

BELIZE NATIONAL STANDARD

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BELIZE NATIONAL STANDARD SPECIFICATION FOR RUM

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**BELIZE NATIONAL STANDARD
SPECIFICATION FOR RUM**

Committee Representation

The preparation of this standard for the Standards Advisory Council established under the Standards Act of 1992, was carried out under the supervision of the Bureau's Technical Committee for Food and Food Related Products, which at the time comprised the following members:

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0.0 FOREWORD

- 0.1** This standard has been prepared by the Caricom Regional Organization for Standards and Quality (CROSQ) with the assistance of the West Indies Rum and Spirits Producers Association (WIRSPA) for use by manufacturers, distillers, bottlers, exporters and importers of rum to ensure that Caribbean consumers get a product of good quality, and also to protect Caricom rum producers in the overseas market.
- 0.2** This standard was adopted from the Caribbean Community Standard Specification for Rum CCS 0025, approved by the Caribbean Community Council of Ministers for implementation as a regional mandatory standard with effect from May 26, 2003.
- 0.3** Rum conforming to the requirements of this standard, which is manufactured under an approved system of quality assurance is eligible to be licensed to use the CARICOM Standard Mark, which is administered by the **CROSQ**.

1.0 SCOPE

- 1.1** This standard provides a definition for the commodity designated as rum and prescribes acceptable methods for sampling and analysis of rum.

2.0 DEFINITIONS

For the purpose of this standard, the following definitions shall apply:

- 2.1 Age:** Means the length of time the rum has been placed in a wooden vat or barrel for continuous maturation.
- 2.2 Alcohol:** The term "alcohol", when used without an adjective, means ethyl alcohol (C₂H₅OH) and includes all concentrations of this substance in water.
- 2.3 Alcohol, methyl (methanol):** The simplest of all alcohols, having the chemical formula CH₃OH.
- 2.4 Alcoholic fermentation:** The process by which fermentable sugars are converted into alcohol by the action of enzymes using microorganisms.
- 2.5 Caramel:** Colouring matter made by heating cane or other sugars under controlled conditions. It is a wholesome colouring matter widely used in the liquor and beverage industry.

- 2.6 Distillation:** The process of purifying a liquid by vaporization followed by condensation.
- 2.7 Ethyl Carbamate:** Also known as carbonic acid ethyl ester, urethane, or ethyl urethane.
- 2.8 Fermentation alcohol:** Alcohol produced by fermentation process from raw materials containing monosaccharides, disaccharides, and polysaccharides after hydrolysis.
- 2.9 Spirit -** As ordinarily used, this word means any distilled liquor (such as brandy, whiskey, rum or gin). It is also employed as a synonym for unflavoured alcohol of any strength.
- 2.10 Spirit caramel:** Caramel for use in rum. It should be negatively charged and specific for the alcoholic strength of the product.
- 2.11 Strength:** the concentration of alcohol present.
- 2.12 Natural flavouring material:** Preparation acceptable for human consumption, obtained exclusively by physical processing from vegetable, and sometimes animal raw materials, either in their natural state or processed for human consumption.

3.0 GENERAL REQUIREMENTS

3.1 Rum is a spirit drink –

- (a) obtained exclusively by alcoholic fermentation and distillation of sugar cane molasses, sugar cane syrups, sugar cane juices or cane sugar produced during the processing of sugar cane;
- (b) distilled at an alcohol content of less than 96.0 percent alcohol by volume at 20 degrees Celsius;
- (c) produced in such a way that the product has the organoleptic characteristics derived from the natural volatile elements contained in the above raw materials or formed during the fermentation or distillation process of the named raw materials; and
- (d) includes mixtures solely of the above distillate.

3.1.1 Flavoured Rum

- (a) flavoured rum is rum to which has been added natural flavouring materials; flavours may be added up to a maximum of 2.5% by volume of

the finished product, with or without the addition of sugar, and bottled in accordance with the excise laws of the country in which it is sold.

- 3.1.1.1** The name of the predominant flavour shall appear as part of the designation.
- 3.2** Ethyl alcohol content shall not be less than 40 percent by volume, but may vary according to the excise legislation of the country of sale and shall be determined according to the method prescribed in **Appendix A** or any other **method deemed suitable by the CARICOM Regional Organization for Standards and Quality (CROSQ)**. The tolerance levels for ethyl alcohol shall be as prescribed by the excise legislation of the country of origin.
- 3.3 Freedom from added colouring matter:** Rum shall be colourless except where the colour is derived from wood during maturation or from caramel produced from sugars.
- 3.4 Flavours and Blenders:** All rums for sale and consumption within the CARICOM area shall comply with the specification in item 3.1.1. Rums for export outside the CARICOM area may have added to them such other blenders and flavours as are permitted by the standards of the importing country (e.g., in the U.S., Alcohol Tobacco Tax and Trade Bureau's Regulation 27 CFR 5.23 (a)).
- 3.5 Freedom from foreign matter:** Bottled rum shall be free from permanent sedimentation or suspended matter of any type.
- 3.6 Hygienic conditions:** Rum shall be manufactured in premises built and maintained in a good hygienic condition satisfactory to the National Authority responsible for food safety.
- 3.7 Reduction in strength:** Rum may only be reduced in strength by the addition of potable water.
- 3.8** Rum shall be produced in accordance with the requirements of the distillation certificate specified under the excise legislation of the country of origin.
- 3.9** Tests for artificial colour, methyl alcohol, ethyl carbamate shall be carried out as prescribed in the appropriate appendices mentioned in Table 1 or by other methods approved by CROSQ.

TABLE 1
SPECIFICATIONS AND METHODS OF TEST FOR RUM

Characteristic	Specifications (at drinking strength 40% alcohol)	Method of Test (Refer to Appendix)
Artificial colour (not including caramel)	NIL	B
Methyl alcohol (max)	Not more than 200mg/Kg (200 ppm)	C
Ethyl carbamate	Not more than 0.15 mg/Kg (150 ppb)	E

4.0 PACKAGING

4.1 Rum may be filled in any suitable container, which does not impair the quality of the rum. However, rum shall not be packaged in containers that are made from, or lined with, a material that may contaminate the product in such a way as to make it injurious to health. The bottles or containers shall be properly sealed.

5.0 LABELLING

5.1 General requirements

5.1.1 The label shall not contain any statement, design, or device which either states or implies that the use of any distilled spirit has curative or therapeutic effects.

5.1.2 Labels shall be so designed that the label statements remain conspicuous and legible under normal conditions of purchase and use. Such statements shall be on a contrasting background.

5.1.3 Labels may contain information other than the mandatory labeling information required by **5.2.1** and **5.2.2**, provided that such information does not conflict with, or in any manner qualify, those statements required by the mandatory labeling standards.

5.1.4 Geographical names shall not be applied to rum produced in any region other than the particular place indicated by the name.

5.2 Detailed requirements

5.2.1 The following shall appear on the principal display panel of the label:

- a) brand name;
- b) product name;
- c) alcoholic content; and
- d) net content.

NOTE: The net contents shall be the average net contents as determined by sampling and measurement in accordance with the procedure set out in the most recent revision of International Recommendation OIML R87 of the International Organization of Legal Metrology (OIML).

5.2.1.1 The brand name shall appear in conjunction with the product name. Where it may be misleading, the word "brand" shall appear in direct conjunction with the brand name.

5.2.1.2 The name of the product shall be "rum" and it shall contain not less than 40 percent alcohol by volume. The alcohol content shall be conspicuously stated on the label. Where the term "white rum" appears, the product shall be colourless.

5.2.1.3 The alcoholic content shall be stated as "% alcohol by volume". The equivalent U.S. Proof may be added in close proximity to the required declaration of alcoholic content. **Where the term "Over Proof" appears, it shall be deemed to be Sykes Proof strength, in which case the product shall contain not less than 57.1 % alcohol by volume.**

5.2.1.4 The net contents shall be stated in appropriate units in accordance with the labeling requirements of the country of origin.

5.2.2 The following additional statements shall appear on any part of the label.

- (a) the name and address of the manufacturer; and
- (b) the country of origin.

5.2.2.1 The name and address of the manufacturer shall be the name and address of the place of business of the distiller or bottler of the product. It shall be preceded by the words “Manufactured by....”, “Distilled by.....”, “Bottled by.....” as applicable.

5.2.2.2 The country of origin shall be prominently and clearly stated and it shall be immediately preceded by the words “Product of”.

5.2.3 The following may appear on the label:

- (a) a statement of age; and
- (b) a statement of maturity.

5.2.3.1 A statement of age may be given on the label in terms such as “___ years old”. Where a statement is given, the age stated shall be that of the youngest distilled spirit in the product.

5.2.3.2 When rum is labeled as "matured", it shall have been suitably matured in wooden vats or barrels for not less than one year.

5.3 The label shall conform to any additional requirements given in the labeling requirements of the country in which the product is to be sold.

6.0 *SAMPLING*

6.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in **Appendix D**.

7.0 *QUALITY OF REAGENTS*

7.1 Unless otherwise specified, distilled water (or water of at least equal purity) and chemicals of reagent or analytical grade shall be used throughout the tests.

APPENDIX A

DETERMINATION OF ETHYL ALCOHOL

Note:

The determination of Ethyl alcohol content may be carried out by the method prescribed below or by any internationally recognised and validated distillation method, e.g. AOAC Manual.

A-1 APPARATUS

A-1.1 Gas chromatograph with flame ionization detector.

A-1.1.1 Column: 1.5m glass - internal diameter 4 mm packed with Poropak Q.

A-1.1.2 Operating conditions: column temperature 160°C, isothermal detector 250°C and injector temperature 200°C.

Carrier gas - nitrogen: flow-rate 40ml/min.

Internal standard - isopropanol - G.C grade.

A-2 PROCEDURE

A.2.1 Prepare a 2.5% (v/v) standard mixture of ethanol and isopropanol (at 20°C) using 2.5ml of each in 100ml water. Inject a 0.4µl volume into the gas chromatograph. Repeat injections to obtain similar peak areas for different injections of the same volume. Measure the peak area for the ethanol and isopropanol.

A-2.2 Prepare a sample mixture containing 5% (v/v) rum and 2.5% (v/v) isopropanol (at 20°C). Inject 0.4 µl into the gas chromatograph. Repeat injections to obtain similar peak areas for different injections of the same volume. Measure the peak area of the ethanol and isopropanol.

A-3 CALCULATIONS

A-3.1 The volume of the component is directly proportional to the area of the peak.

i.e. $V \propto A$

or, $V = kA$

Hence, for ethanol $V_e = k_e A_e$

Hence, for isopropanol $V_p = k_p A_p$

Therefore $V_e / V_p = k_e \cdot A_e / k_p A_p$ equation (1)

Take $k_e / k_p = K$ equation (2)

Therefore $V_e / V_p = K \cdot A_e / A_p$ equation (3)

K can be calculated from the results for the standard mixture used as in A-2.1, where A_e , A_p , are the areas of the peaks for ethanol and propanol.

Using the areas of the peaks for ethanol and propanol obtained in A-2.2 substituted in equation (3), calculate V_e for the rum sample.

APPENDIX B**DETERMINATION OF ARTIFICIAL COLOR****Note:**

Determination of Artificial Color may be carried out by the method prescribed below (MARSH TEST) or by any internationally recognised and validated method, e.g. by high performance liquid chromatography (HPLC).

B-1 REAGENTS

B-1.1 Marsh reagent - Mix together 100ml of amyl alcohol, 3ml of phosphoric acid, and 3ml of water.

B-1.2 Fusel oil or amyl alcohol.

B-1.3 Pectin solution - Dissolve 1g of pectin in 75ml water, add 25ml alcohol to preserve, and shake well before using.

B-1.4 2,4 - DNPH solution - Dissolve 1g of 2,4 dinitrophenylhydrazine in 7.5ml sulphuric acid and dilute to 75ml with alcohol. (If kept in a glass-stoppered bottle, the solution will remain clear and stable for several months.)

B-2 PROCEDURE

B-2.1 To 10ml of the sample in a 20ml test tube, add enough freshly shaken Marsh reagent (B-1.1) to nearly fill the tube and shake several times. Let the layers separate. Colour in the lower layer indicates that the sample has been coloured with caramel, synthetic dye, or extractive material from uncharred white oak chips.

B-2.2 In the absence of any colour, test 10ml in the same manner, using enough Fusel oil (B-1.2) to nearly fill the tube and shaking several times. A deeply coloured lower layer indicates synthetic dye. To confirm caramel, apply the following Mathers test.

B-2.2.1 Place 10ml of the filtered sample in a centrifuge tube. Add 1ml of pectin solution (B-1.3) and mix. Add 3-5 drops hydrochloric acid and mix. Fill the tube with alcohol (about 50ml), mix, centrifuge, and decant. Dissolve the precipitate in 10ml of water, and add hydrochloric acid and alcohol as above. Shake well,

centrifuge and decant. Repeat the operation until the alcoholic liquid is colourless. Finally, dissolve the gelatinous residue in 10-ml hot water.

If the solution is colourless, caramel is absent. If the solution is clear brown, caramel may be present. Confirm this by adding 1ml of 2,4 - DNPH solution (B-1.4), then mix, and heat for 30 minutes in boiling water. A precipitate forms if caramel is present.

APPENDIX C

DETERMINATION OF METHYL ALCOHOL**Note:**

The determination of Methyl alcohol content may be carried out by the method prescribed below or by any internationally recognised and validated method.

C-1 APPARATUS

C-1.1 Gas chromatograph - equipped with flame ionization detector.

C-1.1.1 Column - 23% carbowax 1500 (m/m) on Chromosorb W (60-80 mesh, acid washed). Weigh 9g carbowax 1500 into a 250ml beaker and mix with water on a steam bath. Weigh 30g chromosorb W in a 250ml beaker and combine with the carbowax solution in a large flat-bottom polyethylene container (about 20cm x 25cm). Add water to just cover the solid support and mix thoroughly. Evaporate the water with frequent stirring in a hood. (Gentle steam may be applied to hasten evaporation.) After evaporation of the water, heat the coated support for about 2 hours in a 100°C oven.

Pack 2.4 meters of 6mm diameter copper tubing tightly and evenly by repeated tapping, and condition in a column oven at 150°C with helium flow rate of 150 ml/min until a steady baseline is observed at attenuation 1x at operating parameters (about 24 hours).

C-1.1.2 Approximate parameters - Column temperature 70°C (isothermal) detector and inlet temperature 150°C helium carrier flow 150 ml/mm.

Optimum operating conditions vary with column and instrument, and shall be determined by using standard solutions. Adjust the parameters for maximum peak sharpness and optimum separation. With high-level standard, 1-propanol should give almost complete baseline separation from ethanol.

C-1.2 Syringe – 10 μ l, Hamilton Co No 701, or equivalent.

C-2 REAGENTS

- C-2.1** Alcohol - USP, methanol-free
- C-2.2** Methanol stock solution - Dilute 10ml methanol, 99.9 mol % (Fisher Scientific Company, A-936, or equivalent) to 100ml with 40% alcohol.
- C-2.3** 1 -Butanol internal standard stock solution - Dilute 10ml 1-butanol, 99.9 mol % (Fisher Scientific Company, A-384, or equivalent) to 100ml with 40% alcohol.
- C-2.4** Methanol standard solution - 0.050% methanol plus 0.030% 1-butanol internal standard. Fill a 100ml volumetric flask to about 99ml with 40% alcohol and add, by syringe, 500 μ l methanol stock solution (C-2.2), and 300 μ l 1-butanol stock solution (C-2.3). Mix and dilute to volume with 40% alcohol. Mix again.

C-3 DETERMINATION

- C-3.1** Inject 10 μ l methanol standard solution. Adjust operating parameters and attenuation to obtain measurable peak height (about $\frac{1}{4}$ full-scale deflection). Determine the retention time of methanol and 1-butanol (about 3 and 12 minutes respectively). Inject 10 μ l of the sample to estimate the methanol, using attenuation if necessary, and to check for absence of 1-butanol. On the basis of the presence or absence of 1-butanol in the sample, determine the methanol content from a standard curve prepared according to **C-3.1.1** or **C-3.1.2**.
- C-3.1.1** 1-butanol absent - On the basis of the estimation of methanol, prepare a series of standards (4 or 5) in which the range of concentration includes the methanol concentration in the sample. Add internal standard (**C-2.3**) to both the samples and standard solutions at a concentration similar to that of methanol in the sample. Calculate the peak height ratios of methanol, 1-butanol, using averages of duplicate injections, and plot the ratios against methanol concentration.
- C-3.1.2** 1-butanol present - Prepare a series of methanol standards as in **C-3.1.1**, but do not add the 1-butanol internal standard to the sample or methanol standards. Plot the actual peak height of methanol against the concentrations.

APPENDIX D

SCALE OF SAMPLING

D-1 GENERAL REQUIREMENTS

D-1.1 In drawing, preparing and handling samples, the following precautions and directions shall be observed as far as possible.

D-1.1.1 Sampling shall be done by a qualified person who may be a person with statutory authority and, if desired, may be carried out in the presence of the purchaser (or his representative) and the vendor (or his representative).

D-1.1.2 To obtain a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

D-1.1.3 Precautions shall be taken to protect the sample from extraneous contamination.

D-1.1.4 The sample shall be stored in a cool, dark and dry place.

D-2 SCALE OF SAMPLING

D-2.1 All the cases with containers of the same size, containing material of the same type, grade, class and composition, and manufactured under the same conditions and at the same time, shall constitute a lot or batch.

D-2.2 All the containers drawn from a particular lot shall be considered a sample for testing purposes.

D-2.3 A sample from each lot shall be tested for ascertaining conformity of the material to the requirements of the specification.

D-2.4 The number of containers to be selected from each lot shall be in accordance with Table 2.

D-2.5 The containers shall be selected at random. In order to ensure randomness of selection, random number tables shall be used. If random number tables are not available, the following procedure shall be used:

"Starting from any case, count 1,2,3,4..... "r", in a systematic manner. Every "rth" case thus counted shall be withdrawn until the desired number is obtained "r" being the integral part of N/n , where "N" is the total number of cases in the lot, and "n" is the number of cases to be selected. One container shall be drawn from each case.

- D-2.6** All the cases selected shall be opened and the containers examined visually for the conditions of packing, the external appearance and the fill.
- D-2.7** Sampling shall be done in accordance with Table 2. The effectiveness of the sampling plan is dependent on the execution of proper quality control procedures.

TABLE 2
SAMPLING PLAN FOR PHYSICAL AND
CHEMICAL REQUIREMENTS (AQL = 6.5)

Lot Size (cases)	Sample Size (cases)	Acceptance No. (cases)	Rejection No. (cases)
1 – 50	2	0	1
51 – 500	8	1	2
501 - 3200	13	2	3
3201 - 35000	20	3	4
35001 - 500000	32	5	6
OVER 500000	50	7	8

- D-2.8** Tests shall be made on the composite sample prepared by thoroughly mixing equal quantities from the individual sample containers. The result shall satisfy the requirements of this standard.

APPENDIX E

DETERMINATION OF ETHYL CARBAMATE

Note:

The determination of ethyl carbamate, apart from the method prescribed below, may be carried out using any of the prescribed methods for its determination, as stated in the AOAC Manual.

E-1 APPARATUS

- (a) Gas chromatograph, Varian 6000 or equivalent, equipped with:
- (1) auto-sampler or manual injection system, split-less, for injecting about 2_1 volume.
 - (2) Hall detector in nitrogen mode;

Conditions:

Furnace 840°C

Hydrogen 80ml/min

Helium 30ml/min

Vent Vent at all times, including between samples, except for a 2 – 3 min window for measuring Ethyl carbamate.

- (3) Column: 30 m x 0.32mm DBWAX 0.5 _1 film or equivalent;

Conditions:

Flow rate (helium) about 1ml/min

Temp. program 50°C hold for 2min; 20°C/min to 140°C
and hold 10min; 10°C/min to 250°C and
hold 10min

Injector 240°C

Detector base 250°C

- (b) Centrifuge
- (c) Centrifuge bottles, 250ml, fitted with appropriate polyethylene stoppers
- (d) Graduated centrifuge tubes, 15ml
- (e) Rotary evaporator
- (f) Flasks for rotary evaporator, 500ml.

E-2 REAGENTS

- (a) Sodium chloride, reagent grade
- (b) Methylene chloride, distilled-in-glass grade (see note)
- (c) Ethyl acetate, reagent grade
- (d) Ethanol, absolute, BP-USP
- (e) Sodium sulphate, anhydrous, granular, reagent grade
- (f) Standard solutions of ethyl carbamate

- (1) Stock solution 1 mg/ml (1 μ g/ μ l)

Dissolve 100mg ethyl carbamate in ethyl acetate in a 100ml volumetric flask, dilute to volume and mix well.

- (2) Dilute stock solution (10ng/ μ l)

Pipette 1.0ml of standard stock solution (1 μ g/ μ l) into a 100ml volumetric flask, dilute to volume with ethyl acetate and mix well.

- (3) Working standards

Prepare the following working standards with appropriate serial dilution of

the dilute stock solution (10ng/ μ l) with ethyl acetate: 0.1, 0.25, 0.5, 1.0 and 2.0ng/ μ l.

(4) Spiking solution 2.0 μ g/ μ l

Pipette 1.0ml of standard stock solution 1mg/ml into a 500ml volumetric flask, dilute to volume with ethanol and mix well.

NOTE: Since ethyl carbamate has been found in some lots of methylene chloride, each lot should be tested as follows:

Add 3ml of ethyl acetate to 100ml of methylene chloride, evaporate to about 2ml using a rotary evaporator at 28°C (82°F) and determine the presence or absence of a GC peak corresponding to ethyl carbamate. Use only methylene chloride which is free from ethyl carbamate.

E.3 PROCEDURE

E-3.1 Extraction

E-3.1.1 Weigh about 15g of rum into a 250ml centrifuge bottle. Dilute to 50g with water and mix well.

E-3.1.2 Add about 30g of sodium chloride and swirl 1 min to saturate solution.

E-3.1.3 Add 75ml of methylene chloride, stopper and shake vigorously for about 1 min.

E-3.1.4 Centrifuge until phases separate.

E-3.1.5 Draw off lower layer and filter into a 500ml evaporator flask through 40g anhydrous sodium sulphate on a Reeve Angel No. 202 or Whatman No. 1 filter in a conical glass funnel.

E-3.1.6 Repeat **D-3.1.3** to **D-3.1.5** twice for a total of three extractions.

E-3.1.7 Add about 3ml of ethyl acetate to the extract (about 225ml) then reduce volume to about 2ml using a rotary evaporator at 28°C (82°F).

NOTE: This is a critical step requiring care to ensure that sufficient evaporation takes place to remove all of the methylene chloride, which could damage the Hall detector scrubber. Extracts must not be evaporated to dryness or to near dryness. Adequate venting of the Hall detector, as previously indicated, is another precaution to minimize the effects of even traces of methylene chloride on the Hall scrubber.

E-3.1.8 Transfer to a 15ml graduated centrifuge tube, rinse flask with 2ml x 1ml ethyl acetate, transfer rinses to the centrifuge tube, adjust volume to 5ml and mix well.

E-3.2 Gas chromatography

E-3.2.1 Inject, manually or by using an auto-sampler, precisely measured volumes of about 2 μ l of solutions:

(a) for calibration curve:

standards in increasing order of concentration (0.2, 0.5, 1, 2, and 4 ng);

(b) for sample extracts:

sample extracts interspersed with calibration standards and spiked sample extracts.

E-3.2.2 Compare retention time of samples and standards.

E-3.2.3 Dilute to a known volume any sample extracts which exceeds the range of the calibration curve and inject again.

E-4 CALCULATIONS

Measure response (peak height or peak area) of each standard.

Prepare calibration curve by plotting response of each standard against ng ethyl carbamate injected. Measure response (peak height or peak area) of each sample.

Calculate concentration of ethyl carbamate in sample using the following equation:

$$\text{ppm ethyl carbamate} = C / W_x$$

Where C is ng ethyl carbamate read from calibration curve, and **W_x** is sample weight equivalent in g in volume of sample injected.