

Ciserano, 07/11/2016				
ANALYSIS No. F529/2016		BATCH No. 108116011		
Manufacturing date: <u>October 2016</u>		Retest date: <u>October 2021</u>		Batch size: 412,00 Kg
PRODUCT		KETOPROFEN USP / PH. EUR. CAS N. 22071-15-4		
Physical State: crystalline powder Colour: white or almost white Odour: odourless or almost odourless Solubility: Practically insoluble in Distilled Water; freely soluble in Ethanol 96%, Methylene Chloride and Acetone.		Molecular weight: 254.28 Formula : C ₁₆ H ₁₄ O ₃ Method No. 1081/1		
DETERMINATIONS	RESULTS	LIMITS USP	LIMITS EP	IN-HOUSE LIMITS
IDENTIFICATION				
<i>I.R. spectrum</i>	Positive	Positive	Positive	
<i>Specific absorbance (255 nm)</i>	649		min. 615 max. 680	
<i>Ultraviolet Abs. (USP) difference</i>	1.20 %	n.m.t. 3%		
Appearance of the solution				
<i>Clarity</i>	Clear		Clear	
<i>Colour</i>	n.m.t. yellow 6		n.m.t. yellow degree 6	
Loss on drying	0.04 %	n.m.t. 0,5%	n.m.t. 0,5%	
Sulphated ash	0.049 %	n.m.t. 0,2%	n.m.t. 0,1%	
Melting point				
<i>USP method</i>	94.2 °C - 95.0 °C	min. 92°C max. 97°C		
<i>Ph. Eur. method</i>	95.0 °C		min. 94°C max. 97°C	
Heavy metals	n.m.t. 10 ppm	n.m.t. 20 ppm	n.m.t. 10 ppm	
ASSAY on the dried basis				
<i>NaOH 0,1N</i>	99.66 %	min. 98,5% max. 101,0%	min 99,0% max 100,5%	
<i>HPLC</i>	99.4 %			min. 98,0% max. 102,0%

Qualified Person

Quality Control Manager

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Colour: white or almost white		Formula : C16H14O3		
Odour: odourless or almost odourless		Method No. 1081/1		
Solubility: Practically insoluble in Distilled Water; freely soluble in Ethanol 96%, Methylene Chloride and Acetone.				
DETERMINATIONS	RESULTS	LIMITS USP	LIMITS EP	IN-HOUSE LIMITS
Related substances (HPLC)				
Labelled Impurities				
Carboxyl Ceba	Not detectable	n.m.t 0,2%	n.m.t. 0,2%	n.m.t. 0,1%
Ketoamide	Not detectable	n.m.t 0,2%	n.m.t. 0,2%	n.m.t. 0,10%
Demethyl Keto	0.040 %	n.m.t 0,2%	n.m.t. 0,2%	n.m.t. 0,10%
Methyl Keto	Not detectable	n.m.t 0,2%	n.m.t. 0,2%	n.m.t. 0,1%
3-acetilbenzophenone	Not detectable	n.m.t 0,2%	n.m.t. 0,2%	n.m.t. 0,1%
Ketonitrile	Not detectable	n.m.t 0,2%	n.m.t. 0,2%	n.m.t. 0,1%
Other Impurities				
Ceba	Not detectable	n.m.t 0,2%	n.m.t. 0,10%	n.m.t. 0,10%
Unknown impurity max	0.02 %	n.m.t.0,10%	n.m.t.0,10%	n.m.t.0,10%
Total Impurities	0.06 %	n.m.t. 1%	n.m.t. 0,4%	n.m.t. 0,4%
GLC				
Dicyclohexylamine content	Not detectable			n.m.t. 50 ppm
Specific rotation (in-house)	-0.007 °			min. -1° max. +1°
Specific rotation (USP)	-0.213 °	min. -1° max. +1°		min. -1° max. +1°
Foreign substances	n.m.t. 10 ppm			n.m.t. 10 ppm

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Bulk density	0.20 g/ml			min. 0,19 g/ml max. 0,32 g/ml
Tapped density	0.32 g/ml			min. 0,30 g/ml max. 0,50 g/ml
Particle size (Malvern - Scirocco 2000)				
d(0.1)	1.53 mcm			n.m.t. 2 mcm
d(0.5)	4.33 mcm			n.m.t. 4 mcm
d(0.9)	9.58 mcm			n.m.t. 10 mcm
RESIDUAL SOLVENTS				
Acetone	14.6 ppm			n.m.t. 250 ppm
Toluene	Not detectable			n.m.t. 250 ppm
ORGANIC VOLATILE IMP.				
Benzene	Not detectable			n.m.t. 2 ppm
Enantiomeric purity (HPLC)				
S isomer	50.3 %			min. 48% max. 52%
R isomer	49.7 %			min. 48% max. 52%

We hereby certify that the material identified above has been analysed and found in accordance with the specifications of USP and Ph. Eur..

Qualified Person

Quality Control Manager

Release date: 09/11/2016