

|                     |  |                |                                  |
|---------------------|--|----------------|----------------------------------|
| Rev.12              | <b>METHYLENE CHLORIDE Ph.Eur<br/>SPECIFICATION</b> |                | <b>Reference<br/>Ph.Eur 11.0</b> |
| FG Specification No | Supersedes   | Effective date | Page No                          |
| QC/PH-FG/SPEC/14    | Rev.11   | 01/01/2023     | 1 of 6                           |

| Sr.No. | Test   | Specification  | Method of Analysis No. |
|--------|--|--|------------------------|
| 1      | Appearance   | Clear, colourless, volatile liquid.  | QC/PH-FG/SPEC/14-01    |
| 2      | Solubility   | Sparingly soluble in water, miscible with ethanol (96%).   | QC/PH-FG/SPEC/14-02    |
| 3      | Identification<br>A) Refractive index at 20°C ± 0.5°C  | 1.423 to 1.425 at 20°C ± 0.5°C   | QC/PH-FG/SPEC/14-03    |
|        | B) By IR   | The spectrum obtained with the sample corresponds in position & relative intensity to those in the spectrum obtained with that of Methylene chloride CRS or its Working standard. (Purity index should not be less than 0.99). | QC/PH-FG/SPEC/14-04    |
|        | C) Relative density at 20°C  | 1.320 to 1.332 at 20°C.  | QC/PH-FG/SPEC/14-07    |
|        | D) Chemical Test   | A violet colour is produced.   | QC/PH-FG/SPEC/14-12    |
|        | E) Chemical Test   | Passes the test  | QC/PH-FG/SPEC/14-13    |
| 4      | Appearance of solution   | The substance to be examined is clear and colourless   | QC/PH-FG/SPEC/14-05    |
| 5      | Acidity  | Not more than 0.15 ml of 0.1M sodium hydroxide should require to change the colour of the indicator to blue.   | QC/PH-FG/SPEC/14-06    |
| 6      | Relative density at 20°C   | 1.320 to 1.332 at 20°C   | QC/PH-FG/SPEC/14-07    |
| 7      | Refractive index at 20°C ± 0.5°C   | 1.423 to 1.425 at 20°C ± 0.5°C   | QC/PH-FG/SPEC/14-03    |
| 8      | Ethanol, 2-methylbut-2-ene and volatile impurities (By GC)<br>Ethanol<br>2-methylbut-2-ene<br>Impurity A (Carbon tetrachloride)<br>Impurity B (Chloroform)<br>Total of impurities other than ethanol and 2-methylbut-2-ene | Maximum 2.0% v/v<br>Maximum 300 ppm v/v<br>Maximum 10 ppm v/v<br>Maximum 50 ppm v/v<br>Maximum 0.1% v/v  | QC/PH-FG/SPEC/14-08    |

|           |               |            |                 |
|-----------|---------------|------------|-----------------|
|           | PREPARED BY   | CHECKED BY | AUTHORIZED BY   |
|           | Q.C           | Q.C        | Q.A             |
| SIGNATURE | <i>Uphabe</i> | <i>Q.C</i> | <i>Pspandit</i> |
| DATE      | 26/12/2022    | 27/12/2022 | 27/12/2022      |

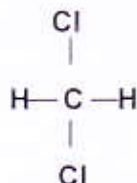
| Rev.12              |   | METHYLENE CHLORIDE Ph.Eur<br>SPECIFICATION   |                     | Reference<br>Ph.Eur 11.0 |
|---------------------|---|--|---------------------|--------------------------|
| FG Specification No | Supersedes  | Effective date   | Page No             |                          |
| QC/PH-FG/SPEC/14    | Rev.11  | 01/01/2023   | 2 of 6              |                          |
| 9                   | Free Chlorine   | No blue colour should develop.   | QC/PH-FG/SPEC/14-09 |                          |
| 10                  | Residue on evaporation  | Maximum 20 ppm.  | QC/PH-FG/SPEC/14-10 |                          |
| 11                  | Water   | Maximum 0.02% m/m.   | QC/PH-FG/SPEC/14-11 |                          |
| 12                  | Residual solvents (By GC)<br>a) Methanol<br>b) Ethanol<br>c) 2-Methyl-2-butene<br>d) Chloroform<br>e) Carbon tetra chloride | Not more than 3000 ppm v/v<br>Not more than 5000 ppm v/v<br>Not more than 300 ppm v/v<br>Not more than 60 ppm v/v<br>Not more than 4 ppm v/v | QC/PH-FG/SPEC/14-14 |                          |

**REFERENCE COPY**



|   |  |                              |                                  |
|---|--|------------------------------|----------------------------------|
| Rev.12                                  | <b>METHYLENE CHLORIDE Ph.Eur<br/>SPECIFICATION</b> |                              | <b>Reference<br/>Ph.Eur 11.0</b> |
| FG Specification No<br>QC/PH-FG/SPEC/14 | Supersedes<br>Rev.11                               | Effective date<br>01/01/2023 | Page No<br>3 of 6                |

### GENERAL INFORMATION

**Structure:**

**Molecular Formula:** CH<sub>2</sub>Cl<sub>2</sub>
**Molecular Weight:** 84.9

**Desirable Pack:**

To be supplied in MS drums/SS containers/HDPE containers/HM-HDPE containers and Glass bottles. properly identified with a label having Name of the material, Name of the Manufacturer, Quantity, Manufacturer's Batch Number, Manufacturing Date, Expiry Date and or Retest Date.

**Storage Condition:**

In an airtight container, protected from light.

**Handling precaution:**

Use PPE (Personal Protective Equipments) during handling of the material.

**Sampling SOP:**

As per the current approved sampling procedure. (SOP/QC/GE/01)

**Quantity to be sampled:**

Analysis Sample: About 575 ml

Control Sample: About 150 ml

Stability Sample: About 6420 ml

**Shelf Life:**

Three years from the date of manufacturing.

**Note:**
**1. For Bullet, Filter, Supporting equipments rinsing and Filter Cleaning-**

- If previous product is any grade of Methylene chloride, then perform Appearance, Solubility, Identification and Water tests as per FG specification.
- If previous product is different then, then perform Appearance, Solubility tests as per FG specification and calculate previous product carry over by using "Purity (By GC)" method from Raw material specification. (Limit - NMT 0.2%) QC/SPEC/MDC\_RM/01

**2. For Tanker Rinsing-**

- Perform Appearance, Solubility, Identification, Relative density at 20°C, Ethanol, 2-methylbut-2-ene and volatile impurities (By GC), Residue on evaporation and Water tests as per FG specification.

**3. Bending and Packing-**

- Perform all tests as per FG specification.
- Residual solvents (By GC) to be perform only for Packing.

REFERENCE COPY

|                     |  |                |                                  |
|---------------------|--|----------------|----------------------------------|
| Rev.12              | <b>METHYLENE CHLORIDE Ph.Eur SPECIFICATION</b> |                | <b>Reference<br/>Ph.Eur 11.0</b> |
| FG Specification No | Supersedes                                     | Effective date | Page No                          |
| QC/PH-FG/SPEC/14    | Rev.11   | 01/01/2023     | 4 of 6                           |

**4. For Stability testing-**

- Perform Appearance, Solubility, Identification, Relative density at 20°C, Ethanol, 2-methylbut-2-ene and volatile impurities (By GC), Residue on evaporation, Water and Residual solvents (By GC) tests as per FG specification. (Stability sample quantity- About 535 ml for single analysis).

| Document number  | Supersedes | Changes made   | Reason for change   |
|------------------|------------|--|---|
| QC/PH-FG/SPEC/14 | Rev.0      | Format change –<br>1. General Information added.<br>2. History page added.<br>3. Reference updated   | As per requirement of Schedule M.<br><br>Ph.Eur-7.0   |
|                  | Rev.1      | RUNA Logo inserted along with name of company.   | As per SOP of Document and Data control   |
|                  | Rev.2      | 1. Reference updated<br>2. Texts revised<br>3. Detector and injector temperature is decreased from 300°C to 230°C and 260°C to 230°C respectively.   | Ph.Eur-8.0<br>Ph.Eur 8.3<br>(Supplement)<br>Refer change control No. RCPL/CC/QC/004-15  |
|                  | Rev.3      | 0.25mm x 60m fused silica column coated with a 1.4µm film of phase G43 used. Column temp. Program is change. Split ratio and column flow increased from 1:40 to 1:50 and 1.0mL/min to 1.07mL/min respectively. Carrier gas Helium is used instead of Nitrogen. | (Refer change control No.RCPL/CC/QC/009-15)<br>(Refer Method validation protocol No. RCPL/QC/VAL-Methylene chloride-BP & Ph.Eur/2015) |
|                  | Rev.4      | Reference updated  | Ph.Eur 9.0<br>(Refer change control No. RCPL/CC/QC/009-16)  |
|                  | Rev.5      | For Ethanol, 2-methylbut-2-ene and volatile impurities (By GC) test- Split Ratio is reduced from 50 to 30  | (Refer change control No. RCPL/CC/QC/004-17)  |
|                  | Rev.6      | 1. Reference updated.<br>2. Mentioned tests to be perform for Bullet Rinsing, Filter Rinsing and Tanker Rinsing.<br>3. Shelf Life is added.  | Ph. Eur -9.5<br>Suppliment.<br>Refer Change Control No. RCPL/CC/QC/008-18   |
|                  | Rev.7      | 1. Mentioned tests to be perform for Supporting equipments<br>2. Quantity of Stability sample is added.  | Refer Change Control No. RCPL/CC/PDN/003-18   |

REFERENCE COPY



|   |  |                              |                                  |
|---|--|------------------------------|----------------------------------|
| Rev.12                                  | <b>METHYLENE CHLORIDE Ph.Eur<br/>SPECIFICATION</b> |                              | <b>Reference<br/>Ph.Eur 11.0</b> |
| FG Specification No<br>QC/PH-FG/SPEC/14 | Supersedes<br>Rev.11                               | Effective date<br>01/01/2023 | Page No<br>5 of 6                |

|  |       |   |   |
|--|-------|---|---|
|  | Rev.8 | Test wise method of analysis (MOA) is prepared.<br>Method of Analysis No. is added.   | Refer change control No. RCPL/CC/QC/003-19                      |
|  | Rev.9 | <ol style="list-style-type: none"> <li>1. Reference updated.</li> <li>2. In MOA No.QC/PH-FG/SPEC/14-01               <ol style="list-style-type: none"> <li>a) Procedure is updated.</li> <li>b) interpretation is added.</li> </ol> </li> <li>3. In MOA No.QC/PH-FG/SPEC/14-02               <ol style="list-style-type: none"> <li>a) Procedure is updated.</li> <li>b) interpretation is added.</li> </ol> </li> <li>4. In MOA No.QC/PH-FG/SPEC/14-03               <ol style="list-style-type: none"> <li>a) "Limit" is added.</li> </ol> </li> <li>5. In MOA No.QC/PH-FG/SPEC/14-04               <ol style="list-style-type: none"> <li>a) Procedure is updated for spectrum range.</li> <li>b) Working standard added with CRS and Purity index added.</li> </ol> </li> <li>6. In MOA No.QC/PH-FG/SPEC/14-05               <ol style="list-style-type: none"> <li>a) "Interpretation" is added.</li> </ol> </li> <li>7. In MOA No.QC/PH-FG/SPEC/14-06               <ol style="list-style-type: none"> <li>a) Solution preparation is updated for addition of volumetric solution.</li> </ol> </li> <li>8. In MOA No.QC/PH-FG/SPEC/14-07               <ol style="list-style-type: none"> <li>a) The term "sample" replaced by "substance to be examine".</li> <li>b) "Limit" is added.</li> </ol> </li> <li>9. In MOA No.QC/PH-FG/SPEC/14-08               <ol style="list-style-type: none"> <li>a) Chromatographic condition is updated for addition of flow Control Mode, Pressure, Purge Flow, Makeup flow, Hydrogen Flow, Air Flow and Equilibration time.</li> <li>b) In procedure "sample" term replaced by "Test solution"</li> </ol> </li> <li>10. In MOA No.QC/PH-FG/SPEC/14-09               <ol style="list-style-type: none"> <li>a) Solution preparation is updated.</li> </ol> </li> <li>11. In MOA No.QC/PH-FG/SPEC/14-10               <ol style="list-style-type: none"> <li>a) In procedure "sample" term replaced by "substance to be examine"</li> <li>b) "Limit" is added.</li> </ol> </li> <li>12. In MOA No.QC/PH-FG/SPEC/14-11               <ol style="list-style-type: none"> <li>a) The term "sample" is replaced by "substance"</li> </ol> </li> <li>13. Following points are added in general information.               <ol style="list-style-type: none"> <li>a) Quantity to be sampled is modified by adding term "About"</li> </ol> </li> </ol> | Ph.Eur -10.0<br>(Refer Change Control No.<br>RCPL/CC/QC/008-19) |

REFERENCE COPY

|   |  |                              |                                  |
|---|--|------------------------------|----------------------------------|
| Rev.12                                  | <b>METHYLENE CHLORIDE Ph.Eur<br/>SPECIFICATION</b> |                              | <b>Reference<br/>Ph.Eur 11.0</b> |
| FG Specification No<br>QC/PH-FG/SPEC/14 | Supersedes<br>Rev.11                               | Effective date<br>01/01/2023 | Page No<br>6 of 6                |

|  |  |   |  |
|--|--|---|--|
|  |  | <ul style="list-style-type: none"> <li>b) Mentioned tests to be perform for stability testing.</li> <li>c) Quantity for stability sample for single analysis is added.</li> <li>d) Desirable pack updated for addition of HM-HDPE containers</li> <li>e) Filter cleaning is added.</li> </ul> |  |
|--|--|---|--|

|  |        |  |  |
|--|--------|--|--|
|  | Rev.10 | <p>1. Identification by Relative density at 20°C, and Chemical tests are added also new MOA No. QC/PH-FG/SPEC/14-12 and QC/PH-FG/SPEC/14-13 added. Same test added in Tanker rinsing and stability testing.</p> <p>2. In MOA No. QC/PH-FG/SPEC/14-08 chromatographic condition is updated for addition of Total Flow. Procedure is updated for injection sequence.</p> <p>3. Residual solvents (By GC) test is added also new MOA No. QC/PH-FG/SPEC/14-14 is added. Same test is added in stability testing.</p> <p>4. Quantity to be sampled and stability quantity for single analysis is updated.</p> | Refer change control No. RCPL/CC/QC/004-21 |
|--|--------|--|--|

|  |         |                       |   |
|--|---------|-----------------------|---|
|  | Rev. 11 | 1. Reference updated. | Ph. Eur 11.0 & (Refer Change control No. RCPL/CC/QC/008-22) |
|--|---------|-----------------------|---|

REFERENCE COPY