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Natural fatty alcohols

天然脂肪醇

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Standardization Administration of the People's Republic of China.

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Natural fatty alcohols

1 Scope

This Standard specifies the product classification, requirements, test methods, inspection rules and marks, packaging, transportation and storage for natural fatty alcohols.

This Standard is applicable to a series of fatty primary alcohol products obtained from natural vegetable oils and their derivatives through alcoholysis or hydrolysis, esterification, hydrogenation and distillation.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 617, *Chemical reagent -- General method for the determination of melting range*

GB/T 6682, *Water for analytical laboratory use -- Specification and test methods*

GB/T 7383-2007, *Non-ionic surface active agents -- Determination of hydroxyl value*

GB/T 9282.1, *Clear liquids -- Estimation of colour by the platinum-cobalt scale -- Part 1: Visual method*

GB/T 11275, *Surface active agents -- Determination of water content*

QB/T 2739-2005, *Preparations of standard volumetric solutions of general test methods for washing products*

3 Product classification

Natural fatty alcohols are divided into ten categories according to the carbon chain length: $C_{8\sim10}$ alcohols, $C_{12\sim14}$ alcohols, $C_{14\sim16}$ alcohols, $C_{16\sim18}$ alcohols, C_8 alcohols, C_{10} alcohols, C_{12} alcohols, C_{14} alcohols, C_{16} alcohols, and C_{18} alcohols. Their molecular formulas and average relative molecular masses are shown in Table 1.

obtained under repeatability conditions shall not be greater than 0.4°C. The premise is that the temperature above 0.4°C does not exceed 5%.

5.4 Color

5.4.1 Specimen processing

The solid fatty alcohol specimen needs to be heated to 75°C±5°C. After it is completely melted, immediately pour it into a pre-warmed colorimetric tube. The liquid specimen also needs to be kept above the melting point to make it transparent and clear, and then placed in the colorimetric tube.

5.4.2 Determination

According to the provisions of GB/T 9282.1. The resulting platinum-cobalt color units are expressed in Hazen.

Precision: The absolute difference between two independent determination results obtained under repeatability conditions shall not be greater than 5 Hazen. The premise is that the case of greater than 5 Hazen does not exceed 5%.

5.5 Acid value

NOTE: The number of milligrams of potassium hydroxide required to neutralize the acidity of 1 g of fatty alcohol specimen is called the acid value.

5.5.1 Reagents

Reagents include:

a) Potassium hydroxide, c(KOH)=0.05mol/L ethanol standard titration solution, prepared and calibrated according to 4.2 in QB/T 2739-2005;

b) 95% ethanol, neutralized with alkali until it is neutral to phenolphthalein;

c) Phenolphthalein, 10 g/L ethanol solution.

5.5.2 Instruments

Commonly used laboratory instruments and the following:

a) Burette without stopper: 10 mL; graduation is 0.02 mL;

b) Erlenmeyer flask: 250 mL.

5.5.3 Test procedures

Weigh about 10 g of specimen (accurate to 0.001 g) and place in a 250 mL conical flask. Add 50 mL of neutral ethanol [5.5.1b)]. After heating to dissolve the specimen, add 2~3

V_2 - the volume of hydrochloric acid standard titration solution consumed for specimen titration, in milliliters (mL);

m_2 - the mass of the specimen, in grams (g);

56.11 - the millimolar mass of potassium hydroxide, in milligrams per millimole (mg/mmol).

Take the arithmetic mean of the two parallel determination results as the saponification value of the specimen.

5.6.5 Precision

The absolute difference between two independent determination results obtained under repeatability conditions shall not be greater than 0.2 mg/g. The premise is that the case of greater than 0.2 mg/g does not exceed 5%.

5.7 Iodine value

NOTE: The number of grams of iodine absorbed per 100 g of fatty alcohol specimen is called iodine value.

5.7.1 Reagents

Reagents include:

a) Carbon tetrachloride or chloroform;

b) Glacial acetic acid;

c) Iodine;

d) Potassium iodide: 100 g/L solution;

e) Sodium thiosulfate, $c(Na_2S_2O_3)$ = 0.1 mol/L standard titration solution: prepared and calibrated according to 4.12 in QB/T 2739-2005;

f) Starch indicator solution, 10 g/L solution: prepared according to 5.3 in QB/T 2739-2005;

g) Chlorine 98.8% or homemade: Add dropwise hydrochloric acid with a density of 1.19 g/L in potassium permanganate. The generated chlorine gas can be passed into the iodine solution after being dried through the scrubber bottle filled with sulfuric acid reagent (density is 1.84 g/L);

h) Iodine chloride, $c(\frac{1}{2}ICl)=0.2\ mol/L$ glacial acetic acid solution (Webster's solution).

Dissolve 16.2 g of iodine chloride in 1000 mL of glacial acetic acid. Or prepare as follows:

Weigh 13 g of iodine and dissolve it in 1000 mL of glacial acetic acid (it can be slightly heated as needed when dissolving). Put in a 1000 mL brown reagent bottle. Cover with a grinding stopper. After cooling, pour 100 mL ~ 200 mL into another brown bottle. Put the cap plug in a dark place for adjusting Wechsler's solution. In the remaining 800 mL ~ 900 mL of iodine solution, pass through the chlorine gas that has been dried in the concentrated sulfuric acid washing bottle until the solution gradually fades from dark color until it is orange-red and transparent. Method for inspecting chlorine gas intake and correction: Respectively sample 25 mL before and after chlorine gas flow. Add 20 mL of 15% potassium iodide solution [5.7.1d)] and 100 mL of water respectively. Use sodium thiosulfate standard titration solution [5.7.1e)] to titrate. When the solution turns light yellow, add 1 mL of starch indicator solution. Continue titration until the blue color disappears as the end point. The volume of sodium thiosulfate standard titration solution consumed after chlorine gas (Wechsler's solution) shall be nearly 2 times that when chlorine gas is not passed. If it exceeds 2 times, the pre-reserved iodine solution shall be added dropwise to adjust.

5.7.2 Instruments

Commonly used laboratory instruments and the following:

a) Iodine measuring bottle: 250 mL;

b) Pipette: 20 mL;

c) Graduated cylinder: 100 mL;

d) Unplugged burette (brown): 50 mL, with a division of 0.1 mL.

5.7.3 Test procedures

Accurately weigh 5 g of specimen (accurate to 0.001 g) in iodine bottle. Use a graduated cylinder to add 20 mL of carbon tetrachloride or chloroform to dissolve. Accurately pipette 20.0 mL of Wechsler's solution. Cork the bottle tightly. Add a small amount of potassium iodide solution to seal. After shaking slowly, place in dark place at room temperature for 60 min. Take out the iodine bottle. Add 25 mL of potassium iodide solution and 50 mL of water. Use 0.1 mol/L sodium thiosulfate standard titration solution to titrate. When the solution is light yellow, add about 1 mL of starch indicator solution. Continue titration until the blue color disappears as the end point.

At the same time, do a blank test under the same conditions.

If the sodium thiosulfate solution consumed by the specimen determination is less than half of the blank test, the specimen weight shall be reduced, and the measurement shall be repeated.

d) Supporting gas: secondary air purification;

e) Support Chromosorb T, or Chromosorb WAW DMCS, or 60-80 mesh 101 silanized white support;

f) Cyclohexane.

5.9.2 Instruments

5.9.2.1 Chromatograph, which has the following parts:

a) Detector: detection limit of hydrogen flame ionization detector (FID): $\leq 1\times10^{-10}$(n-C_{16});

b) Chromatographic column: a packed column or capillary column that can well separate components and impurities in fatty alcohols:

1) Packed column: stainless steel or glass column tube, with an inner diameter of about 2 mm ~ 4 mm and a length of 2 m, with a support [5.9.1e)] and a stationary phase coated with about 5% SE-30 or 3%~5% OV-101 stationary liquid, or other packed columns with equivalent performance;

2) Capillary column: with equivalent or better separation performance, for example: SE-30 or OV-101 (length is 30 m; inner diameter is 0.2 mm ~ 0.32 mm; film thickness is 0.2 μm~0.5 μm).

c) Data processor: chromatography workstation or recorder and electronic integrator.

5.9.2.2 Microsyringe: 1 μL, 5 μL.

5.9.3 Chromatographic analysis conditions

Select the chromatographic conditions according to the column used to obtain the best column efficiency.

The reference conditions of the packed column are as follows:

a) Column temperature: initial temperature is 120°C; heating rate is 4°C/min ~ 6°C/min; final temperature is 240°C; constant temperature is 170°C~200°C.

b) Vaporization chamber temperature: 250°C~300°C.

c) Detector temperature: 250°C~300°C.

d) Gas flow: 30 mL/min ~ 400 mL/min.

e) Assisted gas flow: 300 mL/min ~ 400 mL/min.

f) Carrier gas flow rate: 40 mL/min ~ 60 mL/min.

g) Specimen dilution: dilute according to the ratio of specimen to absolute ethanol at 1:3 (or 1:5).

h) Injection volume: 1 μL ~ 2 μL.

The reference conditions for the capillary column are as follows:

a) Vaporization chamber temperature: 250°C~300°C;

b) Column temperature: initial temperature is 100°C; heating rate is 4°C/min~6°C/min; final temperature is 250°C;

c) Detector temperature: 250°C~300°C;

d) Gas flow: 30 mL/min ~ 40 mL/min;

e) Gas flow rate: 300 mL/min ~ 400 mL/min;

f) Carrier gas flow rate: the flow rate in the column is 1 mL/min ~ 5 mL/min; the split ratio is 20:1~60:1;

g) Specimen dilution: dilute at the ratio of sample to n-pentane of 1:100;

h) Injection volume: 1 μL ~ 2 μL.

5.9.4 Test procedures

5.9.4.1 According to the instructions of the selected instrument, operate according to the chromatographic analysis conditions. It is required that the chromatographic peaks be separated and not overlapped.

5.9.4.2 Use the corrected area normalization method for quantitative analysis.

5.9.4.3 Chromatographic workstation or recorder, electronic integrator can be used to process chromatographic signal data. The minimum peak area is set to 0.

A typical chromatogram is shown in Figure 1.

According to the provisions of GB/T 11275.

5.11 Carbonyl value

NOTE: The total carbonyl content in fatty alcohol samples (calculated as C=O).

5.11.1 Principle

The carbonyl compound in the sample reacts with 2,4-dinitrophenylhydrazine in acidic medium. Generate more stable 2,4-dinitrophenylhydrazone. This product reacts with potassium hydroxide to form a red substance. There is a characteristic peak absorption at a wavelength of 530 nm. Measure the absorbance with a spectrophotometer to obtain the carbonyl compound content.

5.11.2 Reagents

Reagents include:

a) Carbonyl-free ethanol: Take 1500 mL of ethanol and place it in a 2000 mL distillation bottle. Add 15 g of 2,4-dinitrophenylhydrazine and 15 drops of concentrated hydrochloric acid. Reflux for 4 h. Place for more than 4 h. Then change the condenser to a dendritic rectification column for slow distillation. Discard about 100 mL of the initial effluent and about 200 mL of the remaining yellow solution. Collect middle distillate. Seal in a brown bottle. The distillate shall be clear, transparent and colorless, otherwise it shall be re-distilled.

b) Potassium hydroxide.

c) 2,4-Dinitrophenylhydrazine ethanol solution: Weigh 0.03 g of 2,4-dinitrophenylhydrazine. Add 40 mL of carbonyl-free ethanol solution and 0.3 mL of concentrated hydrochloric acid. Continue to dilute to 50 mL with carbonyl-free ethanol. Prepare it when it is needed.

d) Potassium hydroxide ethanol solution: Weigh 10 g of potassium hydroxide. Use 20 mL of distilled water to dissolve. After cooling, dilute to 100 mL with carbonyl-free ethanol.

e) Carbonyl compound standard solution (calculated as C=O), c=0.376 mg/mL: Weigh 0.172 g of 2-octanone. Dissolve it in about 50 mL of carbonyl-free ethanol. Transfer to a 100 mL volumetric flask. Use carbonyl-free ethanol to dilute to the scale. Shake well. This solution is prepared before use.

5.11.3 Instruments

Commonly used laboratory instruments and the following:

a) Spectrophotometer: with a wavelength of 530 nm and an absorbance accuracy of ±0.004(A).

b) Cuvette: 10 mm.

c) Water bath: the temperature can be kept at 60℃±1℃.

d) Volumetric flasks: 50 mL, 100 mL.

e) Pipettes: 1 mL, 2 mL, 5 mL, 10 mL.

f) Stoppered colorimetric tube: 50 mL.

5.11.4 Test procedures

5.11.4.1 Drawing of standard curve

The standard curve is drawn according to the following steps:

a) Respectively pipette 1.0 mL, 2.0 mL, 3.0 mL, 4.0 mL, 5.0 mL of carbonyl compound standard solution [5.11.2e)] into five 50 mL volumetric flasks. Use carbonyl-free ethanol to dilute to the scale. Shake well. Each 1.0 mL of this standard solution contains 7.5 μg, 15.0 μg, 22.5 μg, 30.0 μg, and 37.5 μg of carbonyl compounds, respectively.

b) Pipette 1 mL of standard colorimetric solution into 5 colorimetric tubes respectively [5.11.4.1a)]. Add 1 mL of carbonyl-free ethanol to each. Add 1 mL of 2,4-dinitrophenylhydrazine solution. Shake well and cap. At the same time pipette 1 mL of carbonyl-free ethanol into another colorimetric tube. Add the above reagent. Carry out a blank test under the same conditions. Heat the colorimetric tube in a water bath at 60°C±1°C (slightly loosen the stopper when heating, and reseal it after releasing excess pressure) for 30 min. Take out the colorimetric tube and cool to room temperature. Add 8 mL of potassium hydroxide-ethanol solution to each colorimetric tube. Cover the stopper. Shake well. Place at room temperature for 10 min ± 1 min. Use a cuvette with a 1 cm pathlength. Adjust the zero point of the spectrophotometer with a blank solution at 530 nm. Measure the absorbance of the above solution.

c) Use the absorbance of the standard colorimetric solution as the ordinate, the mass (μg) of the carbonyl compound in the corresponding standard colorimetric solution as the abscissa, to draw the standard curve.

5.11.4.2 Determination of samples

Accurately weigh 0.2 g ~ 0.5 g (accurate to 0.001g) of the fatty alcohol sample to be tested. Place it in the colorimetric tube. Add 1 mL of carbonyl-free ethanol. Operate as specified in [5.11.4.1b)]. At the same time pipette 1.0 mL of carbonyl-free ethanol for blank test. Use the standard curve to convert the measured net absorbance of the sample into carbonyl groups (μg).

If the specimen solution and blank solution appear turbid, they shall be filtered before

proposes to carry out type inspection;

f) When a customer proposes to carry out type inspection for the requirements of Table 3 in Chapter 4.

6.1.2 Exit-factory inspection

The requirements in Table 2 in Chapter 4 are all exit-factory inspection items.

6.2 Batching and sampling rules

6.2.1 Batching

Products are delivered and accepted in batches. Products of the same specification and same batch number delivered at one time constitute one delivery batch.

The products shall first be inspected by the quality inspection department of the manufacturer according to this Standard. Only products that meet this Standard and issue a quality inspection certificate can leave the factory. The product quality inspection certificate shall include manufacturer name, product name, trademark, adopted standard number, batch number, batch size, grade, quality index, production date, etc.

According to the quality certificate, the receiver shall take samples for acceptance or arbitration according to this Standard within one month.

6.2.2 Sampling

The sample size required for the acceptance inspection and arbitration inspection of the receiver shall be determined according to Table 4 according to the batch size of the product. The deliver and receiver will randomly select sample units from the delivery batch at the delivery location.

Table 4 -- Batch and sample size

The unit is barrel or bag or box

批量	≤15	16～25	26～90	91～150	151～500	501～1 200	≥1 201
样本	2	3	5	8	13	20	32

When collecting liquid samples, use a liquid sampler to insert two-thirds of the depth from the center of the packaging to collect samples. When collecting solid samples, use a sampler to insert two-thirds of the depth from the center of the packaging to collect samples. Draw an equal amount of sample from each sample unit. Make the total amount not less than 0.5 kg. Divide into three parts after mixing (solid fatty alcohol shall be melted and mixed). Subpack into three clean sample bottles. Cover and seal. Affix a label and indicate sample name, type, grade, batch number, manufacturer,

production date, sampling date, sampling person. The deliver and the receiver shall hold a copy for inspection respectively. The third copy is kept by the deliver for future arbitration inspection. The storage period is one month.

6.3 Judgment rules and re-inspection

For the test results of physical and chemical indicators, use the rounding value comparison method to determine whether the products are accepted or rejected. If one item fails, twice as many samples can be taken again to reinspect the failed item. If the re-inspection results comply with the provisions of this Standard, the batch of products is judged to be accepted. If it still fails, the batch of products will be judged as rejected.

6.4 Arbitration

If both parties to the delivery still have disputes over the re-inspection results, they can request arbitration for inspection according to this Standard. The arbitration result shall be the final basis.

7 Marks, packaging, transportation, storage

7.1 Marks

Each package of natural fatty alcohol and the accompanying quality certificate shall have the following marks:

a) Product name, trademark, type, grade and execution standard;

b) Net content and gross weight;

c) Production batch number or production date;

d) Words or logos such as waterproof and moisture-proof;

e) Manufacturer's name, address, zip code and contact number.

7.2 Packaging

Bulk fatty alcohol products are generally transported by tank truck or ISOTANK. Generally, fatty alcohol products are packaged in steel drums, composite plastic woven bags or cartons. The barrel mouth of the barreled product shall be sealed. The bagged or boxed product shall be well sealed.

When the receiver has special requirements, it shall be resolved through negotiation between the supplier and the purchaser.

All kinds of packaging shall be undamaged, clean and pollution-free. It shall be well sealed after packaging.

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