

### CERTIFICATE OF ANALYSIS

<b>PRODUCT : DILTIAZEM HCl</b> (MICRONISED)	<b>COMPENDIA : Ph.Eur.-7.7</b>
<b>BATCH NO. : DIL/M-12114</b>	<b>QUANTITY : 500 Kg</b>
<b>MFG. DATE : JUNE.2014</b>	<b>EXP. DATE : MAY.2019</b>
<b>QC A.R. No.: FP/147/2014</b>	
<b>TESTS</b>	<b>RESULTS</b>
	<b>SPECIFICATIONS</b>

**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 212°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**

4.6

4.3 to 5.3

**05. SPECIFIC OPTICAL ROTATION**

+117°

+ 115° to + 120°

**06. RELATED SUBSTANCES**

i. Impurity F

Not Quantified

Not more than 0.3% 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**

0.22%

Not more than 0.50% w/w

**09. SULPHATED ASH**

0.06%

Not more than 0.10% w/w

**10. ASSAY (By Titration)**

100.29%

Not less than 98.50% w/w and

(On dried basis)

Not more than 101.00% w/w

**ADDITIONAL TEST:**
**PARTICLE SIZE**

100%

35 Microns

100% Less than 100 Microns

**Surface Area**

 1.89 m<sup>2</sup>/gm

For Information

**RESIDUAL SOLVENTS ( BY GC-HS)**

Toluene

307 ppm

Not more than 750 ppm

Methanol

107 ppm

Not more than 500 ppm

Isopropyl Alcohol

10 ppm

Not more than 1000 ppm

Ethyl Acetate

2 ppm

Not more than 2500 ppm

Bulk Density

0.24 g/ml

For Information

Tapped Density

0.47 g/ml

For Information

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

*Deew*  
**Prepared by**  
**Executive-QC**

*Lypar*  
**Checked by**  
**Manager-QC**

*N*  
**Approved by**  
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## CERTIFICATE OF ANALYSIS

<b>PRODUCT : DILTIAZEM HCl (MICRONISED)</b>		<b>COMPENDIA : JP XVI</b>
<b>BATCH NO. : DIL/M-12114</b>		<b>QUANTITY : 500 Kg</b>
<b>MFG. DATE : JUNE.2014</b>		<b>EXP. DATE : MAY.2019</b>
<b>QC A.R.No.: FP/147/2014</b>		
TESTS	RESULTS	SPECIFICATIONS
<b>01. DESCRIPTION</b>	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.
<b>02. IDENTIFICATION</b>		
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 exhibit similar intensities of absorption at the same wavelengths.
4. IR Spectrum	Conforms	The IR of the test sample exhibits absorption at the wave numbers of about 1741 cm <sup>-1</sup> , 1678 cm <sup>-1</sup> , 1252 cm <sup>-1</sup> , and 1025 cm <sup>-1</sup>
5. Reaction of chloride	Positive	To comply as per JP
<b>03. OPTICAL ROTATION</b>	+ 117°	Between +115° and +120°
<b>04. MELTING POINT</b>	212° C (with decomposition)	210° to 215° C (with Decomposition)
<b>05. pH</b>	4.6	Between 4.3 to 5.3
<b>06. CLARITY AND COLOUR OF SOLUTION</b>	Clear and colorless	The solution is clear and colourless.
<b>07. SULFATES</b>	Less than 0.024%	Not more than 0.024%
<b>08. HEAVY METALS</b>	Less than 10 ppm	Not more than 10 ppm
<b>09. ARSENIC</b>	Less than 2 ppm	Not more than 2 ppm
<b>10. RELATED SUBSTANCES (By HPLC)</b>		
Total impurities	0.0%	Not more than 0.3%
<b>11. LOSS ON DRYING</b>	0.2%	Not more than 0.5% w/w
<b>12. RESIDUE ON IGNITION</b>	0.0%	Not more than 0.1% w/w
<b>13. ASSAY (On dry basis)</b>	99.9%	Not less than 98.5% w/w
<b>ADDITIONAL TESTS:</b>		
<b>PARTICLE SIZE</b>		
100%	35 Microns	100% Less than 100 Microns
<b>Surface Area</b>	1.89 m <sup>2</sup> /gm	For Information
<b>RESIDUAL SOLVENTS</b>		
Toluene	307 ppm	Not more than 750 ppm
Methanol	107 ppm	Not more than 500 ppm
Isopropyl Alcohol	10 ppm	Not more than 1000 ppm
Ethyl Acetate	02 ppm	Not more than 2500 ppm
<b>Bulk Density</b>	0.24 g/ml	For Information
<b>Tapped Density</b>	0.47 g/ml	For Information

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

  
**Prepared by**  
**Executive-QC**

  
**Checked by**  
**Manager-QC**

  
**Approved by**  
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