



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 1886.68-2015

**National Food Safety Standard -- Food Additives -- Dimethyl
carbonate (also known as Viggo Ling)**

食品安全国家标准 食品添加剂 二甲基二碳酸盐 (又名维果灵)

(English Translation)

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Table of Contents

- 1 Scope
- 2 Chemical name, molecular formula, relative molecular mass and structural formula
 - 2.1 Chemical Name
 - 2.3 Formula
 - 2.4 Relative molecular mass
- 3 Technical requirements
 - 3.1 Sensory requirements
 - 3.2 Physical and Chemical Indicators

1 Scope

This standard applies to methyl chlorine and alkali solution as raw materials in the catalyst, the condensation reaction, separation and purification of the resulting food Dimethyl carbonate additive.

2.1 Chemical Name

Dimethyl carbonate

Formula 2.2

C₄H₆O₅

2.4 Relative molecular mass

134.0874 (according to 2007 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall comply with the requirements of Table 1.

Table 1 Sensory requirements

Project requires test methods Under color is colorless transparent liquid Take appropriate samples were placed in sealed colorless glass container, under natural light, observed Color

NOTE: The product has a pungent odor, not directly sniffing, fume hood operation need to be.

3.2 Physical and Chemical Indicators

Physical and chemical indicators should be consistent with the provisions of Table 2.

Table 2. Physical and chemical indicators

Item Index Test Method Dimethyl carbonate, w /% ≥ 99.8 Appendix A A.4 Dimethyl carbonate, w /% ≤ 0.2 A.5 Appendix A Lead (Pb)/(mg/kg) ≤ 2.0 GB 5009.12 Appendix A Testing method A.1 Warning Dimethyl carbonate to the eyes, skin and respiratory system, corrosive, inhalation or ingestion can cause poisoning. Operation should be in a well-ventilated Under good state, the operator should be good personal protection (such as wearing protective chemical safety glasses, rubber gloves, wear a filter type, if necessary Poison mask).

A.2 General Provisions Unless otherwise indicated, used in the analysis only identified as AR and above reagents, laboratory water should be consistent with GB/T 6682 Three Grade water requirements. Test methods used in the standard titration solution, impurities measured by standard solution, preparations and products, in the absence of other requirements specified when, according to GB/T 601, the provisions of the preparation of GB/T 602 and GB/T 603's. Solution was used in the tests did not indicate what is formulated with solvent when, it refers to an aqueous solution.

A.3 Identification Test A.3.1 solubility Soluble in toluene, methylene chloride and other organic solvents. Decompose rapidly after dilution in water to produce carbon dioxide and trace amounts of methanol.

A.3.2 IR spectrum

At 1130nm and 1800nm accessory area maximum absorption peak in the infrared spectrum in Appendix B of Figure B.1.

A.4 Determination of dimethyl carbonate A.4.1 Reagents and materials A.4.1.1 acetone.

A.4.1.2 dibutylamine dichlorobenzene solution. Weigh dibutylamine 120g, accurate to 0.01g, was dissolved in 1L chlorobenzene.

A.4.1.3 hydrochloric acid standard titration solution. $c(\text{HCl}) = 1\text{mol/L}$.

A.4.2 Instruments and Equipment Potentiometric titrator, Resolution. 1mV or higher.

A.4.3 Analysis step

Weigh 1.0g ~ 1.3g sample, accurate to 0.0001g, has been placed in the addition of about 70mL acetone 150mL beaker, added accurate 20mL dibutylamine dichlorobenzene solution, with stirring, titration with hydrochloric acid standard solution titration, potentiometric titration indicate the end. Meanwhile blank test.

A.4.4 Calculation Results Dimethyl carbonate mass fraction w_1 , according to formula (A.1)

Calculated.

$$(V_1 - V_2) \times c \times M$$

$$m \times 1000 \times$$

$$100\% \text{ (A.1)}$$

Where.

V₁ --- sample consumption hydrochloric acid standard titrant volume in milliliters (mL);

V₂ --- blank test volume of consumption hydrochloric acid standard titration solution, in milliliters (mL);

c --- hydrochloric acid standard titration solution concentration, in units of moles per liter (mol/L);

The M --- dimethyl carbonate molar mass in grams per mole (g/mol), [M (C₄H₆O₅) = 134.09];

M --- the quality of the sample, in grams (g);

1000 --- conversion factor.

A.5 Determination of dimethyl carbonate A.5.1 Reagents and materials Methyl isobutyl ketone.

A.5.2 Instruments and Equipment A.5.2.1 gas chromatograph. equipped with a flame ionization detector.

A.5.2.2 injector. with cool on-column injector type, or quartz micro-syringe needle (for manual injection).

A.5.3 Chromatography conditions Recommended columns and typical operating conditions are shown in Table A.1, approximate retention times of the components shown in Table A.2, dimethyl carbonate content determination A typical chromatogram Appendix B in Figure B.2. Others can achieve the same degree of separation columns and chromatographic operating conditions can be used.

Table A.1 column chromatography and typical operating conditions

Condition of the item Column length 50m, column diameter 0.3mm, coated with a capillary column SE30-D Temperature program the initial temperature 30 °C, maintained 5min, to 40 °C/min heating rate to 120 °C, final Temperature. 120 °C, maintained 5min the gasification chamber temperature/°C room temperature (about 25 °C) Detector temperature/°C 200 the carrier gas (helium) before the column pressure/kPa 300 Column flow rate/(mL/min) 2.0 Gas (hydrogen) flow rate/(mL/min) 35 Supporting gas (air) flow rate/(mL/min) 300 Make-up gas flow rate/(mL/min) 28 the injection volume/μL 2

Table A.2 approximate retention times of the components

Ingredient name Approximate retention times/min Dimethyl carbonate 5.5 Methyl isobutyl ketone 7.2 Dimethyl carbonate 8.8 A.5.4 Analysis step it weighs about 10g sample, accurate to 0.0001g, placed in a covered bottle. Adding a certain amount of methyl isobutyl ketone, and the expected carbonate Dimethyl content consistency (about 20mg, accurate to 0.1mg). Seal, mix thoroughly backward like 2μL.

A.5.5 Calculation Results Dimethyl carbonate mass fraction w_2 , according to equation (A.2) Calculated.

$$A \times m_1 \times f$$

$$A_1 \times m \times$$

$$100\% \text{ (A.2)}$$

Where.

A --- dimethyl carbonate peak area;

m_1 --- methyl isobutyl ketone mass, in milligrams (mg);

f --- correction coefficient dimethyl carbonate, the same mass of methyl isobutyl ketone and dimethyl carbonate (eg. each 20mg, accurate to 0.1mg), was added to a certain amount of dimethyl carbonate (eg. 10.0g), mix well, injection 2μL, gas chromatography Fixed, methyl isobutyl ketone and dimethyl carbonate ratio of peak area, dimethyl carbonate is a correction coefficient;

A_1 --- methyl isobutyl ketone peak area;

M --- the quality of the sample, in milligrams (mg).

Appendix B

Dimethyl carbonate and dimethyl carbonate IR spectra measured chromatograms B.1 dimethyl carbonate IR spectrum Dimethyl carbonate in the infrared spectrum shown in Figure B.1. Sample purity of 99.9%, directly on the sample in the optical system of the sample window, select Reflective mode test the resulting spectra.

Figure B.1 dimethyl carbonate in the infrared spectrum

B.2 dimethyl carbonate measured chromatogram Dimethyl carbonate measured chromatogram shown in Figure B.2.

Figure B.2 dimethyl carbonate measured chromatogram