Japan Customs Analysis Methods

No. 116

Analysis Method for Rum

(Issued in June 1999)

(Updated in May 2014)

1. Scope

This analysis method is applied to goods which is required to conduct analysis for the requirements ハ to ホ of (2) in the Administrative Notification of Classification, *"2208.40 – 1. standards for rums and other spirits obtained by distilling fermented sugar-cane products"*.

2. Outline of Test Method

This method is carried out for checking rum according to the following procedures.

(1) Qualitative analysis of aromatic constituents using gas chromatography

(2) Determination of the ethyl alcohol content

(3) Determination of the contents of n-propyl alcohol, isobutyl alcohol, etc., using gas chromatography

3. Apparatus

(1) Gas Chromatograph

A gas chromatograph, equipped with a flame ionization detector (FID).

(2) Gas chromatography column

Use one of the following columns.

Packed column: a glass column, 3 mm i.d. × 2 m length, packed with 5% PEG-20M Chromosorb WAW DMCS, or its equivalent.

Capillary column: DB-WAX (0.25 mm i.d. × 30 m length, 0.25 μm film thickness) or its equivalent.

4. Reagents

All chemicals must be JIS special reagent grade or equivalent, unless otherwise specified.

4.1. Preparation of standard undiluted solutions and internal standard undiluted solution

Accurately weigh approximately 500 mg each of n-propyl alcohol, isobutyl alcohol, and isoamyl alcohol in separate 100-mL volumetric flasks, add about 5 mL of acetone and dilute to volume with water. They are to be used as standard stock solutions.

Similarly, accurately weigh 500 mg of n-amyl alcohol in a 100-mL volumetric flask. Add 5 mL of acetone and dilute to the volume with water. This is to be used as internal standard stock solution[1)].

Note 1) Each chemical in both the standard stock solutions and the internal standard stock solution is prepared with a concentration of approximately 5,000 ppm.

4.2. Preparation of standard solutions

Take 0.5, 1.0, 2.0 and 2.5 mL of the respective standard stock solutions prepared in 4.1. by whole pipettes into separate 10-mL volumetric flasks and add 2.0 mL of the internal standard stock solution to the respective flasks. Dilute to volume with water. Use them as the standard solutions[2)].

Note 2) A microburette graduated to 0.02 mL may be used to collect the standard stock solutions and the internal standard stock solution.

5. Qualitative Analysis of Aromatic Constituents

5.1. Collection of aromatic constituents

Take 50 mL of a sample in a 200-mL separating funnel and add 50 mL of chloroform to it. Shake the

separating funnel vigorously and separate the chloroform layer. Add another 50 mL of chloroform to the remaining water layer, shake in the same manner and separate the chloroform layer again. Put the chloroform layer fractions together and use it as the chloroform extract (I).

Add 50 mL of 5% sodium hydrogen carbonate aqueous solution to the chloroform extract (I) and shake vigorously. Remove the chloroform layer and use it as the chloroform extract (II).

Add a small amount of sodium sulfate anhydrous to the chloroform extract (II) for dehydration. After filtering it, concentrate it under moderate water aspirator vacuum while warming in a water bath at 50 °C until the volume of the chloroform extract (II) reaches about 0.5 mL. Use the concentrated solution as the analyte for the analysis of aromatic constituents.

From liquor put up in a container for retail sale as rum (control product), collect the aromatic constituents in the same manner above.

5.2. Operating Conditions of Gas Chromatograph

(1) Column temperature

For packed column: 80 - 240 °C, program rate 6 °C/min.

For capillary column: 80 - 40 °C, program rate 10 °C/min.

(2) Inlet port and detector temperature: 250 °C

(3) Other operating conditions of gas chromatograph

Conditions shall be optimized according to the system of analytical devices and for column separation.

5.3. Confirmation of Aromatic Constituents

Inject the aromatic constituents prepared in 5.1. into the gas chromatograph under the prescribed conditions in 5.2. and obtain gas chromatograms.

Identify n-propyl alcohol, isobutyl alcohol, isoamyl alcohol, ethyl caproate, ethyl caprate, ethyl caprylate and β-phenylethyl alcohol by comparing retention times for sample peaks to those for the standard substances; or identify each of the sample peaks by gas chromatography-mass spectrometry (GC-MS).

As to liquor put up in a container for retail sale as rum (control product), carry out analysis in the same manner and compare the similarity of chromatograms[3)].

Note 3) In comparing chromatograms, if main peaks of a sample show an almost similar pattern to those of the control product, it is regarded as similar to the control product (See appendixes 1 and 2).

6. Determination of Alcohols

6.1. Determination of Ethyl Alcohol

Refer to “16. Spirit” of Official Methods of Analysis of National Tax Administration Agency, Japan (National Tax Administration Agency Directive No.1, 1961; Revised National Tax Administration Agency Directive No.6, 2007).

6.2. Determination of n-Propyl Alcohol, Isobutyl Alcohol and Isoamyl Alcohol

6.2.1. Operating Conditions of Gas Chromatograph

(1) Column temperature

For packed column: 80 °C (constant)

For capillary column: 80 °C (constant)

(2) Inlet port and detector temperature: 250 °C

(3) Other operating conditions of gas chromatograph

Conditions shall be optimized according to the system of analytical devices and for column separation.

6.2.2. Preparation of Calibration Curves

Inject about 1 μL of each standard solution prepared in 4.2. into the gas chromatograph under the prescribed conditions in 6.2.1. and measure the peak area of each constituent.

Then, obtain a ratio (Ax/As) of the peak area (Ax) of each constituent to the peak area (As) of n-amyl alcohol used as the internal standard.

Separately, construct calibration curves of n-propyl alcohol, isobutyl alcohol and isoamyl alcohol by plotting a weight concentration ratio (Wx/Ws) of the weight concentration (Wx) of each standard solution to the weight concentration (Ws) of n-amyl alcohol used as the internal standard, both of which were injected into the gas chromatograph, against a ratio of their peak areas (Ax/As).

6.2.3. Sample Measurement

Accurately weigh approximately 20 g of the sample in a 50-mL Erlenmeyer flask. Add precisely 2 mL of the internal standard stock solution prepared in 4.1. with a whole pipette and mix well.

Inject about 1 μL of the mixed solution into the gas chromatograph under the prescribed conditions in 6.2.1. and measure a peak area of each constituent.

Obtain a ratio (Ax/As) of the peak area (Ax) of each constituent to the peak area (As) of n-amyl alcohol used as the internal standard. Using this value, obtain a weight concentration ratio (Wx/Ws) of each alcohol to n-amyl alcohol from the calibration curves constructed in 6.2.2.

6.2.4. Calculation of Alcohol Contents

Calculate the contents of the alcohols in the sample from the following formula.

$$A = \frac{(Wx/Ws) \times Ms}{S} \times 1000$$

Where –

A: Content of each alcohol in the sample (ppm)

Wx/Ws: Concentration ratio obtained from calibration curves

Ms: Amount of a-amyl alcohol used as the internal standard (mg)

S: Amount of the sample collected (g)

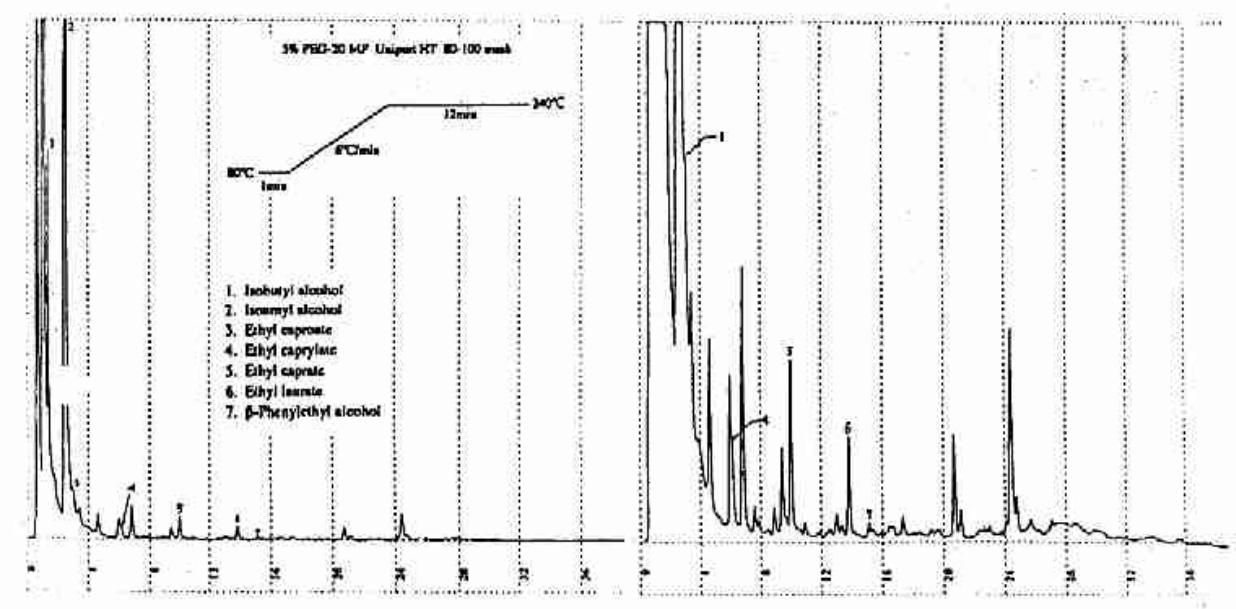
Round off fractions to the first decimal place.

7. Reference

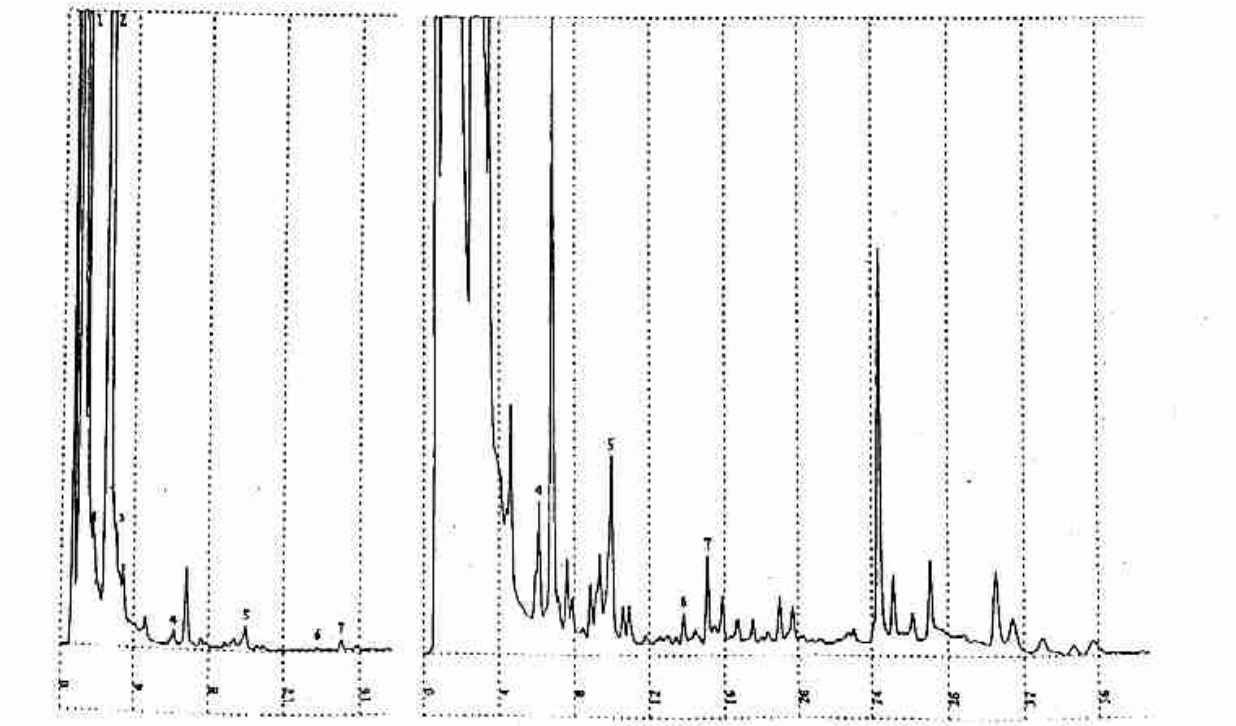
(1) Deki M., Kato T. (1971) Report of the Central Customs Laboratory **11**: 1 (in Japanese).

(2) Official Methods of Analysis of National Tax Administration Agency, Japan

Appendix 1



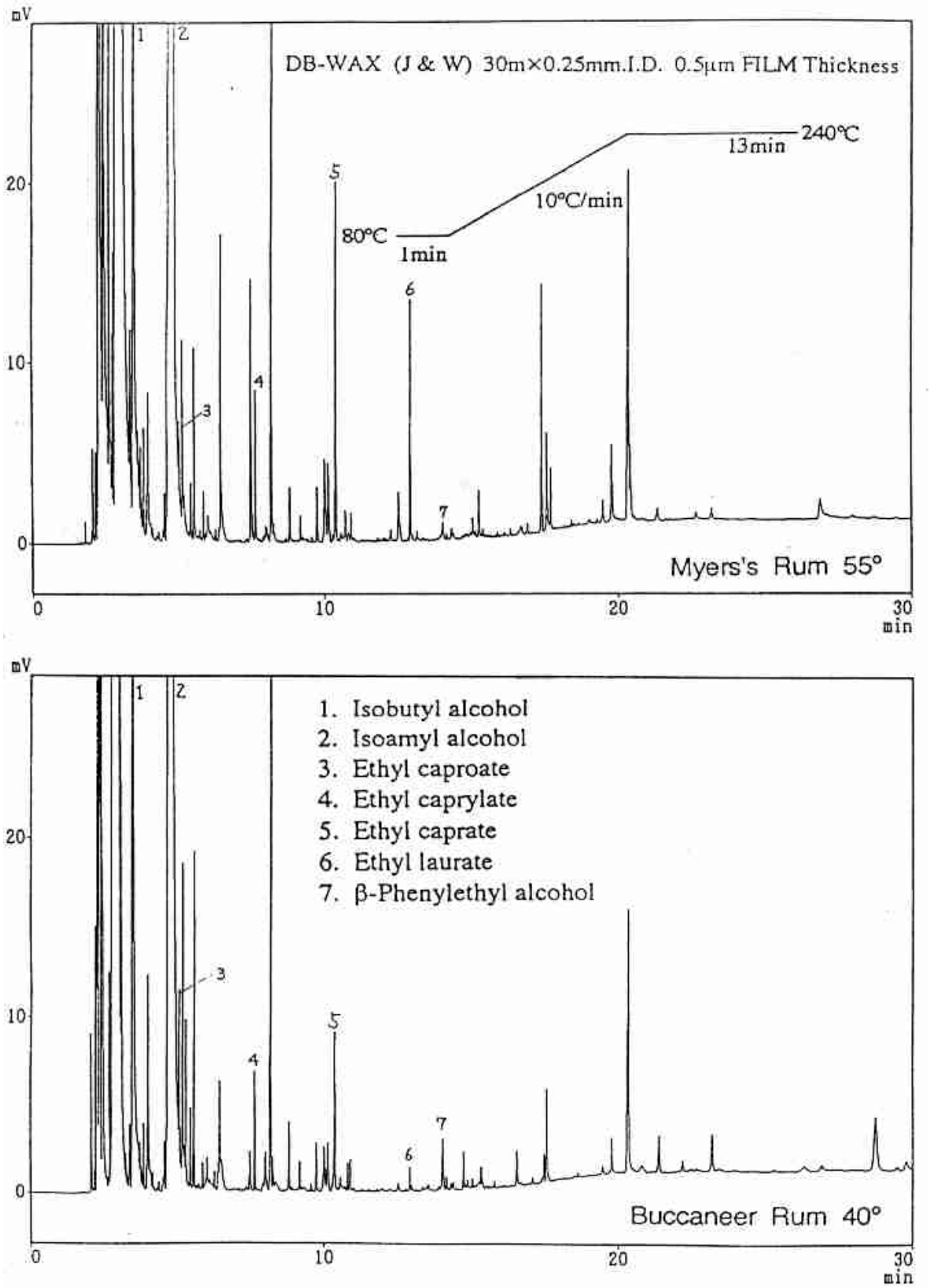
Myers's Rum 55°



Buccaneer Rum 40°

Examples: gas chromatograms of aromatic constituents in liquor sold on the market as rum and imported rum (spirit made from molasses)

Appendix 2



Examples: gas chromatograms of aromatic constituents in two kinds of liquor sold on the market as rum, separated with a capillary column