

CERTIFICATE OF ANALYSIS

PRODUCT : DILTIAZEM HCl (MICRONISED)		COMPENDIA : Ph.Eur.-8.0
BATCH NO. : DIL/M-16814		QUANTITY : 500 Kg
MFG. DATE : JUNE.2014		EXP. DATE : MAY.2019
QC A.R. No.: FP/200/2014		
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	+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	0.20%	Not more than 0.50% w/w
09. SULPHATED ASH	0.02%	Not more than 0.10% w/w
10. ASSAY (By Titration)	99.97%	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	2.15 m ² /gm	For Information
RESIDUAL SOLVENTS (BY GC-MS)		
Toluene	234 ppm	Not more than 750 ppm
Methanol	70 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
Executive-QC

Checked by
Manager-QC

Approved by
General Manager-QC

Piramal Enterprises Limited

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PRODUCT : DILTIAZEM HCl (MICRONISED)		COMPENDIA : JP XVI
BATCH NO. : DIL/M-16814		QUANTITY : 500 Kg
MFG. DATE : JUNE.2014		EXP. DATE : MAY.2019
QC A.R.No.: FP/200/2014		
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 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	Positive	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	0.1%	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%	22 Microns	100% Less than 100 Microns
Surface Area	2.15 m ² /gm	For Information
RESIDUAL SOLVENTS		
Toluene	234 ppm	Not more than 750 ppm
Methanol	70 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-16814 (FP/200/2014)

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

Measured:
27 August 2014 PM 12:37:11
Analysed:
27 August 2014 PM 12:37:12

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

Accessory Name:
Sirocco 2000
Absorption:
0.001
Dispersant RI:
1.000

Analysis model:
General purpose
Size range:
0.020 to 2000.000 μm
Weighted Residual:
1.175 %

Sensitivity:
Normal
Obscuration:
5.32 %
Result Emulation:
Off

Concentration:
0.0004 %Vol

Span :
2.137

Uniformity:
0.657

Result units:
Volume

Specific Surface Area:
2.15 m^2/g

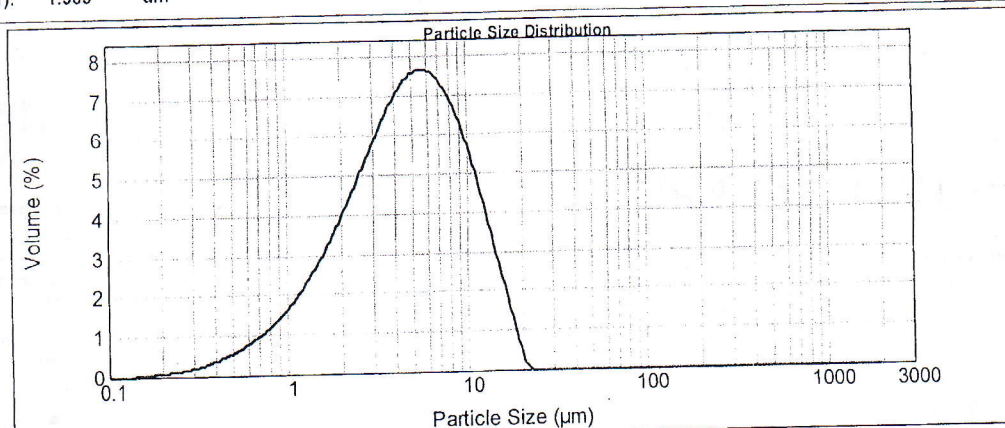
Surface Weighted Mean D[3,2]:
2.795 μm

Vol. Weighted Mean D[4,3]:
5.609 μm

d(0.1): 1.363 μm

d(0.5): 4.663 μm

d(0.9): 11.328 μm



DILTIAZEM HCL, 27 August 2014 PM 12:37:11

Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %	Size (μm)	Vol Under %
0.010	0.00	0.632	4.28	7.458	73.13	22.909	100.00	166.000	100.00
0.011	0.00	0.955	5.50	7.950	76.09	26.303	100.00	174.000	100.00
0.017	0.00	1.096	6.99	8.020	76.49	34.674	100.00	181.970	100.00
0.020	0.00	1.259	8.79	8.640	79.78	39.005	100.00	208.930	100.00
0.026	0.00	1.690	13.57	9.011	81.56	43.452	100.00	239.863	100.00
0.046	0.00	1.905	16.65	9.900	85.30	52.481	100.00	275.423	100.00
0.158	0.01	2.188	20.26	10.400	87.11	79.433	100.00	316.228	100.00
0.209	0.08	2.512	24.44	11.682	90.95	85.000	100.00	363.078	100.00
0.240	0.15	2.884	29.21	12.452	92.78	99.000	100.00	416.869	100.00
0.275	0.26	3.311	34.56	12.600	93.10	99.850	100.00	478.630	100.00
0.316	0.41	3.802	40.46	13.400	94.63	100.000	100.00	549.541	100.00
0.363	0.62	4.365	46.83	13.800	95.30	125.000	100.00	630.957	100.00
0.417	0.91	5.012	53.53	15.060	97.01	125.011	100.00	724.436	100.00
0.479	1.30	5.754	60.42	15.590	97.58	126.251	100.00	831.764	100.00
0.541	1.82	6.426	65.91	16.308	98.24	142.650	100.00	954.993	100.00
0.631	2.47	7.170	71.26	17.950	99.29	158.489	100.00	1006.478	100.00
0.724	3.28	7.390	72.70	22.100	100.00	160.000	100.00	1258.925	100.00

Analysed by: *[Signature]*
27/08/14

Checked by: *[Signature]*
28/08/14