LAMPIRAN

Lampiran 1. Perhitungan Uji Kesesuaian Sistem Teofilin

DATA TEOFILIN						
Replikasi	**RT**	**Area**	**TF**	**K'**	**N Plates**	**Rs**
1	9.67	9028075	1.21	4.07	6214	24.14
2	9.71	9013980	1.21	4.08	6566	24.26
3	9.69	9026300	1.20	4.09	6270	24.38
4	9.69	9020409	1.20	4.09	6286	24.59
5	9.70	9014320	1.20	4.10	6288	24.72
Jumlah	48.46	45103084				
Rata-Rata	9.69	9020616.80	1.20	4.09	6325	24.42
SD	0.013	6551.02				
Syarat	<% 2	<% 2	< 1.5	2-10	>2000	>1.5
RSD		0.07				
Kesimpulan	MS	MS	MS	MS	MS	MS

Simpangan baku relatif luas area = $\frac{\text{SD}}{\text{x}} \times 100\% = \frac{6551,02}{9020616,80} \times 100\%$

$= 0,073\%$

Lampiran 2. Perhitungan Uji Kesesuaian Sistem Efedrin HCl

DATA EFEDRIN HIDROKLORIDA						
Replikasi	**RT**	**Area**	**TF**	**K'**	**N Plates**	**Rs**
1	1.91	6344	1.331	0.75	2507	5.27
2	1.91	6138	1.240	0.76	2518	4.87
3	1.90	6135	1.388	0.75	2563	5.10
4	1.91	6186	1.357	0.76	2789	5.41
5	1.90	6129	1.420	0.75	2877	5.68
Rata-rata	1.91	6186	1.347	0.75	2651	5.26
SD	0.004	91				
SYARAT	<% 2	<% 2	< 1.5	2-10	>2000	>1.5
RSD		1.471				
Kesimpulan	MS	MS	MS	TMS	MS	MS

Simpangan baku relatif luas area $= \frac{SD}{x} \times 100\% = \frac{91}{6186} \times 100\%$

$= 1{,}471$

Lampiran 3. Perhitungan LoD dan LoQ Teofilin

Teofilin			
Konsentrasi	Luas Area	Yi	$(yi-yi)^2$
75	5170250	5150518	389351824
100	6865842	6851268	212401476
125	8452107	8552018	9982207921
150	10291005	10252768	1462068169
175	12020260	11953518	4454494564
200	13615117	13654268	1532800801
Jumlah			18033324755
S			4508331189
SD			67144,11
LoD			2,96
LoQ			9,87

S $= \frac{\Sigma(yi-\bar{y})^2}{N-2}$

$= \frac{18033324755}{6-2} = 4508331189$

S(y/x) $= \sqrt{S}$

$= \sqrt{4508331189}$

$= 67144,11$

LoD $= \frac{3 \times SD}{b}$

$$= \frac{3 \times 67144{,}11}{68030} = 2{,}96$$

$$\text{LoQ} = \frac{10 \times \text{SD}}{\text{b}}$$

$$= \frac{10 \times 67144{,}11}{68030} = 9{,}87$$

Lampiran 4. Perhitungan LoD dan LoQ Efedrin hidroklorida

Efedrin HCl			
Konsentrasi	Luas Area	Yi	$(yi-yi)^2$
7,5	4094	4087	56
10	5557	5496	3727
12,5	6813	6905	8538
15	8229	8315	7370
17,5	9892	9724	28123
20	11076	11134	3335
Jumlah			51150
S			12787
SD			113,08
LoD			0,60
LoQ			2,01

S $= \frac{\Sigma(yi-\bar{y})^2}{N-2}$

$= \frac{51150}{6-2} = 12787$

S(y/x) $= \sqrt{S}$

$= \sqrt{12787}$

$= 113,08$

$$\text{LoD} = \frac{3 \times \text{SD}}{\text{b}}$$

$$= \frac{3 \times 113{,}08}{563{,}78} = 0{,}60$$

$$\text{LoQ} = \frac{10 \times \text{SD}}{\text{b}}$$

$$= \frac{10 \times 113{,}08}{563{,}78} = 2{,}01$$

Lampiran 5. Perhitungan Uji Presisi Teofilin

Teofilin		
Konsentrasi (bpj)	Replikasi	Luas Area
125	1	8069619
	2	8044792
	3	8053334
	4	8044104
	5	8036108
	6	8099879
Rata-rata	8057972,67	
SD	23483,7	
RSD	0,29	
Syarat	< 2%	
Keterangan	MS	

Simpangan baku relatif luas area $= \frac{SD}{x} \times 100\% = \frac{23483,7}{8057972,67} \times 100\%$

$= 0,29\%$

Lampiran 6. Perhitungan Uji Presisi Efedrin Hidroklorida

Efedrin HCl		
Konsentrasi (bpj)	Replikasi	Luas Area
12,5	1	6548
	2	6373
	3	6665
	4	6408
	5	6589
	6	6400
Rata-rata		6497,16
SD		120
RSD		1,85
Syarat		< 2%
Keterangan		MS

Simpangan baku relatif luas area $= \frac{SD}{x} \times 100\% = \frac{120}{6497,16} \times 100\%$

$= 1,85\%$

Lampiran 7. Perhitungan Uji Akurasi Teofilin

Rentang Spesifik (%)	Rep-likasi	Luas Area	Konsentrasi (bpj)		% *Recovery*	Rata-rata (%)
			Teoritis	Pengukuran		
80	1	6786816	100	99,053	99,05	98,2
	2	6815387	100	99,473	99,47	
	3	6547159	100	95,530	95,53	
100	1	8641787	125	126,320	101,06	101,49
	2	8718229	125	127,443	101,95	
	3	8639149	125	126,281	101,02	
120	1	10461456	150	153,068	101,2459	99,83
	2	10719004	150	156,853	101,7352	
	3	9525000	150	139,302	99,8998	
Rata-rata						99,78

Contoh perhitungan:

Konsentrasi Pengukuran : $x = \frac{y-a}{b}$

$= \frac{6786816-48268}{68030}$

= 99,053

% *recovery* : $\frac{\text{kadar pengukuran}}{\text{kadar teoritis}} \times 100\% = \frac{99,053}{100} 100\% = 99,05\ \%$

Lampiran 8. Perhitungan Uji Akurasi Efedrin Hidroklorida

Rentang Spesifik (%)	Replikasi	Luas Area	Konsentrasi (bpj)		% *Recovery*	Rata-rata (%)
			Teoritis	Pengukuran		
80	1	5357	10	9,755	97,54	98,02
	2	5735	10	10,424	104,24	
	3	5277	10	9,612	96,12	
100	1	6053	12,5	10,988	87,90	99,66
	2	7063	12,5	12,780	102,24	
	3	7529	12,5	13,606	108,85	
120	1	7738	15	13,977	93,18	99,10
	2	8024	15	14,484	96,56	
	3	8954	15	16,134	107,56	
Rata-rata						99,35

Contoh perhitungan :

Konsentrasi Pengukuran : $x = \frac{y-a}{b}$

$$= \frac{5357 + 141{,}85}{563{,}78}$$

$$= 9{,}755$$

% *recovery* : $\frac{\text{kadar pengukuran}}{\text{kadar teoritis}} \times 100\% = \frac{9{,}755}{10} 100\% = 97{,}5$

Lampiran 9. Perhitungan Kadar Sampel

Sampel	Nama	Luas Area	Dosis Etiket (mg)	Dosis yang dianalisis (mg)	Kadar sampel (%)
1	Teofilin	8947777	130	130,86	101
	Efedrin HCl	6724	12,5	12,18	97
2	Teofilin	8782973	130	128,39	99
	Efedrin HCl	6816	12,5	12,34	99
3	Teofilin	8628620	130	126,13	97
	Efedrin HCl	6600	12,5	11,96	96

Contoh perhitungan kadar sampel :

Data bobot tablet

No	**Bobot Tablet (mg)**	**No**	**Bobot Tablet (mg)**
1	330	11	331
2	328	12	330
3	330	13	328
4	331	14	329
5	330	15	330
6	330	16	330

7	330	17	328
8	330	18	328
9	329	19	328
10	330	20	330
Rata-rata		**329,5 mg**	

Rata-rata bobot tablet : 329,5 mg

1 tablet mengandung Teofilin 130 mg – Efedrin 12,5 mg dilarutkan dalam labu 25 ml sehingga :

130 mg/25 mL = 5200 bpj

12,5 mg/25 mL = 500 bpj

Diencerkan menjadi konsentrasi 130 ppm dan 12,5 ppm di dalam labu 10 mL

a. Teofilin : $V_1 \times N_1 = V_2 \times N_2$

$V_1 \times 5200$ bpj = 10 mL × 130 bpj

$V_1 = \frac{1300}{5200}$

= 0,25 mL = 250 µL

b. Efedrin HCl : $V_1 \times N_1 = V_2 \times N_2$

$V_1 \times 500$ bpj = 10 mL × 12,5 bpj

$V_1 = \frac{125}{500}$

= 0,25 mL = 250 µL

1. Perhitungan faktor pengenceraan

   Faktor pengenceran $= \frac{\text{V.labu awal}}{\text{V.pemipetan}}$ x vol. labu

   $= \frac{20\ \text{ml}}{0{,}5\ \text{ml}} \times 25$

   =1000

2. Perhitungan kadar sampel

   Perhitungan kadar sampel menggunakan rumus :

   $$\text{Kadar} = \frac{\text{Dosis yang dianalisis}}{\text{Dosis di etiket}} \times 100\%$$

   Bobot sampel yang ditimbang : 334 mg

   Bobot rata-rata tablet : 329,5 mg

   Jumlah zat aktif per tablet : Teofilin = 130 mg

   Efedrin HCl = 12,5 mg

   a. Teofilin

      Persamaan regresi : y = 68046x + 44252

      Luas area : 8947777

      Konsentrasi teoritis : 130 bpj

      Konsentrasi terbaca : $x = \frac{8947777 - 44252}{68046} = 130{,}85$

      Dosis yang dianalisis (mg) = Konsentrasi x Fp

      = 130,85 x 1000

= 130850 µg

= 130,85 mg

Kadar $= \frac{\text{Dosis yang dianalisis}}{\text{Dosis di etiket}} \times 100\%$

$= \frac{130,85}{130} \times 100\%$

= 100,65 %

b. Efedrin HCl

Persamaan regresi : y= 602,27x – 593,28

Luas area : 6724

Konsentrasi teoritis : 12,5 bpj

Konsentrasi terbaca : x = $\frac{6724 + 593,28}{602,27}$ = 12,18

Kadar $= \frac{\text{Dosis yang dianalisis}}{\text{Dosis di etiket}} \times 100\%$

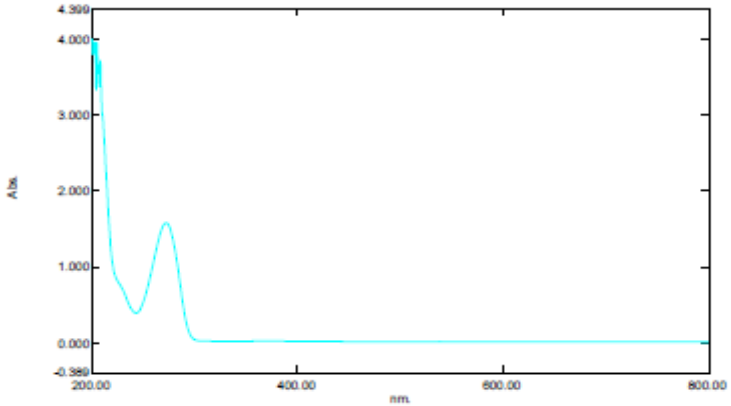
$= \frac{12,18}{12,5} \times 100\%$

= 97,44 %

Lampiran 10. Spektrum Teofilin

Spectrum Peak Pick Report

Data Set: 30 ppm teofilin dalam air - RawData



[Measurement Properties]
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Medium
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Single

[Instrument Properties]
Instrument Type: UV-1700 Series
Measuring Mode: Absorbance
Slit Width: 1.0 nm
Light Source Change Wavelength: 340.8 nm
S/R Exchange: Normal

[Attachment Properties]
Attachment: None

[Operation]
Threshold: 0.0010000
Points: 4
InterPolate: Disabled
Average: Disabled

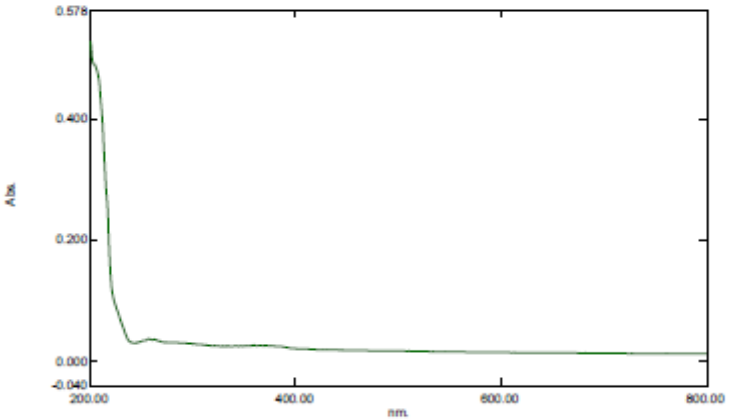
[Sample Preparation Properties]
Weight:
Volume:
Dilution:
Path Length:
Additional Information:

No.	P/V	Wavelength	Abs.	Description
1	P	359.50	0.021	
2	P	341.00	0.020	
3	P	331.50	0.020	
4	P	272.00	1.575	
5	P	202.50	3.957	
6	V	354.50	0.019	
7	V	338.00	0.018	
8	V	242.50	0.390	

Lampiran 11. Spektrum Efedrin HCl

Spectrum Peak Pick Report

Data Set: 10 ppm efedrin hcl dalam air - RawData



[Measurement Properties]
Wavelength Range (nm.): 200.00 to 800.00
Scan Speed: Medium
Sampling Interval: 0.5
Auto Sampling Interval: Enabled
Scan Mode: Single

[Instrument Properties]
Instrument Type: UV-1700 Series
Measuring Mode: Absorbance
Slit Width: 1.0 nm
Light Source Change Wavelength: 340.8 nm
S/R Exchange: Normal

[Attachment Properties]
Attachment: None

[Operation]
Threshold: 0.0010000
Points: 4
InterPolate: Disabled
Average: Disabled

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

No.	P/V	Wavelength	Abs.	Description
1	P	369.50	0.026	
2	P	359.50	0.026	
3	P	341.50	0.026	
4	P	283.00	0.036	
5	P	257.00	0.037	
6	V	336.00	0.024	
7	V	259.50	0.035	
8	V	242.50	0.030	

Lampiran 12. Kromatogram UKS, LoD dan LoQ

a. Replikasi 1

==== POLITEKNIK KESEHATAN BANDUNG ====
UNIT LABORATORIUM - LABORATORIUM TERPADU

Acquired by : DEA NADIA ULFA
Sample Name : STANDAR
Sample ID : TEOFILIN:EFDRIN (125:12.5)-1
Injection Volume : 20
Data File Name : standar t-e (125-12.5)-1.lcd
Method File Name : oka 30042018.lcm
Batch File Name : 20190102.lcb
Report File Name : Default.lcr
Data Acquired : 19 FEBRUARI 2019

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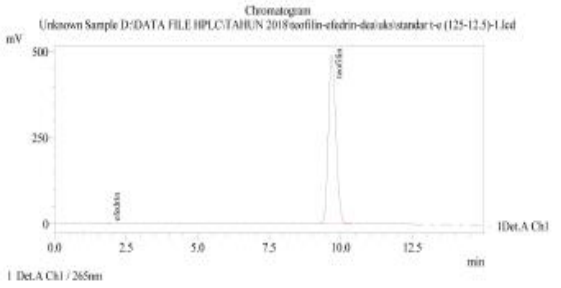

D:\DATA FILE HPLC\TAHUN 2018\teofilin-efedrin-dea\uks\standar t-e (125-12.5)-1.lcd

Detector A

Name	Ret. Time	Area	Theoretical Plate#	Resolution	Tailing Factor	HETP	k'
efedrin	1.909	6344	2506.891	5.269	1.331	59.835	2.752
teofilin	9.674	9028075	6213.976	24.139	1.210	24.139	7.880

b. Replikasi 2

==== POLITEKNIK KESEHATAN BANDUNG ====
UNIT LABORATORIUM - LABORATORIUM TERPADU

Acquired by : DEA NADIA ULFA
Sample Name : STANDAR
Sample ID : TEOFILIN:EFDRIN (125:12.5)-2
Injection Volume : 20
Data File Name : standar t-e (125-12.5)-2.lcd
Method File Name : oka 30042018.lcm
Batch File Name : 20190102.lcb
Report File Name : Default.lcr
Data Acquired : 19 FEBRUARI 2019

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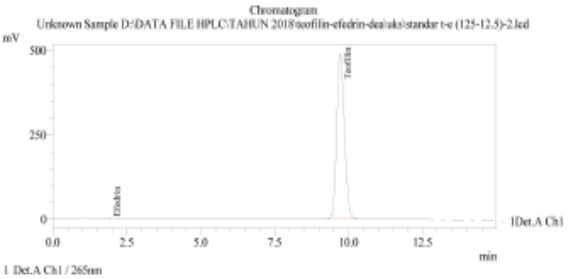

D:\DATA FILE HPLC\TAHUN 2018\teofilin-efedrin-dea\uks\standar t-e (125-12.5)-2.lcd

Detector A

Name	Ret. Time	Area	Theoretical Plate#	Resolution	Tailing Factor	HETP	k'
Efedrin	1.910	6138	2518.075	4.874	1.240	59.569	2.761
Teofilin	9.706	9013980	6266.298	24.261	1.206	23.938	7.948

c. Replikasi 3

==== POLITEKNIK KESEHATAN BANDUNG ====
UNIT LABORATORIUM - LABORATORIUM TERPADU

Acquired by : DEA NADIA ULFA
Sample Name : STANDAR
Sample ID : TEOFILIN:EFDRIN (125:12.5)-3
Injection Volume : 20
Data File Name : standar t-e (125-12.5)-3.lcd
Method File Name : oka 30042018.lcm
Batch File Name : 20190102.lcb
Report File Name : Default.lcr
Data Acquired : 19 FEBRUARI 2019

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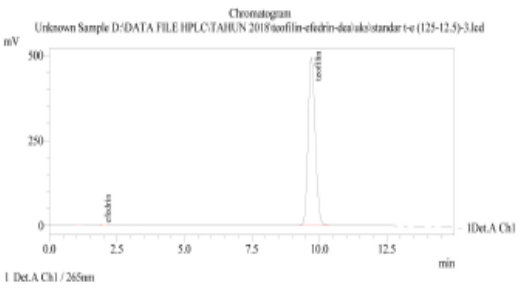

1 Det.A Ch1 / 265nm

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Detector A

Name	Ret. Time	Area	Theoretical Plate#	Resolution	Tailing Factor	HETP	k'
efedrin	1.903	6535	2563.188	5.095	1.388	58.521	2.753
teofilin	9.691	9026300	6292.883	24.377	1.204	23.836	7.927

d. Replikasi 4

==== POLITEKNIK KESEHATAN BANDUNG ====
UNIT LABORATORIUM - LABORATORIUM TERPADU

Acquired by : DEA NADIA ULFA
Sample Name : STANDAR
Sample ID : TEOFILIN:EFDRIN (125:12.5)-4
Injection Volume : 20
Data File Name : standar t-e (125-12.5)-4.lcd
Method File Name : oka 30042018.lcm
Batch File Name : 20190102.lcb
Report File Name : Default.lcr
Data Acquired : 19 FEBRUARI 2019

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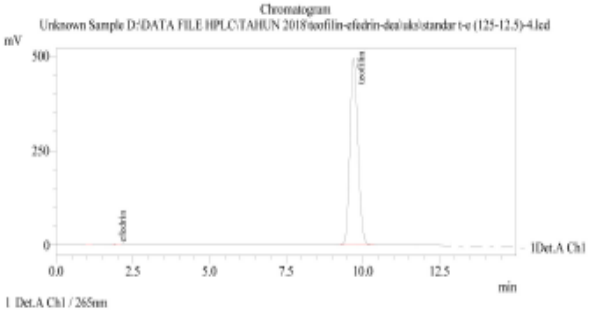

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Detector A

Name	Ret. Time	Area	Theoretical Plate#	Resolution	Tailing Factor	HETP	k'
efedrin	1.905	6186	2789.197	5.407	1.357	53.779	2.759
teofilin	9.686	9020409	6285.854	24.588	1.201	23.863	7.944

e. Replikasi 5

==== POLITEKNIK KESEHATAN BANDUNG ====
UNIT LABORATORIUM - LABORATORIUM TERPADU

Acquired by : DEA NADIA ULFA
Sample Name : STANDAR
Sample ID : TEOFILIN:EFDRIN (125:12.5)-5
Injection Volume : 20
Data File Name : standar t-e (125-12.5)-5.lcd
Method File Name : oka 30042018.lcm
Batch File Name : 20190102.lcb
Report File Name : Default.lcr
Data Acquired : 19 FEBRUARI 2019

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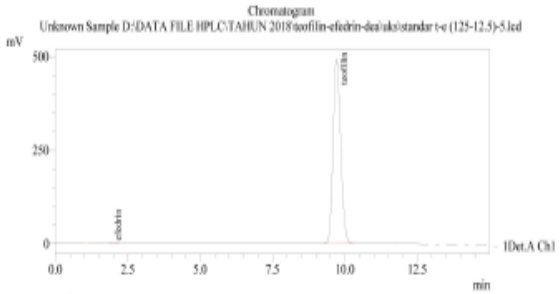

1 Det.A Ch1 / 265nm

D:\DATA FILE HPLC\TAHUN 2018\teofilin-efedrin-dea\uks\standar t-e (125-12.5)-5.lcd

Detector A

Name	Ret. Time	Area	Theoretical Plate#	Resolution	Tailing Factor	HETP	k'
efedrin	1.901	6329	2876.915	5.676	1.420	52.139	2.749
teofilin	9.701	9014320	6288.299	24.722	1.196	23.854	7.929

Lampiran 13. Alat-alat yang digunakan

	KCKT : *Shimadzu Prominance* LC-20AT Kolom : ODS-3 C_{18} (150 × 4,6 mm) Detektor : UV Sistem pompa : *Low pressure gradient* Sistem elusi : Fase terbalik
	Spektrofotometer UV-Vis *Double Beam*

	Sonikator
	Neraca Analitik