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GAIN Report

Global Agricultural Information Network

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China - Peoples Republic of

Post: Beijing

National Food Safety Standard on Methanol

Report Categories:

FAIRS Subject Report

Approved By:

Scott Sindelar

Prepared By:

M. Melinda Meador and Ma Jie

Report Highlights:

On November 18, 2011, China notified the WTO of National Food Safety Standard: Food Additive Methanol as SPS/N/CHN/507. This standard applies to food additive methanol made from coal, natural gas, light oil or heavy oil. It specifies the technical requirements and testing methods for food additive methanol. The date for submission of final comments to China is January 17, 2012. The proposed date of entry is to be determined. Comments can be sent to China's SPS Enquiry Point at sps@aqsiq.gov.cn. This report is an INFORMAL translation of this document.

General Information:
BEGIN TRANSLATION

GB National Food Safety Standard
GB XXXX—XXXX

National Food Safety Standard

Food Additive Methanol (Draft for Soliciting Opinions)

Date of Issue: XXXX-XX-XX

Date of Effectiveness: XXXX-XX-XX

Issued by the Ministry of Health of the People's Republic of China

National Food Safety Standard

Food Additive Methanol

1. Scope

This standard applies to the food additive Methanol synthesized by such raw materials as coal, natural gas, light oil and heavy oil.

2. Molecular formula, constitutional formula and relative molecular mass

2.1 Molecular formula

CH₃OH

2.2 Constitutional formula

H₃C-OH

2.3 Relative molecular mass

32.04 (according to international relative molecular mass in 2007)

3. Technical requirements

Organoleptic requirements: Appearance requirements: should conform to the requirements in table 1.

Table 1: Organoleptic Requirements

Item	Requirement	Method of inspection
Color	Colorless transparent	Apply adequate amount of sample onto a clean, dry and white porcelain dish and make a visual inspection under natural lighting
Odor	Featured by peculiar <u>odor</u>	
Texture	A liquid with low	

	viscosity	
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The physical and chemical indexes: shall conform to the requirements in Table 2.

Table 2. The physical and chemical indexes

Item	Index	Method of inspection
Methanol w/% \geq	99.5	A.4 in Appendix A
Moisture w/% \leq	0.1	Karl -Fischer method in GB5009.
Carbonyl compound (calculated by methanal) w/% \leq	0.010	A.5 in Appendix A
Distillation range / $^{\circ}$ C	64.5-65.5	GB/T75 4
Residue on evaporation / (mg/mL) \leq	3	GB/T6 24.2
Acidity(calculated by methanal) / (mg/kg) \leq	15	A.6 in Appendix A
Alkalinity(calculated by ammonia)/ (mg/kg) \leq	3	A.7 in Appendix A
Lead (Pb) / (mg/kg) \leq	2	GB 5009.12

Appendix A

Method of inspection

A.1 Cautions

Some of the testing processes as stipulated by the testing method may lead to dangerous situations, and adequate safety and protective measures shall be taken by the operator.

A.2 General Provision

Unless otherwise specified, only the reagents that have been identified as A.R and the Class III water as defined in GB/T6682-2008 can be used.

When no other requirement is noted, all the standard titration solution, the standard solution, preparation and products for determination of impurities that will be used by the testing method shall be prepared as prescribed in GB/T 601, GB/ T 602

and GB/T 603; unless otherwise specified, all the solutions to be used shall be aqueous solution.

A.3 Identification test

A.3.1 Dissolubility

It can be easily dissolved in water, ethyl and ethanol.

A.3.2 Refractive index

Determination is conducted as prescribed in GB/T614. The refractive index $n(20, D)$ shall be: 1.328 — 1.330.

A.3.3 Density

20

Determination is conducted as prescribed in GB/T4472. Density (ρ_{20}) shall be (0.790—0.793) g/cm³.

A.3.4 Boiling point

Determination is conducted as prescribed in GB/T 7534. Boiling point shall be about 65°C.

A.4 Determination of Methanol content

A.4.1 Summary of method

Under the selected operating condition, when the sample is boiled off to pass through the chromatographic column and the components are separated from each other, FID is used for determination. The methanol content is determined by area normalization method.

A.4.2 Reagents and materials

Standard solution: Dioxane solution containing 0.4% (V/V) methanol.

A.4.3 Instruments and devices

A.4.3.1 Gas chromatograph: it is provided with FID and the sensitivity and stability of the complete machine shall conform to relevant provisions in GB/T 9722;

A.4.3.2 Graphic instrument: chromatographic data processor or chromatographic work station;

A.4.3.3 Sample injector: micro-injector

A.4.4 Chromatographic column and typical chromatographic operating conditions

The recommended chromatographic column and typical Chromatographic operating conditions are shown in Table A.1. Other chromatographic column and Chromatographic operating conditions that can reach the equal degree of isolation may be used.

Table A.1 Recommended chromatographic column and typical Chromatographic operating conditions

Chromatographic column	Column length: 1.8 m, column inside diameter: 4 mm; filling is 120-150 mesh (105 μm ~120 μm) polar monomer copolymer of Styrene-divinylbenzene (Porapak R).
Column temperature/°C	160
Vaporizer temperature/°C	200
Detector temperature °C	210

Flow rate of carrier gas (nitrogen) (mL min)	25
Sample amount / μ L	5–10

A.4.5 Analysis steps

Adjust the Column temperature and /or the Flow rate of carrier gas to maintain the retention time about (5-7) min. Adjust the chromatographic peak produced from the 8 μ L standard solution to the mid position of the chromatogram range. Inject (5-10) μ L sample to obtain the chromatogram and the content of methanol is determined by using the normalization method.

A.4.6 Result calculation

The mass fraction of methanol is w , and the numerical value is indicated by %, which is calculated according to formula (A.1):

$$w = \frac{A_1}{\sum A_i} \times 100 \dots \dots \dots (A.1)$$

Where: A_1 = the peak area of methanol

$\sum A_i$ = the sum of the peak areas of all the components.

A.5. Determination of the content of the carbonyl compound

It is conducted according to the methods as stipulated in Article 4.2 in GB T 6324.5-2008, of which the wavelength is 430 nm, the carbonyl compound is taken w_3 of the mass fraction of formaldehyde (HCHO) and the numerical value is indicated by %, which is calculated according to formula (A.2):

$$W_3 = \frac{(m_1 - m_2) \times 10^{-6}}{m} \times 100 \dots \dots \dots (A.2)$$

Where:

m_1 = the mass of formaldehyde checked from the standard curve, corresponding to the absorbency of the sample, in the unit of micrograms (μ g);

m_2 = the mass of formaldehyde checked from the standard curve, corresponding to the blank absorbency, in the unit of micrograms (μ g);

m = the numeral value of the sample mass, in the unit of gram (g).

A.6 Determination of acidity

A.6.1 Reagents and devices

A.6.1.1 Ethanol

A.6.1.2 Standard titration solution of sodium hydroxide: $c(\text{NaOH}) = 0.02 \text{ mol/L}$

A.6.1.3 Phenolphthalein indicator solution: 10 g/L.

A.6.2 Analysis step

When blending 10 mL ethanol with 25 mL water, 0.5 mL Phenolphthalein indicator solution is add into, it is titrated by

standard titration solution of sodium hydroxide until the pink color appears first, keeping for at least 30 s. Add 19 mL about 15 g of the sample, mix them evenly, which is titrated by standard titration solution of sodium hydroxide until the pink color appears once again. The amount of standard titration solution of sodium hydroxide shall be no more than 0.25 mL.

A.7 Determination of alkalinity

A.7.1 Reagents and devices

A.7.1.1 Standard titration solution of sulphuric acid: $c(1/2\text{H}_2\text{SO}_4)=0.02$ mol/L

A.7.1.2 Methyl Red indicator solution: 1 g/L.

Add one drop of Methyl Red indicator solution into 25 mL water, which is titrated by standard titration solution of sulphuric acid until the pink color just appears, and then add 29 mL (about 22.5 g) of the sample, mix them evenly, which is titrated by standard titration solution of sulphuric acid until the pink color appears once again, where the amount of the standard titration solution of sulphuric acid shall be no more than 0.2 mL.

END TRANSLATION