DIMETHYL DICARBONATE

Prepared at the 37th JECFA (1990), published in FNP 52 (1990). Metals and arsenic specifications revised at the 63rd JECFA (2004). Considered to be acceptable in accordance with GMP (max 250mg/l) at the 37th JECFA (1990)

SYNONYMS DMDC, dimethyl pyrocarbonate; INS No. 242

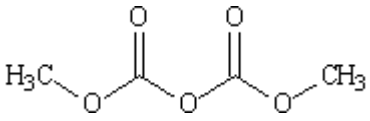
DEFINITION

Chemical names: Dimethyl dicarbonate

C.A.S. number: 004-525-33-1

Chemical formula: $C_4H_6O_5$

Structural formula:



Formula weight: 139.09

Assay: Not less than 99.8%

DESCRIPTION Colourless liquid.
Caution: Corrosive to eyes and skin and toxic by inhalation and ingestion; must be kept in a tightly sealed container to exclude moisture.

FUNCTIONAL USES Preservative

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4): Soluble in water with decomposition; miscible with toluene

Infrared absorption: The infrared spectrum of the sample corresponds with the reference infrared spectrum below

PURITY

Dimethyl carbonate: Not more than 0.2%
See description under TESTS

Lead (Vol. 4): Not more than 2 mg/kg
Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, “Instrumental Methods.”

TESTS

PURITY TESTS

Dimethyl carbonate

Apparatus

Gas chromatograph with a flame ionization detector, equipped for capillary gas chromatography, and inlet part for the "on column" technique (e.g., Carlo Erba Fraktovap 4160, or Hewlett-Packard 5880 or 5792).
- Capillary: 50 m SE 30-D (internal diameter: 0,3 mm).
- Potentiometric recorder, 1 mV.
- Microliter syringes with quartz needles, contents 0,010 ml, suitable for the "on column" technique (e.g., microliter syringe from SGE, type 5 A- SOC), (See Remark 5 below). Rolled rim glass vial, 10 ml content.
- Seal cap for the rolled rim vial with teflon coating.
- Closure tongs for the rolled rim vial
- Auxiliary for the peak area determination (e.g. HP calculator 3353).

Reagents

Methylisobutylketone, purest grade.

Operating conditions

Temperatures:
- Injection block: Room temperature (approximately 25^{o})
- Detector: 200^{o}
- Capillary Tube: Initial temperature 30^{o}; Preliminary phase 5 min, Rate of ascent 40^{o}/min; Final temperature 120^{o}, Final phase 5 min

Carrier gas:
- Preliminary pressure: 3.0 bar (Helium)
- Capillary tubes: Approx. 11 ml/min
- Make-up gas: 28 ml/min

Hydrogen: 35 ml/min
Air: 300 ml/min
Recorder feed: 1 cm/min

Procedure

Accurately weigh about 10 g of the sample to the nearest mg (W_1 mg) into a rolled rim vial. Add in a quantity of dimethyl-isobutylketone (W_2 mg), corresponding to the anticipated dimethylcarbonate content. Seal the rolled rim vial, mix well and inject 0.002 ml.

Retention times:
- Dimethylcarbonate: Approx. 2 min
- Methylisobutylketone: Approx. 4.5 min
- Dimethyldicarbonate: Approx. 8 min

Typical Chromatogram: See Figure below. Determine the peak areas of the standard (F2) and dimethylcarbonate (F1).

Calculation

Calculate the % of dimethyl carbonate from:

$$\frac{W2 \times F1 \times K \times 100}{F2 \times W1}$$

where
K = is a Correction Factor for Dimethylcarbonate
(See Remark 3, below)

Remarks
1. It should be noted the DMDC is sensitive to moisture and heat.
2. If so-called peak splits occur to some extent at the given gaschromatography conditions, then the peak areas are best determined by summation of the areas under both peaks.
3. The correction factor for dimethylcarbonate should be determined with corresponding test solutions in DMDC which is to the greatest extent possible free of dimethylcarbonate.
4. The specimen mixed with the standard must be measured immediately.
5. Hamilton syringes with Metal needles can result in a partial decomposition of the DMDC.

METHOD OF ASSAY

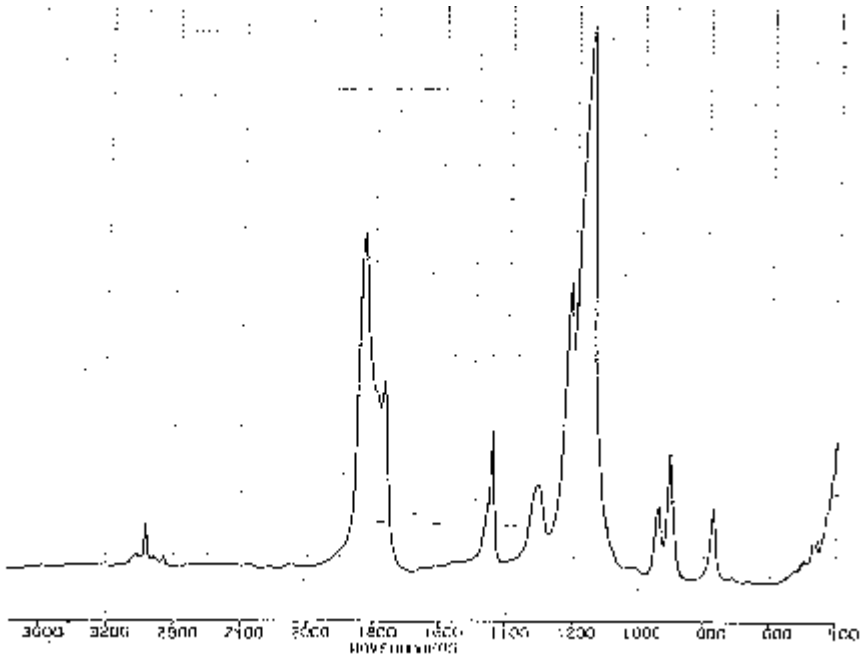
Introduce about 70 ml of pure acetone into a 150-ml glass beaker. Using a disposable 2 ml syringe weigh 1.0-1.3 g of the sample to an accuracy of ±0.1 mg into the glass beaker. Pipette exactly 20 ml dibutyl amine solution (add chlorobenzene to 120 g dibutyl amine until the 1 L mark is reached) while stirring. Titrate the solution potentiometrically with 1N hydrochloric acid. Run a blank test.

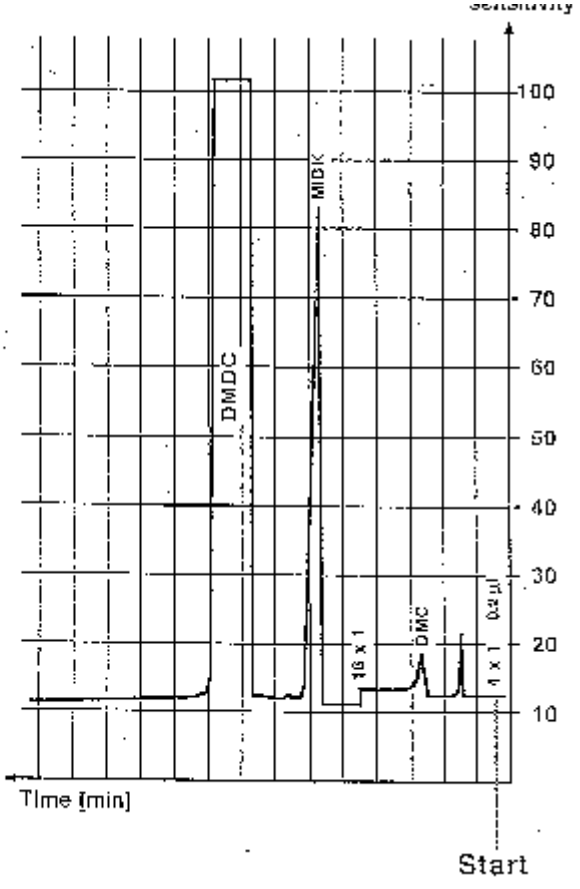
Calculate the % of dimethyldicarbonate from:

$$\frac{(V_2 - V_1) \times t \times 134.1 \times 100}{1000 \times W} = \frac{(V_2 - V_1) \times t \times 13.41}{M}$$

where
V_1 = amount of hydrochloric acid used for titration of the sample (ml)
V_2 = amount of hydrochloric acid used for titration of the blank (ml)
t = normality of hydrochloric acid
W = weight of sample (g)

Infrared spectrum Dimethyl dicarbonate





Typical chromatogram Dimethyl dicarbonate