

CERTIFICATE OF ANALYSIS

PRODUCT : DILTIAZEM HCl (MICRONISED)		COMPENDIA : Ph.Eur.-8.0
BATCH NO. : DIL/M-16914		QUANTITY : 500 Kg
MFG. DATE : JULY.2014		EXP. DATE : JUNE.2019
QC A.R. No.: FP/201/2014		
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	+118°	+ 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	0.19%	Not more than 0.50% w/w
09. SULPHATED ASH	0.02%	Not more than 0.10% w/w
10. ASSAY (By Titration)	99.99%	Not less than 98.50% w/w and Not more than 101.00% w/w
(On dried basis)		
ADDITIONAL TEST:		
PARTICLE SIZE		
100%	23 Microns	100% Less than 100 Microns
Surface Area	2.11 m ² /gm	For Information
RESIDUAL SOLVENTS (BY GC-HS)		
Toluene	238 ppm	Not more than 750 ppm
Methanol	90 ppm	Not more than 500 ppm
Isopropyl Alcohol	2 ppm	Not more than 1000 ppm
Ethyl Acetate	0 ppm	Not more than 2500 ppm
Bulk Density	0.26 g/ml	For Information
Tapped Density	0.45 g/ml	For Information

REMARKS: The product conforms to **Ph.Eur.-8.0 & Customer's Specifications**

Prepared by
**Prepared by
Executive-QC**

Checked by
**Checked by
Manager-QC**

Approved by
**Approved by
General Manager-QC**

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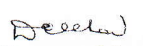
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PRODUCT : DILTIAZEM HCl (MICRONISED)		COMPENDIA : JP XVI
BATCH NO. : DIL/M-16914		QUANTITY : 500 Kg
MFG. DATE : JULY.2014		EXP. DATE : JUNE.2019
QC A.R.No.: FP/201/2014		
TESTS	RESULTS	SPECIFICATIONS
01. DESCRIPTION	White crystalline powder. It is 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 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 ⁻¹ , 1678 cm ⁻¹ , 1252 cm ⁻¹ , and 1025 cm ⁻¹
5. Reaction of chloride	Positive	To comply as per JP
03. OPTICAL ROTATION	+ 118°	Between +115° and +120°
04. MELTING POINT	213° C (with decomposition)	210° to 215° C (with Decomposition)
05. pH	4.9	Between 4.3 to 5.3
06. CLARITY AND COLOUR OF SOLUTION	Clear and colorless	The solution is clear and colourless.
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	0.1%	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)	99.8%	Not less than 98.5% w/w
ADDITIONAL TESTS:		
PARTICLE SIZE		
100%	23 Microns	100% Less than 100 Microns
Surface Area	2.11 m ² /gm	For Information
RESIDUAL SOLVENTS		
Toluene	238 ppm	Not more than 750 ppm
Methanol	90 ppm	Not more than 500 ppm
Isopropyl Alcohol	2 ppm	Not more than 1000 ppm
Ethyl Acetate	0 ppm	Not more than 2500 ppm
Bulk Density	0.26 g/ml	For Information
Tapped Density	0.45 g/ml	For Information

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


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PIRAMAL ENTERPRISES LIMITED

Result Analysis Report

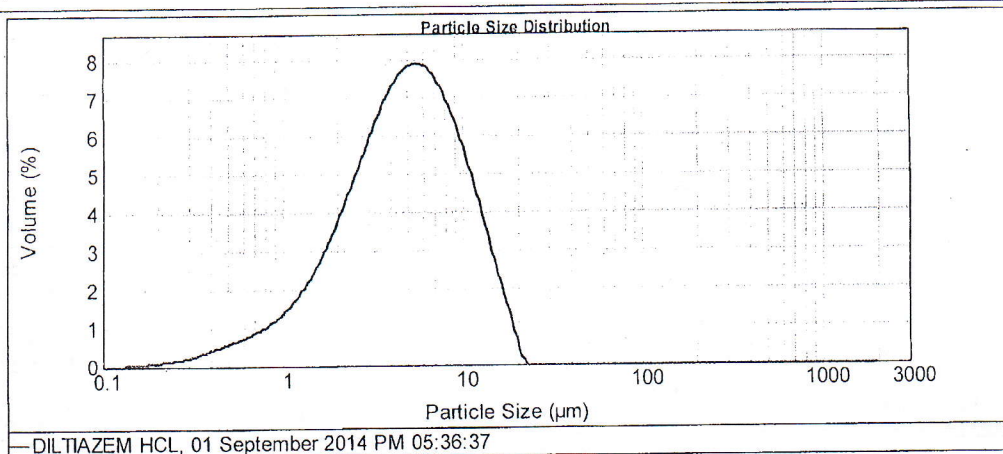
Sample Name:
DILTIAZEM HCL
Sample Source & type:
Factory = PHL
Sample bulk lot ref:
DIL/M-16914(p-201-2014)

SOP Name:
DILTIAZEM HCL
Measured by:
MAHESH
Result Source:
Measurement

Measured:
01 September 2014 PM 05:36:37
Analysed:
01 September 2014 PM 05:36:39

Particle Name: DILTIAZEM HCL	Accessory Name: Sciocco 2000	Analysis model: General purpose	Sensitivity: Normal
Particle RI: 1.500	Absorption: 0.001	Size range: 0.020 to 2000.000 um	Obscuration: 5.78 %
Dispersant Name:	Dispersant RI: 1.000	Weighted Residual: 0.498 %	Result Emulation: Off
Concentration: 0.0005 %Vol	Span : 2.089	Uniformity: 0.641	Result units: Volume
Specific Surface Area: 2.11 m ² /g	Surface Weighted Mean D[3,2]: 2.841 um	Vol. Weighted Mean D[4,3]: 5.718 um	

d(0.1): 1.452 um d(0.5): 4.778 um d(0.9): 11.436 um



Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %	Size (µm)	Vol Under %
0.010	0.00	1.096	6.52	8.020	76.09	26.303	100.00	174.000	100.00
0.011	0.00	1.259	8.05	8.640	79.43	34.674	100.00	181.970	100.00
0.017	0.00	1.660	12.25	9.011	81.23	39.005	100.00	208.930	100.00
0.020	0.00	1.905	15.09	9.900	84.98	43.452	100.00	239.883	100.00
0.026	0.00	2.188	18.52	10.400	86.81	52.481	100.00	275.423	100.00
0.046	0.00	2.512	22.60	11.682	90.66	79.433	100.00	316.228	100.00
0.159	0.02	2.884	27.36	12.452	92.48	85.000	100.00	363.078	100.00
0.275	0.33	3.311	32.81	12.600	92.80	99.000	100.00	416.869	100.00
0.316	0.51	3.802	38.88	13.605	94.69	99.850	100.00	478.630	100.00
0.363	0.75	4.365	45.47	13.800	95.01	100.000	100.00	549.541	100.00
0.417	1.08	5.012	52.44	14.440	95.96	125.000	100.00	630.957	100.00
0.479	1.51	5.754	59.57	15.000	96.68	125.011	100.00	724.436	100.00
0.550	2.03	6.425	65.25	15.400	97.14	126.251	100.00	831.764	100.00
0.631	2.65	7.170	70.75	15.590	97.34	142.650	100.00	954.993	100.00
0.724	3.38	7.390	72.22	16.308	98.01	158.489	100.00	1096.478	100.00
0.832	4.25	7.459	72.66	17.950	99.13	160.000	100.00	1258.925	100.00
0.955	5.28	7.950	75.68	22.909	100.00	166.000	100.00	1445.440	100.00

Analysed by: 01/09/14

Checked by: 03/09/14