

D51-1

4
D51-1Cipla
Kirkumbh

TEST STANDARD CERTIFICATE

QUALIFICATION OF TEST STANDARD

ITEM	: DARUNAVIR ETHANOLATE
DATE OF QUALIFICATION	: 21.01.2015
DIRECTION FOR STORAGE	: Store in tightly closed, light resistant containers enclosed in triple laminated high barrier bags. Recommended storage condition is 15°C to 30°C. #
DIRECTION FOR USE	: Determine water content and ethanol content for quantitative application.
VALIDITY OF USE	: 20.01.2017
TEST STANDARD NO.	: TS/C/D70/04
A. R. NO.	: KA1500247
SOURCE B. NO.	: B140690
	SOURCE A.R.No.: KA1404813

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REFERENCE PROTOCOL No. : TP/TS/C/D70, Version 01.

Sr.No.	TESTS	STANDARDS	RESULTS
1	Description	A White to off white crystalline powder.	Meets the requirement.
2	Identification [A] By IR	The infrared spectrum of the sample is concordant with the spectrum obtained from the similar determination of Darunavir Ethanolate test standard.	Meets the requirement
3	Identification [B] By HPLC	The retention time of the principal peak in the chromatogram obtained from the Assay solution is corresponds with that in the chromatogram obtained from standard solution in the test of Assay.	Meets the requirement
4	Polymorphic Identification (By IR)	The infrared spectrum pattern of sample is concordant with that of Darunavir Ethanolate standard showing presence of characteristic peaks at $3429 \text{ cm}^{-1} \pm 7 \text{ cm}^{-1}$ and $876 \text{ cm}^{-1} \pm 7 \text{ cm}^{-1}$.	Meets the requirement
5	Identification By NMR Spectroscopy	The characterisation of sample by NMR spectrum should conform to the structural identity of the molecule.	Meets the requirement

HEAD QUALITY CONTROL

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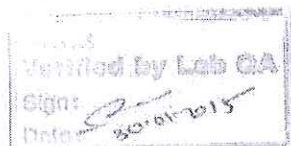
DATE :

21.01.2015

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QCP14/F2/3



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REFERENCE PROTOCOL No. : TP/TS/C/D70, Version 01.

Sr.No.	TESTS	STANDARDS	RESULTS
6	Identification By Mass Spectroscopy	The characterisation of sample by Mass spectrum should obtain similar molecular weight obtained by empirical formula.	Meets the requirement.
7	Water content (By KF)	Not more than 2.00 % w/w	0.44 % w/w
8	Specific optical rotation	Not less than +35.00° and Not more than +41.00° on anhydrous and ethanol free basis.	+38.52°
9	Residue on ignition	Not more than 0.10 % w/w	0.04 % w/w
10	*Ethanol Content (By Gas Chromatography)	Not less than 5.0 % and Not more than 8.5 % on as such basis.	7.3 %
11	Related Compounds (By HPLC) Di-furofuranyl impurity Individual impurity Total impurities	Not more than 0.08 % Not more than 0.08 % Not more than 0.70 %	Below LOQ (0.04 %) Below LOQ (0.05 %) Below LOQ

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Sr.No.	TESTS	STANDARDS	RESULTS
12	Assay (By HPLC)	Not less than 98.0 % w/w and not more than 102.0 % w/w calculated on anhydrous and Ethanol free basis.	101.3 % w/w
13	*Residual solvents (By GC) Methanol Methylene dichloride Ethyl acetate Tetrahydrofuran Triethylamine Toluene Other class 1,2,3 residual solvent and other organic solvents.	Not more than 3000 ppm Not more than 600 ppm Not more than 5000 ppm Not more than 720 ppm Not more than 320 ppm Not more than 890 ppm Meets the requirement of other Class 1, 2 and 3 residual solvents as per USP chapter <467> and no other organic solvents are present.	Below LOD (100 ppm) 268 ppm Below LOD (20 ppm) Not detected Not detected 195 ppm Meets the requirement

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Sr.No.	TESTS	STANDARDS	RESULTS
14	Purity (By mass balance method)	Not Applicable	99.9 % (On Anhydrous and ethanol free basis)
			92.2 % (On such basis)

REMARK: Material qualifies above tests and is certified to be used as Test Standard. *Results reported from previous analysis report.

NOTE: Third parties may store as per the product specifications.

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