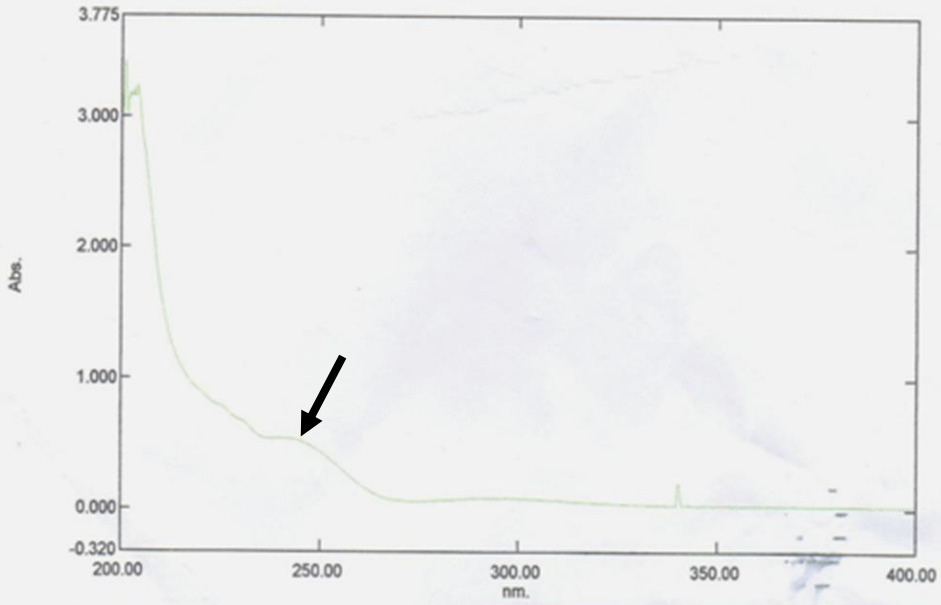
LAMPIRAN

Lampiran 1. Spektrum Panjang Gelombang Maksimal

Spectrum Peak Pick Report

01/12/2017 02:16:07 PM

Data Set: File_170112_135731 - RawData



[Measurement Properties]
Wavelength Range (nm.): 200.00 to 400.00
Scan Speed: Fast
Sampling Interval: 0.2
Auto Sampling Interval: Enabled
Scan Mode: Single

[Instrument Properties]
Instrument Type: UV-1800 Series
Measuring Mode: Absorbance
Slit Width: 1.0 nm
Light Source Change Wavelength: 350.0 nm
S/R Exchange: Normal

[Attachment Properties]
Attachment: 6-Cell
Number of cells: 4

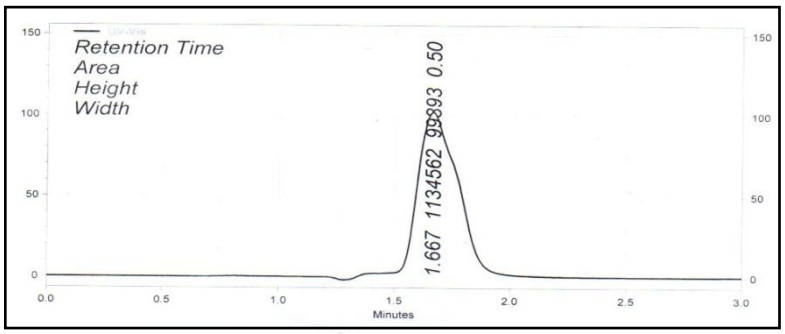
[Operation]
Threshold: 0.0010000
Points: 2
InterPolate: Disabled
Average: Disabled

[Sample Preparation Properties]
Weight:
Volume:
Dilution:
Path Length:
Additional Information:

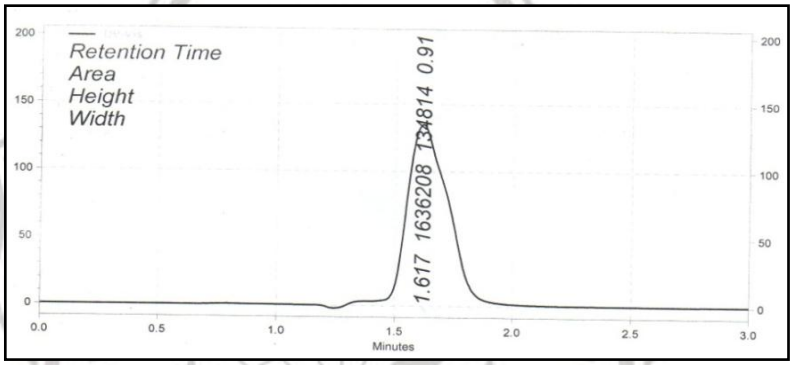
No.	P/V	Wavelength	Abs.	Description
25	↑	317.60	0.051	
26	↑	315.60	0.054	
27	↑	310.20	0.067	
28	↑	306.00	0.072	
29	↑	304.60	0.077	
30	↑	302.00	0.079	
31	↑	299.20	0.080	
32	↑	297.00	0.081	
33	↑	294.00	0.080	
34	↑	290.60	0.083	
35	↑	278.20	0.060	
36	↑	269.60	0.065	
37	↑	242.40	0.533	
38	↑	239.60	0.537	panjang gelo
39	↑	204.00	3.244	
40	↑	201.00	3.434	
41	↓	391.80	0.023	
42	↓	389.40	0.023	
43	↓	383.40	0.024	
44	↓	375.40	0.024	
45	↓	367.80	0.025	
46	↓	366.00	0.026	
47	↓	361.80	0.026	
48	↓	353.80	0.026	

Lampiran 2. Kurva Baku Ketokonazol

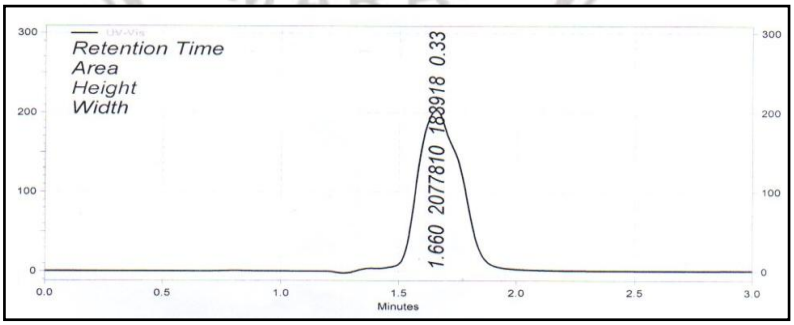
a. Larutan standar baku ketokonazol 2 µg/mL



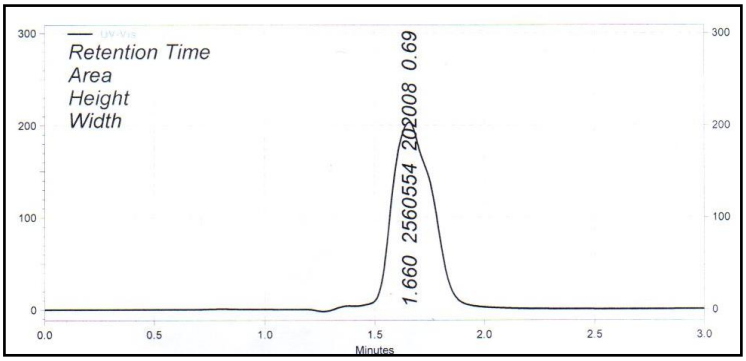
b. Larutan standar baku ketokonazol 4 µg/mL



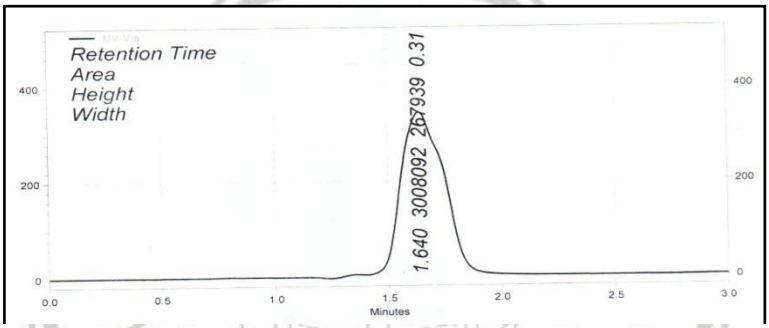
c. Larutan standar baku ketokonazol 6 µg/mL



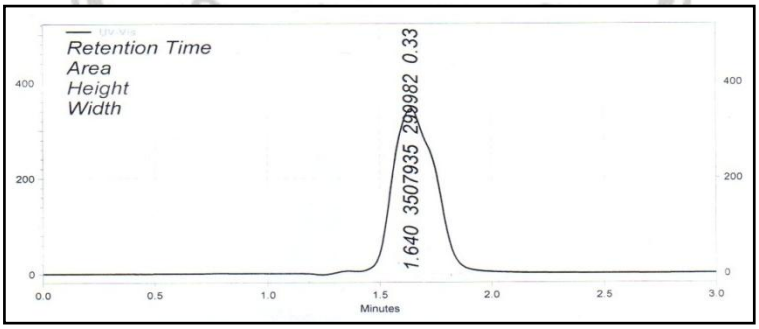
d. Larutan standar baku ketokonazol 8 µg/mL



e. Larutan standar baku ketokonazol 10 µg/mL

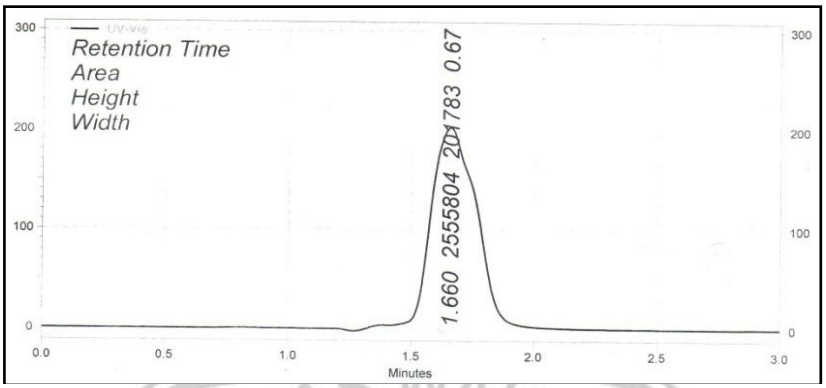


f. Larutan standar baku ketokonazol 12 µg/mL

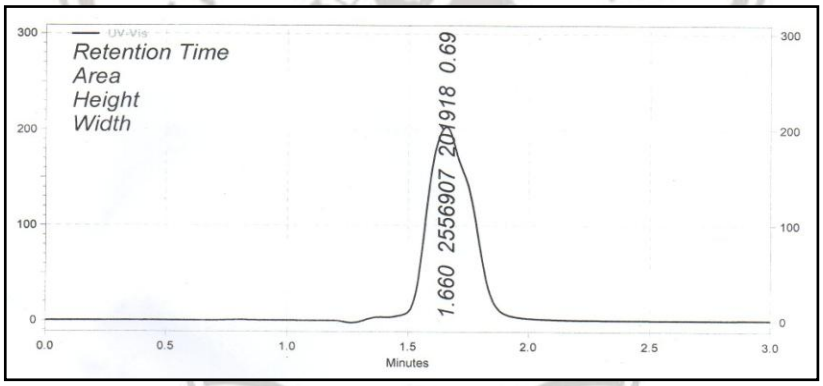


Lampiran 3. Contoh Kromatogram Sampel Ketokonazol Replikasi 6 kali

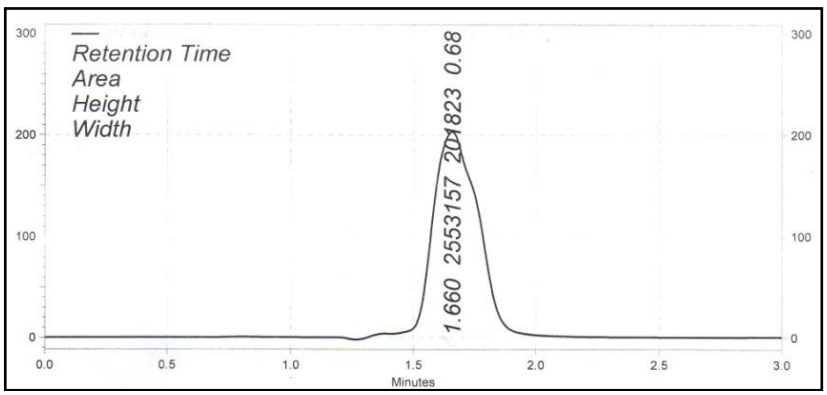
a. Kromatogram Sampel Ketokonazol Replikasi 1



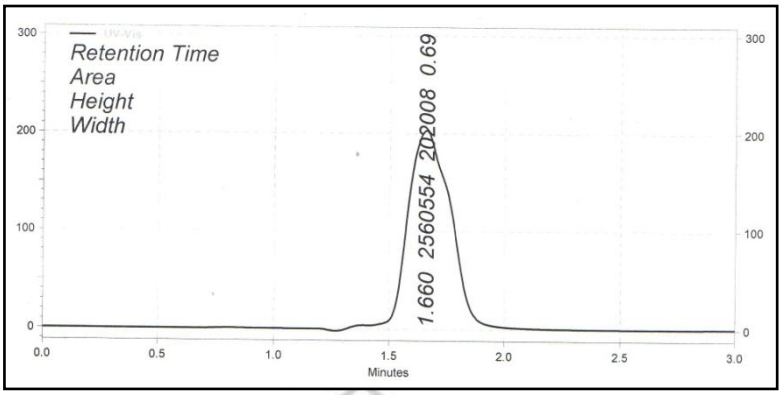
b. Kromatogram Sampel Ketokonazol Replikasi 2



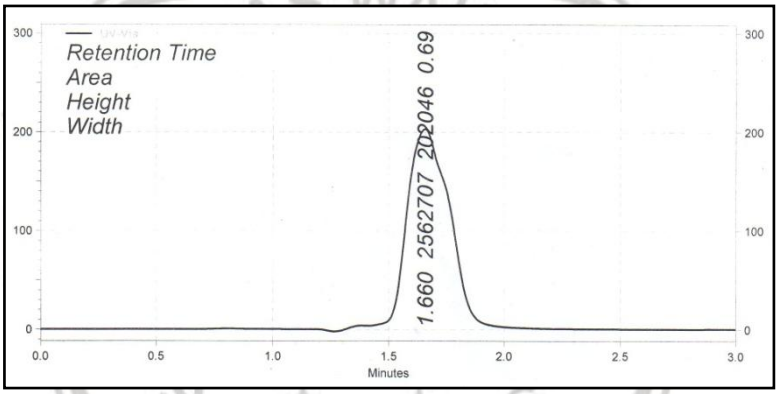
c. Kromatogram Sampel Ketokonazol Replikasi 3



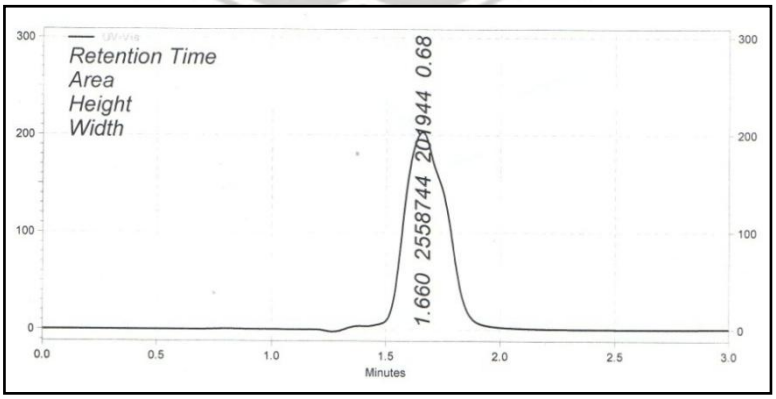
d. Kromatogram Sampel Ketokonazol Replikasi 4



e. Kromatogram Sampel Ketokonazol Replikasi 5



f. Kromatogram Sampel Ketokonazol Replikasi 6



Lampiran 4. Contoh Perhitungan Perolehan Kembali Ketokonazol dengan Metode *Standartd Addition Method*

1. Perolehan kembali pada larutan pembanding yang ditambah bahan baku sejumlah 80% dari target kadar analit dalam larutan pembanding
   a. Konsentrasi larutan pembanding sebelum penambahan bahan baku (B)
      1) Luas puncak ketokonazol = 1615967
      2) Kadar ketokonazol berdasarkan persamaan garis Y=235218x+674334,07 adalah 4.003 µg/mL
   b. Konsentrasi bahan baku yang ditambahkan (C)
      1) Luas puncak ketokonazol = 1134562
      2) Kadar ketokonazol berdasarkan persamaan garis Y = 235218x + 674334,07 adalah 1.957µg/mL
   c. Konsentrasi larutan pembanding yang diperoleh setelah penambahan bahan baku (A)
      1) Luas puncak total analit 1 = 2079420

         Luas puncak total analit 2 = 2084932

         Luas puncak total analit 3 = 2080649
      2) Berdasarkan persamaan garisY = 235218x + 674334,07 maka :

         Kadar total analit 1 = 5,974 µg/mL

         Kadar total analit 2 = 5,997 µg/mL

         Kadar total analit 3 = 5,979 µg/mL

2. Perhitungan perolehan kembali

$$\% \text{ perolehan kembali} = \frac{A-B}{C} \text{ x } 100\ \%$$

a. Analit 1

$$\% \text{ perolehan kembali} = \frac{5.974 - 4.003}{1,957} \text{ x } 100\ \%$$

$$= 100,7\%$$

b. Analit 2

$$\% \text{ perolehan kembali} = \frac{5,997\ - 4,003}{1,957} \text{ x } 100\ \%$$

$$= 101,8\%$$

c. Analit 3

$$\% \text{ perolehan kembali} = \frac{5,979 - 4,003}{1,957} \text{ x } 100\ \%$$

$$= 100,9\%$$

Lampiran 5. Perhitungan LOD dan LOQ Ketokonazol

X	Xi^2	$Xi-\bar{X}$	$(Xi-\bar{X})^2$	Yi	Yc	(Yi-Yc)	$(Yi-Yc)^2$
2	4	-5	25	1134562	1144770	-10208,1	104204693,1
4	16	-3	9	1636208	1615206	21001,93	441081063,7
6	36	-1	1	2077810	2085642	-7832,07	61341320,48
8	64	1	1	2560554	2556078	4475,93	20033949,36
10	100	3	9	3008092	3026514	-18422,1	339372663,1
12	144	5	25	3507935	3496950	10984,93	120668687,1
7	364		70				1086702377

Dari persamaan Y = 235218x + 674334,07 maka Yc dapat dihitung :

1. Y = 235218x + 674334,07
   Y = 235218 (2) + 674334,07
   Y = 1144770
2. Y = 235218x + 674334,07
   Y = 235218 (4) + 674334,07
   Y = 1615206
3. Y = 235218x + 674334,07
   Y = 235218 (6) + 674334,07
   Y = 2085642
4. Y = 235218x + 674334,07
   Y = 235218 (8) + 674334,07
   Y = 2556028
5. Y = 235218x + 674334,07
   Y = 235218 (10) + 674334,07
   Y = 3026514
6. Y = 235218x + 674334,07
   Y = 235218 (12) + 674334,07
   Y = 3496950
7. Persamaan kurva baku : Y = 235218x + 674334,07 (r = 0,99986)

$$S_{y/x} = \{\frac{\Sigma(Y_i - Y_c)^2}{n-2}\}^{1/2}$$

$= (1086702377/4)^{½}$

$= (271675594,3)^{½}$

$= 16482,58457$

$$S_a = S_{y/x}\sqrt{\frac{\sum X_i^2}{n\sum(X_i - X_{rata-rata})^2}}$$

$= 16482,58457 \text{ x}\sqrt{\frac{364}{6 \text{ x } 70}}$

$= 16482,58457 \text{ x } 0,931$

$= 15345,28624$

Perhitungan nilai LOD :

Nilai Y pada batas deteksi ditentukan dengan persamaan $Y = Y_B + 3\ S_B$

Y = nilai intersept (a) pada persamaan kurva kalibrasi

S_B = simpangan baku intersept (a) (S_a)

Y = 674334,07 + 3 (15345,28624)

= 720369,9287

Y =235218x + 674334,07

720369,9287=235218x + 674334,07

LOD = X = 0,196 µg/mL

Perhitungan nilai LOQ :

Nilai Y pada batas kuantifikasi ditentukan dengan persamaan $Y = Y_B + 10\ S_B$

Y = nilai intersept (a) pada persamaan kurva kalibrasi

S_B = simpangan baku intersept (a) (S_a)

Y = 674334,07 + 10 (15345,28624)

= 827786,9324

Y = 222167x + 228729

827786,9324 = 235218x + 674334,07

LOQ = X = 0,652 µg/mL

Lampiran 6. Contoh Perhitungan Kadar Ketokonazol dalam Sediaan Krim

Persamaan regresi linier kurva baku adalah

Y = bx+ a

Y = 235218x + 674334,07

Replikasi 1

Y = 235218x + 674334,07

2555804 = 235218x + 674334,07

X = 7,998 µg/mL

Faktor pengenceran 50x, sehingga kadar krim ketokonazol adalah

X = 7,998 µg/mL x 50

= 399,9 µg/mL

Kadar ketokonazol dalam 1 gram sampel = 399,9 µg/mL x 50

= 19995 g

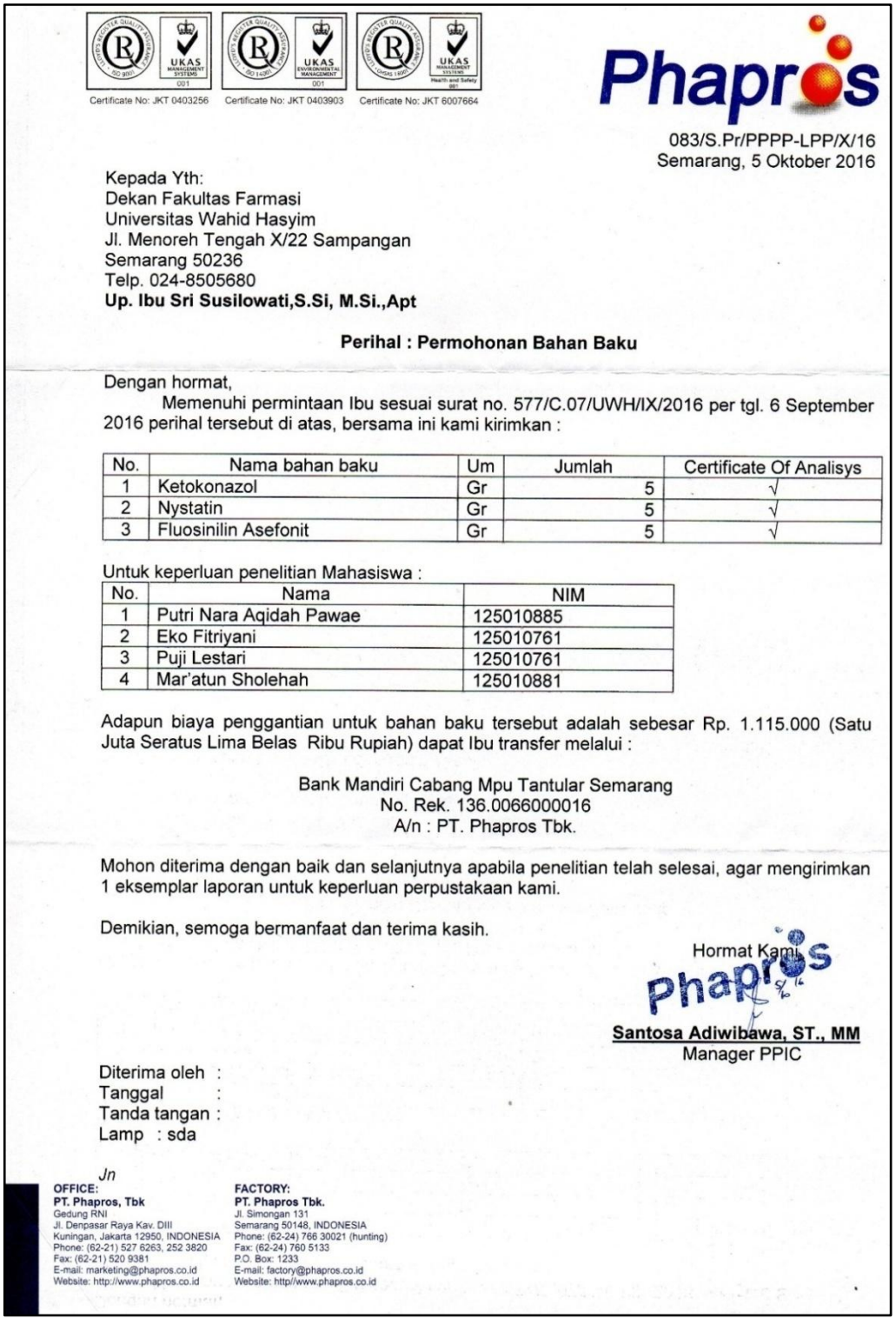
=19,995 mg

% Kadar krim ketokonazol = $\frac{19{,}995 \text{ mg}}{20\ mg}$ x 100%

= 99,975 %

Lampiran 7. Instrumen KCKT

Lampiran 8. Surat Penerimaan Bahan Baku Ketokonazol



Certificate No: JKT 0403256 Certificate No: JKT 0403903 Certificate No: JKT 6007664

Phapros

083/S.Pr/PPPP-LPP/X/16
Semarang, 5 Oktober 2016

Kepada Yth:
Dekan Fakultas Farmasi
Universitas Wahid Hasyim
Jl. Menoreh Tengah X/22 Sampangan
Semarang 50236
Telp. 024-8505680
Up. Ibu Sri Susilowati,S.Si, M.Si.,Apt

Perihal : Permohonan Bahan Baku

Dengan hormat,
Memenuhi permintaan Ibu sesuai surat no. 577/C.07/UWH/IX/2016 per tgl. 6 September 2016 perihal tersebut di atas, bersama ini kami kirimkan :

No.	Nama bahan baku	Um	Jumlah	Certificate Of Analisys
1	Ketokonazol	Gr	5	√
2	Nystatin	Gr	5	√
3	Fluosinilin Asefonit	Gr	5	√

Untuk keperluan penelitian Mahasiswa :

No.	Nama	NIM
1	Putri Nara Aqidah Pawae	125010885
2	Eko Fitriyani	125010761
3	Puji Lestari	125010761
4	Mar'atun Sholehah	125010881

Adapun biaya penggantian untuk bahan baku tersebut adalah sebesar Rp. 1.115.000 (Satu Juta Seratus Lima Belas Ribu Rupiah) dapat Ibu transfer melalui :

Bank Mandiri Cabang Mpu Tantular Semarang
No. Rek. 136.0066000016
A/n : PT. Phapros Tbk.

Mohon diterima dengan baik dan selanjutnya apabila penelitian telah selesai, agar mengirimkan 1 eksemplar laporan untuk keperluan perpustakaan kami.

Demikian, semoga bermanfaat dan terima kasih.

Hormat Kami,
Phapros
Santosa Adiwibawa, ST., MM
Manager PPIC

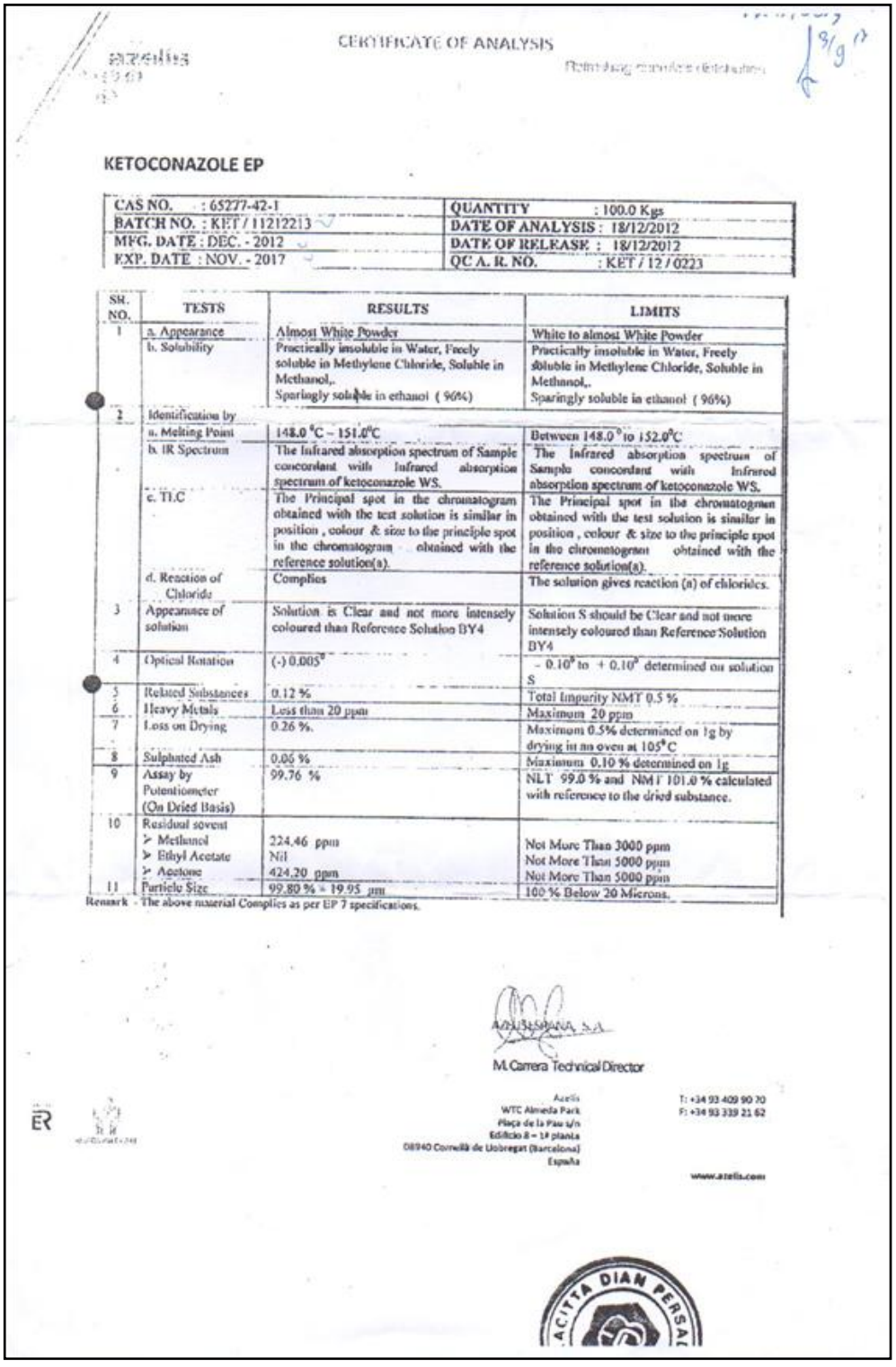
Diterima oleh :
Tanggal :
Tanda tangan :
Lamp : sda

Jn

OFFICE:
PT. Phapros, Tbk
Gedung RNI
Jl. Denpasar Raya Kav. DIII
Kuningan, Jakarta 12950, INDONESIA
Phone: (62-21) 527 6263, 252 3820
Fax: (62-21) 520 9381
E-mail: marketing@phapros.co.id
Website: http://www.phapros.co.id

FACTORY:
PT. Phapros Tbk.
Jl. Simongan 131
Semarang 50148, INDONESIA
Phone: (62-24) 766 30021 (hunting)
Fax: (62-24) 760 5133
P.O. Box: 1233
E-mail: factory@phapros.co.id
Website: http//www.phapros.co.id

Lampiran 9. Sertifikat Analisis Ketokonazol



CERTIFICATE OF ANALYSIS

KETOCONAZOLE EP

CAS NO. : 65277-42-1	QUANTITY : 100.0 Kgs
BATCH NO. : KET / 11212213	DATE OF ANALYSIS : 18/12/2012
MFG. DATE : DEC. - 2012	DATE OF RELEASE : 18/12/2012
EXP. DATE : NOV. - 2017	QC A. R. NO. : KET / 12 / 0223

SR. NO.	TESTS	RESULTS	LIMITS
1	a. Appearance	Almost White Powder	White to almost White Powder
	b. Solubility	Practically insoluble in Water, Freely soluble in Methylene Chloride, Soluble in Methanol,. Sparingly soluble in ethanol (96%)	Practically insoluble in Water, Freely soluble in Methylene Chloride, Soluble in Methanol,. Sparingly soluble in ethanol (96%)
2	Identification by		
	a. Melting Point	148.0 °C – 151.0°C	Between 148.0 ° to 152.0°C
	b. IR Spectrum	The Infrared absorption spectrum of Sample concordant with Infrared absorption spectrum of ketoconazole WS.	The Infrared absorption spectrum of Sample concordant with Infrared absorption spectrum of ketoconazole WS.
	c. TLC	The Principal spot in the chromatogram obtained with the test solution is similar in position , colour & size to the principle spot in the chromatogram obtained with the reference solution(a).	The Principal spot in the chromatogram obtained with the test solution is similar in position , colour & size to the principle spot in the chromatogram obtained with the reference solution(a).
	d. Reaction of Chloride	Complies	The solution gives reaction (a) of chlorides.
3	Appearance of solution	Solution is Clear and not more intensely coloured than Reference Solution BY4	Solution S should be Clear and not more intensely coloured than Reference Solution BY4
4	Optical Rotation	(-) 0.005°	- 0.10° to + 0.10° determined on solution S
5	Related Substances	0.12 %	Total Impurity NMT 0.5 %
6	Heavy Metals	Less than 20 ppm	Maximum 20 ppm
7	Loss on Drying	0.26 %.	Maximum 0.5% determined on 1g by drying in an oven at 105°C
8	Sulphated Ash	0.06 %	Maximum 0.10 % determined on 1g
9	Assay by Potentiometer (On Dried Basis)	99.76 %	NLT 99.0 % and NMT 101.0 % calculated with reference to the dried substance.
10	Residual solvent ➢ Methanol ➢ Ethyl Acetate ➢ Acetone	 224.46 ppm Nil 424.20 ppm	 Not More Than 3000 ppm Not More Than 5000 ppm Not More Than 5000 ppm
11	Particle Size	99.80 % - 19.95 µm	100 % Below 20 Microns.

Remark : The above material Complies as per EP 7 specifications.

M.Carrera Technical Director

Azelis
WTC Almeda Park
Plaça de la Pau s/n
Edificio 8 – 1ª planta
08940 Cornellà de Llobregat (Barcelona)
España

T: +34 93 409 90 70
F: +34 93 339 21 62

www.azelis.com

Lampiran 10. Surat Keterangan telah Melakukan Penelitian di Laboratorium Kimia Fakultas Wahid Hasyim Semarang

UNIVERSITAS WAHID HASYIM
FAKULTAS FARMASI
Jl. Menoreh Tengah X / 22 Sampangan - Semarang 50236 Telp. (024) 8505680 - 8505681 Fax. (024) 8505680

SURAT KETERANGAN
No 09/Lab. Kimia Farmasi/ C.05/UWH/IV/ 2017

Assalamu'alaikum Wr. Wb.

Yang bertanda tangan dibawah ini, Kepala Bagian Kimia Farmasi Fakultas Farmasi Universitas Wahid Hasyim Semarang menerangkan bahwa :

Nama : Eko Fitriyani
NIM : 125010814
Fak/ Univ/ Sekolah : Farmasi / Universitas Wahid Hasyim

Telah melakukan Penelitian Validasi menggunakan Spektrofotometer UV-Vis dan KCKT di Laboratorium Kimia Analisa, Fakultas Farmasi Universitas Wahid Hasyim Semarang, dengan judul penelitian :
"Validasi Metode Penetapan Kadar Ketokonazol Menggunakan Kromatografi Cair Kinerja Tinggi serta Aplikasinya Dalam Sediaan Krim"

Demikian surat keterangan ini dibuat untuk dipergunakan semestinya.

Wassalamu'alaikum Wr. Wb.

Semarang, April 2017
Ka.Bag Kimia Farmasi

Mirna Lailah, M.Sc, Apt