16.2.2 *Reagents*

a) *Ethanol* — Methanol-free.

b) *N-Pentanol internal standard* — 0.05 percent v/v n-pentanol in 40 percent v/v ethanol (methanol-free).

c) *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).

d) *Methanol stock solution* — Dilute 10 ml of methanol stock solution see **16.2.2**(c)] 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 5 ml of the resulting solution into a 10 ml-stoppered test tube, add 1 ml of n-pentanol internal standard solution and mix well.

16.2.3 *Procedure*

Transfer 5 ml of sample into a 10-ml stoppered test tube, add 1 ml of n-pentanol internal standard solution and mix well. Inject 2µl of methanol standard solution into chromatograph and record the chromatogram. Adjust the operating parameters and attenuation to obtain measurable peaks (at least 25 percent of full- scale deflection). Determine the retention time of methanol and n-pentanol (Hgnd. Inject 2µl sample solution into chromatograph and record the chromatogram (adjust attenuation, if necessary).

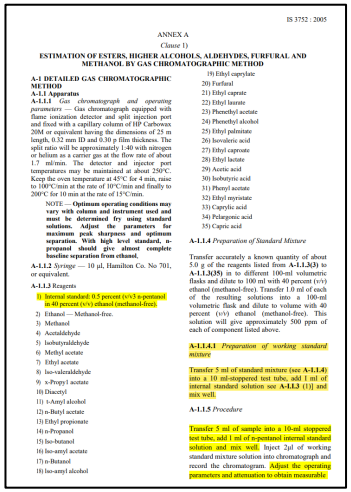
16.2.4 *Calculation*

Calculate methanol content in grams per 100 liters of absolute alcohol as follows:

$$\text{Methanol} = \frac{R_2 \times C \times D \times 1000 \times 100 \times 100}{R_1 \times S}$$

$$\text{Methanol} = \frac{R_2 \times 5}{R_1}$$

where
R_2 = peak ratio of methanol to n-pentanol ethanol for sample solution;
C = concentration of methanol standard solution, in g/ml, in g per 100 liters of absolute alcohol;
D = dilution factor for sample solution;
R_1 = peak ratio of methanol to n-pentanol ethanol for standard solution; and
S = ethanol content of liquor sample in percent (v/v).


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 injection port and fixed with a capillary column of HP Carbowax 20M or equivalent having the dimensions of 25 m length, 0.32 mm ID and 0.30 µ 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.

NOTE — **Optimum operating conditions may vary with column and instrument used and must be determined fry using standard solutions. Adjust the parameters for maximum peak sharpness and optimum separation. With high level standard, n-propanol should give almost complete baseline separation from ethanol.**

A-1.1.2 *Syringe* — 10 µl, Hamilton Co. No 701, or equivalent.

A-1.1.3 Reagents

1) Internal standard: 0.5 percent (v/v) n-pentanol in 40 percent (v/v) ethanol (methanol-free).
2) Ethanol — Methanol-free.
3) Methanol
4) Acetaldehyde
5) Isobutyraldehyde
6) Methyl acetate
7) Ethyl acetate
8) Iso-valeraldehyde
9) n-Propyl acetate
10) Diacetyl
11) t-Amyl alcohol
12) n-Butyl acetate
13) Ethyl propionate
14) n-Propanol
15) Iso-butanol
16) Iso-amyl acetate
17) n-Butanol
18) Iso-amyl alcohol
19) Ethyl caprylate
20) Furfural
21) Ethyl caprate
22) Ethyl laurate
23) Phenethyl acetate
24) Phenethyl alcohol
25) Ethyl palmitate
26) Isovaleric acid
27) Ethyl caproate
28) Ethyl lactate
29) Acetic acid
30) Isobutyric acid
31) Phenyl acetate
32) Ethyl myristate
33) Caprylic acid
34) Pelargonic acid
35) Capric acid

A-1.1.4 *Preparation of Standard Mixture*

Transfer accurately a known quantity of about 5.0 g of the reagents listed from **A-1.1.3**(3) to **A-1.1.3**(35) 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-1.1.4.1 *Preparation of working standard mixture*

Transfer 5 ml of standard mixture (see **A-1.1.4**) into a 10 ml-stoppered test tube, add 1 ml of internal standard solution see **A-1.1.3** (1)] and mix well.

A-1.1.5 *Procedure*

Transfer 5 ml of sample into a 10-ml stoppered test tube, add 1 ml of n-pentanol internal standard solution and mix well. Inject 2µl of working standard mixture solution into chromatograph and record the chromatogram. Adjust the operating parameters and attenuation to obtain measurable peaks (at least 25 percent of full-scale deflection). Determine the retention time of methanol and n-pentanol ethanol. Inject 2µl sample solution into chromatograph and record the chromatogram (adjust attenuation, if necessary).

NOTE — Identify the individual components by injecting respective component standard solutions to the gas chromatograph and record the retention times.

A-1.1.6 *Calculation*

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

$$\text{Individual component} = \frac{R_2 \times C \times D \times 1000 \times 100 \times 100}{R_1 \times S}$$

$$\text{Individual component} = \frac{R_2 \times 5}{R_1}$$

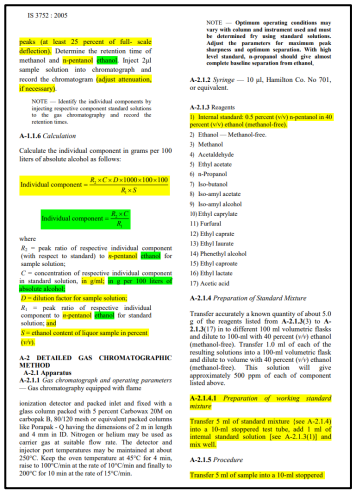
where
R_2 = peak ratio of respective individual component (with respect to standard) to n-pentanol ethanol for sample solution;
C = concentration of respective individual component in standard solution, in g/ml, in g per 100 liters of absolute alcohol;
D = dilution factor for sample solution;
R_1 = peak ratio of respective individual component to n-pentanol ethanol for standard solution; and
S = ethanol content of liquor sample in percent (v/v).

A-2 DETAILED GAS CHROMATOGRAPHIC METHOD

A-2.1 Apparatus

A-2.1.1 *Gas chromatograph and operating parameters* — Gas chromatography equipped with flame

ionization detector and packed inlet and fixed with a glass column packed with 5 percent Carbowax 20M on carbopak B, 80/120 mesh or equivalent packed columns like Porapak - Q having the dimensions of 2 m in length and 4 mm in ID. Nitrogen or helium may be used as carrier gas at suitable flow rate. 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.


IS 3752 : 2005

peaks (at least 25 percent of full- scale deflection). Determine the retention time of methanol and n-pentanol ethanol. Inject 2µl sample solution into chromatograph and record the chromatogram (adjust attenuation, if necessary).

NOTE — Identify the individual components by injecting respective component standard solutions to the gas chromatograph and record the retention times.

A-1.1.6 Calculation

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

$$\text{Individual component} = \frac{R_2 \times C \times D \times 1000 \times 100 \times 100}{R_1 \times S}$$

$$\text{Individual component} = \frac{R_2 \times 5}{R_1}$$

where
R_2 = peak ratio of respective individual component (with respect to standard) to n-pentanol ethanol for sample solution;
C = concentration of respective individual component in standard solution, in g/ml, in g per 100 liters of absolute alcohol;
D = dilution factor for sample solution;
R_1 = peak ratio of respective individual component to n-pentanol ethanol for standard solution; and
S = ethanol content of liquor sample in percent (v/v).

NOTE — Optimum operating conditions may vary with column and instrument used and must be determined fry using standard solutions. Adjust the parameters for maximum peak sharpness and optimum separation. With high level standard, n-propanol should give almost complete baseline separation from ethanol.

A-2.1.2 Syringe — 10 µl, Hamilton Co. No 701, or equivalent.

A-2.1.3 Reagents

1) Internal standard: 0.5 percent (v/v) n-pentanol in 40 percent (v/v) ethanol (methanol-free).
2) Ethanol — Methanol-free.
3) Methanol
4) Acetaldehyde
5) Ethyl acetate
6) n-Propanol
7) Iso-butanol
8) Iso-amyl acetate
9) Iso-amyl alcohol
10) Ethyl caprylate
11) Furfural
12) Ethyl caprate
13) Ethyl laurate
14) Phenethyl alcohol
15) Ethyl caproate
16) Ethyl lactate
17) Acetic acid

A-2.1.4 Preparation of Standard Mixture

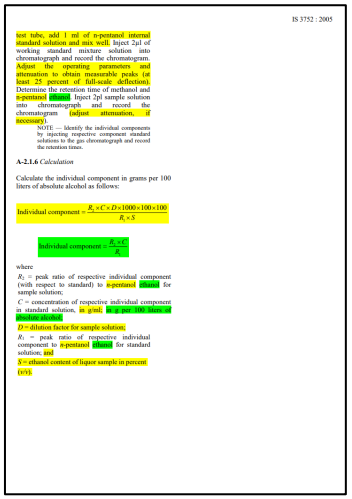
Transfer accurately a known quantity of about 5.0 g of the reagents listed from A-2.1.3(3) to A-2.1.3(17) in to different 100 ml volumetric flasks and dilute to 100-ml with 40 percent (v/v) ethanol (methanol-free). Transfer 1.0 ml of each of the resulting solutions into a 100-ml volumetric flask and 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-2.1.4.1 Preparation of working standard mixture

Transfer 5 ml of standard mixture [see A-2.1.4] into a 10-ml stoppered test tube, add 1 ml of internal standard solution [see A-2.1.3(1)] and mix well.

A-2.1.5 Procedure

Transfer 5 ml of sample into a 10-ml stoppered


IS 3752 : 2005

test tube, add 1 ml of n-pentanol internal standard solution and mix well. Inject 2µl of working standard mixture solution into chromatograph and record the chromatogram. Adjust the operating parameters and attenuation to obtain measurable peaks (at least 25 percent of full-scale deflection). Determine the retention time of methanol and n-pentanol ethanol. Inject 2µl sample solution into chromatograph and record the chromatogram (adjust attenuation, if necessary).

NOTE — Identify the individual components by injecting respective component standard solutions to the gas chromatograph and record the retention times.

A-2.1.6 Calculation

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

$$\text{Individual component} = \frac{R_2 \times C \times D \times 1000 \times 100 \times 100}{R_1 \times S}$$

$$\text{Individual component} = \frac{R_2 \times 5}{R_1}$$

where
R_2 = peak ratio of respective individual component (with respect to standard) to n-pentanol ethanol for sample solution;
C = concentration of respective individual component in standard solution, in g/ml, in g per 100 liters of absolute alcohol;
D = dilution factor for sample solution;
R_1 = peak ratio of respective individual component to n-pentanol ethanol for standard solution; and
S = ethanol content of liquor sample in percent (v/v).

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		
	Liqueurs								
	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		
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		
Δ, %	**0.8**	**0.7**	**0.4**	**0.8**	**0.5**	**-1.1**	**0.5**		

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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