

CERTIFICATE OF ANALYSIS

| | |
|---|--------------------------------|
| PRODUCT : DILTIAZEM HCl (MICRONISED) | COMPENDIA : Ph.Eur.-7.7 |
| BATCH NO. : DIL/M-13614 | QUANTITY : 360 Kg |
| MFG. DATE : JUNE.2014 | EXP. DATE : MAY.2019 |
| QC A.R. No.: FP/164/2014 | |
| 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

Positive

It gives reaction to chlorides.

03. APPEARANCE OF SOLUTION

Clear and colourless

Clear and colourless.

04. pH

4.9

4.3 to 5.3

05. SPECIFIC OPTICAL ROTATION

+116°

+ 115° to + 120°

06. RELATED SUBSTANCES

i. Impurity F

Not Quantified

Not more than 0.3% 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

0.14%

Not more than 0.50% w/w

09. SULPHATED ASH

0.02%

Not more than 0.10% w/w

10. ASSAY (By Titration)

99.86%

Not less than 98.50% w/w and

(On dried basis)

Not more than 101.00% w/w

ADDITIONAL TEST:
PARTICLE SIZE

100%

23 Microns

100% Less than 100 Microns

Surface Area

 1.94 m²/gm

For Information

RESIDUAL SOLVENTS (BY GC-HS)

Toluene

393 ppm

Not more than 750 ppm

Methanol

346 ppm

Not more than 500 ppm

Isopropyl Alcohol

0 ppm

Not more than 1000 ppm

Ethyl Acetate

1 ppm

Not more than 2500 ppm

Bulk Density

0.27 g/ml


For Information

Tapped Density


0.46 g/ml

For Information

REMARKS: The product conforms to **Ph.Eur.-7.7 & Customer's Specifications**



**Prepared by
Executive-QC**



**Checked by
Manager-QC**



**Approved by
General Manager-QC**

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CERTIFICATE OF ANALYSIS

| PRODUCT : DILTIAZEM HCl (MICRONISED) | | COMPENDIA : USP-37 |
|---|--|---|
| BATCH NO. : DIL/M-13614 | | QUANTITY : 360 Kg |
| MFG. DATE : JUNE.2014 | | EXP. DATE : MAY.2019 |
| QC A.R. No. : FP/164/2014 | | |
| TESTS | RESULTS | SPECIFICATION |
| 1. DESCRIPTION | White crystalline powder. Melts at 213°C with decomposition. | White, odorless, crystalline powder or small crystals. Melts at about 210° C with decomposition. |
| 2. SOLUBILITY | Freely soluble in water, in formic acid, in methanol and in chloroform. Sparingly soluble in dehydrated alcohol; insoluble in ether. | Freely soluble in water, in formic acid, in methanol and in chloroform. Sparingly soluble in dehydrated alcohol; insoluble in ether. |
| 3. IDENTIFICATION | | |
| A. IR Absorption | Concordant with working standard. | The IR of the test sample is concordant with that of the working standard/Reference standard. |
| B. HPLC | Conforms | The retention time of the Diltiazem Hydrochloride peak in the chromatogram of the assay preparation corresponds to that of the chromatogram of the standard preparation, obtained under assay test. |
| C. Chlorides. | Positive | Gives positive test for chloride. |
| 4. SPECIFIC ROTATION | + 111° | Between +110° to +116° |
| 5. LOSS ON DRYING | 0.14% | Not more than 0.50% w/w |
| 6. RESIDUE ON IGNITION | 0.02% | Not more than 0.10% w/w |
| 7. HEAVY METALS | Less than 20 ppm | Not more than 20 ppm |
| 8. RELATED SUBSTANCES (By HPLC) | | |
| 1. Desacetyl Diltiazem HCl. | 0.03% | Not more than 0.10% |
| 2. Unknown Individual impurity | 0.00% | Not more than 0.10% |
| 3. Total impurities | 0.04% | Not more than 0.30% |
| 9. RESIDUAL SOLVENTS (By GC-HS) | | |
| CLASS - 2. Residual Solvents | | |
| a) Toluene | 393 ppm | Not more than 50 ppm |
| b) Methanol | 346 ppm | Not more than 500 ppm |
| CLASS - 3. Residual Solvents | | |
| a) Isopropyl Alcohol | 0 ppm | Not more than 500 ppm |
| b) Ethyl Acetate | 1 ppm | Not more than 250 ppm |
| 10. ASSAY (By HPLC) | 100.3% | Not less than. 98. 0% w/w and Not more than. 102. 0% w/w of C ₂₂ H ₂₆ N ₂ O ₄ S. HCl On dried basis. |
| ADDITIONAL TEST: | | |
| PARTICLE SIZE BY MALVERN | | |
| 100% | 23 Microns | 100% Less than 100 Microns |
| Surface Area | 1.94 m ² /gm | For Information |
| Bulk Density | 0.27 g/ml | For Information |
| Tapped Density | 0.46 g/ml | For Information |

Remarks: The product conforms to **USP-37 & Customer's** specifications.

Sealed
Prepared by
Executive-QC

[Signature]
Checked by
Manager-QC

[Signature]
Approved by
General Manager-QC

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