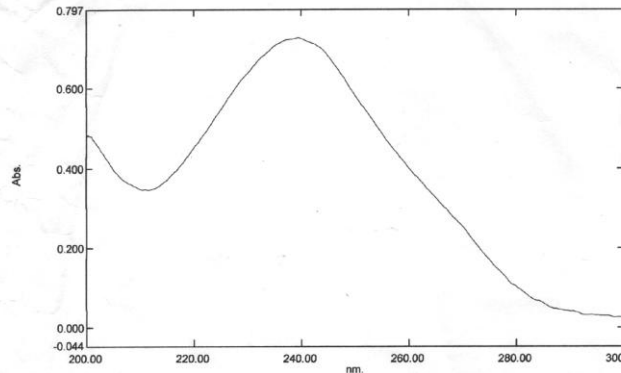


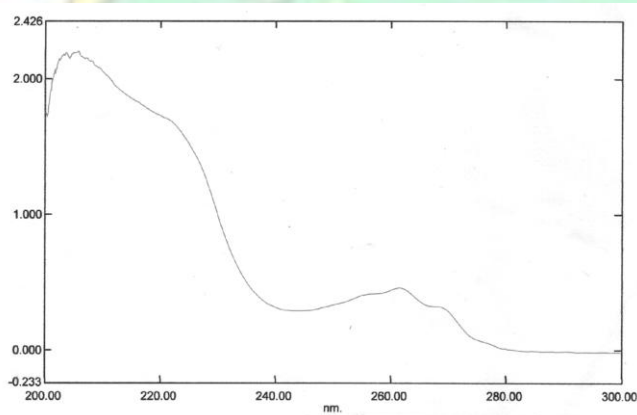
## Lampiran 1. Hasil Scanning Panjang Gelombang Betametason dan Deksklorfeniramin Maleat

### a. Panjang Gelombang Maksimum Betametason



No.	P/V	Wavelength	Abs.	Description
1	⊙	290.50	0.043	
2	⊙	287.40	0.049	
3	⊙	239.50	0.727	
4	⊙	211.10	0.348	
5	⊙	200.60	0.481	
6	⊙	298.30	0.027	
7	⊙	292.90	0.032	
8	⊙	290.10	0.042	
9	⊙	287.20	0.049	
10	⊙	211.70	0.346	
11	⊙	210.50	0.346	

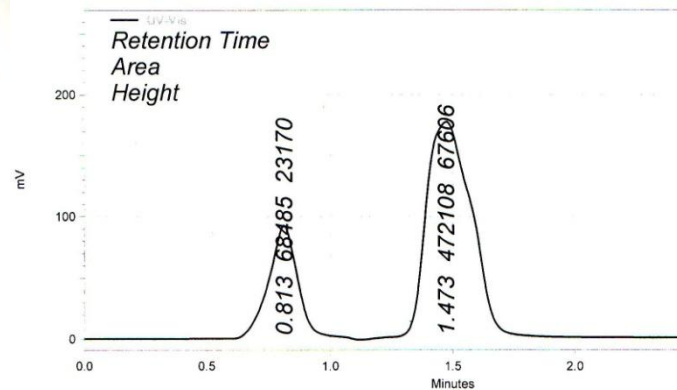
### b. Panjang Gelombang Maksimum Deksklorfeniramin Maleat



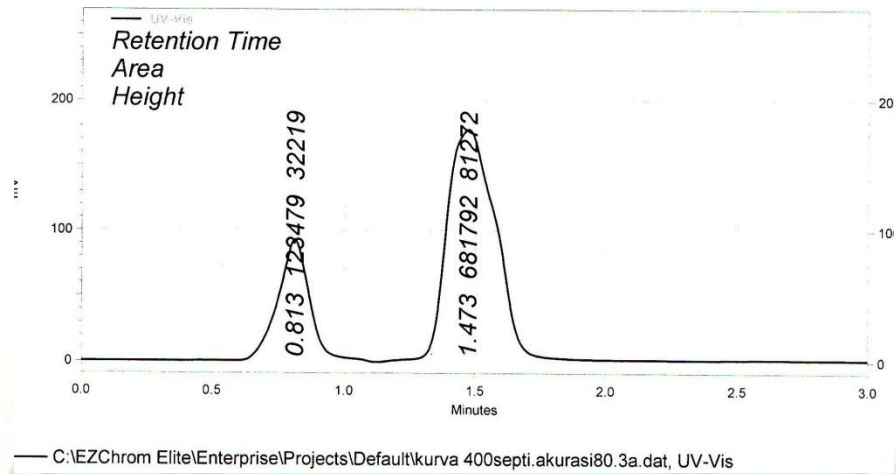
No.	P/V	Wavelength	Abs.	Description
1	⊙	297.00	-0.009	
2	⊙	294.00	-0.009	
3	⊙	290.50	-0.004	
4	⊙	288.20	-0.005	
5	⊙	287.40	-0.005	
6	⊙	284.60	-0.002	
7	⊙	261.60	0.462	
8	⊙	205.80	2.204	
9	⊙	203.70	2.195	
10	⊙	298.30	-0.012	
11	⊙	289.00	-0.007	
12	⊙	244.90	0.294	
13	⊙	242.10	0.296	
14	⊙	204.30	2.152	
15	⊙	200.40	1.726	

## Lampiran 2. Kurva Baku Betametason dan Deksklorfeniramin Maleat

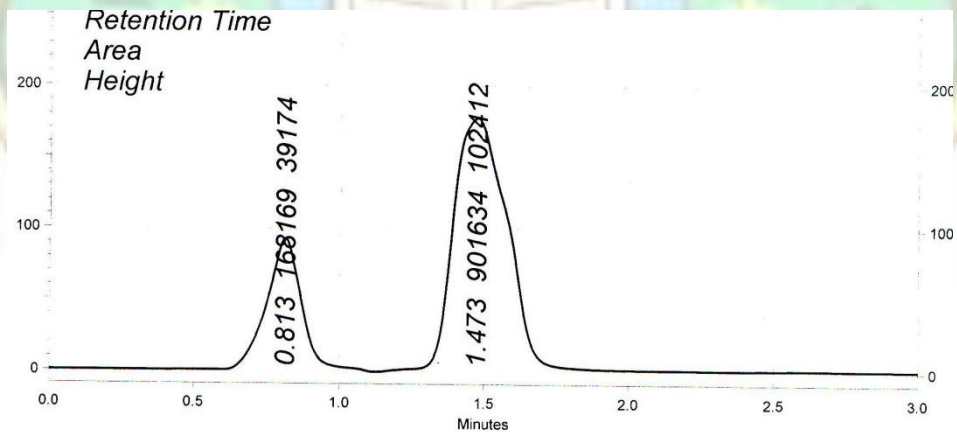
### a. Larutan Standar Baku Betametason dan Deksklorfeniramin Maleat 20 µg/mL



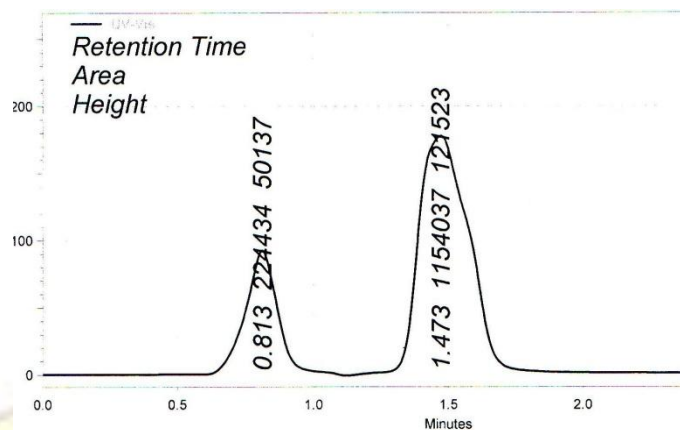
b. Larutan standar baku betametason dan deksklorfeniramin maleat 40



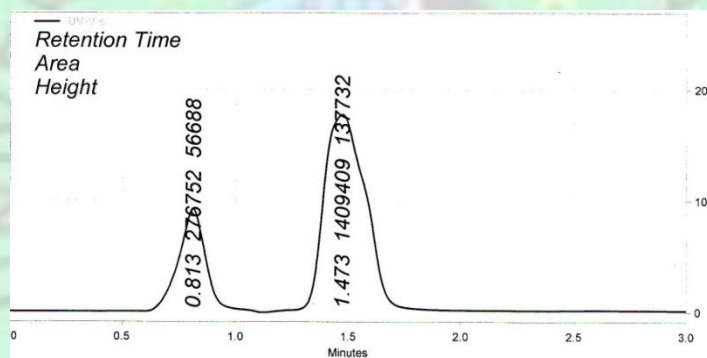
c. Larutan standar baku betametason dan deksklorfeniramin maleat 60 µg/mL



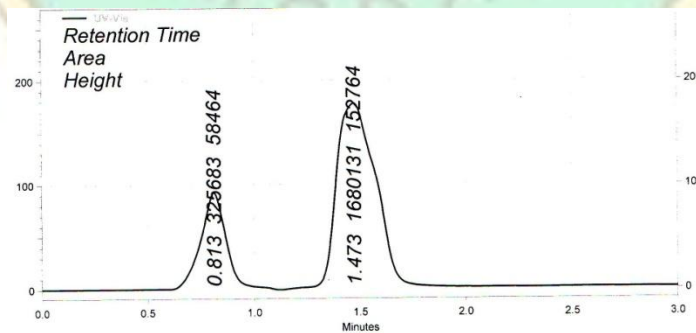
- d. Larutan standar baku betametason dan deksklorfeniramin maleat 80  $\mu\text{g/ml}$



- e. Larutan standar baku betametason dan deksklorfeniramin maleat 100  $\mu\text{g/ml}$

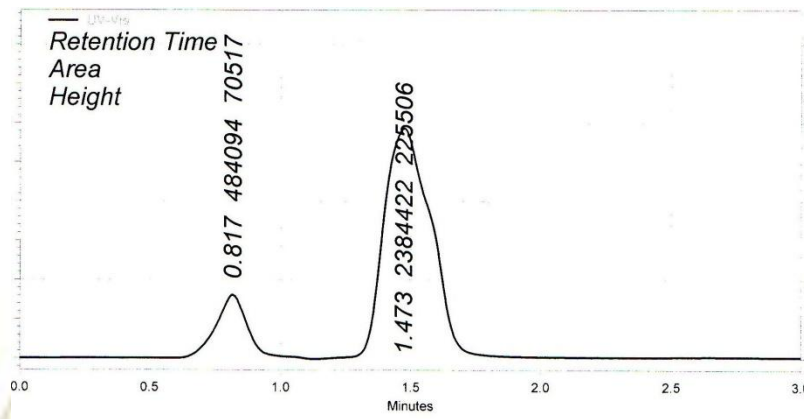


- f. Larutan standar baku betametason dan deksklorfeniramin maleat 120  $\mu\text{g/ml}$

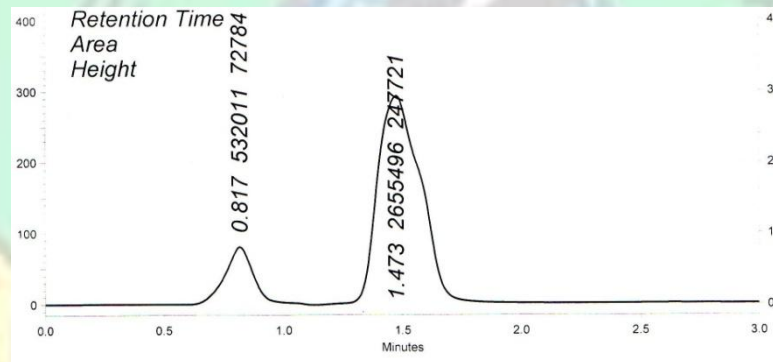


### Lampiran 3. Contoh kromatogram akurasi betametason dan deksklorfeniramin maleat

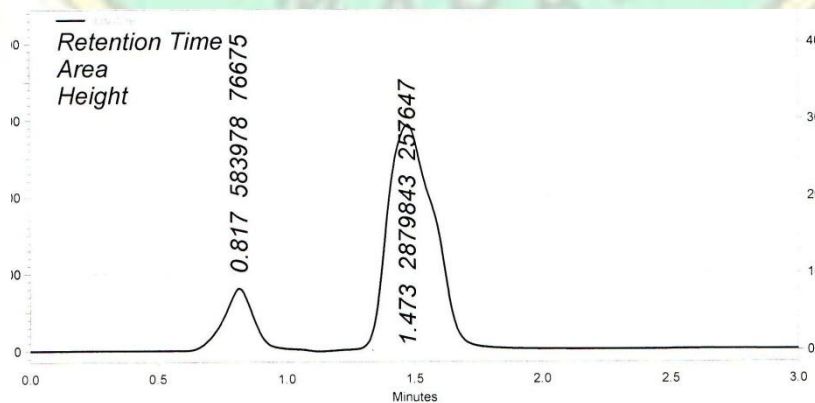
a. Akurasi betametason dan deksklorfeniramin maleat 80



b. Akurasi betametason dan deksklorfeniramin maleat 100

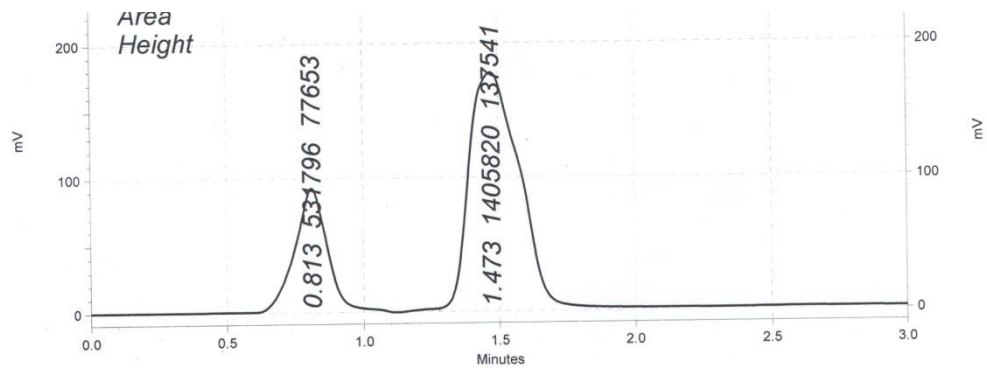


c. Akurasi betametason dan deksklorfeniramin maleat 120

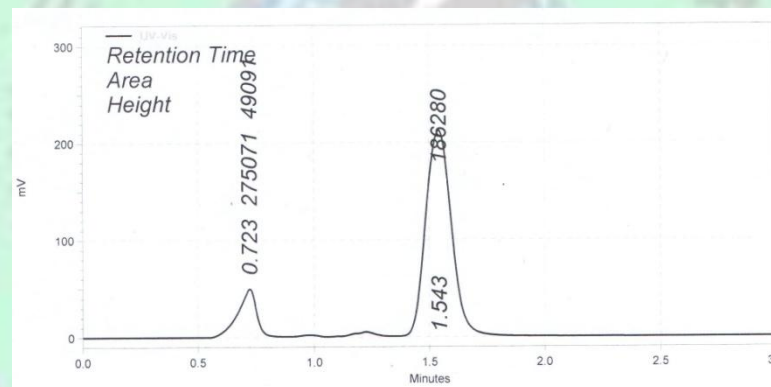


#### Lampiran 4. Kromatogram sampel betametason dan dekslorfeniramin maleat

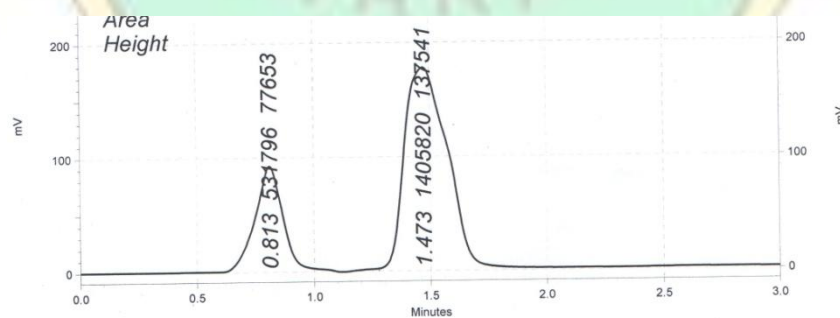
a. Kromatogram sampel betametason dan dekslorfeniramin maleat sirup A



b. Kromatogram sampel betametason dan dekslorfeniramin maleat sirup B



c. Kromatogram sampel betametason dan dekslorfeniramin maleat sirup C



**Lampiran 5. Contoh perhitungan perolehan kembali betametason dengan metode *standard addition method* sirup A**

1. Perolehan kembali pada sampel yang ditambah baku sejumlah 80% dari target kadar analit dalam sampel

a. Konsentrasi sampel sebelum penambahan bahan baku (B)

1) Luas puncak Betametason = 1413321

2) Kadar Betametason berdasarkan persamaan garis  $Y = 12107.67x + 202314.933$  adalah 100,020  $\mu\text{g/mL}$

b. Konsentrasi bahan baku yang ditambahkan (C)

1) Luas puncak Betametason = 1180121

2) Kadar Betametason berdasarkan persamaan garis  $Y = 12107.67x + 202314.933$  adalah 80,759  $\mu\text{g/mL}$

c. Konsentrasi sampel yang diperoleh setelah penambahan bahan baku (A)

1) Luas puncak total analit 1 = 2384422

Luas puncak total analit 2 = 2385269

Luas puncak total analit 3 = 2384065

2) Berdasarkan persamaan garis  $Y = 12107.67x + 202314.933$  maka dihasilkan :

Kadar total analit 1 = 180,23  $\mu\text{g/mL}$

Kadar total analit 2 = 180,30  $\mu\text{g/mL}$

Kadar total analit 3 = 180,20  $\mu\text{g/mL}$

2. Perhitungan perolehan kembali



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

a. Analit 1

$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{180,225 - 100,020}{80,759} \times 100 \% \\ &= 99,31\% \end{aligned}$$

b. Analit 2

$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{180,295 - 100,020}{80,759} \times 100 \% \\ &= 99,40\% \end{aligned}$$

c. Analit 3

$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{180,293 - 100,020}{80,759} \times 100 \% \\ &= 99,278\% \end{aligned}$$

#### Lampiran 6. Perhitungan LOD dan LOQ betametason

No	X	$X_i^2$	$X_i - X_{\square}$	$(X_i - X_{\square})^2$	$Y_i$	$Y_c$	$(Y_i - Y_c)$	$(Y_i - Y_c)^2$
1	20	400	-50	2500	472108	444468.3	27639.67	763951191.9
2	40	1600	-30	900	681792	686621.7	-4829.73	23326320.85
3	60	3600	-10	100	901634	928775.1	-27141.1	736641100.5
4	80	6400	10	100	1154037	1170929	-16891.5	285323887.1
5	100	10000	30	900	1409409	1413082	-3672.93	13490436.82
6	120	14400	50	2500	1680131	1655235	24895.67	619794235.4
$X_{\square}$	70	36400		7000				2442527173

Dari persamaan  $Y = 12107.67x + 202314.933$  maka  $Y_c$  dapat dihitung :

$$1. Y = 12107.67x + 202314.933$$

$$Y = 12107.67(20) + 202314.933$$

$$Y = 444468,33$$

$$2. Y = 12107.67x + 202314.933$$

$$Y = 12107.67(40) + 202314.933$$

$$Y = 686621,73$$

$$3. Y = 12107.67x + 202314.933$$

$$Y = 12107.67(60) + 202314.933$$

$$Y = 202314,933$$

$$4. Y = 12107.67x + 202314.933$$

$$Y = 12107.67(80) + 202314.933$$

$$Y = 1170928,53$$

$$5. Y = 12107.67x + 202314.933$$

$$Y = 12107.67(100) + 202314.933$$

$$Y = 1413081,93$$

$$6. Y = 12107.67x + 202314.933$$

$$Y = 12107.67(120) + 202314.933$$

$$Y = 1655235,33$$

7. Persamaan kurva baku :  $Y = 12107.67x + 202314.933 (r = 0,998)$

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

$$= (2442527174.1/4)^{1/2}$$

$$= 24710.97$$

$$S_a = S_y \times \frac{\sqrt{\sum X_i^2}}{n \sqrt{(\sum X_i - X_{rata-rata})^2}}$$

$$= 24710.97 \times \frac{\sqrt{36400}}{6 \times 7000}$$

$$= 24710.97 \times 0.930949336$$

$$= 23004.66$$



**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) ( $S_a$ )

$$Y = 202314.933 + 3 (23004.66)$$

$$= 271328.91$$

$$Y = 12107.67x + 202314.933$$

$$271328.91 = 12107.67x + 202314.933$$

$$\text{LOD} = X = 5,70 \mu\text{g/mL}$$

**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) ( $S_a$ )

$$Y = 202314.933 + 10 (23004.66)$$

$$= 69013,68$$

$$Y = 12107.67x + 202314.933$$

$$234451,54 = 12107.67x + 202314.933$$

$$\text{LOQ} = X = 19,00 \mu\text{g/mL}$$

**Lampiran 7. Perhitungan selektivitas betametason dan deksklorfeniramin  
maleat**

$$R = 2 \frac{(tR2 - tR1)}{W1 + W2}$$

$$R = 2 \frac{(1,417 - 0,893)}{0,9 + 1,2}$$

$$R = 3,34$$

### Lampiran 8. Contoh Perhitungan Betametason

Persamaan regresi linier kurva baku adalah

$$Y = BX + A$$

$$Y = 12107.67x + 202314.933$$

Replikasi 1

$$Y = 12107.67x + 202314.933$$

$$1405820 = 12107.67x + 202314.933$$

$$X = 99,400 \mu\text{g/mL}$$

Faktor pengenceran 10x, sehingga kadar betametason adalah

$$X = 99,400 \mu\text{g/mL} \times 10$$

$$= 994,00 \mu\text{g/mL}$$

$$\text{Kadar betametason dalam 1 ml sampel (0,25mg)} = \frac{0,25 \text{ mg}}{1000 \mu\text{g/mL}} \times 994,00 \mu\text{g/mL}$$

$$= 0,2485 \text{ mg}$$

$$\% \text{ Kadar betametason} = 0,994 \times 100\% = 99.4\%$$

### Lampiran 9. Certificate of Analysis betametason

15/0756  
No. 021209  
May. 11. 2015  
01/02

## CERTIFICATE OF ANALYSIS

Product : Dexchlorpheniramine Maleate		Quantity : 75 kg
Standard : USP38		Manufacturing Date : Dec. 11. 2014
Lot No. : 4Y010		Retest Date : Dec. 11. 2017
Evaluation : PASSED		Evaluation Date : Dec. 11. 2014

Test Item	Specification	Result
Description	White, odorless, crystalline powder	White, odorless crystalline powder
Identification		
A. IR		
	The IR absorption spectrum exhibits maxima only at the same wavelengths as that of a similar preparation of USP-RS.	Conform
B. The Retention Times of the Maleic Acid and Dexchlorpheniramine Peaks		
	Those of the Sample solution correspond to those of the Standard solution, as obtained in the Assay.	Conform
Assay	98.0 - 102.0 %	99.5 %
Impurities		
Residue on Ignition	Not more than 0.2 %	0.0 %
Organic Impurities		
Pheniramine	Not more than 0.4 %	0.0 %
Any other unspecified impurity	Not more than 0.10 %	0.07 %
Total impurities	Not more than 1 %	0 %
Enantiomeric Purity		
R-enantiomer	Not more than 2 %	1 %
Specific Tests		
Optical Rotation	+ 39.5 ° to + 43.0 °	+42.2 °
pH	4.0 - 5.0	4.9

Continued on next page

Note

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KONGO CHEMICAL CO.,LTD.  
NO.3,HIMATA,TOYAMA,930-0912,JAPAN  
TEL (076)-423-3131

*J. Yamafuji*  
Tomoko Yamafuji  
Release Decision Manager

Lampiran 10. *Certificate of Analysis* dekslorfeniramin maleat



B0. 05/04/01  
09/06/09

 **Crystal pharma**

**CERTIFICATE of ANALYSIS**

**BETAMETHASONE VALERATE USP37, MICRONIZED**

**BATCH N.:** B0073/1 13071    **MAN. DATE:** Apr/14    **RE-TEST DATE:** Apr/19

**DESCRIPTION:** White or almost white, odorless powder. Practically insoluble in water; freely soluble in acetone, in methylene chloride and in chloroform; soluble in alcohol; slightly soluble in benzene and in ether.

TEST	SPECIFICATIONS	RESULTS
1. Description	White or almost white, odorless powder	Complies
2. Identification	IR TLC	Concordant Concordant
3. Specific optical rotation	+ 75° to + 82° (c=1, dioxane, 25 °C)	+ 78 °
4. Loss on drying	≤ 0.5 %	0.1 %
5. Residue on ignition	≤ 0.2 % (platinum crucible)	< 0.2 %
6. Chromatographic purity HPLC	Individual ≤ 1.0 % Total ≤ 2.0 %	0.10 % 0.35 %
7. HPLC assay (dried basis)	97.0 - 103.0 %	99.0 %
8. Residual solvents	Methanol < 3000 ppm Acetone < 5000 ppm Tetrahydrofuran < 720 ppm Heptane < 5000 ppm	18 ppm 560 ppm 11 ppm 28 ppm
9. Particle size	99 % ≤ 20 µm 90 % ≤ 10 µm	9 µm 5 µm

21/05/14  
Quality Assurance Department

MARAI

Lampiran 11. Surat keterangan laboratorium



**UNIVERSITAS WAHID HASYIM**  
**FAKULTAS FARMASI**  
**BAGIAN KIMIA FARMASI**

Jl. Menoreh Tengah X / 22 Sampangan – Semarang 50236 Telp. (024) 8505680 – 8505681 fax. (024) 8505680

**SURAT KETERANGAN**

No. 03/Lab. Kimia Farmasi/ C.05/UWH/X/ 2016

Assalamu'alaikum Wr. Wb.

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

Nama : Septi Ayu Dianti  
NIM : 125010788  
Fak/ Univ/ Sekolah : Farmasi / Universitas Wahid Hasyim Semarang

Telah melakukan Penelitian Validasi menggunakan alat Spektrofotometer UV-Vis dan HPLC di Laboratorium Kimia Analisa, Fakultas Farmasi Universitas Wahid Hasyim Semarang, dengan judul penelitian :

“ Validasi Metode Penetapan Kadar Betametason dan Deksklorfeniramin Maleat Menggunakan Kromatografi Cair Kinerja Tinggi Serta Aplikasinya dalam Sediaan Sirup”

Demikian surat keterangan ini dibuat untuk dipergunakan semestinya.

Wassalamu'alaikum Wr. Wb.

Semarang, Oktober 2016

Ka. Bag Kimia Farmasi

  
Maria Liliati, M.Sc, Apt



## Lampiran 12. Gambar Alat-Alat yang Digunakan Penelitian



## Kromatografi Cair Kinerja Tinggi (Jasco) Spektrofotometer UV-Vis (Shimadzu)





Lampiran 13. Gambar Sampel Sirup A, B dan C

