RESOLUTION OENO 25/2004

DIMETHYL DICARBONATE (DMDC)

THE GENERAL ASSEMBLY,

IN VIEW of Article 2 paragraph iv of the Agreement of 3 April 2001 establishing the International Organisation of Vine and Wine,

UPON THE PROPOSAL by the Sub-Commission of methods of analysis and appraisal of wine,

DECIDES to add to the International Oenological Codex, the following monograph:

DIMETHYL DICARBONATE (DMDC)

Dimethyl pyrocarbonate

N° SIN = 242

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

EINECS	224-859-8
Chemical formula	$C_4H_6O_5$ H_3C-O-(C=O)-O-(C=O)-O-CH_3
Molecular weight	134.09

1. OBJECT, ORIGIN AND FIELD OF APPLICATION

Antiseptic mainly active against yeast. Synthetic product.

2. LABELLING

The name “Dimethyl dicarbonate”, the batch number, the date of expiry, storage temperature (20°C–30°C) and safety precautions must be indicated on the label.

3. CHARACTERISTICS

Colourless liquid that decomposes in an aqueous solution. Corrosive for skin and eyes.

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Toxic in case of inhalation and ingestion.
After dilution in water, CO_2 is formed which can be caracterised.
Melting point: 17°C.
Boiling point: 172°C with decomposition.
Density at 20°C: about 1.25.
Infrared spectrum: maximum absorption at 1156 nm and 1832 nm.

4. CHARACTERISATION

4.1. Principle of the method

The sample is mixed with an excess of dibutylamine with which it reacts directly. The excess of amine is determined by back titration.

4.2. Apparatus

4.2.1. 150 ml cylindrical vase

4.2.2. 100 ml graduated test tube

4.2.3. 20 ml pipette

4.2.4. Glass electrode/reference electrode

4.2.5. pH metre

4.2.6. 20 ml plunger burette

4.2.7. Magnetic stirrer

4.2.8. 2 ml disposable syringe.

4.3. Reagents

4.3.1. Pure acetone

4.3.2. Dibutylamine solution [$C_8H_{19}N$] = 1 mole/l

Weigh 128 g of dibutylamine into a 1 l volumetric flask and fill to the mark with chlorobenzene.

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4.3.3. Molar hydrochloric acid solution [HCl] = 1 mole/l

Determine the mass concentration by titration with sodium carbonate. Titre: t

4.3.4. Anhydrous sodium carbonate, dried in incubator at 110°C.

4.4. Procedure

Pour about 70 ml of acetone (4.3.1) in a 150 ml cylindrical vase.

Place a cylindrical vase (4.2.1) and introduce 1.0 to 1.3 g (W) of sample by using a disposable syringe (4.2.8) (precision of ± 0.1 mg).

Add exactly 20 ml of the dibutylamine solution (4.3.2) using a pipette (4.2.3) and shake vigorously.

4.4.1. Titrate by potentiometry the excess of amine with hydrochloric acid (4.3.4).

Consumption of HCl solution = V1 ml.

4.4.2. Perform a control trial according to 4.4 but without adding the sample.

Consumption of HCl solution = V2 ml.

4.5. Result

$$\frac{(V2-V1)\,.\,t\,.\,134.1\,.\,100}{1000\,.\,W} = \frac{(V2-V1)\,.\,t.\,13.41}{W} = \%\ dimethyl\ dicarbonate$$

DMDC content should be more than or equal to 99.8%.

5. DMDC HEAVY METAL, CONTENT (EXPRESSED IN LEAD), MERCURY AND CHLORIDE

5.1. Buffer solution

pH= 3.5 Dissolve 6.25 of ammonium acetate in 6 ml water. Add 6.4 ml of hydrochloric acid and dilute water to 25 ml.

5.2. Solution for trials:

Pour 5 ml of buffer solution in a conical flask, 25.0 g of sample and approximately 15 ml of water. Let the sample hydrolyze for 3 days, shaking from time to time. Transfer the

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solution to a 50 ml graduated cylinder and fill up with water to indicator.

5.3. Heavy metals

Determine the heavy metal content according to the method in chapter II of the International

Oenological Codex.

The contents of heavy metals must be less than 10 mg/kg.

5.4. Mercury

Using the solution for trials (5.2) measure the mercury according to the method in chapter II of

the International Oenological Codex.

The contents of mercury must be less than 1 mg/kg.

5.5. Chloride

Using the trial solution 5.2 (diluted two times compared to initial contents) measure the chloride

according to the method in chapter II of the International Oenological Codex.

The contents of chloride must be less than 3 mg/kg.

6. DETERMINATION OF ARSENIC, LEAD AND CADMIUM BY ATOMIC ABSORPTION SPECTROMETRY

6.1. Preparation of the test trial solution

For the determination of arsenic, lead, and cadmium.

Weigh about 100 g of the sample with a precision of ▫ 0.1 g in a cylindrical vase.

Add 200 ml of water and 5 ml of pure sulphuric acid (R) and concentrate on a hot plate until the first vapours of sulphuric acid appear.

Re-dilute the solution with water and add 1 ml of pure hydrochloric acid (R). Pour while washing into a 50 ml volumetric flask and bring to mark.

6.2. Arsenic

Using the trial solution (6.1) determine the arsenic content according to the method in chapter II of the International Oenological Codex.

Arsenic content should be less than 3 mg/kg.

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6.3. Lead

Using the trial solution (6.1), determine the lead content according to the method in chapter II of the International Oenological Codex.

Lead content should be less than 5 mg/kg.

6.4. Cadmium

Using the trial solution (6.1), determine the cadmium content according to the method in chapter II of the International Oenological Codex.

Cadmium content should be less than 0.5 mg/kg.

7. DETERMINATION OF DIMETHYL CARBONATE

Dimethyl carbonate content should be less than 0.2%.

7.1. Principle of the method

The concentration of dimethyl carbonate is determined by chromatography in gaseous phase. The quantitative evaluation is performed by using methyl-isobutylcetone as an internal standard.

7.2. Apparatus

7.2.1. Chromatograph in gaseous phase with a flame ionisation detector and capillary column (apolar type "SE 30" or other; a polar column can also be used such as the Carbowax type 20 M), 50 m x 0.3 mm.

7.2.2. Data acquisition system.

7.2.3. A 10 µl quartz needle syringe suitable for an on column injection (injection "on column" (cf. remark 7.7).

7.2.4. 10 ml antibiotic flask with a Teflon stopper that can be sealed with a aluminium capsule with the top part that can be torn off.

7.3. Internal standard

Ultra pure methyl-isobutylcetone.

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7.4. Procedure

7.4.1. Weigh about 1 g of the sample at ± 1 mg (W1 mg) in a flask 7.2.4.

7.4.2. Add a quantity of internal standard (W2 mg) of methyl-isobuthylcetone (7.3) corresponding to 10 mg/kg after addition (10 µl for example).

7.4.3. Seal the flask, mix vigorously and inject 0.2 µl.

7.4.4. Determine the peak area corresponding to the internal standard (F 2) and corresponding to dimethyl carbonate (F 1).

7.5. Result

$$\frac{W2.\ F1\ .\ K\ .\ 100.}{F2\ .W1} = \%mass\ of\ dimethyl\ carbonate$$

K = Factor for the dimethyl carbonate calculated using reference solutions of this substance preferably prepared in DMDC free from dimethyl carbonate.

7.6. Remark 1

The sample prepared with the standard should be immediately analysed.

7.7. Remark 2

A partial decomposition of DMDC can occur when in contact with the metal needles of traditional syringes.

8. STORAGE

The DMDC must be stored in perfectly watertight containers at a temperature between 20°C and 30°C. Its shelf life is 12 months.

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