



Piramal Healthcare Limited


knowledge action care

CERTIFICATE OF ANALYSIS

| PRODUCT : DILTIAZEM HCl (MICRONISED) | | COMPENDIA : Ph.Eur.-7.4 |
|---|--|---|
| BATCH NO. : DIL/M-11912 | | QUANTITY : 500 Kg |
| MFG. DATE : DECEMBER.2012 | | EXP. DATE : NOVEMBER.2017 |
| QC A.R. No.: FP/305/2012 | | |
| TESTS | RESULTS | SPECIFICATIONS |
| 01. CHARACTERS | | |
| i. Appearance | A white, crystalline powder. | White or almost white, crystalline powder. |
| ii. Solubility | Freely soluble in water, in methanol and in methylene chloride, slightly soluble in anhydrous ethanol. | Freely soluble in water, in methanol and in methylene chloride, slightly soluble in anhydrous ethanol. |
| iii. Melting point | Melted at 213°C with decomposition. | Melts at about 213° C with decomposition. |
| 02. IDENTIFICATION | | |
| IR Absorption | Concordant with working standard | IR Absorption spectrum of the sample is concordant with that of the Diltiazem hydrochloride CRS / working standard. |
| CHLORIDES | Conforms | It gives reaction to chlorides. |
| 03. APPEARANCE OF SOLUTION | Clear and colorless | Clear and colourless. |
| 04. pH | 4.9 | 4.3 to 5.3 |
| 05. SPECIFIC OPTICAL ROTATION | +117° | + 115° to + 120° |
| 06. RELATED SUBSTANCES | | |
| i. Impurity F | Not Quantified | Not more than 0.2% w/w |
| ii. Unspecified Impurities | Not Quantified | Not more than 0.10% w/w each |
| iii. Total Impurities | Not Quantified | Not more than 0.30% w/w |
| 07. HEAVY METALS | Less than 10 ppm | Not more than 10 ppm |
| 08. LOSS ON DRYING | 0.18% | Not more than 0.50% w/w |
| 09. SULPHATED ASH | 0.03% | Not more than 0.10% w/w |
| 10. ASSAY (By Titration) | 99.89% | Not less than 98.50% w/w and Not more than 101.00% w/w |
| (On dried basis) | | |
| ADDITIONAL TEST: | | |
| PARTICLE SIZE | | |
| 100% | 82 Microns | 100% Less than 100 Microns |
| Surface Area | 1.69 m ² /gm | For Information |
| RESIDUAL SOLVENTS | | |
| Toluene | 243 ppm | Not more than 750 ppm |
| Methanol | 135 ppm | Not more than 500 ppm |
| Isopropyl Alcohol | 7 ppm | Not more than 1000 ppm |
| Ethyl Acetate | 10 ppm | Not more than 2500 ppm |
| Bulk Density | 0.29 g/ml | For Information |
| Tapped Density | 0.50 g/ml | For Information |

REMARKS: The product conforms to **Ph.Eur.-7.4 & Customer's Specifications**


**Prepared by
Executive-QC**


**Checked by
Executive-QC**


**Approved by
Manager-QC**

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CERTIFICATE OF ANALYSIS

| PRODUCT : DILTIAZEM HCl (Micronised) | | COMPENDIA : JP XVI |
|---|---|--|
| BATCH NO. : DIL/M-11912 | | QUANTITY : 500 Kg |
| MFG. DATE : DECEMBER.2012 | | EXP. DATE : NOVEMBER.2017 |
| QC A.R.No.: FP/305/2012 | | |
| TESTS | RESULTS | SPECIFICATIONS |
| 01. DESCRIPTION | White crystalline powder. It is very soluble in formic acid, freely soluble in water, in methanol and in chloroform, sparingly soluble in acetonitrile, slightly soluble in acetic anhydride and in ethanol (99.5%) and practically insoluble in diethyl ether. | White crystals or crystalline powder. It is very soluble in formic acid, freely soluble in water, in methanol and in chloroform, sparingly soluble in acetonitrile, slightly soluble in acetic anhydride and in ethanol (99.5%), and practically insoluble in diethyl ether. |
| 02. IDENTIFICATION | | |
| 1. Reaction with Ammonium thiocyanate cobaltous nitrate | Conforms | To comply as per JP |
| 2. Reaction of sulphates | Conforms | To comply as per JP |
| 3. UV | Conforms | UV spectrum of sample and standard should exhibits similar intensities of absorption at the same wavelengths. The IR of the test sample exhibits absorption at the wave numbers of about 1741 cm ⁻¹ , 1678 cm ⁻¹ , 1252 cm ⁻¹ , and 1025 cm ⁻¹ |
| 4. IR Spectrum | Conforms | To comply as per JP |
| 5. Reaction of chloride | Conforms | Between +115° and +120° |
| 03. OPTICAL ROTATION | + 117° | 210° to 215° C(with Decomposition) |
| 04. MELTING POINT | 213° C (with decomposition) | Between 4.3 to 5.3 |
| 05. pH | 4.9 | The solution is clear and colourless. |
| 06. CLARITY AND COLOUR OF SOLUTION | Clear and colorless | |
| 07. SULFATES | Less than 0.024% | Not more than 0.024% |
| 08. HEAVY METALS | Less than 10 ppm | Not more than 10 ppm |
| 09. ARSENIC | Less than 2 ppm | Not more than 2 ppm |
| 10. RELATED SUBSTANCES (By HPLC) | | |
| Total impurities | 0.0% | Not more than 0.3% |
| 11. LOSS ON DRYING | 0.2% | Not more than 0.5% w/w |
| 12. RESIDUE ON IGNITION | 0.0% | Not more than 0.1% w/w |
| 13. ASSAY (On dry basis) | 100.0% | Not less than 98.5% w/w |
| ADDITIONAL TESTS: | | |
| PARTICLE SIZE | | |
| 100% | 82 Microns | 100% Less than 100 Microns |
| Surface Area | 1.69 m ² /gm | For Information |
| RESIDUAL SOLVENTS | | |
| Toluene | 243 ppm | Not more than 750 ppm |
| Methanol | 135 ppm | Not more than 500 ppm |
| Isopropyl Alcohol | 7 ppm | Not more than 1000 ppm |
| Ethyl Acetate | 10 ppm | Not more than 2500 ppm |
| Bulk Density | 0.29 g/ml | For Information |
| Tapped Density | 0.50 g/ml | For Information |

REMARKS: The product conforms to JP XVI & Customer's Specifications.

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