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Methylene Chloride

CH_2Cl_2 84.93
Methane, dichloro-;
Dichloromethane CAS RN®: 75-09-2.

DEFINITION

Methylene Chloride contains NLT 99.0% of methylene chloride (CH_2Cl_2). [**Caution—**Perform all steps involving evaporation of methylene chloride in a well-ventilated fume hood.]

IDENTIFICATION

• **A.**

Sample: 5 mL

Analysis: Place the *Sample* into a glass-stoppered, 10-mL conical flask, and shake for several min. Remove the stopper, quickly withdraw a portion of the vapor into a 50-mL syringe that is not fitted with a needle, and inject the vapor into a suitable evacuated gas cell.

Acceptance criteria: The IR absorption spectrum of the vapor shows strong doublet peaks at 7.8 and 7.9 µm and at 13.2 and 13.4 µm, and relatively few minor peaks.

ASSAY

• **Procedure**

System suitability solution: Methylene chloride and chloroform (3:7)

Chromatographic system

(See *Chromatography ⟨621⟩, System Suitability*.)

Mode: GC

Detector: Thermal conductivity (under typical conditions)

Column: 4-mm × 1.8-m; packed with 15% liquid phase G18 on 30- to 60-mesh S1C unsilanized support

Temperatures

Injection port: 200°

Detector: 250°

Column: 60°

Carrier gas: Helium

Flow rate: 20 mL/min

Injection volume: 1 µL

System suitability

Sample: *System suitability solution*

Suitability requirements

Resolution: NLT 4.0 between methylene chloride and chloroform

Tailing factor: NMT 1.4

Relative standard deviation: The peak response ratio does not exceed 2% for five replicate injections.

Analysis

Sample: Methylene Chloride

Inject the *Sample*, and determine the peak responses by any convenient means. [Note—The order of elution is amylenes (5 or 6 peaks), if present, and then methylene chloride.]

Calculate the percentage of methylene chloride (CH_2Cl_2) in the portion of sample taken:

$$\text{Result} = (r_U/r_T) \times 100$$

r_U = peak response of methylene chloride

r_T = sum of all the peak responses

Acceptance criteria: NLT 99.0%

IMPURITIES

• **Limit of Nonvolatile Residue**

Sample: 50 g

Analysis: Evaporate the *Sample* in a platinum or porcelain dish on a steam bath, and dry at 105° for 30 min.

Acceptance criteria: NMT 0.002%; NMT 1 mg of residue

SPECIFIC TESTS

• **Limit of Hydrogen Chloride**

Sample: 20.0 mL

Analysis: Into each of two glass-stoppered, 50-mL color-comparison cylinders having an internal diameter of 20 mm, place 10 mL of water, 2 drops of phenolphthalein TS, and sufficient 0.010 N sodium hydroxide to produce a pink color that persists after vigorous shaking for 30 s and is of equal intensity in each cylinder.

[Note—In the following step, take special care to avoid contamination with carbon dioxide.]

Into one of the cylinders, place the *Sample* and 0.70 mL of 0.010 N sodium hydroxide, and shake again.

Acceptance criteria: NMT 0.001%; the pink color in the sample cylinder is at least as intense as that in the comparison cylinder, and the color persists for NLT 15 min.

• **Specific Gravity ⟨841⟩:** 1.318–1.322

• **Water Determination, *Method I* ⟨921⟩:** NMT 0.02%

• **Free Chlorine**

Sample: 10 mL

Analysis: To the *Sample* add 10 mL of water and 0.1 mL of potassium iodide TS, shake for 2 min, and allow the liquids to separate.

Acceptance criteria: The lower layer does not show a violet tint.

ADDITIONAL REQUIREMENTS

• **Packaging and Storage:** Preserve in tight containers.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
METHYLENE CHLORIDE	Documentary Standards Support	SE2020 Simple Excipients
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SE2020 Simple Excipients

Chromatographic Database Information: Chromatographic Database

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