LAMPIRAN

Lampiran 1. Certificate of Analysis Adapalen

AJANTA INDUSTRIES LIMITED

Factory : Unit – IV, Plot No. E-56, MIDC, Tarapur,
Taluka & District- Palghar, Pin - 401506, Maharashtra, INDIA.
Tel.: +91 8983035402, 8983069452, Telefax : (02525) 661082

CERTIFICATE OF ANALYSIS

ADAPALENE BP

Batch No.	: AJS-[illegible]	Date of Mfg.	: JUN-2019
A/R. No.	: FO/Sales/[illegible]	Date of Exp.	: MAY-2021
Batch Qty.	: 22.27 Kg	Date of Release	: [illegible]

Sr. #	TESTS	RESULTS	SPECIFICATION
1.	Description	Almost white powder	White or almost white powder
2.	Solubility	Complies	Sparingly soluble in Tetrahydrofuran, Practically insoluble in water and ethanol (96%)
3.	Identification A)By IR	Complies	The infra red spectrum of Sample should concordant with similar preparation of that of Adapalene WS
	B)By HPLC	Complies	The retention time of the major peak of the sample solution corresponds to that of standard solution, as obtained in the assay
4.	Appearance of Solution	Complies	The solution is clear and not more intensely coloured than reference solution BY5
5.	Related substances (By HPLC)	Below disregard limit	Impurity A : Not more than 0.2%
		Below disregard limit	Impurity C : Not more than 0.15%
		Below disregard limit	Impurity D : Not more than 0.2%
		Below disregard limit	Unspecified impurities : Not more than 0.10%
		Below disregard limit	Total impurities : Not more than 0.5%
6.	Heavy metals	Complies	Not more than 20 ppm
7.	Loss on drying (By 105°C for 4 hours)	0.14 %	Not more than 0.5 %
8.	Sulphated ash	0.04%	Not more than 0.1 %
9.	Assay (By HPLC)	100.27 %	Between 98.0 % to 102.0 % on dried substance
10.	Residual solvents (By HSGC)	Not detected	Dichloromethane : NMT 600 ppm
		77 ppm	Ethyl acetate : NMT 5000 ppm
		113 ppm	Tetrahydrofuran : NMT 720 ppm
		940 ppm	Toluene : NMT 890 ppm
		Not detected	Cyclohexane : NMT 3880 ppm
11.	Triethylamine content (By HPLC)	18 ppm	Not more than 50 ppm

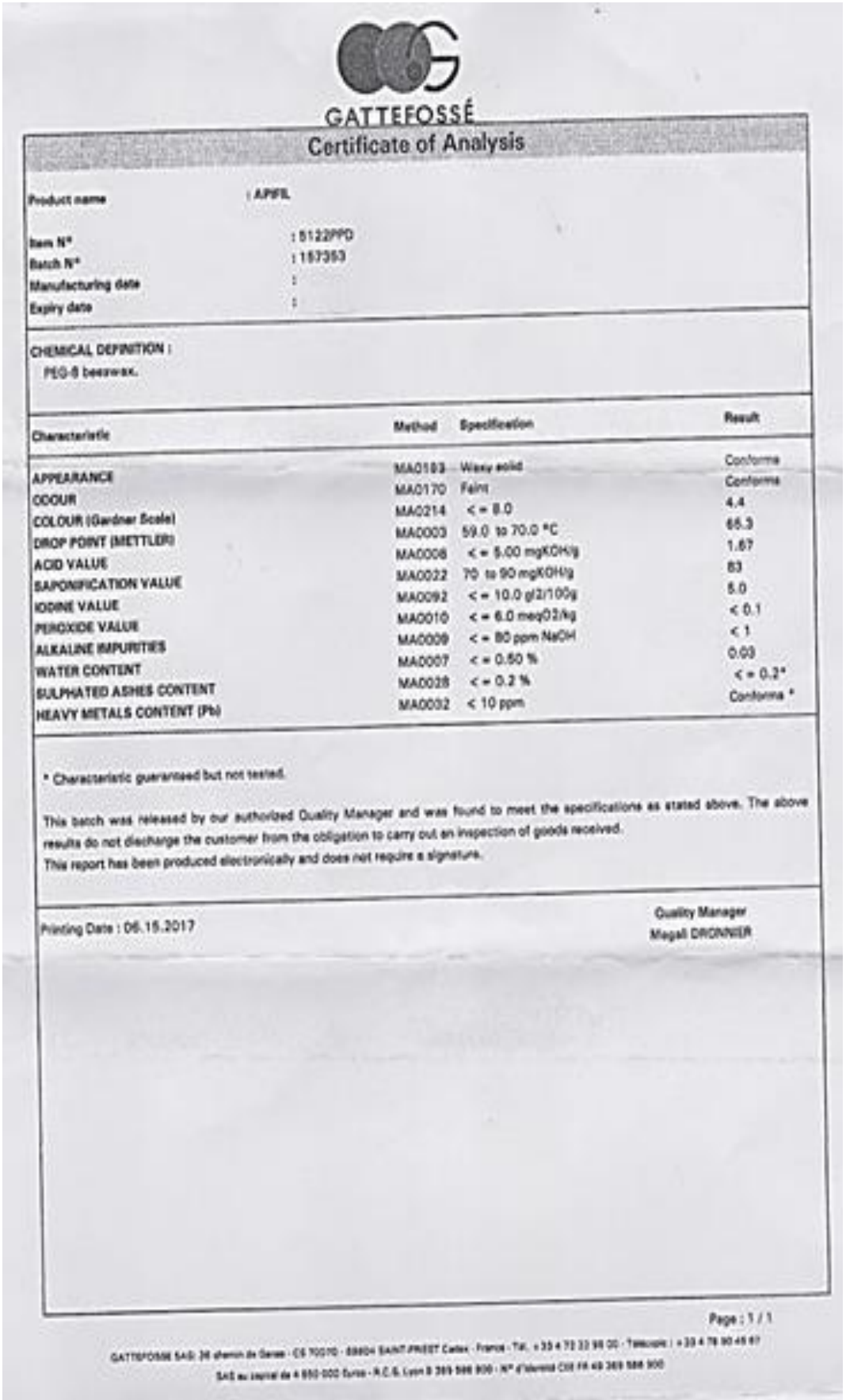
Remark: The material complies as per BP and above specifications

Analyst	Checked By	Approved By
Vikas Pardeshi Analyst	Mahendra Girase Head Quality Control	Shyamsundar Dasal Head Quality Assurance

AJANTA INDUSTRIES LTD.
Unit – IV, Plot No. E-56,
MIDC, Tarapur, Tal. & Dist. Palghar,
Pin – 401 506. Maharashtra. India.

HEAD OFFICE :
71, Udyog Kshetra, 2nd Floor, Mukund Goregaon Link Road, Mulund (W), Mumbai – 400 080. INDIA
Tel. : 00 - 91 - 22 - 67978666, 67970097, 21619185 • Fax : 00 - 91 - 22 - 25904806, 25653185 / 3234
Regd. Office : Plot No. B/1/23, G.I.D.C. Estate, Phase – III, Vapi-396195, Dist- Valsad, Gujarat, INDIA.

Lampiran 2. Certificate of Analysis Apifil



GATTEFOSSÉ

Certificate of Analysis

Product name : APIFIL

Item N° : 5122PPD
Batch N° : 157353
Manufacturing date :
Expiry date :

CHEMICAL DEFINITION :
PEG-8 beeswax.

Characteristic	Method	Specification	Result
APPEARANCE	MA0193	Waxy solid	Conforms
ODOUR	MA0170	Faint	Conforms
COLOUR (Gardner Scale)	MA0214	< = 8.0	4.4
DROP POINT (METTLER)	MA0003	59.0 to 70.0 °C	65.3
ACID VALUE	MA0006	< = 5.00 mgKOH/g	1.67
SAPONIFICATION VALUE	MA0022	70 to 90 mgKOH/g	83
IODINE VALUE	MA0092	< = 10.0 gI2/100g	5.0
PEROXIDE VALUE	MA0010	< = 6.0 meqO2/kg	< 0.1
ALKALINE IMPURITIES	MA0009	< = 80 ppm NaOH	< 1
WATER CONTENT	MA0007	< = 0.50 %	0.03
SULPHATED ASHES CONTENT	MA0028	< = 0.2 %	< = 0.2*
HEAVY METALS CONTENT (Pb)	MA0032	< 10 ppm	Conforms *

* Characteristic guaranteed but not tested.

This batch was released by our authorized Quality Manager and was found to meet the specifications as stated above. The above results do not discharge the customer from the obligation to carry out an inspection of goods received.
This report has been produced electronically and does not require a signature.

Printing Date : 06.15.2017

Quality Manager
Magali DRONNIER

Page : 1 / 1

GATTEFOSSE SAS: 36 chemin de Genas - CS 70010 - 69804 SAINT-PRIEST Cedex - France - Tel. : + 33 4 72 22 98 00 - Telefax : + 33 4 78 90 45 67
SAS au capital de 4 950 000 Euros - R.C.S. Lyon B 389 588 900 - N° d'identité CEE FR 49 389 588 900

Lampiran 3. Karakterisasi SLN Adapalen

formula	Ukuran partikel (nm)	Pdi (nm)	Zp (mV)
AAC 2%	132,47±65,71	0.30±0,09	-21,8±1,57
AAC 3%	88,52±0,79	0,21±0,01	-20,5±2,19
AAC 4%	98,51±1,16	0,24±0,02	-21,53±0,86
AAC 5%	101,4±1,25	0,20±0,01	-18,83±0,47
AAC 6%	124,56±0,20	0,28±0,01	-19,23±1,56

4.Perhitungan pembuatan larutan Adapalen

4.1 Larutan induk

Konsentrasi larutan = $\frac{bobot\ adapalene}{volume}$

100 ppm . = $\frac{bobot\ adapalene}{100\ mL}$

= 100 ppm x 100 mL

Bobot Adapalen = 10.000 µg = 10 mg

4.2 Pengenceran larutan

a. Konsentrasi 1 ppm

V_1 X C_1 = V_2 X C_2

V_1 X 100 ppm = 10 mL X 1 ppm

V1 = $\frac{10\ mL\ x\ 1\ ppm}{100\ ppm}$

V1 = 0,1 mL = 100 µL

b. Konsentrasi 2 ppm

V_1 X C_1 = V_2 X C_2

V_1 X 100 ppm = 10 mL X 2 ppm

V1 = $\frac{10\ mL\ x\ 2\ ppm}{100\ ppm}$

V1 = 0,2 mL = 200 µL

c. Konsentrasi 3 ppm

V_1 X C_1 = V_2 X C_2

V_1 X 100 ppm = 10 mL X 3 ppm

$V1 = \frac{10\ mL\ x\ 3\ ppm}{100\ ppm}$

V1 = 0,3 mL = 300 µL

d. Konsentrasi 4 ppm

V_1 X C_1 = V_2 X C_2

V_1 X 100 ppm = 10 mL X 4 ppm

$V1 = \frac{10\ mL\ x\ 4\ ppm}{100\ ppm}$

V1 = 0,4 mL = 400 µL

e. Konsentrasi 5 ppm

V_1 X C_1 = V_2 X C_2

V_1 X 100 ppm = 10 mL X 5 ppm

$V1 = \frac{10\ mL\ x\ 5\ ppm}{100\ ppm}$

V1 = 0,5 mL = 500 µL

f. Konsentrasi 6 ppm

V_1 X C_1 = V_2 X C_2

V_1 X 100 ppm = 10 mL X 6 ppm

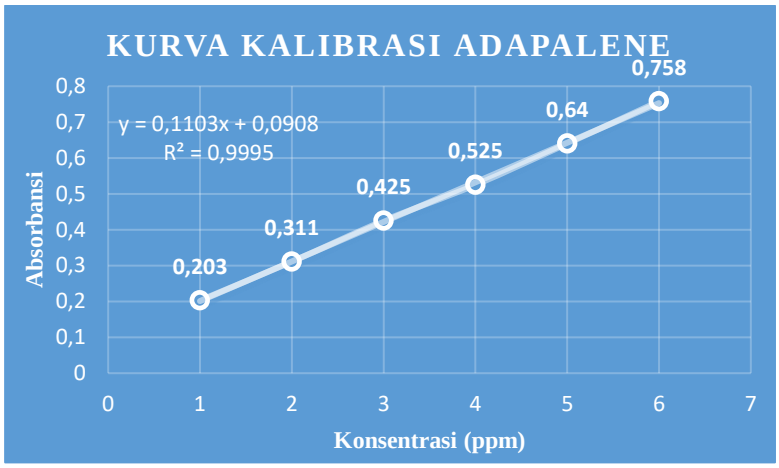
$V1 = \frac{10\ mL\ x\ 6\ ppm}{100\ ppm}$

V1 = 0,6 mL = 600 µL

4.3 Kurva kalibrasi Adapalen

Konsentrasi	Absorbansi
1 ppm	0,203
2 ppm	0,311
3ppm	0,425
4ppm	0,525
5ppm	0,640
6ppm	0,758

Lampiran 4. Kurva kalibrasi SLN Adapalen



Perhitungan %EE SLN Adapalen

Konsentrasi Larutan

$$\text{Konsentrasi} = \frac{y-a}{b}$$

Bobot Zat Aktif bebas

Bobot Zat Aktif Bebas = Konsentrasi X Volume X fp

Rumus EE

$$\%\ \text{EE} = \frac{Total\ zat\ aktif - zat\ aktif\ bebas}{Total\ zat\ aktif} \times 100\%$$

Lampiran 5. Perhitungan %EE SLN Adapalen

Formula	Absorbansi	EE%
AAC 2%	0,492	98,78%
AAC 3%	0,713	98,12%
AAC 4%	0,502	98,75%
AAC 5%	0,546	98,62%
AAC 6%	0,565	98,56%

1. AAC 2%

   Konsentrasi $= \frac{0,492-0,0908}{0,1103}$ = 3,64 ppm

   Bobot Zat Aktif Bebas = 3,64 ppm x 10 x 10 = 0,364 mg

   % EE = $\frac{30\ mg-0,364\ mg}{30\ mg}$ x 100%= 98,78 %

2. AAC 3%

   Konsentrasi $= \frac{0,713-0,0908}{0,1103}$ = 5,64 ppm

   Bobot Zat Aktif Bebas = 5,64 ppm x 10 x 10 = 0,564 mg

   % EE = $\frac{30\ mg-0,564\ mg}{30\ mg}$ x 100% = 98,12 %

3. AAC 4%

   Konsentrasi $= \frac{0,502-0,0908}{0,1103}$ = 3,73 ppm

   Bobot Zat Aktif Bebas = 3,73 ppm x 10 x 10 = 0,373 mg

   % EE = $\frac{30\ mg-0,373\ mg}{30\ mg}$ x 100% = 98,75 %

4. AAC 5%

   Konsentrasi $= \frac{0,546-0,0908}{0,1103}$ = 4,13 ppm

   Bobot Zat Aktif Bebas = 4,13 ppm x 10 x 10 = 0,413 mg

   % EE = $\frac{30\ mg-0,413mg}{30\ mg}$ x 100% = 98,62 %

5. AAC 6%

   Konsentrasi $= \frac{0,565-0,0908}{0,1103}$ = 4,3 ppm

   Bobot Zat Aktif Bebas = 4,3 ppm x 10 x 10 = 0,43 mg

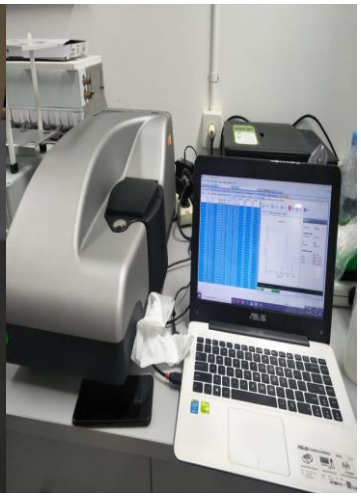
   % EE = $\frac{30\ mg-0,43\ mg}{30\ mg}$ x 100% = 98,56 %

Lampiran 6. Dokumentasi penelitian

Uji kelarutan

Uji solidifikasi

Sonikator *Probe*



Sentrifuga

Paticle Size Analyzer