RESOLUTION OENO 11/2000

INTERNATIONAL OENOLOGICAL CODEX

RECTIFIED ALCOHOL OF AGRICULTURAL ORIGIN

1. OBJECTIVE, ORIGIN AND SCOPE OF APPLICATION

Rectified, or "neutral," alcohol obtained by distilling and rectifying alcohol from wine, wine sediments or alcoholic fermentation products derived from from grape or raisin marcs, and all other plant-based substances of agricultural origin.

Rectified alcohol of agricultural origin forms an ingredient of some spirits and special wines.

2. COMPOSITION

At a temperature of 20 °C, 100 parts of this alcohol contain at least 96 parts ethanol.

Note: The tests and controls described below in italics are not mandatory and are performed only upon request.

3. PROPERTIES

Colorless, clear, volatile liquid with a penetrating odor and fiery flavor. It is flammable and burns without smoke and with a blue flame.

It should be distilled completely at between 78 and 79 °C.

3.1. Solubility

Neutral alcohol is miscible in water in all proportions with a notable release of heat and contraction of volume. It is also mixable with in acetone, chloroform, ethyl ether, glycerol, and an equal volume of castor oil.

3.2. Characterization Procedure

- Slowly heat a mixture of 1 ml neutral alcohol, twenty drops of concentrated sulfuric acid (R) and 10 g of sodium acetate (R) in a test tube. A strong, characteristic odor of ethyl acetate will be released.

Certified in conformity Paris, 23th June 2000
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- Mix several drops of alcohol and 1 ml of concentrated sulfuric acid (R), then add several drops of 10 pp 100 potassium dichromate solution. The liquid will become green and emit the odor of ethanal
- Dilute 0.5 ml of alcohol with 4.5 ml of water. Add 1 ml of 1M sodium hydroxide solution, then slowly add 2 ml of iodized potassium iodide (R). An odor of iodoform will be produced, following by the formation of a yellow precipitate.

3.3. Analysis of Agricultural Origin

This analysis is carried out by measuring the ethanol 14C/12C ratio (scintillation) in accordance with the method described in the Spirits Compendium.

4. TESTS

4.1. Appearance

Take two identical test tubes made of alkali-lime glass about 250 mm high, fill one with alcohol, the other with water, which will serve as a control. Examine the liquids along the cylinder's axis. The alcohol should not exhibit any noticeable coloration.

In one test tube about 250 mm high and 25 mm in diameter, pour 40 ml of alcohol, then dilute it with 80 ml of water. The mixture should not cloud nor present any odor or foreign taste.

4.2. Foreign Odoriferous Substances

Let 10 ml of alcohol evaporate spontaneously on a strip of white filter paper. No foreign odor should be perceived during or after evaporation.

4.3. Dry Extract or Non-Volatile Residue

In a 25 ml calibrated dish, heat to 100 °C in a water bath, then slowly evaporate 100 ml of alcohol. Weigh. The dry extract should be less than 1.5 g/hl 100% ethanol by volume.

4.4. Heavy Metals

Take up, using 10 ml dilute chlorhydric acid (R), any residue left from the evaporation of 100 ml alcohol during the dry extract analysis. After heating for several minutes in a 100 °C water bath to stimulate dissolution of this residue, decant the acid solution in a 25 ml volumetric flask after the calibrated dish has been washed three times with 5 ml of water and the volume raised to 25 ml. Take a 5 ml sample of this solution in a test tube. Add 2 ml of a pH 3.5 (R) buffer solution, 7.5 ml of water and 1.2 ml of thioacetamide reagent (R). The solution should not produce any white or black precipitates nor any brown or other coloration. At the very least, any coloration produced should be no more intense than that obtained using the general method . (Heavy metal conent expressed in lead, after 50% concentration of the alcohol, should be 0.5 mg/l).

4.5. Lead

Using the method set forth in the Compendium, perform the lead analysis lead in the solution obtained in the previous paragraph. (Lead content should be less than 0.5 mg/l).

4.6. Mercury

Using the method described in the annex, carry out the mercury analysis in the solution obtained in Paragraph 4.4. (Mercury content should be less than 0.2 mg/l).

4.7. Arsenic

Using the method described in the annex, carry ou the arsenic analysis in the solution obtained in Paragraph 4.4. (Arsenic content should be less than 0.5 mg/kg after 50% alcohol concentration).

4.8. Ketones, propan-2-ol and 2-methylpropan-1-ol

Add 3 ml of water and 10 ml of mercury sulfate (II) solution (R) to 1 ml of alcohol, then heat in a 100 °C water bath. No precipitate should form in the first three minutes.

4.9. Permanganate Decolorization Time (Barbet Test)

Pour 50 ml of the alcohol sample into a flask. Add 2 ml of freshly prepared potassium permanganate solution to 0.20 g/l (R). Place the container in a 15 °C water bath and start a stopwatch. Avoid directly exposing the sample to natural or artificial light during the test.

Simultaneously, place 50 ml of the comparison solution in the 15 °C water bath. This solution is obtained by mixing 3 ml of 5 pp 100 cobalt chloride solution (R), 4.2 ml of 4 pp 100 uranyl nitrate solution (R) and filling to 50 ml with distilled water. Compare the test color to the standard. Stop the timer when the colors are identical. Note the amount of time elapsed. The decolorization time of the permanganate should be at least 20 minutes.

4.10. Sulfured Derivatives

Add approximately 1 ml of mercury, then 20 ml of alcohol to a test tube. Agitate for 1-2 minutes. The surface of the mercury should remain brilliant with no black clouding.

4.11. Methanol

4.11.1. Colorimetric Analysis

Standard solution: weigh 5 g of methanol in a 50 ml volumetric flask, then top off to the line with ethanol (free of methanol).

In a 1-liter volumetric flask, place 1 g of the preceding solution (i.e., 1.25 ml) containing 125 mg of methanol, 250 ml of pure alcohol (methanol free). Top off with water to 1000 ml.

Test technique: place 1250/A ml of alcohol in a volumetric flask (A is the alcoholmetric titer of the alcohol to be tested.) and fill to the gauge line with water. Place 1 ml of alcohol, diluted to 25 pp 100 in a test tube. Add four drops of 50 pp 100 (m/m) phosphoric acid (R), four drops of 5 pp 100 (m/m) potassium permanganate solution (R), then stir and let sit 10 minutes. Decolorize the permanganate with several (typically 8) drops of 2 pp 100 (m/v) of potassium anhydrous sulfite (metabisulfite) (R), avoiding any excess. Add 5 ml of chromotropic sulfuric acid solution (R). Place in a 70 °C water bath for 20 minutes. No violet color should appear, or in the event it does appear, it should not be more intense than that of a control prepared using the same technique and the same reagents, with 1 ml of the aforementioned standard solution (maximum methanol content is 50 g/hl at 100% vol.).

4.11.2. Gas phase chromatography Analysis

Equipment (example):

Gas phase chromatograph with a flame ionization detector

Semi-polar capillary columns, for example Carbowax 20 M □.

<u>Test technique:</u>

Prepare a water-alcohol solution using 1 g per liter of the internal standard (4-methylpentane-2-ol) in 50 pp 100 alcohol by volume.

Prepare the solution to be analyzed by adding 5 ml of this solution to 50 ml of alcohol reduced to 50 pp 100 by volume.

Prepare a reference solution of methanol at 100 mg per liter of alcohol at 50 pp 100 by volume. Add 5 ml of the internal standard solution to 50 ml of this solution.

Inject 2 microliters of the solution to be analyzed added to the internal standard solution, into the chromatograph.

The oven temperature should be 90 °C and the supporting gas flow rate should be 25 ml per minute. These settings are given as an example.

S: surface of the methanol peak of the reference solution

Sx: surface of the methanol peak of the solution to be analyzed

i: surface of the internal standard solution peak in the solution to be analyzed

I: surface of the internal standard solution peak in the reference solution

The methanol content, expressed in milligrams per liter of alcohol at 50 pp 100 by volume, is given by the formula:

$$C = 100(I/i)(S_x/S)$$

The content in grams per hectoliter of pure alcohol is 0.20C (maximum content in methanol 50 g/hl of ethanol at 100% by volume).

4.12. Ammonium Hydroxide and Nitrogenous Bases

Pour 50 ml of the alcohol to be examined into a 200 ml flask. Add 40 ml of water and two drops of phosphoric acid (ρ 20 = 1.58). Distill and collect the 80 ml that are returned. Add 2 ml of 10 pp 100 sodium hydroxide ® to the cooled residue. Distill again and collect approximately 7 ml of distillate in a test tube to which had previously been added 2 ml of water and one drop of methyl red solution ®. The distillate should be drawn to the bottom of the tube using a slender tube. Titrate using a solution of 0.01 M hydrochloric acid until the indicator turns to red. Let n be the number of milliliters of 0.01 M hydrochloric acid solution used.

1 ml of 0.01 M hydrochloric acid solution corresponds to 0.00014 g of nitrogen (ammoniacal or volatile nitrogen bases).

The quantity of ammoniacal nitrogen or nitrogenous bases expressed in milligrams of nitrogen per liter of ethanol is:

- 280n/A

Where A is the alcohometric titer by volume of the alcohol studied.

Neutral alcohol should not contain more than 1 mg of nitrogen (ammoniacal or of volatile nitrogenous bases) per liter of ethanol.

(Maximum ammonium hydroxide and nitrogenous base content is expressed in terms of nitrogen is 0.1 g/hl of ethanol at 100% by volume).

4.13. Acidity

Place 100 ml strengthened of 50 pp 100 by volume alcohol in a 250 ml conical flask. Add one drop of phenol red solution (R) and add 0.01 M sodium hydroxide, one drop at a time, until red, where n is the number of milliliters used.

1 ml of 0.01 M sodium hydroxide corresponds to 0.0006 g of acetic acid.

Acidity expressed in milligrams of acetic acid per liter of ethanol is equal to 12n.

This acidity should be less than 15 mg/l of ethanol (or 1.5 g/hl) at the time the alcohol is delivered.

(Maximum acidity expressed in terms of acetic acid is 1.5 g/hl of ethanol at 100% by volume).

Note: Indicator movement should be stable and clear cut during quantitative analysis of the acidity. If it is not, and especially if the acidity exceeds 15 mg/l, a new test should be conducted after the sample is degassed using the following technique.

100 ml of alcohol at 50 pp 100 by volume is placed in a 250 ml flask whose stopper has two tubes through it.

One tube permits the flask to be kept under a vacuum using a glass filter pump. Pressure is kept between 55 and 65 cm of mercury.

During the procedure, the other tube allows air bubbling from which carbon dioxide is removed by using a sode wash bottle. To accomplish this, the tube has a capillary portion which is submerged in the alcohol. The rate of air flow through the wash bottle is approximately 1 ml per second.

The procedure should last between 3 and five minutes. Titration is accomplished in the same flask.

4.14. Esters

Add 10 ml of 0.1 M sodium hydroxide solution measured with precision to the solution prepared to analyze acidity as detailed under 4.13 (or 100 ml of alcohol at 50% by volume). Cork the flask and stir while maintaining a temperature equal to or slightly higher than 20 °C. After 24 hours of contact, titrate the excess sodium hydroxide using a 0.1 M solution of hydrochloric acid, where n is the number of milliliters used.

To determine the quantity of 0.1 M hydrochloric acid solution which will neutralize 10 ml of 0.1 M sodium hydroxide solution in the presence of the same quantity of alcohol and of the same indicator movement obtained by decreased pH intervals, perform the following test: place 100 ml of degasified 50 pp 100 alcohol in a 250 ml conical flask. Add one drop of phenol red solution (R) and n milliliters of 0.1 M sodium hydroxide. Which cuase the indicator to turn to red. Add 10 ml of the 0.1 M sodium hydroxide solution, and, immediately thereafter, add 0.1 M hydrochloric acid solution to obtain the same movement of the indicator, that is, n^n of the volume used.

1 ml of 0.1 M sodium hydroxide solution corresponds to 0.0088 g of ethyl acetate. The ester concentration, expressed in milligrams of ethyl acetate contained in 1 liter of ethanol is:

$$176(n^n - n_,)$$

This content level should not exceed 13 mg for 1 liter of ethanol (or 1.3 g/hl) at the time the alcohol is delivered.

(Maximum ester content expressed in terms of ethyl acetate is 1.3 g/hl of ethanol at 100% of volume).

4.15. Aldehydes

Standard solution: Place 268.3 mg of pure acetal (boiling point : 102 °C) in a 100 ml volumetric flask. Top off to the line with 50 pp 100 alcohol by volume, free of aldehydes.

Dilute this solution to 1/10 in 50 pp 100 alcohol by volume , is free of aldehydes. The solution obtained contains 100 mg of ethanal per liter of 50 pp 100 alcohol by volume, or 20 g in 100 liters of ethanol.

Test procedure: Place 10 ml of alcohol reduced to 50 pp 100 by volume in a test tube. In a second test tube, place 5 ml of the solution containing 100 mg of ethanal per liter of alcohol at 50 pp 100 and 5 ml of alcohol at 50 pp 100 by volume which is free of aldehydes. Add to the two tubes 4 ml aniline red chlorhydrate solution decolorized by sulfuric acid (R), stir, and compare the colorations obtained after 20 minutes.

The alcohol to be tested should have a color approximately equal to that of the standard solution.

(Maximum aldehyde content expressed in ethanal is 0.5 g/hl at 100% of volume).

Note concerning 50 pp 100 alcohol by volume without aldehydes: Place 100 ml of alcohol diluted to 50 pp 100 by volume in a 250 ml flask with 2 g of metaphenylene diamine (R) and two pieces of pumice stone. Connect the flask to a reflux condenser and maintain a gentle boil for one hour. After cooling, connect the flask to the distilling apparatus and slowly distill without overheating the walls. Collect 75 ml of distillate in a 100 ml volumetric flask. Fill to the line with distilled water.

4.16. Superior Alcohols

Propan-1-ol, 2-methylpropan-1-ol, 2- and 3-methylbutan-1-ol.

Quantitative analysis by gas phase chromatography (see methanol).

Maximum content for the sum of each of the alcohols: 0.5 g/hl of ethanol at 100% of volume.

4.17. Furfural

Place 10 ml of alcohol reduced to 50% by volume in a test tube with an emery stopper. Add 0.5 ml of aniline (R) and 2 ml of crystallizable acetic acid (R). Stir. No salmon pink coloration should be perceptible after 20 minutes.

5. STORAGE

The alcohol should be stored in inert containers which will not give off metals, ions or plastic constituents.

The containers, as well as storage methods, must be in compliance with safety standards.

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