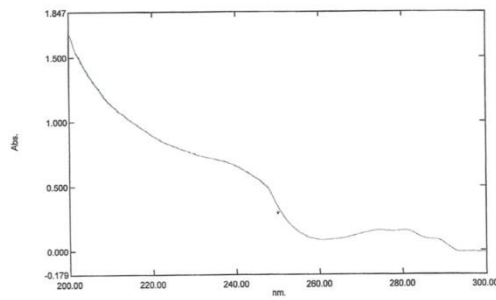


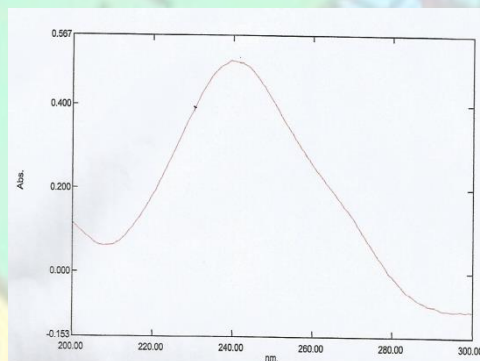
Lampiran 1. Hasil *Scanning* Panjang Gelombang Deksametason dan Deksklorfeniramin Maleat

a. Deksametason



No.	P/V	Wavelength	Abs.	Description
1	⊕	297.00	-0.007	
2	⊕	294.00	-0.007	
3	⊕	290.50	-0.002	
4	⊕	287.50	-0.002	
5	⊕	284.60	0.003	
6	⊕	279.70	0.019	
7	⊕	261.60	0.237	
8	⊕	256.20	0.237	
9	⊕	255.10	0.239	
10	⊕	242.70	0.165	
11	⊕	298.30	-0.010	
12	⊕	290.10	-0.004	
13	⊕	289.00	-0.005	
14	⊕	279.50	0.019	
15	⊕	258.40	0.229	
16	⊕	255.80	0.237	
17	⊕	242.90	0.164	
18	⊕	241.60	0.162	

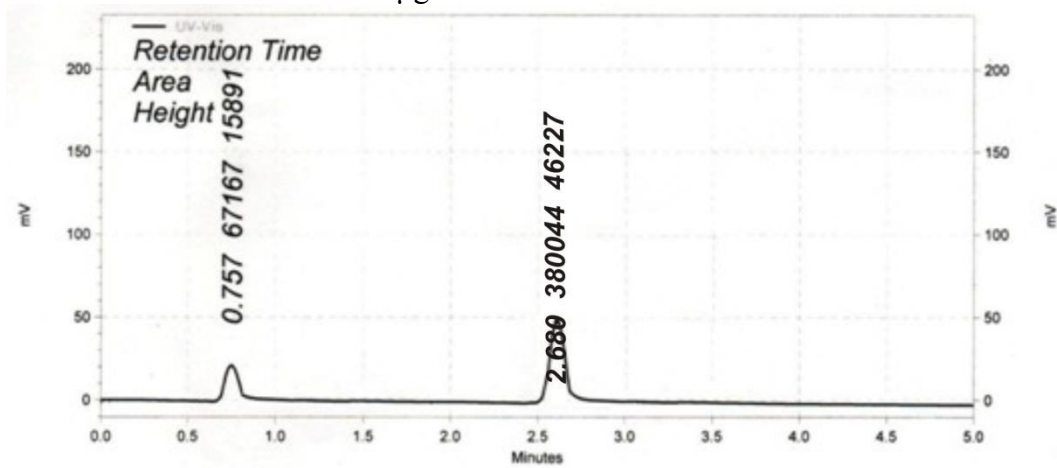
b. Deksklorfeniramin maleat



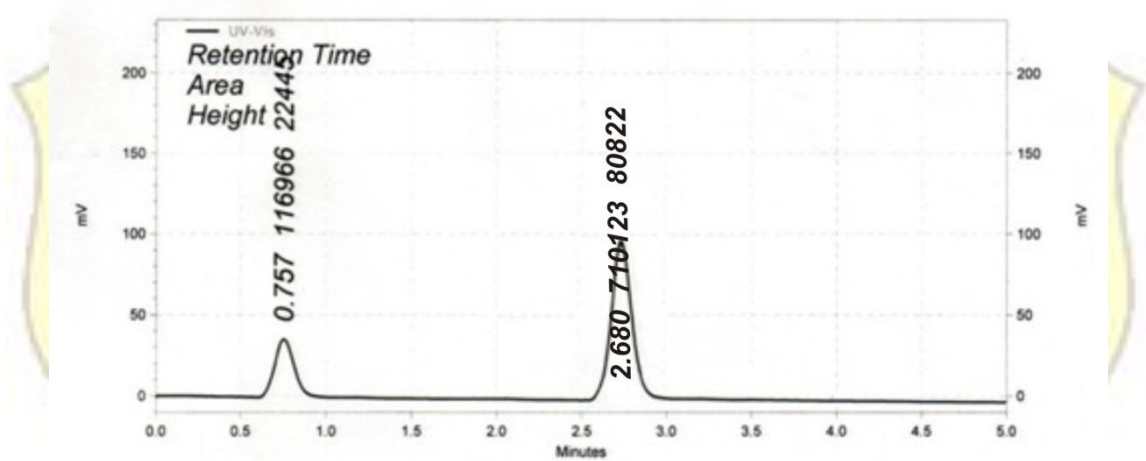
No.	P/V	Wavelength	Abs.	Description
1	⊕	299.20	-0.091	
2	⊕	297.00	-0.090	
3	⊕	290.50	-0.080	
4	⊕	287.40	-0.071	
5	⊕	242.30	0.501	
6	⊕	239.70	0.507	
7	⊕	298.30	-0.093	
8	⊕	294.80	-0.092	
9	⊕	292.90	-0.090	
10	⊕	290.10	-0.081	
11	⊕	287.20	-0.071	
12	⊕	209.60	0.065	
13	⊕	207.90	0.063	

Lampiran 2. Kromatogram Hasil Uji Linieritas

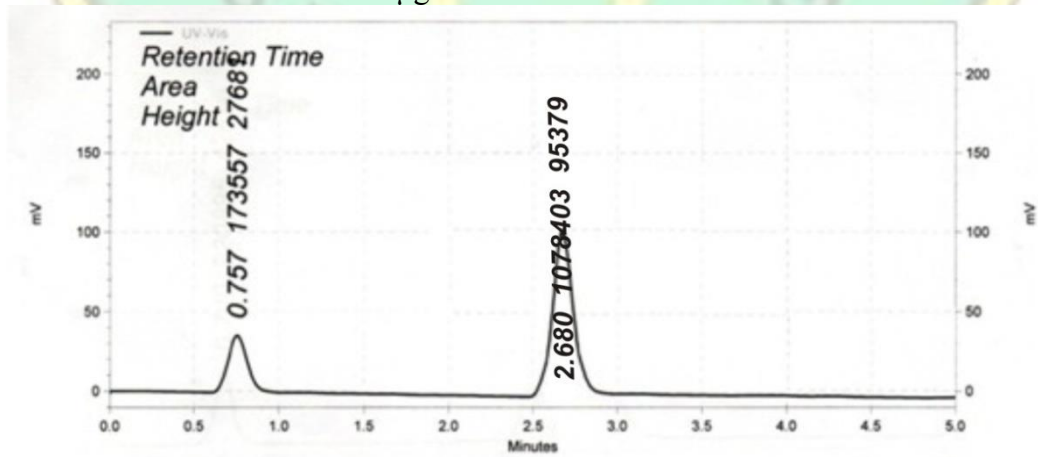
a. Kadar DK dan DM 10 $\mu\text{g/ml}$



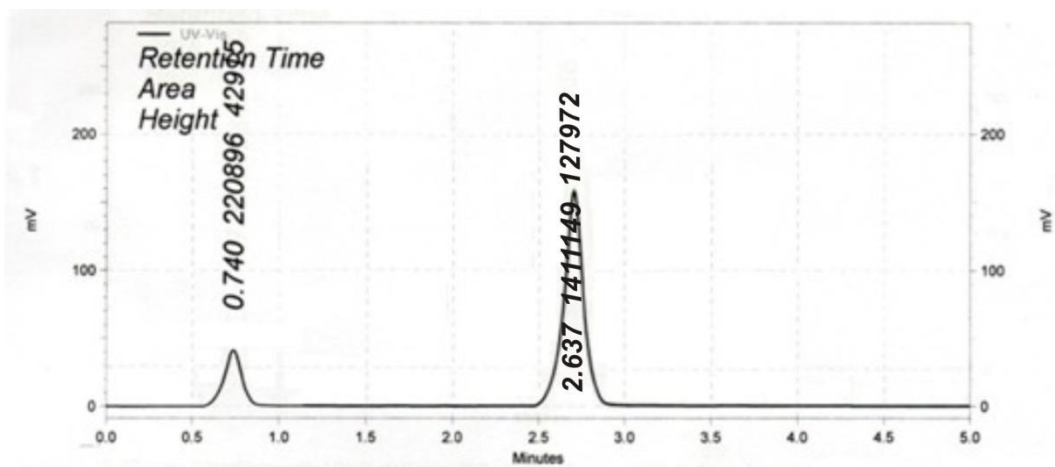
b. Kadar DK dan DM 20 $\mu\text{g/ml}$



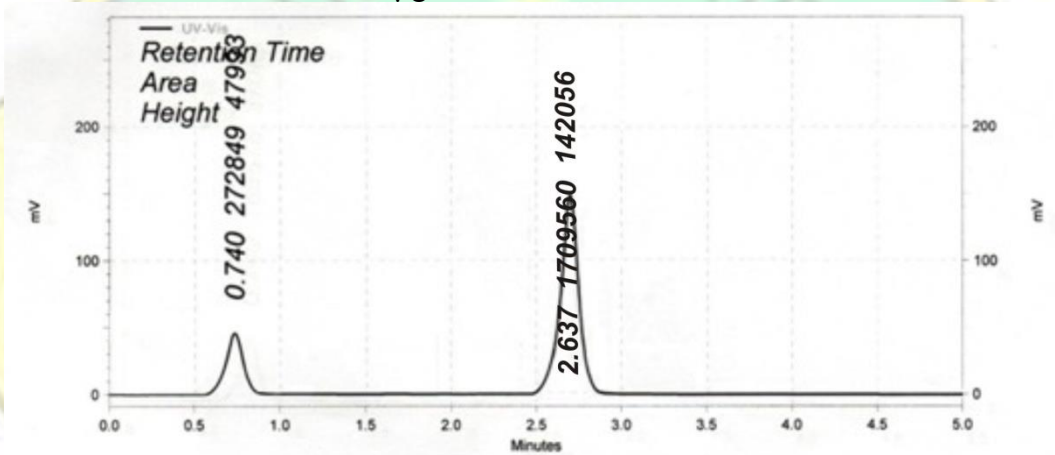
c. Kadar DK dan DM 30 $\mu\text{g/ml}$



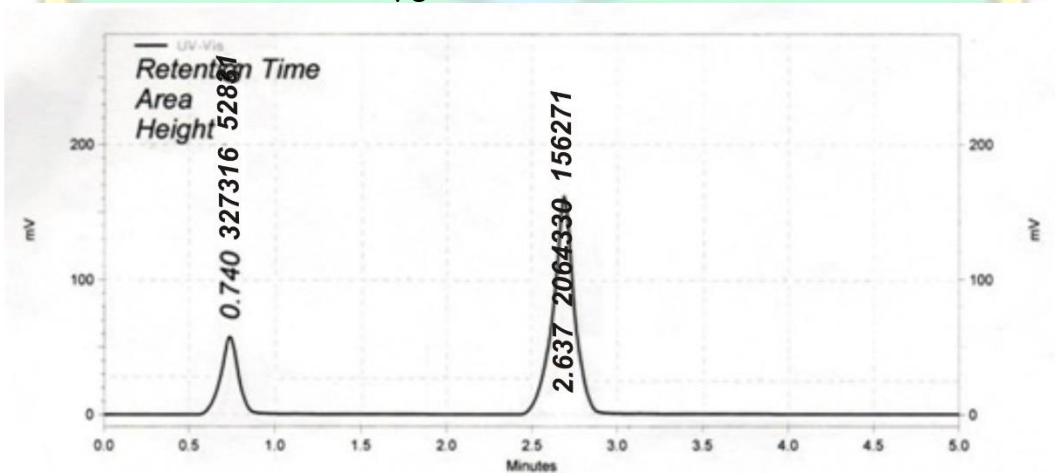
d. Kadar DK dan DM 40 $\mu\text{g/ml}$



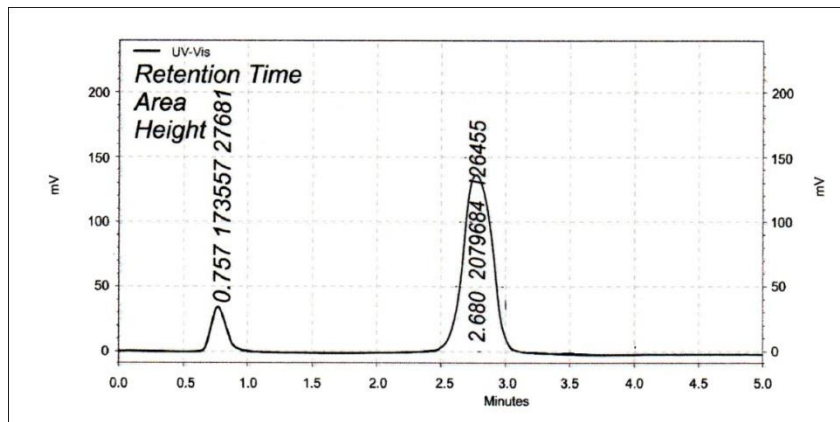
e. Kadar DK dan DM 50 $\mu\text{g/ml}$



f. Kadar DK dan DM 60 $\mu\text{g/ml}$



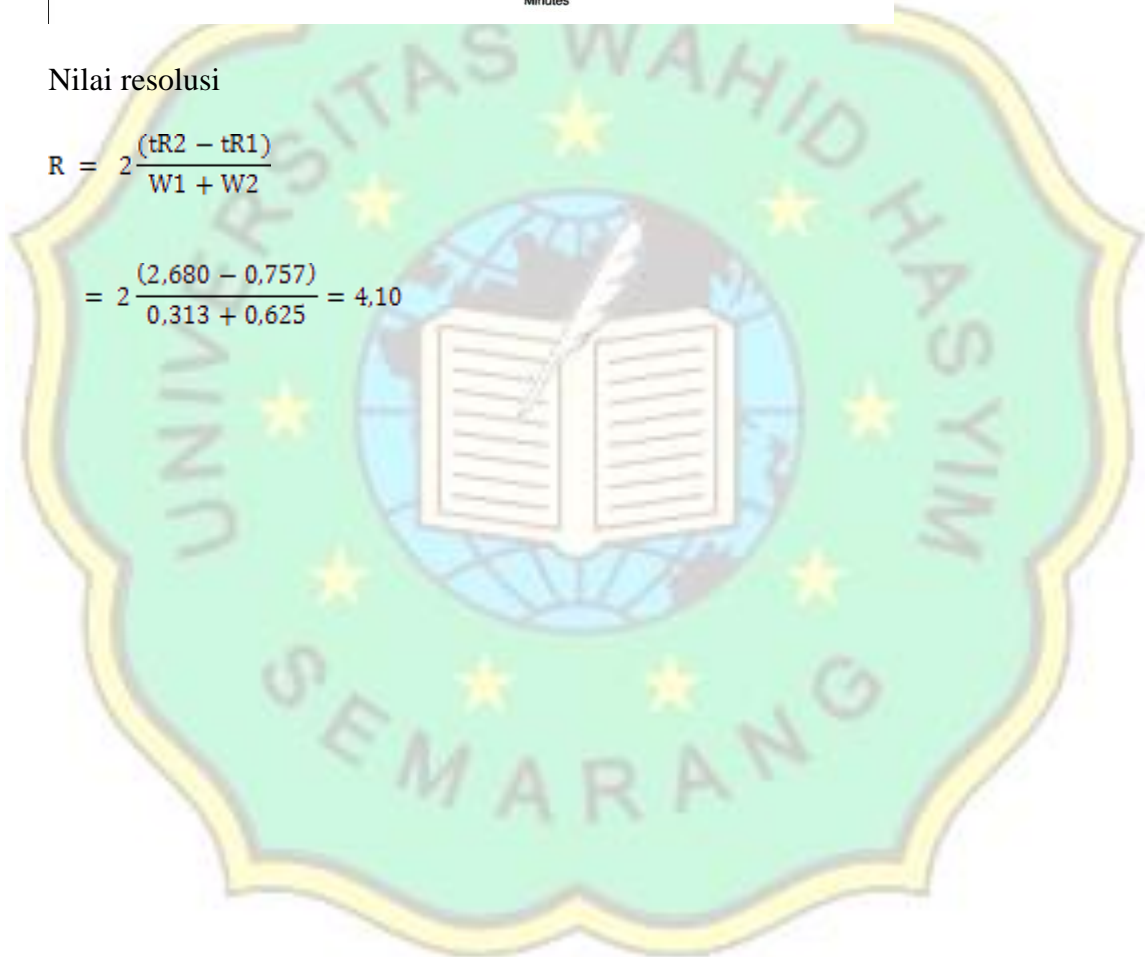
Lampiran 3. Contoh Perhitungan Nilai Resolusi (R)



Nilai resolusi

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

$$= 2 \frac{(2,680 - 0,757)}{0,313 + 0,625} = 4,10$$



Lampiran 4. Contoh Perhitungan Perolehan Kembali Deksklorfeniramin Maleat 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 DK = 276737

2) Kadar DK berdasarkan persamaan garis $Y = 5187,81x + 14885,20$ adalah 50,47 $\mu\text{g/mL}$

b. Konsentrasi bahan baku yang ditambahkan (C)

1) Luas puncak DK = 225233

2) Kadar DK berdasarkan persamaan garis $Y = 5187,81x + 14885,20$ adalah 40,55 $\mu\text{g/mL}$

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

1) Luas puncak total analit 1 = 484393

Luas puncak total analit 2 = 485429

Luas puncak total analit 3 = 487672

2) Berdasarkan persamaan garis $Y = 5187,81x + 14885,20$ maka :

Kadar total analit 1 = 90,50 $\mu\text{g/mL}$

Kadar total analit 2 = 90,70 $\mu\text{g/mL}$

Kadar total analit 3 = 91,13 $\mu\text{g/mL}$

2. Perhitungan perolehan kembali

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

a. Analit 1

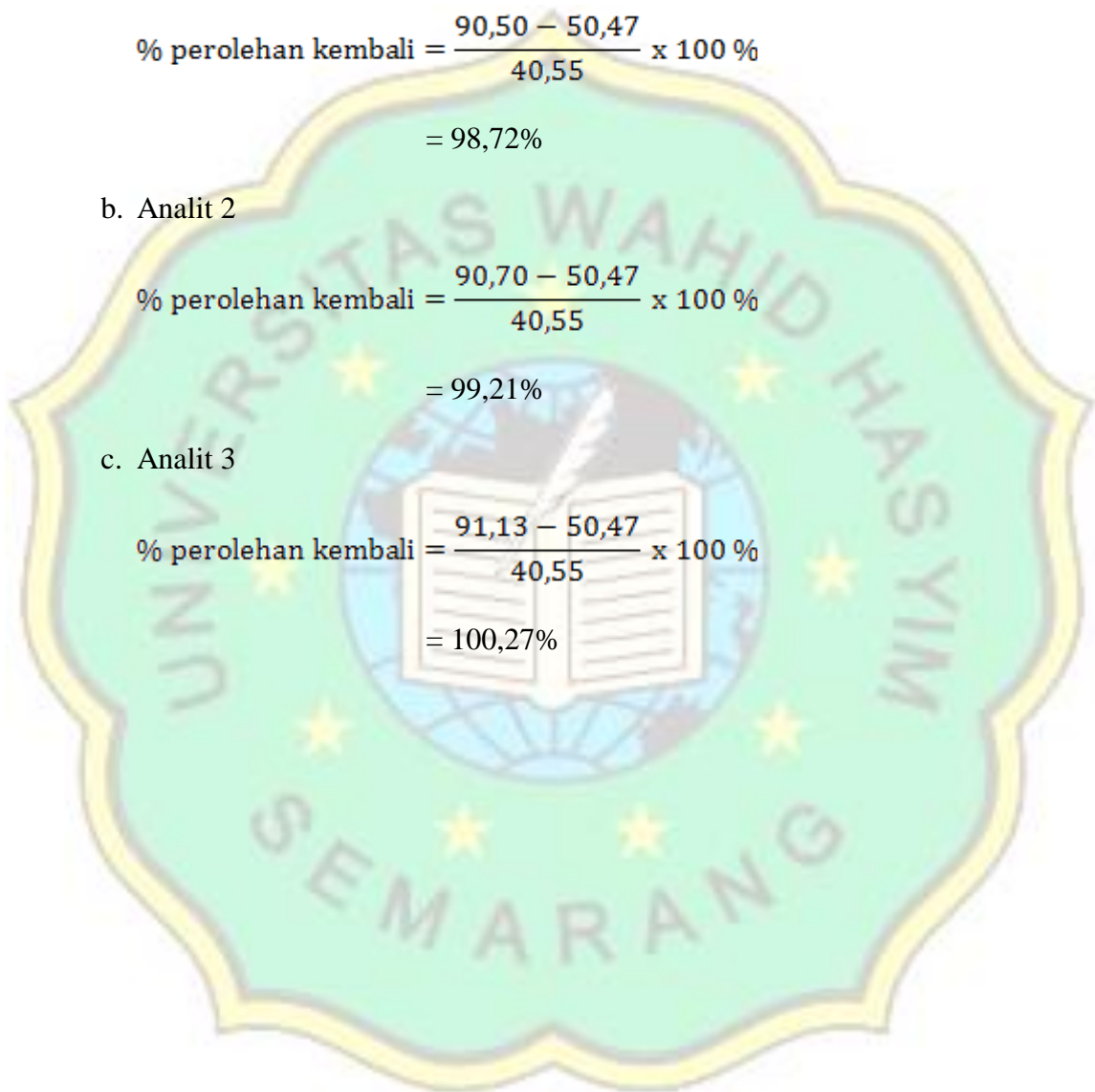
$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{90,50 - 50,47}{40,55} \times 100 \% \\ &= 98,72\% \end{aligned}$$

b. Analit 2

$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{90,70 - 50,47}{40,55} \times 100 \% \\ &= 99,21\% \end{aligned}$$

c. Analit 3

$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{91,13 - 50,47}{40,55} \times 100 \% \\ &= 100,27\% \end{aligned}$$



Lampiran 5. Perhitungan LOD dan LOQ Deksklorfeniramin Maleat

no	X	X_i^2	$X_i - \bar{X}$	$(X_i - \bar{X})^2$	Y_i	Y_c	$(Y_i - Y_c)$	$(Y_i - Y_c)^2$
1	10	100	-25	625	67167	66763,29	403,71	162981,8
2	20	400	-15	225	116966	118641,4	-1675,38	2806898
3	30	900	-5	25	173557	170519,5	3037,53	9226589
4	40	1600	5	25	220896	222397,6	-1501,56	2254682
5	50	2500	15	225	272849	274275,7	1426,65	2035330
6	60	3600	25	625	327316	326153,7	1162,26	1350848
\bar{X}	35	$\Sigma 9100$		$\Sigma 1750$				$\Sigma 1783732$ 9

Dari persamaan $Y = 5187,81x + 14885,20$ maka Y_c dapat dihitung :

1. $Y = 5187,81x + 14885,20$

$$Y = 5187,81(10) + 14885,20$$

$$Y = 66763,30$$

2. $Y = 5187,81x + 14885,20$

$$Y = 5187,81(20) + 14885,20$$

$$Y = 118641,40$$

3. $Y = 5187,81x + 14885,20$

$$Y = 5187,81(30) + 14885,20$$

$$Y = 170519,50$$

4. $Y = 5187,81x + 14885,20$

$$Y = 5187,81(40) + 14885,20$$

$$Y = 222397,60$$

5. $Y = 5187,81x + 14885,20$

$$Y = 5187,81(50) + 14885,20$$

$$Y = 274275,70$$

$$6. Y = 5187,81x + 14885,20$$

$$Y = 5187,81(60) + 14885,20$$

$$Y = 326153,80$$

$$7. \text{ Persamaan kurva baku : } Y = 5187,81x + 14885,20 \text{ (} r = 0,999 \text{)}$$

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

$$= (17837329/4)^{1/2}$$

$$= 2111,71$$

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

$$= 2111,71x \sqrt{\frac{9100}{6 \times 1750}}$$

$$= 2111,71x0,93$$

$$= 1963,90$$

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)

$$Y = 14885,20 + 3 (1963,90)$$

$$= 20776,90$$

$$Y = 5187,81x + 14885,20$$

$$20776,90 = 5187,81x + 14885,20$$

$$\text{LOD} = X = 1,14 \mu\text{g/mL}$$

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)

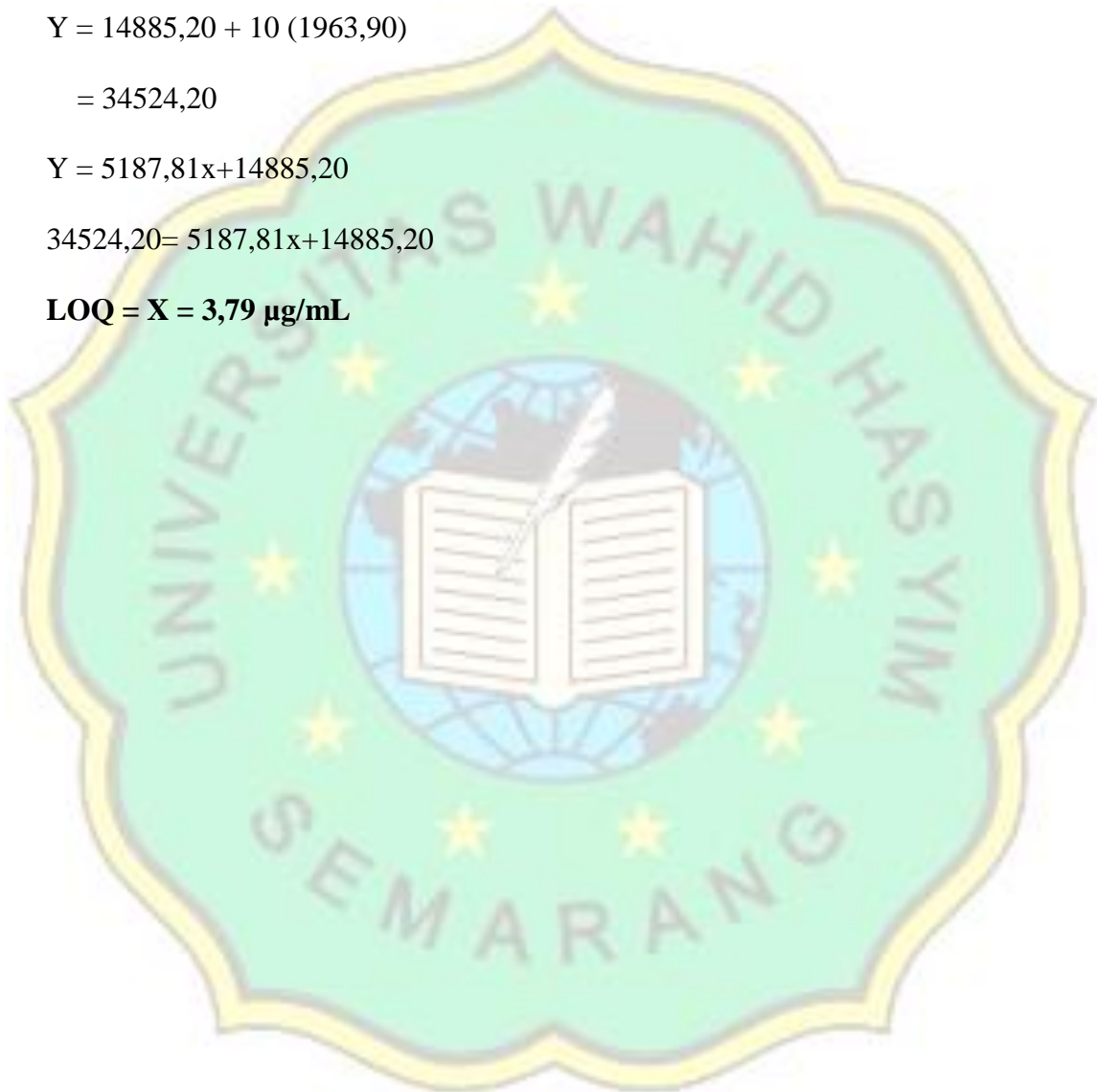
$$Y = 14885,20 + 10 (1963,90)$$

$$= 34524,20$$

$$Y = 5187,81x + 14885,20$$

$$34524,20 = 5187,81x + 14885,20$$

$$\mathbf{LOQ = X = 3,79 \mu\text{g/mL}}$$



Lampiran 6. Contoh Perhitungan Kadar Deksklorfeniramin Maleat

Persamaan regresi linier kurva baku adalah

$$Y = BX + A$$

$$Y = 5187,81x + 14885,20$$

Replikasi 1

$$Y = 5187,81x + 14885,20$$

$$533637 = 5187,81x + 14885,20$$

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

Faktor pengenceran 10x, sehingga kadar DK adalah

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

$$= 999,90 \mu\text{g/mL}$$

$$\begin{aligned} \text{Kadar DK dalam 1 ml sampel (0,4 mg)} &= \frac{0,4 \text{ mg}}{1000 \mu\text{g/mL}} \times 999,90 \mu\text{g/mL} \\ &= 0,39996 \text{ mg} \end{aligned}$$

$$\% \text{ Kadar DK} = \frac{0,39}{0,4} \times 100\%$$

$$= 99,99\%$$

Lampiran 7. Surat Permohonan Bahan Baku



Certificate No: JKT 0403256

Certificate No: JKT 0403903

Certificate No: JKT 6007664

Phapros

047/S.P/PPPP-LPP/IV/16
Semarang, 11 April 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. 238/C.07/UWH/II/2016 per tgl. 31 Maret 2016 perihal tersebut di atas, bersama ini kami kirimkan :

No.	Nama bahan baku	Um	Jumlah	Certificate Of Analysis
1	Dexamethasone	Gr	10	√
2	Betamethason	Gr	10	√
3	Dexchlorpheniramine maleat	Gr	10	√
4	Acetaminophen	Gr	10	√
5	Chlorpheniramine maleat	Gr	10	√

Untuk keperluan penelitian Mahasiswa :

No.	Nama	NIM
1	Ira Afifah	125010823
2	Nur aini	125010793
3	Tri wahyuni lestari	125010778
4	Septi ayu dianti	125010788

Perlu kami informasikan bahwa permintaan bahan baku **PPA** tidak dapat kami penuhi karena kami tidak mempunyai persediaan bahan baku tersebut.

Adapun biaya penggantian untuk bahan baku tersebut adalah sebesar Rp. 700.000 (Tujuh ratus 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
Mr. 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 50145, 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 8. Certificate of Analysis Deksklorfeniramin Maleat

No. 021209
May.11.2015
01/02

CERTIFICATE OF ANALYSIS

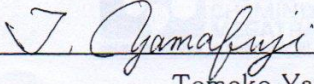
Product : Dexchlorpheniramine Maleate	Quantity : 75 kg
Standard : USP38	Manufacturing Date : Dec. 11. 2014
Lot No. : 4Y010	Retest Date : Dec. 11. 2017
Evaluation : PASSED	Evaluation Date : Dec. 11. 2014

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


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


Tomoko Yamafuji
 Release Decision Manager

Lampiran 9. Certificate of Analysis Deksmetason

20/03 2015 15:02 882224134098 TIANFA #1423 P.008
 15/03/2015

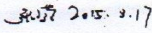

 **天津天药药业股份有限公司**
 TIANJIN TIANYAO PHARMACEUTICALS CO., LTD.

 **CERTIFICATE OF ANALYSIS** **REVIEWED**
 By Mahanie at 4:54 pm, Mar 31, 2015

Page: 1 / 1

Product	Dexamethasone	Manufacturing date	2015.02.12
Batch No.	ND 150204	Report date	2015.03.17
Test criteria	BP1993	Expiry date	2020.02.12
Test	Specifications	Results	
Appearance	A white or almost white, crystalline powder	White crystalline powder	
Melting point	About 255°C	259.5 - 260.5°C	
Identification	B: IR; C: TLC	Conforms	
Light absorption	380 ~ 410	390	
Specific optical rotation	+75° ~ +80°	+77°	
Related substances	1.0% < Only one impurity spot ≤ 2.0%	Conforms	
Loss on drying	≤ 0.5%	0.24%	
Assay*	96.0% ~ 104.0%	96.9%	
Particle size	Micronized	Conforms	
Batch size	69.78kg		
Quantity	65kg		
Conclusion	The above product conforms to BP1993.		

*Storage conditions: Kept in a well-closed container and protected from light
 *Calculated on the dried basis

Checked by:  2015.3.17 Approved by:  2015.3.17

**Lampiran 10. Surat Keterangan Telah Melakukan Penelitian di
Laboratorium Kimia, Fakultas Farmasi, Universitas Wahid
Hasyim**



**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. 04/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 : Tri Wahyuni Lestari
NIM : 125010778
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 Deksametason 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 Elifah, M.Sc, Apt

Lampiran 11. Gambar Alat-Alat yang Digunakan pada saat Penelitian

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



Digital Ultrasonic Cleanser (Jeken)



Timbangan Analitik (Ohaus)



Vakum pump (Rocker)



Vortex Mixer (Health)



Lampiran 12. Gambar Sampel Penelitian