

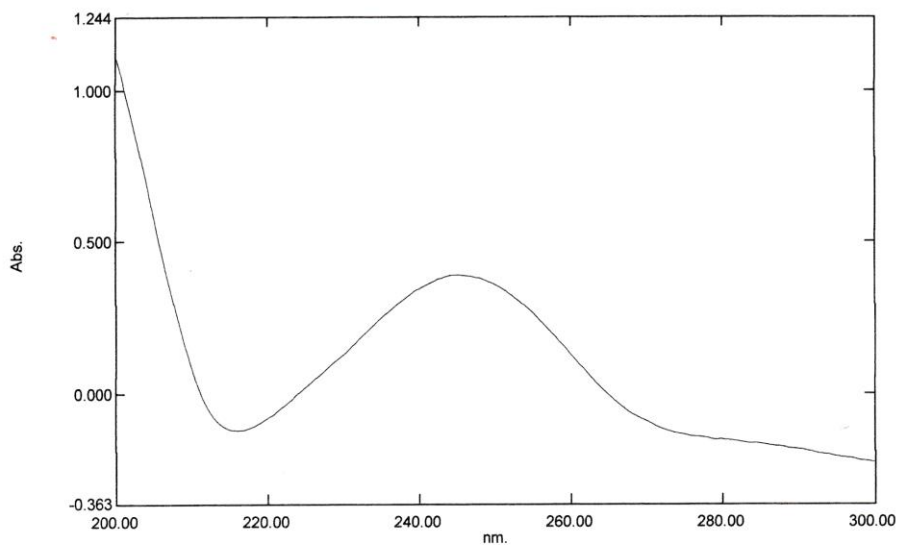
Lampiran 1. Hasil *Scanning* Penentuan Panjang Gelombang PCT dan CTM

1. Parasetamol

Spectrum Peak Pick Report

05/26/2016 11:16:23 AM

Data Set: File_160526_111323 - RawData



[Measurement Properties]
 Wavelength Range (nm.): 200.00 to 300.00
 Scan Speed: Fast
 Sampling Interval: 0.1
 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:

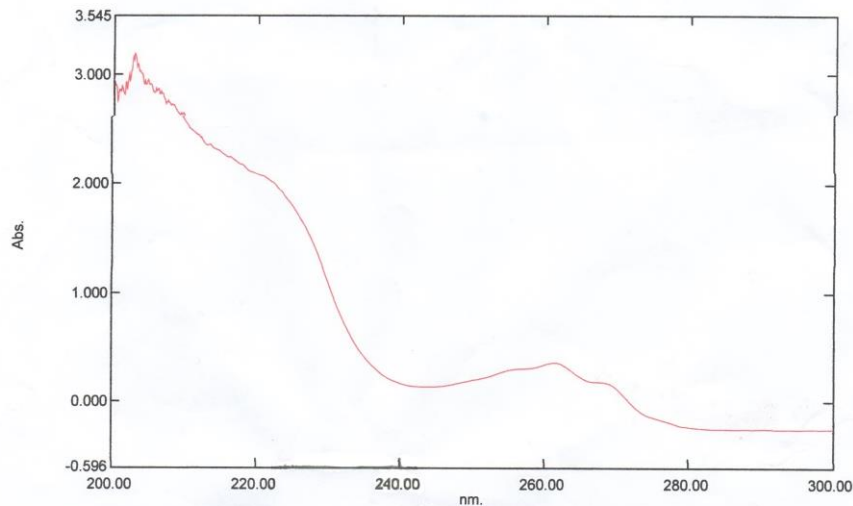
No.	P/V	Wavelength	Abs.	Description
1	⊕	290.50	-0.185	
2	⊕	287.40	-0.175	
3	⊕	284.30	-0.165	
4	⊕	279.90	-0.152	
5	⊕	245.30	0.391	
6	⊕	287.20	-0.175	
7	⊕	279.40	-0.154	
8	⊕	216.70	-0.122	
9	⊕	216.00	-0.123	

2. Klorfeniramin Maleat

Spectrum Peak Pick Report

05/26/2016 11:17:23 AM

Data Set: File_160526_111520 - RawData



[Measurement Properties]
Wavelength Range (nm.): 200.00 to 300.00
Scan Speed: Fast
Sampling Interval: 0.1
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

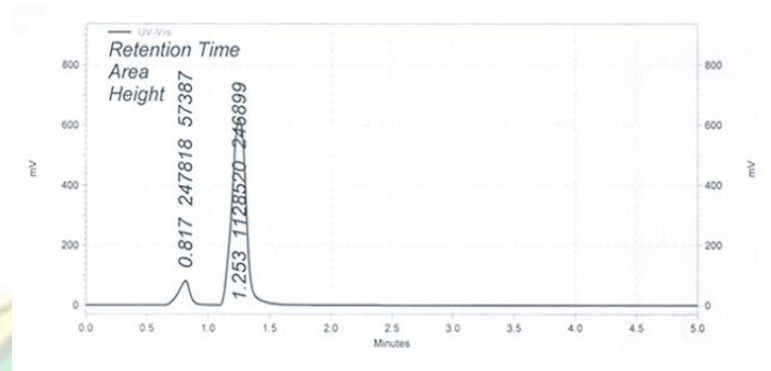
[Operation]
Threshold: 0.0010000
Points: 2
InterPolate: Disabled
Average: Disabled

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

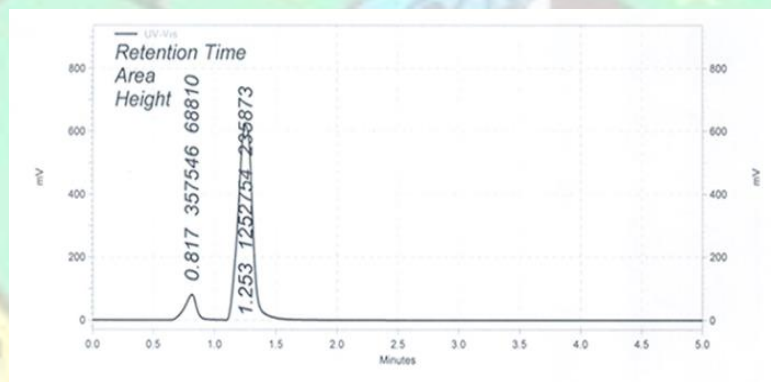
No.	P/V	Wavelength	Abs.	Description
1	●	299.40	-0.249	
2	●	297.30	-0.248	
3	●	294.00	-0.250	
4	●	290.50	-0.245	
5	●	288.40	-0.248	
6	●	284.70	-0.246	
7	●	261.60	0.363	
8	●	216.50	2.254	
9	●	213.60	2.367	
10	●	209.60	2.656	
11	●	207.50	2.771	
12	●	206.80	2.831	
13	●	206.10	2.876	
14	●	205.10	2.918	
15	●	204.70	2.960	
16	●	202.90	3.200	
17	●	201.10	2.902	
18	●	298.30	-0.251	
19	●	288.70	-0.249	
20	●	244.90	0.140	
21	●	216.30	2.252	
22	●	213.30	2.360	
23	●	209.10	2.635	
24	●	207.20	2.737	

Lampiran 2. Kromatogram Larutan Standar Baku PCT dan CTM

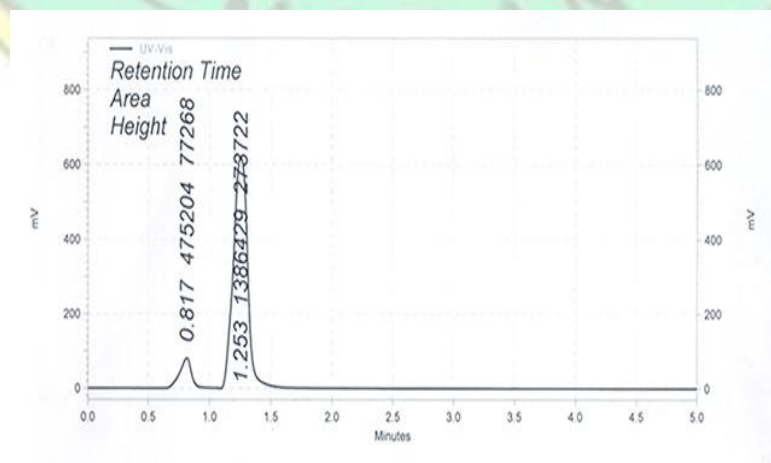
a. Larutan standar baku 20 $\mu\text{g/mL}$.



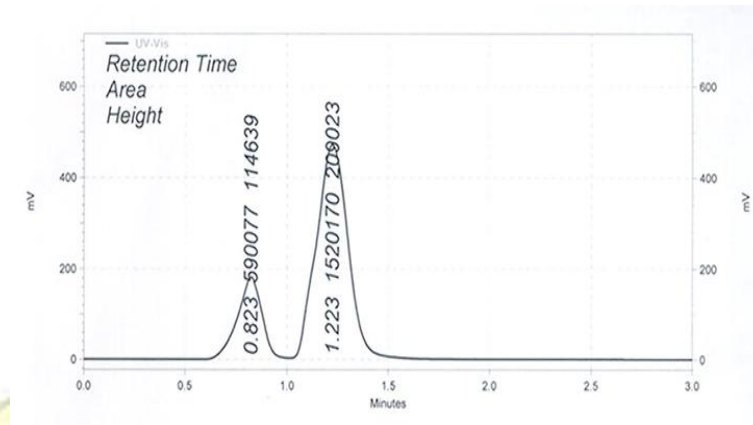
b. Larutan standar baku 40 $\mu\text{g/mL}$.



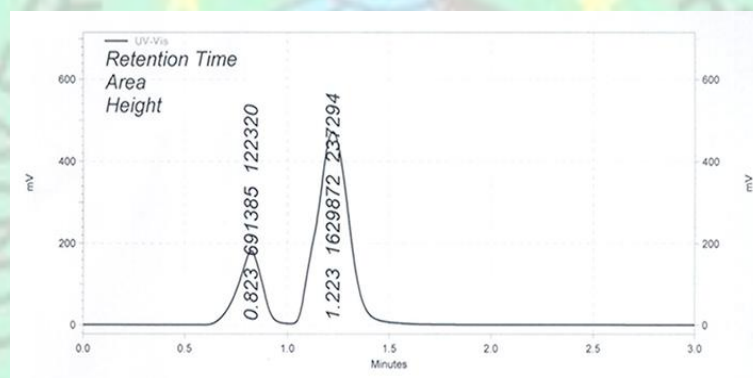
c. Larutan standar baku 60 $\mu\text{g/mL}$.



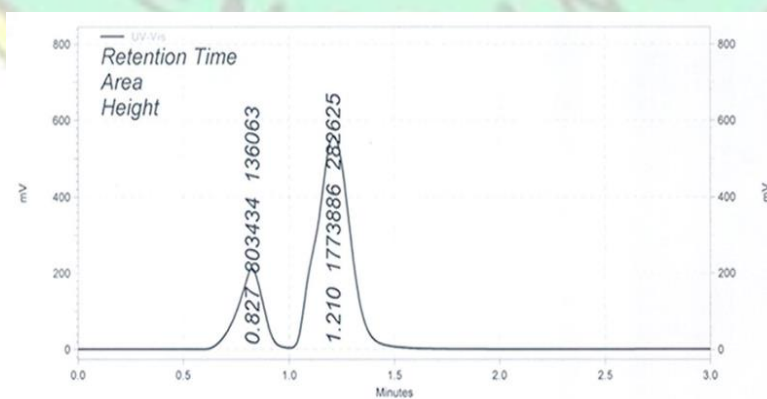
d. Larutan standar baku 80 µg/mL.

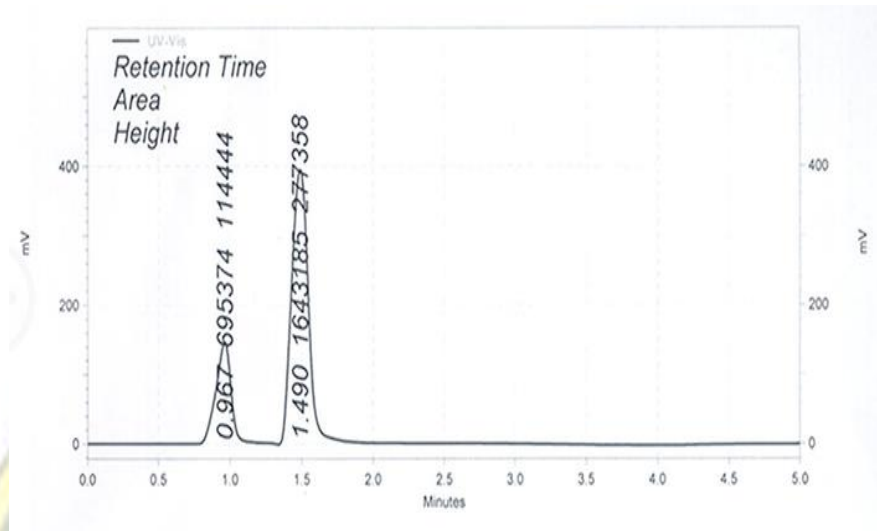


e. Larutan standar baku 100 µg/mL.



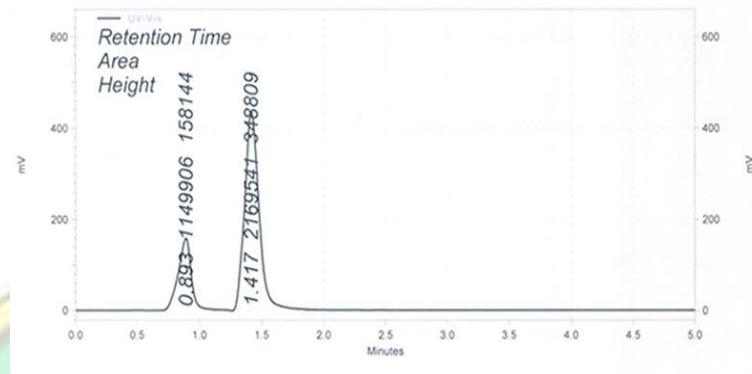
f. Larutan standar baku 120 µg/mL.



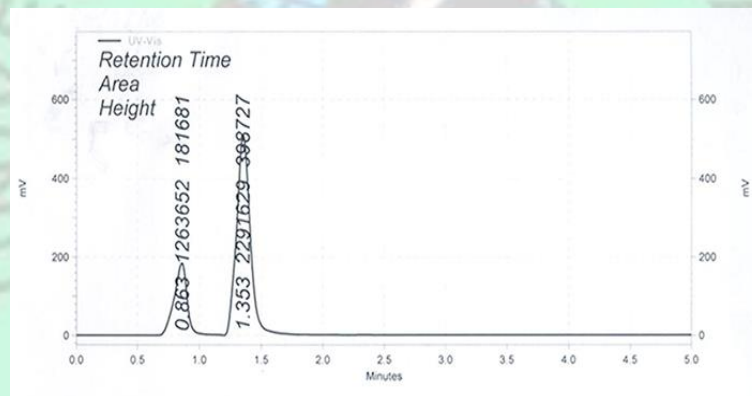
Lampiran 3. Kromatogram Presisi PCT dan CTM

Lampiran 4. Kromatogram Uji Akurasi PCT dan CTM

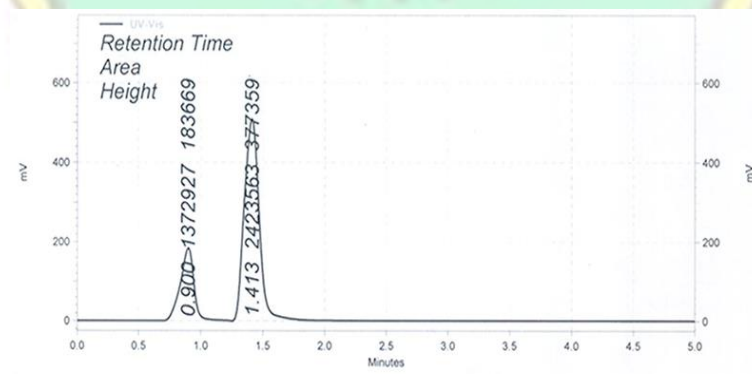
a. Uji akurasi 80%.



b. Uji akurasi 100%.

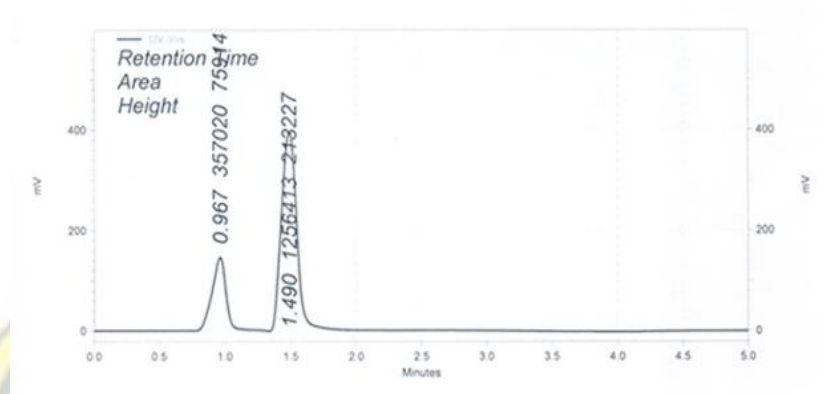


c. Uji akurasi 120%.

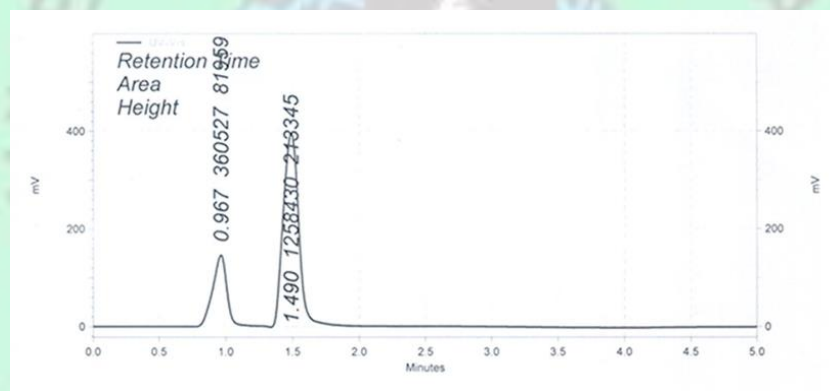


Lampiran 5. Kromatogram Sampel PCT dan CTM dalam Sediaan Sirup.

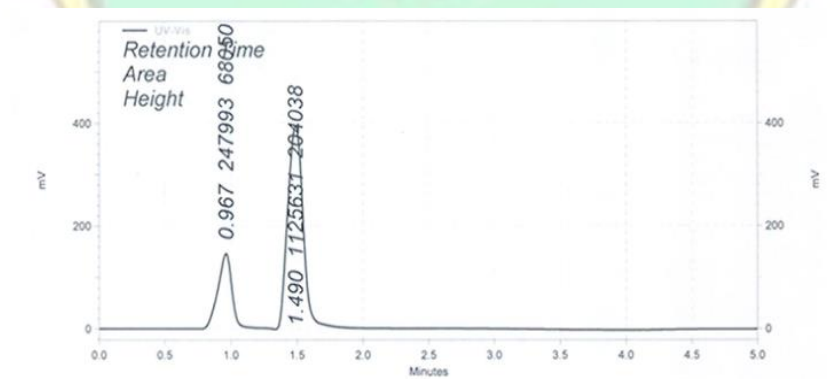
a. Sampel Obat A.



b. Sampel Obat B.



c. Sampel Obat C.



Lampiran 6. Contoh Perhitungan Perolehan Kembali Dosis PCT 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 parasetamol = 1647195

2) Kadar parasetamol berdasarkan persamaan garis $Y = 6417,036 X + 999412,667$ adalah 100,95 $\mu\text{g/mL}$

b. Konsentrasi bahan baku yang ditambahkan (C)

1) Luas puncak parasetamol = 1517070

2) Kadar parasetamol berdasarkan persamaan garis $Y = 6417,036 X + 999412,667$ adalah 80,67 $\mu\text{g/mL}$

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

1) Luas puncak total analit 1 = 2169541

Luas puncak total analit 2 = 2171730

Luas puncak total analit 3 = 2172112

2) Berdasarkan persamaan garis $Y = 6417,036 X + 999412,667$

Kadar total analit 1 = 182,35 $\mu\text{g/mL}$

Kadar total analit 2 = 182,69 $\mu\text{g/mL}$

Kadar total analit 3 = 182,75 $\mu\text{g/mL}$

2. Perhitungan perolehan kembali

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

a. Analit 1

$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{182,35 - 100,95}{80,67} \times 100 \% \\ &= 100,90 \% \end{aligned}$$

b. Analit 2

$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{182,69 - 100,95}{80,67} \times 100 \% \\ &= 101,33 \% \end{aligned}$$

c. Analit 3

$$\begin{aligned} \% \text{ perolehan kembali} &= \frac{182,78 - 100,95}{80,67} \times 100 \% \\ &= 101,44 \% \end{aligned}$$

Lampiran 7. Perhitungan LOD dan LOQ PCT

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	20	400	-50	2500	112850	1127753	766.613	587695.498
2	40	1600	-30	900	125274	1256094	-3340.11	11156314.7
3	60	3600	-10	100	138649	1384435	1994.13	3976725.94
4	80	6400	10	100	152010	1512776	7394.43	54677935.7
5	100	10000	30	900	162982	1641116	-11244.3	126433540.4
6	120	14400	50	2500	177386	1769457	4429.03	19616156.5
$\bar{X}$	70	36400		7000				216448367.9

Dari persamaan $Y = 6417,036x + 999412,667$ maka Y_c dapat dihitung

1. $Y = 6417,036x + 999412,667$

$$Y = 6417,036(20) + 999412,667$$

$$Y = 1127753,39$$

2. $Y = 6417,036x + 999412,667$

$$Y = 6417,036(40) + 999412,667$$

$$Y = 1256094,11$$

3. $Y = 6417,036x + 999412,667$

$$Y = 6417,036(60) + 999412,667$$

$$Y = 1384434,83$$

4. $Y = 6417,036x + 999412,667$

$$Y = 6417,036(80) + 999412,667$$

$$Y = 1512775,55$$

5. $Y = 6417,036x + 999412,667$

$$Y = 6417,036(100) + 999412,667$$

$$Y = 1641116,27$$

6. $Y = 6417,036x + 999412,667$

$$Y = 6417,036(120) + 999412,667$$

$$Y = 1769456,99$$

7. Persamaan kurva baku $Y = 6417,036x + 999412,667$ ($r = 0,99$)

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

$$= (216448367,9/4)^{1/2}$$

$$= 7356,09$$

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

$$= 7356,09 \times \sqrt{\frac{36400}{6 \times 7000}}$$

$$= 7356,09 \times 0,93$$

$$= 6841,16$$

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)

$$Y = 999412,667 + 3 (6841,16)$$

$$= 1019936,15$$

$$Y = 6417,036x + 999412,667$$

$$1019936,15 = 6417,036x + 999412,667$$

$$\text{LOD} = X = 3,20 \mu\text{g/mL}$$

Perhitungan nilai LOQ :

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

Y = nilai intercept (a) pada persamaan kurva kalibrasi

S_B = simpangan baku intercept (a) (S_a)

$$Y = 999412,667 + 10 (6841,16)$$

$$= 1067824,27$$

$$Y = 6417,036x + 999412,667$$

$$1067824,27 = 6417,036x + 999412,667$$

$$\text{LOQ} = X = 10,67 \mu\text{g/mL}$$

Lampiran 8. Contoh Perhitungan Kadar PCT

Persamaan regresi linier kurva baku adalah

$$Y = BX + A$$

$$Y = 6417,036x + 999412,667$$

Replikasi 1

$$Y = 6417,036x + 999412,667$$

$$1256413 = 6417,036x + 999412,667$$

$$X = 40,05 \mu\text{g/mL}$$

Faktor pengenceran 25x, sehingga kadar PCT adalah

$$X = 40,05 \mu\text{g/mL} \times 25$$

$$= 1001,25 \mu\text{g/mL}$$

$$\text{Kadar PCT dalam 1 ml sampel} = \frac{25 \text{ mg}}{1000 \mu\text{g/mL}} \times 1001,25 \mu\text{g/mL}$$

$$= 25,03 \text{ mg}$$

$$\% \text{ kadar PCT} = \frac{25,03 \text{ mg}}{25 \text{ mg}} \times 100 \%$$

$$= 100,12 \%$$

Lampiran 9. Perhitungan Selektivitas PCT dan CTM

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

$$R = 2 \frac{(1,417 - 0,893)}{0,285 + 0,214}$$

$$R = 2,10$$



Lampiran 10. Instrument KCKT



Lampiran 11. Certificate of Analysis PCT

10/2/14 82/1/2/14

常熟华港制药有限公司
CHANGSHU HUAGANG PHARMACEUTICALS CO.,LTD
检验报告单
CERTIFICATE ANALYSIS

品名 Product Name	扑热息痛 微粉 Paracetamol Micro grade	包装规格 Packing	25kgs/drum
批号 Batch No.	KLB1312351 ✓	数量 Quantity	1000kg
生产日期 Manufacture Date	04-12-2013	有效期 Expiry Date	03-12-2017
检验标准 Standard	BP2010/USP32		

检验结果
Examination

L/C NO.:225LC30502B
DATE:SEP 9,2013
INVOICE NO.:CSHG131205
DATE:DEC 5,2013

项目 Contents	标准 Specification	结果 Results
性状 Characters	White,crystalline powder,sparingly soluble in water,freely soluble in alcohol,very slightly soluble in ether and in methylene chloride.	White,crystalline powder,sparingly soluble in water,freely soluble in alcohol,very slightly soluble in ether and in methylene chloride.
鉴别 Identification	A:Melting range168-172℃ B.C.D.E Positive	170.1-170.7℃ B.C.D.E Positive
相关物质 Related substance	Impurity X(chloracetanilide) ≤10ppm	<10ppm
	Impurity K(4-aminophenol) ≤50ppm	<50ppm
	Impurity F(4-nitrophenol) ≤0.05%	<0.05%
	Any other impurity ≤0.05%	<0.05%
	Total of other impurity ≤0.1%	<0.1%
氯化物 Chloride	≤0.014%	<0.014%
硫酸盐 Sulfate	≤0.02%	<0.02%
硫化物 Sulfide	Complies	Complies
重金属 Heavy metals	≤0.001%	<0.001%
游离对氨基酚 Free p-Aminophenol	≤0.005%	<0.005%
对氯苯乙酰胺 chloracetanilide 易炭化物	≤0.001%	<0.001%
Readily carbonizable substances	Complies	Complies
溶剂残留 Residual solvent	Residual content of acetic acid ≤0.5%	<0.09%
有机挥发性杂质 Organic volatile impurities	Complies	Complies
水份 Water	≤0.5%	0.09%
炽灼残渣 Residue on ignition	≤0.1%	0.05%
含量 Assay	99.0-101.0%(Dried substance)	100.2%

结论: 按 BP2010/USP32 版检验符合规定
Conclusion: It complies with requirements of the BP2010/USP32

质量部 QAD:  检验者: Inspector: Zhao liqin Heng qing
 

KONGO CHEMICAL CO.,LTD.
PAGE : 1 OF 1

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Lampiran 12. Certificate of Analysis CTM

MR 13/0752
QV 8/13


SUPRIYA LIFESCIENCE LTD.
(Formerly known as Supriya Chemicals)

CERTIFICATE OF ANALYSIS

Product Name	Chlorpheniramine Maleate EP	A.R. Number	SL/ QC/ FF/ 13/ 0396
Batch No.	SL/ C/ 0613082	Dispatch Quantity	40 x 25 kg = 1000kg
Batch Size	1000.0 kgs	Expiry Date	May-2018
Manufacturing Date	June-2013		

Tests	Specification & Limits	Results
Appearance	White or almost white crystalline powder.	White crystalline powder
Solubility	Freely soluble in water, soluble in ethanol (96%)	Freely soluble in water, soluble in ethanol (96%)
Identification		
A) Melting point	130°C to 135°C	133.8°C
B) IR Absorption	The infra red absorption spectrum should be concordant with the reference spectrum of chlorpheniramine maleate	The infra red absorption spectrum is concordant with the reference spectrum of chlorpheniramine maleate.
C) Optical rotation	-0.10° to +0.10°	+0.002°
Appearance of solution	Solution should be clear and not more intensely coloured than reference solution BY6	Solution is clear and not more intensely coloured than reference solution BY6
Optical rotation	-0.10° to +0.10°	+0.002°
Related Substances		
1) Impurity A	Not more than 0.2%	Below disregard limit
2) Impurity B	Not more than 0.1%	Below disregard limit
3) Impurity C	Not more than 0.1%	Below disregard limit
4) Impurity D	Not more than 0.1%	Not detected
5) Unspecified Impurity	Not more than 0.10%	Below disregard limit
6) Total impurities	Not more than 0.5%	Below disregard limit
Heavy metals	Not more than 20ppm	Less than 20ppm
Loss on drying	Not more than 0.5%	0.20%
Sulphated Ash	Not more than 0.1%	0.05%
Assay (on dried basis)	98.0% to 101.0%	99.73%
Residual Solvents		
Methanol	Not more than 3000ppm	812 ppm
Isopropanol	Not more than 5000ppm	487 ppm
O-xylene	Not more than 2170ppm	Not detected

Date of Release :07/06/2013

REMARKS: Chlorpheniramine maleate complies / ~~does not comply~~ with respect to above mentioned test as per EP Specification

 07/06/13 PREPARED BY	 07/06/13 REVIEWED BY	 07/06/13 APPROVED BY
--	--	---

Customer Name: PT. Global Chemindo Megatrade

Ref.SOP No.SOP/SL/ QC065

SQCF/108-02

Corporate Office : 207/208, Udyog Bhavan, Sonawala Road, Goregaon (East), Mumbai - 400 063, Maharashtra, India.
Tel: +91 22 40332727 / 66942507 Fax: +91 22 26860011
E-mail: supriya@supriyalifescience.com Website: www.supriyalifescience.com

Factory : A 5/2, Lote Parshuram Industrial Area, M.I.D.C. Tal - Khed, Dist-Ratnagiri, 415 722, Maharashtra, India.
Tel: +91 2356 272299 Fax: +91 2356 272178
E-mail: factory@supriyalifescience.com

GOVT. RECOGNISED EXPORT HOUSE

PAGE : 1 OF 1


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Lampiran 13. 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. 05/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 : Nur Aini
NIM : 125010793
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 Klorfeniramin Maleat dan Parasetamol Menggunakan Kromatografi Cair Kinerja Tinggi Serta Aplikasinya dalam Sediaan Sirup”

Demikian surat keterangan ini dibuat untuk dipergunakan semestinya.

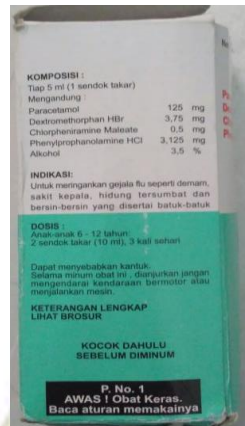
Wassalamu'alaikum Wr. Wb.

Semarang, Oktober 2016
Kep. Bag Kimia Farmasi

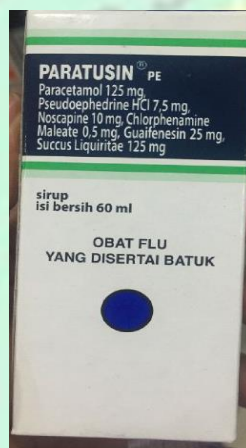
Maria Ulfah, M.Sc, Apt

Lampiran 14. Gambar Sampel

SIRUP A



SIRUP B



SIRUP C

