



Piramal Healthcare Limited

knowledge action care

CERTIFICATE OF ANALYSIS

PRODUCT : DILTIAZEM HCl (MICRONISED)	COMPENDIA : Ph.Eur.-7.4
BATCH NO. : DIL/M-12312	QUANTITY : 300 Kg
MFG. DATE : DECEMBER.2012	EXP. DATE : NOVEMBER.2017
QC A.R. No.: FP/316/2012	
TESTS	RESULTS
01. CHARACTERS	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	+119°	+ 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	0.17%	Not more than 0.50% w/w
09. SULPHATED ASH	0.03%	Not more than 0.10% w/w
10. ASSAY (By Titration)	100.12%	Not less than 98.50% w/w and Not more than 101.00% w/w
(On dried basis)		
ADDITIONAL TEST:		
PARTICLE SIZE		
100%	22 Microns	100% Less than 100 Microns
Surface Area	2.03 m ² /gm	For Information
RESIDUAL SOLVENTS		
Toluene	10 ppm	Not more than 50 ppm
Methanol	199 ppm	Not more than 500 ppm
Isopropyl Alcohol	Not detected	Not more than 500 ppm
Ethyl Acetate	16 ppm	Not more than 250 ppm
Bulk Density	0.28 g/ml	For Information
Tapped Density	0.46 g/ml	For Information

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

[Signature]
Prepared by
Executive-QC

[Signature]
Checked by
Executive-QC

[Signature]
Approved by
Manager-QC

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CERTIFICATE OF ANALYSIS

PRODUCT : DILTIAZEM HCl (MICRONISED)		COMPENDIA : USP-35
BATCH NO. : DIL/M-12312		QUANTITY : 300 Kg
MFG. DATE : DECEMBER.2012		EXP. DATE : NOVEMBER.2017
QC A.R. No.: FP/316/2012		
TESTS	RESULTS	SPECIFICATION
1. DESCRIPTION	White, odorless, 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.	Conforms	Gives positive test for chloride.
4. SPECIFIC ROTATION (at 25° C)	+ 114°	Between +110° to +116°
5. LOSS ON DRYING	0.17%	Not more than 0.50% w/w
6. RESIDUE ON IGNITION	0.03%	Not more than 0.10% w/w
7. HEAVY METALS	Less than 20 ppm	Not more than 20 ppm
8. RELATED COMPOUNDS (By HPLC)		
1. Desacetyl Diltiazem HCl.	0.01%	Not more than 0.10%
2. Unknown Individual impurity	Not detected	Not more than 0.10%
3. Total impurities	0.01%	Not more than 0.30%
9. RESIDUAL SOLVENTS (By GC-MS)		
CLASS - 2. Residual Solvents		
a) Toluene	10 ppm	Not more than 50 ppm
b) Methanol	199 ppm	Not more than 500 ppm
CLASS - 3. Residual Solvents		
a) Isopropyl Alcohol	Not detected	Not more than 500 ppm
b) Ethyl Acetate	16 ppm	Not more than 250 ppm
10. ASSAY (By HPLC)	100.0%	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		
100%	22 Microns	100% Less than 100 Microns
Surface Area	2.03 m ² /gm	For Information
Bulk Density	0.28 g/ml	For Information
Tapped Density	0.46 g/ml	For Information

Remarks: The product conforms to USP-35 & 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 : BP-2012	
BATCH NO. : DIL/M-12312		QUANTITY : 300 Kg	
MFG. DATE : DECEMBER.2012		EXP. DATE : NOVEMBER.2017	
QC A.R. No.: FP/316/2012			
TESTS		RESULTS	SPECIFICATIONS
01. CHARACTERS			

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

03. APPEARANCE OF SOLUTION

Conforms

It gives reaction to chlorides.

04. pH

Clear and colorless

Clear and colourless.

05. SPECIFIC OPTICAL ROTATION

4.9

4.3 to 5.3

06. RELATED SUBSTANCES

+119°

+ 115° to + 120°

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

0.17%

Not more than 0.50% w/w

09. SULPHATED ASH

0.03%

Not more than 0.10% w/w

10. ASSAY (By Titration)

100.12%

Not less than 98.50% w/w and

(On dried basis)

Not more than 101.00% w/w

ADDITIONAL TEST:

PARTICLE SIZE

100%

22 Microns

100% Less than 100 Microns

Surface Area

2.03m²/gm

For Information

RESIDUAL SOLVENTS

Toluene

10 ppm

Not more than 50 ppm

Methanol

199 ppm

Not more than 500 ppm

Isopropyl Alcohol

Not detected

Not more than 500 ppm

Ethyl Acetate

16 ppm

Not more than 250 ppm

Bulk Density

0.28 g/ml

For Information

Tapped Density

0.46 g/ml

For Information

REMARKS: The product conforms to **BP-2012 & Customer's** Specifications

Salu
Prepared by
Executive-QC

Seew
Checked by
Executive-QC

Aranya
Approved by
Manager-QC

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Result Analysis Report

Sample Name:
DILTIAZEM HCL
Sample Source & type:
Factory = PHL
Sample bulk lot ref:
DIL/M-12312(FP/316/2012)

SOP Name:
DILTIAZEM HCL
Measured by:
R.Damodaram
Result Source:
Measurement

Measured:
Friday, December 28, 2012 02:27:17 PM
Analysed:
Friday, December 28, 2012 02:27:18 PM

Particle Name:
DILTIAZEM HCL
Particle RI:
1.500
Dispersant Name:

Accessory Name:
Scirocco 2000 (A)
Absorption:
0.001
Dispersant RI:
1.000

Analysis model:
General purpose
Size range:
0.020 to 2000.000 μm
Weighted Residual:
0.766 %
Sensitivity:
Normal
Obscuration:
5.11 %
Result Emulation:
Off

Concentration:
0.0004 %Vol

Span :
2.103

Uniformity:
0.643

Result units:
Volume

Specific Surface Area:
2.03 m^2/g

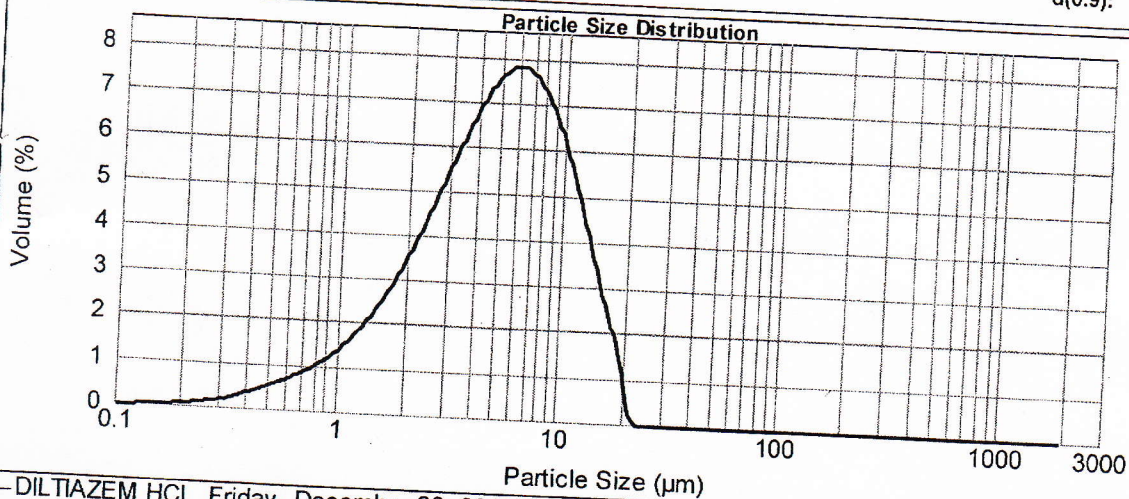
Surface Weighted Mean D[3,2]:
2.956 μm

Vol. Weighted Mean D[4,3]:
6.035 μm

d(0.1): 1.473 μm

d(0.5): 5.059 μm

d(0.9): 12.115 μm



DILTIAZEM HCL, Friday, December 28, 2012 02:27:17 PM

Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %
0.010	0.00	7.096	67.16	11.120	87.10	18.000	98.63	41.000	100.00	112.468	100.00
1.000	5.49	7.200	67.89	11.600	88.57	18.900	99.21	46.000	100.00	122.000	100.00
1.125	6.62	7.300	68.58	11.700	88.86	20.000	99.73	50.238	100.00	126.191	100.00
1.900	14.56	7.410	69.32	12.800	91.67	22.440	100.00	56.368	100.00	200.000	100.00
3.170	29.35	7.520	70.05	12.900	91.90	23.900	100.00	65.000	100.00	220.404	100.00
3.991	38.78	7.700	71.21	13.000	92.12	24.100	100.00	68.400	100.00	251.785	100.00
4.477	44.06	7.866	72.25	13.200	92.55	24.950	100.00	70.000	100.00	316.979	100.00
5.024	49.65	7.962	72.84	13.655	93.44	27.300	100.00	73.000	100.00	355.656	100.00
5.637	55.44	7.985	72.97	13.700	93.53	28.251	100.00	75.000	100.00	360.656	100.00
6.120	59.64	8.255	74.56	13.900	93.90	30.000	100.00	76.000	100.00	362.666	100.00
6.350	61.53	9.000	78.53	14.000	94.07	31.950	100.00	77.000	100.00	389.052	100.00
6.700	64.26	9.150	79.26	14.300	94.58	32.700	100.00	79.000	100.00	447.744	100.00
6.750	64.64	10.000	83.03	14.700	95.20	34.000	100.00	80.000	100.00	502.377	100.00
6.825	65.20	10.480	84.89	14.800	95.35	35.566	100.00	81.000	100.00	632.456	100.00
6.890	65.68	10.550	85.14	14.955	95.58	38.300	100.00	82.000	100.00		
6.950	66.12	10.600	85.32	15.420	96.20	39.905	100.00	100.000	100.00		
6.990	66.41	10.925	86.45	16.335	97.23	40.000	100.00	100.237	100.00		

Analysed by:

Checked by:

Malvern Instruments Ltd.
Malvern, UK

Mastersizer 2000 Ver. 5.22
Serial Number: MAL100042

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File name: DILTIAZEM HCL
Record Number: 52
28 Dec 2012 02:42:04 PM

Refer HS 1 2194, 064