WRS
I20-3

Cipla
Bangalore

WORKING STANDARD CERTIFICATE
(Amendment)

QUALIFICATION OF WORKING STANDARD

ITEM :LOPINAVIR USP EVALUATED WITH : USPRS/BL/L36/01 & 02
BATCH No. : F0I127

DATE OF QUALIFICATION :19.05.2014
DIRECTION FOR STORAGE :Store between 2°C to 8°C in well closed container and protected from light#
DIRECTION FOR USE : Determine the water content at the time of analysis.
DATE OF AMMENDMENT-I :30.05.2014
VALIDITY OF USE :08.05.2016
WORKING STANDARD No. :WS/C/L19/03
A.R. No. :BE1402834
SOURCE B. No. :FWX130002 SOURCE AR.No.:BE1301521 PAGE 1 of 3
AMMENDED FOR THE TEST : As per USP 37 standards updated.

REFERENCE : USP 37 , **Protocol No.**: WP/USP/WS/C/L19

Sr. No.	TESTS	STANDARDS	RESULTS
1)	DESCRIPTION	A white powder.	Complies
2)	SOLUBILITY	Freely soluble in methanol, Alcohol and soluble in Isopropanol.	Complies Complies
3)	*IDENTIFICATION A (By IR Spectrophotometry)	The infrared spectrum of the sample is concordant with the spectrum obtained from the similar determination of USP Lopinavir RS.	Complies
	*IDENTIFICATION B (By HPLC)	The retention time of the major peak in the chromatogram of the Sample solution corresponds to that in the chromatogram of the Standard solution, as obtained in the test for Assay.	Complies
4)	*ASSAY (By HPLC)	Lopinavir contains not less than 98.0 % w/w and not more than 102.0 % w/w of $C_{37}H_{48}N_4O_5$, calculated on anhydrous basis.	98.9 % w/w (On anhydrous basis)
5)	IMPURITIES RESIDUE ON IGNITION	Not more than 0.20 % w/w	0.03 % w/w
6)	HEAVY METALS	Not more than 20 µg/g.	Complies

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Sr. No.	TESTS	STANDARDS	RESULTS
7)	*ORGANIC IMPURITIES (By HPLC)		
		Lopinavir free amine : Not more than 0.10 %	Not detected
		Lopinavir N-formylaminoalcohol: Not more than 0.20 %	Not detected
		Lopinavir divalinate : Not more than 0.10 %	0.00 %
		Lopinavir phenoxyacetamide : Not more than 0.10 %	0.00 %
		Lopinavir N-formyl phenoxyacetamide : Not more than 0.10 %	Not detected
		Lopinavir N-acetyl phenoxyacetamide : Not more than 0.10 %	0.01 %
		Lopinavir oxazine : Not more than 0.10 %	Not detected
		Isolopinavir : Not more than 0.20 %	0.05 %
		Lopinavir 2,4-phenoxy isomer : Not more than 0.10 %	Not detected
		Lopinavir D-Valine diastereomer : Not more than 0.10 %	0.03 %
		Lopinavir (2R,4R) diastereomer : Not more than 0.10 %	Below LOD (0.02)
		Lopinavir (4R) epimer : Not more than 0.10 %	0.00 %
		Lopinavir O-acyl : Not more than 0.10 %	Not detected
		Lopinavir (2R) epimer : Not more than 0.10 %	Not detected
		Lopinavir diamide : Not more than 0.10 %	0.00 %
		Lopinavir N-acyl : Not more than 0.10 %	0.00 %
		Lopinavir O-phenoxyacetyl : Not more than 0.10 %	Not detected
		Lopinavir amino alcohol urea : Not more than 0.10 %	Not detected
		Any other individual impurity : Not more than 0.10 %	0.06 %
		Total impurities : Not more than 0.70 %	0.26 %
8)	SPECEFIC TESTS *WATER DETERMINATION (By KF)	Not more than 4.40 % w/w	2.99 % w/w

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QUALIFICATION OF WORKING STANDARD

ITEM :LOPINAVIR USP EVALUATED WITH : USPRS/BL/L35/01 & 02
BATCH No. : F0I127

DATE OF QUALIFICATION :19.06.2014
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DIRECTION FOR USE : Determine the water content at the time of analysis.
DATE OF AMMENDMENT-I :30.06.2014
VALIDITY OF USE :08.06.2016
WORKING STANDARD No. :WS/C/L19/03
A.R. No. :BE1402834
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AMMENDED FOR THE TEST : As per USP 37 standards updated.

REFERENCE : USP 37 , Protocol No.: WP/USP/WS/C/L19

Sr. No.	TESTS	STANDARDS	RESULTS
		IN-HOUSE STANDARDS	
1)	RESIDUAL SOLVENTS (By GC)	Methanol : Not more than 3000 ppm Isopropyl alcohol : Not more than 5000 ppm Methylene chloride : Not more than 600 ppm Ethyl acetate : Not more than 5000 ppm n-Hexane : Not more than 290 ppm n-Butanol : Not more than 5000 ppm n-Heptane : Not more than 5000 ppm Dimethyl formamide : Not more than 880 ppm Toluene : Not more than 890 ppm Diisopropyl ethylamine: Not more than 320 ppm	Below LOD(129) Not detected Not detected Not detected Not detected Not detected Not detected Not detected Below LOQ(37) Not detected
2)	SPECIFIC OPTICAL ROTATION	Not less than -22.00° and Not more than -28.00°, calculated on anhydrous basis.	-24.46°
3)	*POLYMORPHIC IDENTITY	The X-ray Powder diffractogram of Lopinavir sample should be concordant with that of the Lopinavir standard determined with similar conditions.	Complies
4)	**PURITY (By mass balance method)	Not applicable	96.7 % (On as such basis) 99.7 % (On anhydrous basis)

REMARKS: *Test/s qualified as per USPRS. All other tests complies with USP 37 and certified to be used as Working standard
NOTE: #Third party may store as per specification.

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