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QUALITY CONTROL DEPARTMENT

CERTIFICATE OF ANALYSIS

glenmark

Working Standard	: Imiquimod	**Effective Date**	: 19/03/2016
WS Number	: GAWS0241601	**Valid Up to**	: 10/12/2016
Source	: Glenmark Pharmaceuticals Ltd., Ankleshwar	**Quantity**	: 50 g
Vendor Batch No	: 80155317	**AR No. of API**	: 080000023007

TEST	SPECIFICATION	OBSERVATION
	In House Test	
* **Clarity and color of solutions**	A solution of Imiquimod having a concentration of 10 mg per mL in 10% acetic acid in water is not less clear than an equal volume of 10% acetic acid in water in a test tube of similar size. The absorbance of the solution determined at 440 nm in a 1-cm cell, using 10% acetic acid in water as the blank is not more than 0.1.	Meets the requirement
* **Melting range**	Between 294.0°C to 302.0°C	295.6°C – 296.1°C
* **Residual solvents**	N,N-Dimethyl formamide : NMT 880 ppm Ethyl acetate : NMT 5000 ppm Methanol : NMT 3000 ppm Methylene chloride : NMT 600 ppm Toluene : NMT 890 ppm	Not Detected Not Detected 297 ppm Not Detected Not Detected
* **Particle size (By Malvern)**	50% of particles below 100µ 90% of particles below 200µ	54 µ 119 µ

LOD: Limit of Detection

LOD for Impurity Related compound C: 0.004%

* Results transcribed from Raw material Certificate of Analysis Analytical Reference Number 080000023007

Remarks: The Working Standard is qualified against Imiquimod USPRS Lot No. F0K063 and complies with the laid down specification. Working standard is to be stored at 2°C – 8°C.

COMPILED BY: DGamas
NAME: Datta K. Gamas
DESIGNATION: Sr. officer
DATE: 19/03/2016

CHECKED BY:
NAME: Kundan Talvile.
DESIGNATION: Executive
DATE: 19/03/16

APPROVED BY:
NAME: Mangesh Patil
DESIGNATION: Sr. Officer
DATE: 19/03/2016

Glenmark Pharmaceutical Ltd. Mumbai 400 099

Glenmark Pharmaceuticals Ltd.
Corporate Office: Glenmark House, HDO-Corporate Building,Wing A, B D Sawant Marg, Chakala, Off Western Express Highway Andheri (E), Mumbai - 400 099. T : 91 22 4018 9999 F: 91 22 4018 9986 www.glenmarkpharma.com
Registered Office: B/2, Mahalaxmi Chambers, 22, Bhulabhai Desai Road, Mumbai 400 026
Manufacturing Site: Plot No S-7, Colvale Industrial Estate, Colvale, Bardez, Goa - 403 513 T: 91 0832 6652 222 F: 91 0832 6652 323

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TEST	SPECIFICATION	OBSERVATION
Description	White or almost white powder or crystalline powder.	Almost white crystalline powder.
*** Solubility**	Very slightly soluble in methanol; Practically insoluble in water and acetone.	Very slightly soluble in methanol; Practically insoluble in water and acetone.
Identification	**A. By IR:** The Infrared absorption spectrum of substance being examined must be concordant with the IR spectrum obtained from USP Imiquimod Reference Standard.	Meets the Requirement
	***B. By HPLC:** In the test for assay, the retention time of principal peak from the sample should match with that from USP Imiquimod RS/Working Standard.	Meets the Requirement
Loss on drying	NMT 0.50%w/w	0.19 % w/w
*** Residue on Ignition**	NMT 0.20%w/w	0.05 % w/w
*** Heavy Metals**	NMT 20 ppm	Less than 20 ppm
*** Organic Impurities (By HPLC)**	**Impurity related compound A:** 1-Isobutyl-1H-imidazo[4,5-c]quinoline : NMT 0.15%	Not Detected
	Impurity related compound B: 1-Isobutyl-1H-imidazo[4,5-c]quinolin-5-oxide :NMT 0.15%	Not Detected
	Impurity related compound C: 4-Chloro-1-Isobutyl-1H-imidazo[4,5-c] quinoline : NMT 0.15%	BLOD
	Any other unknown individual impurity : NMT 0.10%	0.06 %
	Total impurities : NMT 0.50%	0.15 %
Assay	NLT 98.0% and NMT 102.0%w/w on dried basis	99.6 % w/w **(On as is basis)**
		99.7 % w/w **(On dried basis)**

COMPILED BY: Dgamad
NAME: Dalla K. Gamad
DESIGNATION: Sr. Officer
DATE: 19/03/2016

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