

Attachment

Dimethyl Dicarbonate

二炭酸ジメチル

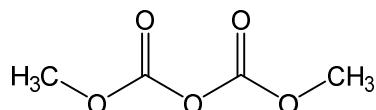
Standards for use

Dimethyl Dicarbonate is not permitted to use for food products other than fruit wines and nonalcoholic beverages (excluding mineral waters). The amount of Dimethyl Dicarbonate shall not be exceeding 0.25 g/kg in fruit wines (excluding grape wine) and nonalcoholic beverages, and not be exceeding 0.20 g/kg in grape wine.

Compositional specifications

Substance name Dimethyl Dicarbonate

Structural formula



Molecular formula C₄H₆O₅

Molecular weight 134.09

Chemical name [CAS number]

Dimethyl dicarbonate [4525-33-1]

Content Dimethyl Dicarbonate contains not less than 99.8% of dimethyl dicarbonate (C₄H₆O₅).

Description Dimethyl Dicarbonate is a colorless liquid.

Identification test

Determine the absorption spectrum of Dimethyl Dicarbonate as directed in the Liquid Film Method under Infrared Spectrophotometry, and compare with the Reference Spectrum. Both spectra exhibit similar intensities of absorption at the same wave-numbers.

Purity test

(1) Lead Not more than 1 µg/g as Pb (Electrothermal Method).

Test Solution Weigh accurately about 1.5 g of Dimethyl Dicarbonate into a container made of polyethylene, quartz, or hard-glass, and add 0.75 mL of nitric acid (for trace metals determination). Cover its container loosely, heat up gradually with stirring or occasional shaking, heat at 90°C to 30 minutes, and cool. Add dropwise 0.85 mL of hydrogen peroxide, heat at 95°C to 5–10 minutes with stirring or occasional shaking, and cool. Add dropwise hydrogen peroxide, heat in the same manner, and cool. Transfer this solution into a 25-mL volumetric flask, wash the container with a small amount of water, combine the washings with the solution, and add water to make 25 mL.

Standard Solutions Prepare four standard solutions with different concentrations. Transfer exactly 1, 2.5, 5, 10 mL of Lead Standard Solution into four separate 100-mL volumetric flasks, and to them, add a diluted solution (3 in 100) of nitric acid (for trace metals determination) to make exactly 100 mL.

Procedure Measure exactly a constant portion of the test solution and the standard solutions, and to each, add a quarter volume of a solution of magnesium nitrate hexahydrate (1 in 50), prepared before use. Analyze 25-µL portions of these solutions as directed in Lead Limit Test (Atomic Absorption Spectrophotometry) using the operating conditions given below. Determine the concentration (ng/mL) of lead in the test solution from the calibration curve prepared using the standard solutions. Calculate the amount of lead in the sample by the formula given below. Perform a blank test using the blank test solution prepared with water instead of Dimethyl Dicarbonate in the same manner as for the test solution, and make any necessary correction.

$$\text{Amount } (\mu\text{g/g}) \text{ of lead} = \frac{\text{Concentration } (\mu\text{g/mL}) \times 25}{\text{Weight } (\text{g}) \text{ of the sample}}$$

Operating Conditions

Light source: Lead hollow cathode lamp.

Wavelength: 283.3 nm.

Temperature for drying: A constant temperature of 200–250°C.

Temperature for incineration: A constant temperature of 700–750°C.

Temperature for atomization: A constant temperature of 1800–2000°C.

(2) Dimethyl carbonate Not more than 0.2%.

The procedure given here should be done as quickly as possible in order to avoid humidity.

Test Solution Weigh accurately about 5 g of Dimethyl Dicarbonate, add 0.5 mL of the internal standard solution, and add *tert*-butyl methyl ether to make exactly 5 mL.

Standard Solution Weigh accurately about 10 mg of dimethyl carbonate, add exactly 0.5 mL of the internal standard solution, and add *tert*-butyl methyl ether to make exactly 5 mL.

Internal Standard Solution Dissolve 50 mg of 3-pentanone in *tert*-butyl methyl ether to make exactly 5 mL.

Procedure Analyze 0.5- μ L portions of the test solution and the standard solution by gas chromatography using the operating conditions given below. Determine the peak area ratios (Q_T and Q_S) of dimethyl carbonate to 3-pentanone for the test solution and the standard solution, respectively. Determine the amount of dimethyl carbonate by the formula:

$$\begin{aligned} \text{Amount (\%)} \text{ of dimethyl carbonate (C}_3\text{H}_6\text{O}_3) \\ = \frac{\text{Weight (mg) of dimethyl carbonate}}{\text{Weight (g) of the sample} \times 1000} \times \frac{Q_T}{Q_S} \times 100 \end{aligned}$$

Operating Conditions

Detector: Flame ionization detector.

Column: A fused silica tube (0.53 mm internal diameter and 60 m length) coated with a 1.5 μ m thick layer of dimethylpolysiloxane for gas chromatography.

Column temperature: Maintain the temperature at 45°C for 7.5 minutes, raise at 10°C/minute to 75°C, then raise at 25°C/minute to 125°C, and maintain the temperature at 125°C for 2 minutes. Raise at 30°C/minute to 260°C, and maintain the temperature at 260°C for 4.5 minutes.

Detector temperature: 300°C.

Carrier gas: Helium.

Flow rate: Adjust so that the peak of 3-pentanone appears in 4–8 minutes after injection.

Injection method: Cold on-column injection method.

Assay The procedure given here should be done as quickly as possible in order to avoid humidity.

Weigh accurately 2 g of Dimethyl Dicarbonate, add 100 mL of acetone (dehydrated), and mix. To this solution, add exactly 20 mL of dibutylamine–toluene TS (1 mol/L), stir, and immediately titrate the excess dibutylamine with 1 mol/L hydrochloric acid using an automatic potentiometric titrator. Perform a blank test in the same manner, and determine the content of dimethyl dicarbonate by the formula:

Content (%) of dimethyl dicarbonate ($C_4H_6O_5$)

$$= \frac{(a - b) \times 0.1341}{\text{Weight (g) of the sample}} \times 100$$

a = volume (mL) of 1 mol/L hydrochloric acid consumed in the blank test.

b = volume (mL) of 1 mol/L hydrochloric acid consumed in the test.

Storage Standards Store in a hermetic container at 20–30°C.

Reagents, Solutions, and Other Reference Materials

Acetone (dehydrated) CH_3COCH_3 [67-64-1] A colorless, clear liquid.

Content Not less than 99.5% of acetone (CH_3COCH_3).

Specific gravity d_{20}^{20} : 0.788–0.792.

Water Not more than 0.001%.

Assay Analyze 0.2- μ L portions of acetone by gas chromatography using the operating conditions given below. Measure the peak area of each peak to determine the percentage of the main peak by the peak area percentage method.

Operating conditions

Detector: Flame ionization detector.

Column: A fused silica tube (0.53 mm internal diameter and 30 m length)

coated with a 5.0 μ m thick layer of dimethylpolysiloxane for gas chromatography.

Column temperature: Maintain the temperature at 40°C for 5 minutes, raise at 5°C/minute to 90°C, and maintain the temperature at 90°C for 2 minutes.

Injection port temperature: 150°C.

Detector temperature: 150°C.

Carrier gas: Helium.

Flow rate: 5 mL/min.

tert-Butyl Methyl Ether C₅H₁₂O [1634-04-4] A colorless liquid.

Content Not less than 99.5% of *tert*-butyl methyl ether (C₅H₁₂O).

Specific gravity d₂₀²⁰: 0.738–0.744.

Water Not more than 0.08%.

Assay Analyze 0.2-μL portions of *tert*-butyl methyl ether by gas chromatography using the operating conditions given below. Measure the peak area of each peak to determine the percentage of the main peak by the peak area percentage method.

Operating conditions

Detector: Flame ionization detector.

Column: A fused silica tube (0.53 mm internal diameter and 15 m length) coated with a 5.0 μm thick layer of a mixture of 5% phenyl/95% methylpolysiloxane for gas chromatography.

Column temperature: Maintain the temperature at 40°C for 10 minutes, raise at 20°C/minute to 260°C, and maintain the temperature at 260°C for 4 minutes.

Injection port temperature: 200°C.

Detector temperature: 260°C.

Carrier gas: Helium or Nitrogen.

Flow rate: A constant rate of about 4 mL/min.

Injection method: Split.

Split ratio: 1 : 50.

Dibutylamine C₈H₁₉N [111-92-2] A colorless, clear liquid.

Content Not less than 99.0% of dibutylamine (C₈H₁₉N).

Specific gravity d₂₀²⁰: 0.756–0.764.

Water Not more than 0.3%.

Assay Analyze 0.2-μL portions of dibutylamine by gas chromatography using the operating conditions given below. Measure the peak area of each peak to determine the percentage of the main peak by the peak area percentage method.

Operating conditions

Detector: Flame ionization detector.

Column: A fused silica tube (0.32 mm internal diameter and 25 m length) coated with a 1.2 μm thick layer of polyethylene glycol for gas chromatography.

Column temperature: Maintain the temperature at 60°C for 2 minutes, raise at 5°C/minute to 100°C, and maintain the temperature at 100°C

for 20 minutes.

Injection port temperature: A constant temperature of 150–170°C.

Detector temperature: 200°C.

Carrier gas: Nitrogen.

Flow rate: Adjust the retention time of dibutylamine to about 20 minutes.

Injection method: Split.

Split ratio: 1 : 80.

Dibutylamine–Toluene TS (1 mol/L) Dissolve 129.3 g of dibutylamine in toluene to make 1000 mL. Prepare before use.

Dimethyl Carbonate $C_3H_6O_3$ [616-38-6] A colorless to slightly light yellow liquid.

Content Not less than 98.0% of dimethyl carbonate ($C_3H_6O_3$).

Refractive index n_D^{20} : 1.365–1.372.

Water Not more than 0.2%.

Assay Analyze 0.2- μ L portions of dimethyl carbonate by gas chromatography using the operating conditions given below. Measure the peak area of each peak to determine the percentage of the main peak by the peak area percentage method.

Operating conditions

Detector: Flame ionization detector.

Column: A fused silica tube (0.32 mm internal diameter and 15 m length) coated with a 5.0 μ m thick layer of dimethylpolysiloxane for gas chromatography.

Column temperature: Maintain the temperature at 50°C for 10 minutes, raise at 20°C/minute to 250°C, and maintain the temperature at 250°C for 5 minutes.

Injection port temperature: 200°C.

Detector temperature: 260°C.

Carrier gas: Helium.

Flow rate: A constant rate of about 1.5 mL/min.

Injection method: Split.

Split ratio: 1 : 200.

Nitric Acid (for trace metal determination) HNO_3 [K8541, for trace metal determination] [7697-37-2] Use a reagent with a nitric acid concentration of 69–70%, unless otherwise specified.

3-Pentanone C₅H₁₀O [96-22-0] A colorless to light yellow liquid.

Content Not less than 98.0% of 3-pentanone (C₅H₁₀O).

Refractive index n_D²⁰: 1.390 – 1.396.

Water Not more than 0.2%.

Assay Analyze 0.2-μL portions of 3-pentanone by gas chromatography using the operating conditions given below. Measure the peak area of each peak to determine the percentage of the main peak by the peak area percentage method.

Operating conditions

Detector: Flame ionization detector.

Column: A fused silica tube (0.32 mm internal diameter and 15 m length) coated with a 5.0 μm thick layer of a mixture of 5% phenyl/95% methylpolysiloxane for gas chromatography.

Column temperature: Maintain the temperature at 70°C for 10 minutes, raise at 20°C/minute to 250°C, and maintain the temperature at 250°C for 6 minutes.

Injection port temperature: 250°C.

Detector temperature: 260°C.

Carrier gas: Helium.

Flow rate: A constant rate of about 1.5 mL/min.

Injection method: Split.

Split ratio: 1 : 300.

Infrared Reference Spectrum

