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

Foreword

This standard took references from the Vodka section of the 1576/89 Regulation (EC) No. 110/2008 of the European Parliament and of the Council on the Definition, Description, Presentation, Labelling and the Protection of Spirit Drinks.

This standard replaces the earlier version GB/T 11858-2000 Vodka.

As compared with GB/T 11858-2000, key changes are as follows:

- Description of scope of application had been amended;
- Section on “Terms and Definition” was added;
- Requirements on raw and supplementary ingredients were removed;
- Made appropriate amendments to alcohol content index;
- Made appropriate amendments to inspection guidelines.

Appendix A and B in this standard are normative appendices.

This standard was proposed by the National Food Industry Standardization Management Committee.

This standard is under the jurisdiction of the National Brewers Standardization Management Committee.

The organizations involved in the drafting of this standard: China Food Fermentation Industry Research Institute, Jilin Tuopai Agricultural Product Development Co., Ltd.

The key personnel involved in the drafting of this this standard: Wei Zhang, Xuebo Bai, Yongpu Kang, Yuxing Liu and Xingguang Guo.

This standard will replace the earlier versions:

- GB/T 11858-1989, GB/T 11858-2000.

National Food Safety Standards

Vodka

1. Scope

This standard specified the terms and definition, requirements, analysis methods, inspection guidelines as well as labelling, packaging, transportation & storage for vodka.

This standard applies to the production, inspection and sales/distribution of vodka.

2. Normative References

Clauses involved in the following documents constitute the ones in this standard through reference in this standard. If any reference is dated, the following amendment or revised versions (excluding errata) are not applicable to this standard. However, the study of whether the latest version of these documents can be used by all parties who reach agreement according to this standard is encouraged. Any latest version of the non-dated reference is applicable to this standard.

GB/T 191 *Illustration and Logo for Packaging, Storage and Transportation* (GB/T 191-2008.ISO 780:1197, MOD)

GB/T 601 *Chemical Reagents – Preparation of Standard Titration Solution*

GB/T 603 *Chemical Reagents – Preparation of Reagents and Substances Used in Tests* (GB/T 603-2002, ISO 6353-1:1982, NEQ)

GB 2757 *Hygienic Standard for Distilled Spirits and Liquor*

GB/T 6682 *Specifications and Testing Methods for Water Used in Analysis Experiments* (GB/T 6682-2008, ISO 3696:1987, MOD)

GB 10344 *General Principles of Prepackaged Wine Beverage Labels*

3. Terms and Definition

The following terms and definitions will apply for this standard.

3.1 Vodka

Refers to distilled spirits, products of special refining processes of edible alcohol that is the result of fermentation and distillation of grains, tubers, molasses and other similar agricultural products as its key ingredients.

3.2 Flavored Vodka

Refers to vodka products that highlight the flavor of the specific food flavoring added with plain vodka described above as base alcohol.

4. Requirements

4.1 Sensory Requirements

Should comply with the requirements listed in Table 1.

Table 1 Sensory Requirements

Items	Vodka	Flavored Vodka
Appearance	Colorless, clear, transparent without any suspended substances or precipitation	
Smell	Aromatic, no unusual odor	Aromatic and has the smell of the food flavoring added
Taste	Gentle, fruity, distinct sweetness, no unusual taste	Has obvious flavor of the specific flavoring added
Style	Has the style unique to the product	

4.2 Physical-Chemical Index Requirements

Physical-chemical indexes should comply with the requirements listed in Table 2.

Table 2 Physical-Chemical Index Requirements

Items		Superior Grade	First Grade	Second Grade
Alcohol Content[a] / (%vol)	≥	37.0		
Alkalinity / mL	≤	2.5	3.0	3.5
Total Aldehyde (Acetaldehyde) / [mg/L (100%vol Ethyl Alcohol)]	≤	4	6	8
Total Ester (Ethyl Acetate) / [mg/L (100%vol Ethyl Alcohol)]	≤	10	15	25
Methyl Alcohol / [mg/L (100%vol Ethyl Alcohol)]	≤	50		
High Quality Alcohols / [mg/L (100%vol Ethyl Alcohol)]	≤	4	6	8
[a]Discrepancy between actual alcohol content and alcohol content value indicated on product label should be within the ±1.0 %vol range.				

4.3 Hygiene Requirements

Should comply with the requirements specified in GB 2757.

5. Analysis Methods

The water used in this standard, unless otherwise stated, all refers to water that complies with the requirements specified in GB/T 6682.

The chemical reagents used in this standard, unless otherwise stated, all are analytically pure (AR). The formulated “solutions”, unless otherwise stated, all are aqueous solutions.

If there are two or more methods of analysis for the same test item, laboratory can choose which method to adopt according to individual circumstances, although the first method is always the method to reference under situation of conflicting results.

Ethanol content (alcohol content) mentioned in this standard is represented in fraction of total volume (%vol), hereinafter simply indicated to as %.

5.1 Sensory Analysis

5.1.1 Preparation of Alcohol Samples

Mark each alcohol sample with a code number, then place the sample in a water bath adjusted to 20°C~25°C. Pour 45 mL of each alcohol samples into corresponding clean and dry tasting glasses.

5.1.2 Appearance

Place the tasting glass with alcohol samples in a bright area, and raise it up to eyebrow level. Observe the color, luster, degree of transparency and clarity of the sample in the glass as well as to check for the presence of precipitation and suspended substance with your sense of sight. Make proper records of the findings.

5.1.3 Smell

Hold by the stem of the tasting glass with your hands, slowly move the glass under your nostrils and smell the aroma emanating from the sample. Thereafter, slowly swirl the glass and smell the aroma again after the diffusion of air into the alcohol sample after the swirling. Add a lip to the cup and hold the bowl of the glass for 2 mins, smell the aroma again after swirling. Analyze and determine the aroma of the ingredients, flavorings added or if there is any unusual odor. Write a report on the analysis.

5.1.4 Taste

Drink and place a small amount of the sample (~2 mL) into the mouth, uniformly distribute the liquid across the entirety of the taste buds and taste carefully. Once a distinct impression has been formed, swallow to determine the palate and make appropriate records of the palate and taste features of the vodka.

5.1.5 Style

Analyze and evaluate the specific style and traditional level of strength/weakness of the alcohol collectively based on the appearance, aroma and taste characteristics determined as abovementioned. Write a report on the conclusion of the evaluation.

5.2 Alcohol Content

5.2.1 Density Bottle Method

5.2.1.1 Principle

Remove substances that are not volatile with distillation and then use the density bottle method, electronic density apparatus to determine the density of the sample (aqueous solution of alcohol) at 20°C. Match results against a table of specific fraction of volume of ethanol at 20°C in Appendix A and the value will be the alcohol content.

5.2.1.2 Apparatus

5.2.1.2.1 All-glass Distillation Apparatus: 500 mL.

5.2.1.2.2 Thermostatic Water Bath: Precision of Temperature Control ±0.1°C.

5.2.1.2.3 Density Bottle with Thermometer Attachment: 25 mL or 50 mL.

5.2.1.3 Preparation of Sample Solution

Use a clean and dry 100 mL volumetric flask to extract 100 mL of alcohol sample (solution temperature at 20°C) accurately into a 500 mL distillation flask. Use total 50 mL water to wash the volumetric flask 3 times and combine the washing solution together with the existing solution in the distillation flask. Add a few zeolites (or glass beads) and attach the flask with a condenser pipe. Use the original volumetric flask as the receiver (along with an ice bath) and start the cooling process (cooling water should be less than 15°C). Gradually increase the temperature and begin distillation, collect the distillates. When it is near full, detach the volumetric flask, cover it and keep its temperature maintained in a 20°C water bath for 30 mins. Add water to full, mix and prepare for use later.

5.2.1.4 Analysis Procedure

Wash the density bottle clean and heat it up repeatedly to dry it. Take weight after each attempt until its weight (m) stabilizes, i.e. does not change anymore.

Remove the cork that is attached with a thermometer and then fill the density bottle to full with water that had been previously boiled and then cooled to 15°C. Reattach the cork (ensure there is no air bubbles in the bottle), and immediately submerge the bottle into a thermostatic water batch at 20°C±0.1°C. Once temperature of the bottle's content reached and maintained at 20°C for 20 mins, swiftly removed any overflowing liquid from the sides of the tube with filter paper and immediately cover the small lid on the tube's side branch. Remove the density bottle, clean the external surfaces of the bottle of any liquid with filter paper and measure weight (m_1) immediately.

Pour away the water, use non-aqueous ethanol and then diethyl ether to wash the density bottle. Blow dry (or bake dry in an oven), then use test reagent (prepared as in 5.2.1.3) to wash the density bottle repeatedly for 3~5 times, and fill it up to full. Repeat abovementioned operation and weigh again (m_2).

5.2.1.5 Result Calculation

Density of sample solution at 20°C can be computed with the following formula (1) and (2).

$$\rho_{20}^{20} = \frac{m_2 - m + A}{m_1 - m + A} * \rho_0 \quad \text{..... (1)}$$

$$A = \rho_a * \frac{m_1 - m_2}{997.0} \quad \text{..... (2)}$$

In the formula:

ρ_{20}^{20} – Density of sample solution at 20°C, unit is gram per liter (g/L);

m_2 – Weight of density bottle with samples, unit is gram (g);

m – Weight of density bottle, unit is gram (g);

A – Air buoyancy correction value;

m_1 – Weight of density bottle with water, unit is gram (g);

ρ_0 – Density of distilled water in 20°C (998.20 g/L);

ρ_a – Density value of dry air at 20°C, 1013.25 hP_a (~1.2 g/L);

997.0 - Density deviation between that of distilled water and that of dry air at 20°C, unit is gram per liter (g/L).

According to the density of the sample solution ρ_{20}^{20} determined, refer to the corresponding alcohol content value of the sample listed in Appendix A at 20°C.

Result should be presented in one decimal place format.

5.2.1.6 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 0.5% range.

5.2.2 Digital Densimeter Method

5.2.2.1 Principle

Pour samples into a “U” shaped tube, compute the density value through the comparison of the vibration frequencies of the two standards at 20°C and thus calculate the fraction of volume of ethanol in the sample at 20°C, i.e. the alcohol content.

5.2.2.2 Apparatus

5.2.2.2.1 Digital Densimeter: Mettler/KEM DA-210 DMA 55D, with No.5771 connecting pipe, that can allow the sample to pass through the “U” shape tube continuously. Or use a digital densimeter with similar effect of analysis, then setting up, adjusting, calibrating and measuring according to specific instructions provided by its corresponding user manual.

5.2.2.2.2 Thermostatic Water Bath: Precision of temperature control ±0.01°C.

5.2.2.2.3 Injector: 10 mL, Lucr accessory No.15 needle.

5.2.2.3 Reagents and Solutions

Water: Redistilled water, filtered through a 0.2 μm film.

5.2.2.4 Instruments Calibration

5.2.2.4.1 Observe and make records of the “T” value of the air inside the “U” shaped tube (clean and dry) at temperature 20.00°C±0.01°C.

5.2.2.4.2 Connect the injector with no.15 needle with the plastic pipe at the outlet of the top end of the “U” shaped tube. Submerge the plastic pipe at the outlet of the bottom end of the “U” shaped tube in redistilled water that had previously been newly boiled, cooled and filtered. Inject the “U” shaped tube full with water (there should be no air bubbles) and then make records of the readings of the “T” value after the water temperature have stabilized at 20.00°C±0.01°C and the “T” value remained unchanged within a 2 min~3 min period.

5.2.2.4.3 Constants A, B of the apparatus can be computed with the following formula (3) and (4).

$$A = T_{\text{water}}^{2} - T_{\text{air}}^{2} \quad \text{..... (3)}$$

$$B = T_{\text{air}}^{2} \quad \text{..... (4)}$$

Input values of constants A, B into the memory unit of the apparatus. Adjust the switch to the ρ(density) position. Inspect density value of water. Pour out the water in "U" shaped tube, dry the tube and inspect the density value of air. The readings should be at 1.00000 (density of water) and 0.00000 (density of air) respectively. If discrepancy between the above stated value and the actual value reading in any of the 1st to 5th decimal place is more than 1, then the temperature of the thermostatic water bath and the "T" values of water and air will need to be reevaluated.

5.2.2.5 Analysis Procedure

Inject the "U" shaped tube (ensure there is no air bubbles) to full with sample solution (prepared as in 5.2.1.3), make records of the density of the sample solution after the temperature of the water bath and the sample solution had stabilized (2 min~3 min). Match the density result with the table in Appendix A to attain the alcohol content of the sample at 20°C.

Result should be presented in one decimal place format.

5.2.2.6 Precision

Discrepancies between the results of two independent tests conducted under iterative conditions should be smaller or equal to ±0.000 01.

5.2.3 Alcohol Meter Method

5.2.3.1 Principle

Use a precise alcohol meter to determine the fractional value of volume of alcohol and then match the reading with the table in Appendix B to make adjustment for temperature to attain the fractional volume of ethanol in the sample at 20°C, i.e. the alcohol content.

5.2.3.2 Apparatus

Precise Alcohol Meter: Graduation at 0.1%.

5.2.3.3 Analysis Procedure

Inject sample solution (prepared as in 5.2.1.3) into a clean and dry measuring cylinder and let it settle for a few minutes. Wait for the air bubbles in the alcohol disappears and then transfer the measuring cylinder into a clean and dry alcohol meter. Press lightly, ensuring that there is no contact with the walls of the measuring cylinder, inserting in the thermometer at the same time. Balance the cylinder for ~5 mins and visually observe the water level. Read off and make records of the scale value at the tangent of the meniscus. According to the reading of the alcohol meter and the temperature, match the reading with the table in Appendix B to make adjustment for temperature to attain the fractional volume of ethanol in the sample at 20°C, i.e. the alcohol content.

Result should be presented in one decimal place format.

5.2.3.4 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 0.5% range.

5.3 Alkalinity

5.3.1 Principle

Conduct neutralization titration with acid to determine the amount of basic substances (such as carbonate, bicarbonate) in sample.

5.3.2 Apparatus

5.3.2.1 Micro Burette: 5 mL.

5.3.2.2 Conical Flask: 250 mL.

5.3.3 Reagents and Solutions

5.3.3.1 Hydrochloric Acid Standard Solution [c(HCl) = 0.1mol/L]: Formulate and label according to guidelines specified in GB/T 601.

5.3.3.2 Hydrochloric Acid Standard Titration Reagent [c(HCl) = 0.05mol/L]: Dilute the abovementioned hydrochloric acid standard solution to half its initial concentration.

5.3.3.3 Methyl Red Indicator Solution (2 g/L): Formulate according to guidelines specified in GB/T 603.

5.3.4 Analysis Procedure

Extract 100 mL of sample solution (prepared as in 5.2.1.3) into a 250 mL conical flask and then add 2 drops of methyl red indicator solution. Titrate with hydrochloric acid standard titration reagent (prepared as in 5.3.3.2) with the mixture turning pink as an end point. Record the volume of titration solution used.

5.3.5 Result Calculation

Alkalinity of sample can be computed with the following formula (5).

$$X = \frac{V * c}{0.1} \quad \text{..... (5)}$$

In the formula:

X – Volume of 0.1 mol/L hydrochloric acid standard solution used in 100 mL of sample solution, unit is milliliter (mL);

V - Volume of hydrochloric acid standard titration reagent used, unit is milliliter (mL);

c – Concentration of hydrochloric acid standard titration reagent, unit is mol per liter (mol/L);

Result should be presented in one decimal place format.

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_0): 220°C.

Temperature of Sample Inlet (T_1): 220°C.

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 stabilized~~, 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).

$$f = \frac{A_1}{A_2} * \frac{d_2}{d_1} \quad \text{..... (6)}$$

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

$$X_1 = f * \frac{A_3}{A_4} * X_4 \quad \text{..... (7)}$$

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

~~$X_2 = \frac{X_1 * 100}{E}$ ..... (8)~~

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

$$X_3 = X_5 + X_6 * 0.37 \quad \text{..... (9)}$$

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_2 – Peak area ~~(or peak height)~~ of acetal during the determination of standard sample f value;

d_2 – ~~Relative concentration of acetal;~~ Concentration of acetaldehyde (or acetal) in standard sample, unit is milligram per liter of 100% ethanol (mg/L AA);

d_1 – ~~Relative concentration of internal standard;~~ Concentration of internal standard (ethanol) in standard sample, d_1 = 789270 mg/L;

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

A_3 – Peak area ~~(or peak height)~~ of acetaldehyde (or acetal) in sample;

A_4 – Peak area ~~(or peak height)~~ of internal standard added in the alcohol sample;

X_4 – Internal standard ~~(added in the alcohol sample)~~ (ethanol) content, unit is milligram per liter (mg/L), X_4 = 789270 mg/L;

X_2 – 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_5 – Acetaldehyde content in a liter of 100% ethanol in the sample, unit is milligram per liter (mg/L);

X_6 – 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

Sodium hydrogen sulfite will go through addition reaction with aldehyde, producing α-sodium phenolsulphonate in the process. Remove the excess sodium hydrogen sulfite by iodine oxidation. Add in excess amount of sodium bicarbonate into the resultant solution, so as to disintegrate the α-sodium phenolsulphonate and release sodium hydrogen sulfite. Titrate the resultant solution with iodine standard titration reagent.

5.4.2.2 Apparatus

Iodine Flask: 250 mL.

5.4.2.3 Reagents and Solutions

5.4.2.3.1 Hydrochloric Acid Solution [c(HCl) = 0.1mol/L]: Formulate in accordance with GB/T 601.

5.4.2.3.2 Sodium Hydrogen Sulfite Solution (12 g/L): Weigh and extract 6 g sodium hydrogen sulfite, dissolve in the water, fill up to 500 mL volume.

5.4.2.3.3 Sodium Bicarbonate Solution [$c(NaHCO_3) = 1mol/L$].

5.4.2.3.4 Iodine Standard Reagent [$c(1/2I_2) = 0.1mol/L$]: Formulate and label according to GB/T 601.

5.4.2.3.5 Iodine Standard Titration Reagent [$c(1/2I_2) = 0.1mol/L$]: Dilute the above iodine standard solution to 10% of its original concentration.

5.4.2.3.6 Starch Indicator Solution (10 g/L): Formulate in accordance with GB/T 601.

5.4.2.4 Preparation of Sample Solution

In the same way as section 5.2.1.3.

5.4.2.5 Analysis Procedure

Extract 30.0 mL sample solution (prepared as in 5.2.1.3) into a 250 mL iodine flask, add 15 mL sodium hydrogen sulfite solution (prepared as in 5.4.2.3.2), 7 mL hydrochloric acid solution (prepared as in 5.4.2.3.1), shake evenly and set it aside in a dark place for 1 hour. Remove from dark place, wash the flask stopper with a minute amount of water and titrate with iodine standard reagent (prepared as in 5.4.2.3.4). Upon nearing the end point of the test, add 0.5 mL of starch indicator solution and start titration with the iodine standard titration reagent (prepared as in 5.4.2.3.5) instead till the appearance of a pale blue color (recording not required). Add 20 mL sodium bicarbonate solution (prepared as in 5.4.2.3.3), open the stopper slightly and shake for 0.5 min (appears colorless). Use the iodine standard titration reagent (5.4.2.3.5) till the mixture turns purplish-blue as the end point. Conduct a control experiment concurrently.

5.4.2.6 Result Calculation

a) Total acetaldehyde content in the sample can be calculated with the following formula (10).

$$X_1 = \frac{(V_1 - V_2) * c * 22}{V} * 1000 \quad \text{..... (10)}$$

b) Total acetaldehyde content in a liter of 100% ethanol can be calculated with the following formula (11).

$$X_2 = \frac{X_1 * 100}{E} \quad \text{..... (11)}$$

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_2 – 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

specific exception that the standard sample used will be replaced by ethyl acetate solution (prepared as in 5.5.1.3.2) instead.

5.5.1.6 Result Calculation

Same as 5.4.1.6.

5.5.1.7 Precision

Same as 5.4.1.7.

5.5.2 Colorimetric Method

5.5.2.1 Principle

Esters and hydroxylamines will result in a quantitative reaction when added into basic solution, in the process producing hydroxamic acid. Hydroxamic acid in acidic solution will react with iron ions then to produce yellow complexes. Under certain alcohol concentration, light absorbance of ester concentration and the yellow complexes will be directly proportional to each other at a wavelength of 525 nm.

5.5.2.2 Apparatus

5.5.2.2.1 Spectrophotometer: Visible light range, cuvette at 1 cm.

5.5.2.2.2 All-glass Distillation Apparatus: Distillation Flask 500 mL.

5.5.2.2.3 All-glass Backflow Device: Conical flask 1000 mL, 250 mL (length of condenser pipe not shorter than 45 cm).

5.5.2.2.4 Colorimetric Tube with Stopper: 25 mL.

5.5.2.2.5 Micro Burette: 5 mL.

5.5.2.3 Reagents and Solutions

5.5.2.3.1 Hydroxylamine Hydrochloride Solution (2 mol/L): Weigh and extract 13.9 g hydroxylamine hydrochloride then dissolve in 100 mL water. Store in refrigerated container.

5.5.2.3.2 Sodium Hydroxide Solution [c(N_aOH) = 3.5 mol/L]: Formulate according to GB/T 601.

5.5.2.3.3 Hydrochloric Acid Solution [c(HCl) = 4 mol/L]: Formulate according to GB/T 601.

5.5.2.3.4 Ferric Chloride Solution [100 g/L]: Weigh and extract 50 g ferric chloride ($F_eCl_3 \cdot 6H_2O$), dissolve with 400 mL water, then add in 12.5 mL hydrochloric acid (prepared as in 5.5.2.3.3), lastly fill it to 500 mL with water (if precipitation is still observed in the mixture, filter before use).

5.5.2.3.5 40% Ethanol (Without Ester) Solution: Weigh and extract 600 mL 95% ethanol into 1000 mL conical flask and add in 5 mL sodium hydroxide solution (prepared as in 5.5.2.3.2). Perform heated backflow saponification on the mixture for 1 hour. Thereafter transfer the mixture into the distillation apparatus for re-distillation and then formulate into 40% ethanol solution.

5.5.2.3.6 Ethyl Acetate Standard Storage Reagent: Weigh and extract 0.1667 g ethyl acetate, fill it up to 500 mL with 40% ethanol solution. Formulated solution in this case should have 0.3334 mg of ethyl acetate per milliliter of the solution.

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. 2 mL N-butanol (chromatographically pure), then titrate it with 40% ethanol solution till it reaches 100 mL volume.~~

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.

~~5.6.3.3 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.

5.7.7 Precision

Same as 5.4.1.7.

6. Testing Methods

6.1 Batches

Products filled and manufactured during every shift, in the same product category, of same quality and specifications, packaged and meant for out-factory shipping will be classified under the same production batch.

6.2 Sampling

6.2.1 Draw samples (in boxes) according to guidelines listed in Table 3 and then draw unitary samples (in bottles) from random positions of each box. Sample quantity can be increased proportionally if unitary sample packaging net content is less than 500 mL or if total sampling volume does not meet the 1,500 mL mark.

Table 3 Sampling Table

Range of Batch Quantity / No. of Boxes	Sample Quantity / No. of Boxes	Unitary Sample Quantity / No. of Bottles
＜50	3	3
51~1,200	5	2
1,201~35,000	8	1
Above 35,000	13	1

6.2.2 After samples have been drawn, immediately label the samples with the following information indicated: sample name, product specifications, quantity, manufacturer name, sampling time and place, sampling personnel. Seal and safe keep 2 bottles of sample for the next 2 months for further reference. Other samples should be sent to the laboratory immediately for inspections on items such as sensory, physical-chemical, hygiene.

6.3 Inspection Classifications

6.3.1 Out-factory Inspection

6.3.1.1 Products should be inspected batch-by-batch by the manufacturing factory's internal quality supervision and inspection department according to this standard before out-factory shipping. If the products are qualified, qualified certification should be issued for the production batch before shipping out. The certificate can be placed in the packaging boxes or within individual product packaging. Alternatively, equivalence of stamping wordings like "qualified" or "qualified by inspection" on the labels or packaging boxes is also allowed.

6.3.1.2 Inspection items: Sensory requirements, alcohol content, alkalinity, total aldehyde, total ester, high quality alcohols.

6.3.2 Type Inspection

6.3.2.1 Inspection items: All inspection items required by this standard.

6.3.2.2 Under normal circumstances, type inspection for the same product category need only to be

conducted semiannually. Yet, type inspection should also be conducted if any of the following situations arises:

a) When there are significant changes in the main or supplementary ingredients used;

b) When key processes or equipment used changes;

c) When new products are being manufactured or when there is resumption of production after stoppage of routine production for 3 months or more;

d) When material discrepancies are observed between the results of the last type inspection and those of the out-factory inspection;

e) When it is specifically required by the State quality supervision and inspection institutions according to relevant regulations.

6.4 Judging Guidelines

6.4.1 If inspection results show that there are less than 2 test items (incl. 2) failing to meet the corresponding product requirements, a second round of inspection should be conducted on the same production batch, though with double the quantity of samples as compared to the first round. The basis of judging if this production batch is qualified will be the results of the second round of inspection and tests.

6.4.2 If there is still one (or more) item that fail to meet the corresponding requirement after the second round of inspection, the entire production batch will be deemed unqualified.

6.4.3 When there are disagreements on the results of the inspection between the supply and demand sides of the transaction, it can be resolved through negotiation between related parties or resolved by relevant authorities on behalf of related parties through the use of arbitration inspection methods, with the arbitration inspection results as the basis for the final decision.

7. Labelling, Packaging, Transportation & Storage

7.1 Labelling

7.1.1 Labels for prepackaged vodka products should comply with relevant requirements stipulated in the standard GB 10344.

7.1.2 Besides clearly indicating the product name, manufacturer name and address on the external packaging boxes, net content of unitary packaging and total quantity should also be shown clearly.

7.1.3 Transportation logos and shipping marks should comply with the requirements of GB/T 191.

7.2 Packaging

7.2.1 Packaging material should comply with food hygienic requirements.

7.2.2 Packaging container should be positioned upright, clean, tightly sealed with no signs of any leakage.

7.2.3 Qualified packaging materials should be used for external packaging, while there should be anti-shock buffers that comply with relevant standards incorporated within the container.

7.3 Transportation & Storage

7.3.1 Use cork (or similar substitutes) to seal the vodka bottles. The bottles should be stored and transported in an “upside down” or “lying down” position.

7.3.2 Products should be kept clean, avoiding any strong shaking, direct sunlight or rain and prevented from freezing during storage and transportation. They should be handled gently during loading and unloading.

7.3.3 Storage venues should be shady, cool, dry, well ventilated. Products should be kept strictly away from direct sunlight, rain or any potential fire hazard.

7.3.4 End products should not have direct contact with wet/moist floor and they should not be stored or transported together with poisonous, hazardous, foul-smelling and easily corrosive substances.

7.3.5 Temperature during transportation should be kept at 5^{o}C~35^{o}C, while storage temperature should be kept at 5^{o}C~25^{o}C.

Supplementary:

GB/T 11858-2008 Vodka

First Amendment Article of National Standard

This article of amendment was approved by the National Standardization Management Committee on the 27th, May 2009, and subsequently implemented on 1st, June 2009.

The specific content amended for the standard GB/T 11858-2008 *Vodka* is as follows:

Table 2 Physical-Chemical Index Requirements Covered in Section 4.2

Items		Superior Grade	First Grade	Second Grade
Total Ester (Ethyl Acetate) / [mg/L (100%vol Ethyl Alcohol)]	≤	4	6	8
Total Aldehyde (Acetaldehyde) / [mg/L (100%vol Ethyl Alcohol)]	≤	10	15	25

Amended to:

Table 2 Physical-Chemical Index Requirements

Items		Superior Grade	First Grade	Second Grade
Total Aldehyde (Acetaldehyde) / [mg/L (100%vol Ethyl Alcohol)]	≤	4	6	8
Total Ester (Ethyl Acetate) / [mg/L (100%vol Ethyl Alcohol)]	≤	10	15	25