The World Class Active Pharmaceutical Ingredients Company

Responsabilidad Integral

Lote: 0/907/1.

SIGNA, S.A. DE C.V.
CERTIFICATE OF ANALYSIS

CERTIFICATE NUMBER: XXXVII-002-03 ISSUE DATE: 2011-06-15

PRODUCT:	NADOLOL USP	
LOT NUMBER:	NADFA00311	MANUFACTURING DATE: JUNE-2011
RETEST DATE:	JUNE-2016	

TEST	METHOD	SPECIFICATIONS		RESULTS
APPEARANCE	Visual	White to off-white crystalline powder		White crystalline powder
IDENTIFICATION - IR	USP <197M>	Sample spectrum conforms to that of standard preparation spectrum		Conforms
RACEMATE COMPOSITION	USP - IR	The percentage of racemate "A" is between 40% and 60 %		53 %
LOSS ON DRYING	USP <731>	Not more than 2.0%		0.1 %
HEAVY METALS	USP <231> Method II	Not more than 0.003%		Less than 0.003%
MELTING POINT	USP <741> Class I, apparatus II	Between 124°C and 136°C		130 °C
RESIDUE ON IGNITION	USP <281>	Not more than 0.1%		0.0 %
SOLUBILITY	USP	Freely soluble in methanol and insoluble in acetone		Conforms
CHROMATOGRAPHIC PURITY (TLC)	TLC - USP	Total impurities: Not more than 2.0 %		ND
ASSAY	USP - Nonaqueous Titration	Not less than 98.0% and not more than 101.5% calculated on the dried basis		99.0 %
CHROMATOGRAPHIC PURITY	HPLC - House Method	Purity:	NLT 99.5%	99.74 %
		Identified impurities:		
		Tetra-ol:	NMT 0.2%	BRT
		4-Methoxy:	NMT 0.2%	BRT
		Penta-ol:	NMT 0.2%	0.09%
		Dimer:	NMT 0.2%	0.06%
		Alpha:	NMT 0.2%	ND
		Dideoxy:	NMT 0.2%	ND
		Largest unidentified impurity:	NMT 0.1%	0.11%
		(*)Total impurities:	NMT 0.5%	0.26%

CHECKED BY QC: Date: 2011-06-15

APPROVED BY QC: Date: 2011 06 15

APPROVED BY QA: V. Gurrola Ramos Date: 2011-06-15

Av. Industria Automotriz N° 301, Zona Industrial Toluca, Toluca, Estado de México, C.P. 50071, MEXICO Tel. (52) (722) 226-11-00 Fax (52) (722) 226-11-01

Signa S.A. de C.V.
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RESIDUAL SOLVENTS	Headspace GC House Method	Methanol:	NMT 0.1%	ND
		Acetone:	NMT 0.1%	0.06%
		Methyl isobutyl ketone:	NMT 0.1%	0.05%
		Toluene:	NMT:0.089%	Less than 0.001%
PARTICLE SIZE	Laser diffraction (wet dispersion) - House Method	% below 50 µm:	Not less than 90 %	99 %
BULK DENSITY	USP <616> Method I	Between 0.1 and 0.4 g/mL		0.2 g/mL
TAPPED DENSITY	USP <616> Method I	Between 0.3 and 0.7 g/mL		0.5 g/mL

CHECKED BY QC: V. Canal

Date: 2011-06-15

APPROVED BY QC: A. Sanchez M

Date: 2011-06-15

APPROVED BY QA: V. Guadarrama

Date: 2011-06-15

(*) Legend
BRT: Below Reporting Threshold
ND: Not Detected
Reporting Threshold: 0.05%

Storage Conditions: Store in a well closed container at 15° - 30°C

Packing Conditions: Pack in double anti-static polyethylene bag closed with a cable tie within a fiber drum.

CHROMATOGRAPHIC PURITY (IMPURITY PROFILE)

TETRA-OL: 5-(2,3-DIHYDROXYPROPOXY)-*CIS*-1,2,3,4-TETRAHYDRO-2,3-NAPHTHALENEDIOL
4-METHOXY: 5-(2-HYDROXY-3-METHOXYPROPOXY)-*CIS*-1,2,3,4-TETRAHYDRO-2,3-NAPHTHALENEDIOL
PENTA-OL: 5,5'-(2-HYDROXY-1,3-PROPANEDIYLDIOXY)-BIS(*CIS*-1,2,3,4-TETRAHYDRO-2,3-NAPHTHALENEDIOL)
DIMER: *N*,*N*-BIS[3-(1,2,3,4-TETRAHYDRO-2,3-*CIS*-DIHYDROXY-1-NAPHTHYLOXY)-2-HYDROXYPROP-1-YL] TERT-BUTYLAMINE
ALPHA: 1-[3-[(1,1-DIMETHYLETHYL)AMINO]-2-HYDROXYPROPOXY]NAPHTHALENE
DIDEOXY: 5-[3-[(1,1-DIMETHYLETHYL)AMINO]-2-HYDROXYPROPOXY]-1,2,3,4-TETRAHYDRONAPHTHALENE

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