Lampiran 1. Certifcate of Analys Paracetamol

安丘市鲁安药业有限责任公司

ANQIU LU'AN PHARMACEUTICAL CO., LTD.

No. 35, Weixu North Road, Anqiu City, Shandong Province, China

山东省安丘市潍徐北路 35 号

Certificate of Analysis

Product:	Paracetamol	COA No.:	20173301
Batch No.	1730550	Manufacturing Date:	2017.10.29
Quantity:	5000kg	Expiry Date:	2021.10.29
Packing:	25kg/woven bag	Standard:	USP35

Tests	Specification	Test Reference	Results
Appearance:	A white, crystalline powder; odourless; slight bitterness.	USP monograph for Acetaminophen	Conforms
Solubility**:	1g of the specimen dissolves in 20ml boil water, 15ml 1mol/L sodium Hydroxide solution, freely soluble in alcohol.	USP monograph for Acetaminophen	Conforms
Identification	IR spectrum conforms to that of the reference standard. UV spectrum conforms to that of the reference standard. TLC spectrum conforms to that of the reference standard.	USP Monograph for Acetaminophen	Conforms Conforms Conforms
Melting point:	168-172°C	USP <741>	169-170°C
Free p-Aminophenol:	≤ 0.005%	EP monograph for Paracetamol, HPLC method	4.1ppm
Related Substances (4-Chloroacetanilide):	≤ 0.001%	EP monograph for Paracetamol, HPLC method	ND*
Chloride**:	≤ 0.014%	USP <221>	<0.014%
Sulfate**:	≤ 0.02%	USP <221>	<0.02%
Sulfide**:	No coloration or spotting of the test paper occurs.	USP Monograph for Acetaminophen	Conforms
Loss on Drying:	≤0.5%	EP 2.2.32	0.12%
Residue on Ignition:	<0.1%	USP <281>	0.04%
Heavy Metals:	≤10ppm	USP<231> method II	<10ppm
Readily carbonizable substances:	The solution has no more color than Matching Fluid A.	USP <271>	Conforms
Assay:	98.0-101.0%	USP monograph for Acetaminophen, UV method	100.0%
Particle Size:	Min.80% pass through 100 mesh	Air Jet Method	Conforms
Residual Solvents	Glacial acetic acid is used in Acetaminophen production, and it can be determined by Loss on Drying not more than 0.5%.		
Conclusion:	It conforms to USP35.		

QA Manager: Analyst: Checker:

*: *ND means not detected.*

**: *Skip test.*

Manufacturer: Anqiu Lu'an Pharmaceutical Co., Ltd.

Lampiran 2. Tabel koefisien sederhana, r tabel pada taraf kepercayaan 5% dan 1%

d.b	5%	1%	df	5%	1%
1	0,997	1,000	26	0,374	0,478
2	0,950	0,999	27	0,367	0,470
3	0,878	0,959	28	0,361	0,463
4	0,811	0,917	29	0,355	0,456
5	0,754	0,874	30	0,349	0,449
6	0,707	0,834	32	0,339	0,437
7	0,666	0,798	34	0,329	0,424
8	0,632	0,765	36	0,321	0,413
9	0,602	0,735	38	0,312	0,403
10	0,576	0,708	40	0,304	0,393
11	0,553	0,684	45	0,288	0,372
12	0,532	0,661	50	0,273	0,354
13	0,514	0,641	55	0,262	0,340
14	0,497	0,623	60	0,250	0,325
15	0,482	0,606	70	0,232	0,302
16	0,468	0,590	80	0,217	0,283
17	0,456	0,575	90	0,205	0,267
18	0,444	0,561	100	0,195	0,254
19	0,433	0,549	125	0,174	0,228
20	0,423	0,537	150	0,159	0,208
21	0,413	0,526	175	0,148	0,194
22	0,404	0,515	200	0,138	0,181
23	0,396	0.505	300	0,113	0,148
24	0,388	0,496	400	0,098	0,128
25	0,381	0,487	500	0,088	0,115

d.b = n- 2 sedangkan n adalah jumlah sampel (Gomez & Gomez, 1995)

Lampiran 3. Data Perhitungan Batas Deteksi dan Batas Kuantitiasi

No	Konsentrasi	Intensitas (y)	ȳ	(ȳ-y)	$(ȳ-y)^2$
1.	5	56,871	53,6944	-3,1766	10,090
2.	50	517,179	531,3874	14,2084	201,878
3.	100	1037,032	1062,1574	25,1254	631,265
4.	200	2200,110	2123,6974	-76,4126	5838,885
5.	300	3144,998	3185,2374	40,2394	1619,177
				Σ =	8301,295

y = bx + a

= 10,6154 x + 0,617400

y = 10,6154 (5) + 0,617400 = 53,6944

y = 10,6154 (50) + 0,617400 = 531,3874

y = 10,6154 (100) + 0,617400 = 1062,1574

y = 10,6154 (200) + 0,617400 = 2123,6974

y = 10,6154 (300) + 0,617400 = 3185,2374

Sb = $\frac{\sqrt{\Sigma(y-\bar{y})2}}{n-2} = \frac{8301,295}{3}$ = 52,6032

LOD = $\frac{3.Sb}{b} = \frac{3.52,6032}{10,6154}$ = 14,866 ppm

LOQ = $\frac{10.Sb}{b} = \frac{10.52,6032}{10,6154}$ = 49,553 ppm

Lampiran 4. Data Perhitungan Presisi

No	Intensitas	x	(x-$\tilde{x}$)	(x-$\tilde{x}$)^2
1.	1035,057	97,447	0,033	0,001089
2.	1031,087	97,073	-0,341	0,1162
3.	1036,174	97,552	0,138	0,0190
4.	1035,470	97,485	0,071	0,005041
5.	1037,100	97,639	0,225	0,0506
6.	1033,412	97,292	0,122	0,0148
		$\tilde{x}$ = 97,414		Σ = 0,206964

$x = \frac{y-a}{b}$

$x_1 = \frac{1035,057-0,617400}{10,6154} = 97,447$

$x_2 = \frac{1031,087-0,617400}{10,6154} = 97,073$

$x_3 = \frac{1036,174-0,617400}{10,6154} = 97,552$

$x_4 = \frac{1035,470-0,617400}{10,6154} = 97,485$

$x_5 = \frac{1037,100-0,617400}{10,6154} = 97,639$

$x_6 = \frac{1033,412-0,617400}{10,6154} = 97,292$

SD $= \sqrt{\frac{\sum(x-\tilde{x})^2}{n-1}} = \sqrt{\frac{0,206964}{5}} = \sqrt{0,0413925} = 0,20345$

RSD $= \frac{SD}{\tilde{x}}$ x 100%

$= \frac{0,20345}{97,414}$ x 100%

= 0,00208 x 100%

= 0,208%

Ketelitian alat= 100% - $\frac{SD}{\tilde{x}}$

= 100% - $\frac{0,20345}{97,414}$

= 100% - 0,208%

= 99,8%

Lampiran 5. Data Penimbangan Bobot Tablet Parasetamol

Menimbang bobot rata-rata atau keseragaman bobot

Replikasi	Bobot Tablet (mg)
1	680
2	650
3	670
4	660
5	670
6	670
7	680
8	690
9	660
10	670
Rata-rata	670

Kemudian dihitung setara 100 ppm → 5 mg ad 50 ml

Bobot setara $= \frac{bobot\ yang\ diminta}{bobot\ dietiket}$ x bobot rata-rata tablet

$= \frac{5\ mg}{500\ mg}$ x 670 mg

= 6,7 mg g ad 50 ml → setara dengan 100 ppm

= 0,0067 g ad 50 ml → setara dengan 100 ppm

Menimbang sebanyak 0,0067 gram tablet parasetamol yang telah digerus dan dilarutkan kedalam 50 ml labu takar (menjadi larutan 100 ppm)

Lampiran 6. Data Hasil Perhitungan Akurasi

Hasil Akurasi Baku 50 ppm

Replikasi	Intensitas Non Baku	Intensitas Baku
1	521,390	787,773
2	549,671	805,052
3	524,183	800,871
Rata-rata	531,748	797,898

Perhitungan Akurasi baku 50 ppm

y = **x̃ intensitas baku - x̃ intensitas non baku**

= 797,898 – 531,748

= 266,15

y = bx + a

266,15 = 10,6154 x + 0,617400

266,15 – 0,617400 = 10,6154x

265,532 = 10,6154x

$$x = \frac{265,532}{10,6154}$$

= 25,013 ppm

$$\% \text{ recovery} = \frac{\text{kadar terukur}}{\text{kadar sebenarnya x fp}} \text{ x } 100\%$$

$$= \frac{25,013}{50 \; x \; 0,5} \text{ x } 100\%$$

= 100,05 %

Hasil Akurasi Baku 100 ppm

Replikasi	Intensitas Non Baku	Intensitas Baku
1	521,390	1062,979
2	549,671	1064,450
3	524,183	1062,641
Rata-rata	531,748	1063,464

Perhitungan Akurasi baku 100 ppm

y = **x̃ intensitas baku - x̃ intensitas non baku**

= 1063,464 – 531,748

= 531,716

y = bx + a

531,748 = 10,6154 x + 0,617400

531,748 – 0,617400 = 10,6154x

531,098 = 10,6154x

$$x = \frac{531,098}{10,6154}$$

= 50,030 ppm

$$\% \text{ recovery} = \frac{\text{kadar terukur}}{\text{kadar sebenarnya x fp}} \text{ x } 100\%$$

$$= \frac{50,030}{100 \; x \; 0,5} \text{ x } 100\%$$

= 100,06 %

Hasil Akurasi Baku 200 ppm

Replikasi	Intensitas Non Baku	Intensitas Baku
1	521,390	1599,641
2	549,671	1536,065
3	524,183	1665,076
Rata-rata	531,748	1600,260

Perhitungan Akurasi baku 200 ppm

y = **x̃ intensitas baku - x̃ intensitas non baku**

= 1600,260 – 531,748

= 1068,512

y = bx + a

1068,512= 10,6154 x + 0,617400

1068,512 – 0,617400 = 10,6154x

1067,895 = 10,6154x

$$x = \frac{1067,895}{10,6154}$$

= 100,600 ppm

$$\% \text{ recovery} = \frac{\text{kadar terukur}}{\text{kadar sebenarnya x fp}} \text{ x } 100\%$$

$$= \frac{100,600}{200 \; x \; 0,5} \text{ x } 100\%$$

= 100,6 %

Rata-rata % recovery $= \frac{100,05+100,06+100,60}{3}$

$= \frac{300,71}{3}$

= 100,23%

Lampiran 7. Data Hasil Perhitungan Penetapan Kadar

Rep	Konsentrasi (ppm)	Intensitas	Kadar parasetamol (mg/tab)	% kadar (x)
1	51,722	549,671	549,671	103,44%
2	49,058	521,390	521,390	98,11%
3	49,321	524,183	524,183	98,64%
$\tilde{x}$				100,07%

Penetapan kadar memenuhi syarat apabila berada pada kisaran 90-110% (Depkes RI, 2014). Dimana kadar parasetamol dietiket adalah 500 mg, maka:

500-50 = 450

500+50 = 550

Jadi kadar parasetamol mg/tablet yang memenuhi persyaratan apabila mencapai 450-550 mg/tablet

Hasil Penetapan Kadar 1 :

Y = bx + a

549,671 = 10,6154 x + 0,617400

549,671 - 0,617400 = 10,6154 x

x = 51,722

Kadar mg/tab = $\frac{x\ (ppm) x\ Fp\ x\ Vp}{mg\ serbuk\ x\ 1000}$ x bobot rata-rata tablet (mg/mL)

= $\frac{51,722\ x\ 50\ x\ 2}{6,7\ mg\ x\ 1000}$ x 670 mg

= $\frac{5172,2}{6700}$ x 670 mg

= 517,22 mg/tab

% kadar = $\frac{kadar\ mg/tab}{bobot\ etiket}$ x 100%

= $\frac{517,22}{500}$ x 100%

= 103,44 %

Hasil Penetapan Kadar II :

Y = bx + a

521,390 = 10,6154 x + 0,617400

521,390 - 0,617400 = 10,6154 x

x = 49,058

Kadar mg/tab = $\frac{x\ (ppm) x\ Fp\ x\ Vp}{mg\ serbuk\ x\ 1000}$ x bobot rata-rata tablet (mg/mL)

= $\frac{49{,}058\ x\ 50\ x\ 2}{6{,}7\ mg\ x\ 1000}$ x 670 mg

= $\frac{4905{,}8}{6700}$ x 670 mg

= 490,58 mg/tab

% kadar = $\frac{kadar\ mg/tab}{bobot\ etiket}$ x 100%

= $\frac{490{,}58}{500}$ x 100%

= 98,11 %

Hasil Penetapan Kadar III :

Y = bx + a

524,183 = 10,6154 x + 0,617400

524,183 - 0,617400 = 10,6154 x

x = 49,321

Kadar mg/tab = $\frac{x\ (ppm) x\ Fp\ x\ Vp}{mg\ serbuk\ x\ 1000}$ x bobot rata-rata tablet (mg/mL)

= $\frac{49{,}321\ x\ 50\ x\ 2}{6{,}7\ mg\ x\ 1000}$ x 670 mg

= $\frac{4932{,}1}{6700}$ x 670 mg

= 493,21 mg/tab

% kadar = $\frac{kadar\ mg/tab}{bobot\ etiket}$ x 100%

= $\frac{493{,}21}{500}$ x 100%

= 98,642 %

Rata-rata % penetapan kadar = $\frac{103{,}44+98{,}11+98{,}642}{3}$

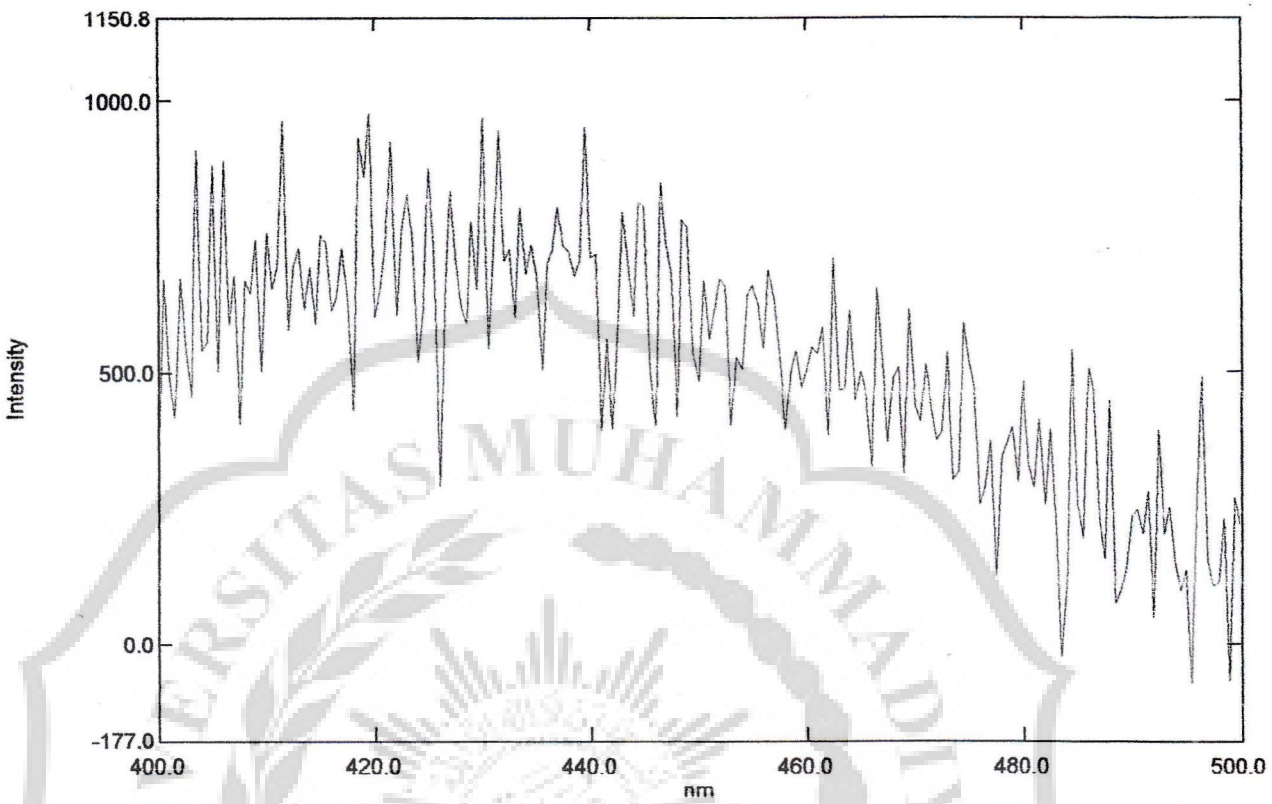
= $\frac{300{,}192}{3}$

= 100,07

Point Pick Report

Print Date: 30/06/2018 11:24:50 AM

File Name: OPTIMASI EMISI 100 ppm (20 maret) - CorrectionData



Date/Time: 20/03/2018 09:53:53 AM
Analyst:
Sample Name:
SampleID:
Option:
Comment:

[Measurement]
Spectrum Type: Emission
EX Wavelength: 330.0 nm
EM Wavelength Start: 400.0 nm
EM Wavelength End: 500.0 nm
Data Interval: 0.5 nm
Scan Speed: 6000 nm/min

[Instrument]
EX Bandwidth: 5.0 nm
EM Bandwidth: 5.0 nm
Sensitivity: Low

[Attachment]
Attachment: None

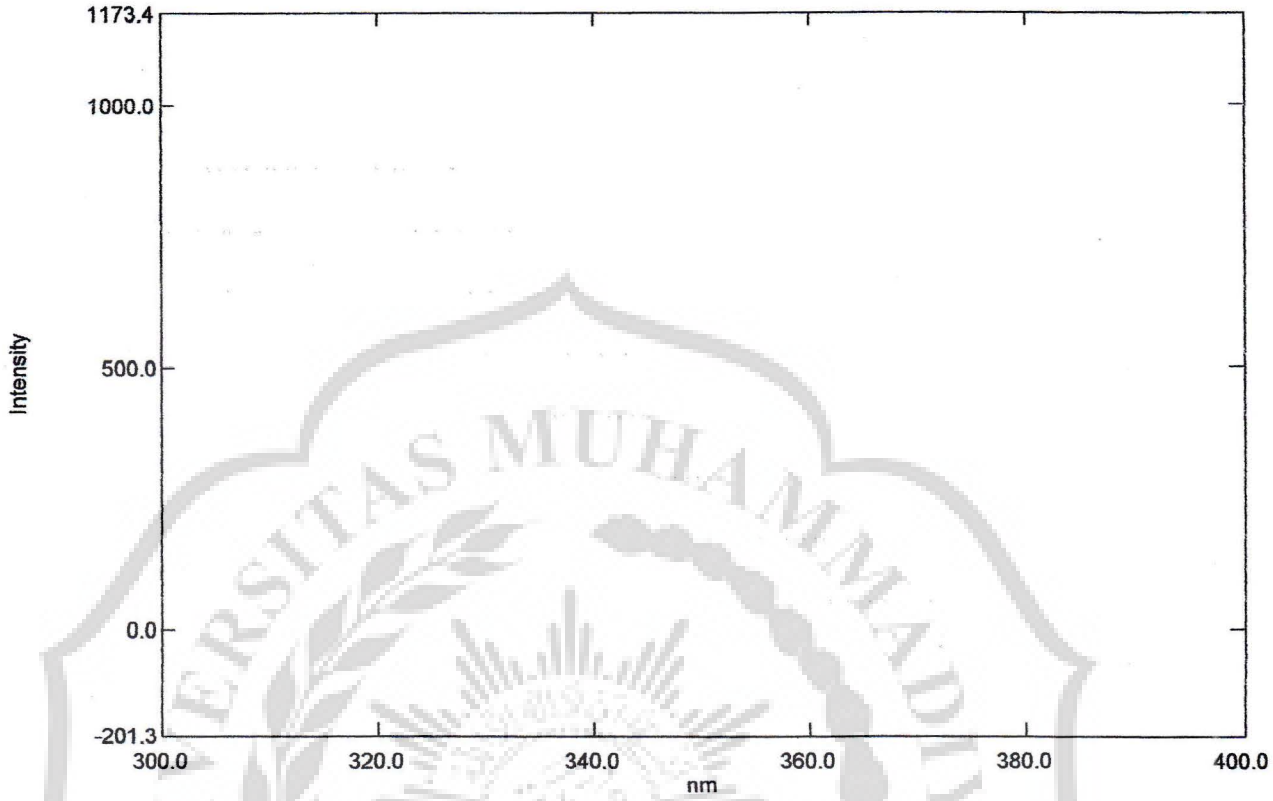
[Point Pick Table]

No.	Wavelength	Intensity	Description
1	400.0	352.6	
2	420.0	601.3	
3	427.0	832.7	
4	430.0	967.6	
5	440.0	710.4	
6	450.0	482.8	
7	460.0	504.2	
8	470.0	442.2	
9	480.0	482.1	
10	500.0	215.9	
11	490.0	233.5	

Point Pick Report

Print Date: 30/06/2018 11:26:15 AM

File Name: OPTIMASI EKSITASI 100 ppm (20 maret) - CorrectionData



Date/Time: 20/03/2018 09:57:57 AM
Analyst:
Sample Name:
SampleID:
Option:
Comment:

[Measurement]
Spectrum Type: Excitation
EX Wavelength Start: 300.0 nm
EX Wavelength End: 400.0 nm
EM Wavelength: 430.0 nm
Data Interval: 1.0 nm
Scan Speed: 6000 nm/min

[Instrument]
EX Bandwidth: 5.0 nm
EM Bandwidth: 5.0 nm
Sensitivity: Low

[Attachment]
Attachment: None

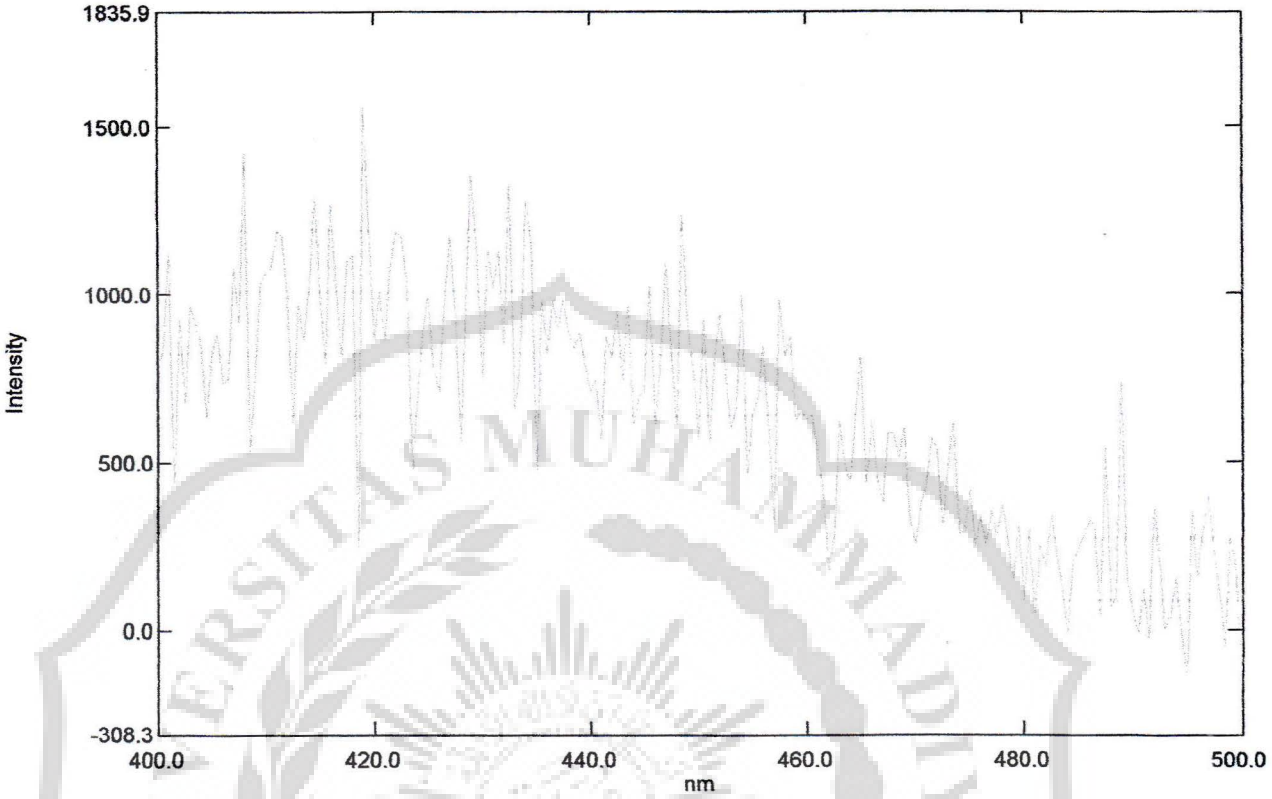
[Point Pick Table]

No.	Wavelength	Intensity	Description
1	300.0	860.3	
2	310.0	923.3	
3	320.0	877.6	
4	335.0	526.7	
5	330.0	523.5	
6	350.0	267.5	
7	360.0	121.5	
8	370.0	166.7	
9	380.0	118.2	
10	390.0	-32.8	

Point Pick Report

Print Date: 30/06/2018 11:27:33 AM

File Name: OPTIMASI EMISI 100 ppm (20 maret) (1) - CorrectionData



Date/Time: 20/03/2018 10:01:12 AM
Analyst:
Sample Name:
SampleID:
Option:
Comment:

[Measurement]
Spectrum Type: Emission
EX Wavelength: 310.0 nm
EM Wavelength Start: 400.0 nm
EM Wavelength End: 500.0 nm
Data Interval: 0.5 nm
Scan Speed: 6000 nm/min

[Instrument]
EX Bandwidth: 5.0 nm
EM Bandwidth: 5.0 nm
Sensitivity: Low

[Attachment]
Attachment: None

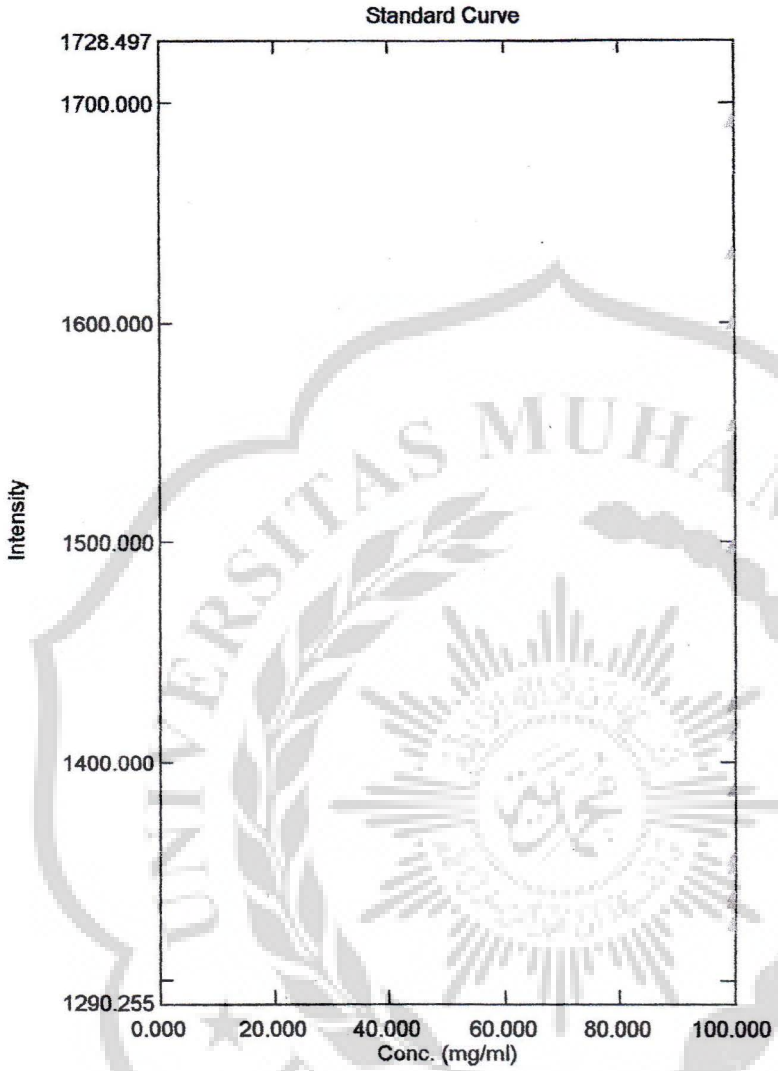
[Point Pick Table]

No.	Wavelength	Intensity	Description
1	400.0	769.5	
2	410.0	1064.7	
3	420.0	867.9	
4	427.0	1170.9	
5	430.0	751.0	
6	440.0	712.1	
7	450.0	578.2	
8	460.0	627.6	
9	470.0	255.5	
10	480.0	85.5	
11	490.0	53.1	
12	500.0	-90.7	

Standard Table Report

Print Date: 30/06/2018 11:37:09 AM

File Name: D:\IKA FAJAR RAKHMAWATI 1408010109\VARIASI pH\VARIASI pH BARU.fqqf



Date/Time: 20/05/2018 12:22:58 PM
Analyst:
Comment:

[Instrument Parameter]
EX Bandwidth: 5.0 nm
EM Bandwidth: 5.0 nm
Sensitivity: High
Accumulation Time: 10 ms

[Wavelengths]
Column Name1: EX310.0_EM427.0
EX Wavelength: 310.0 nm
EM Wavelength: 427.0 nm

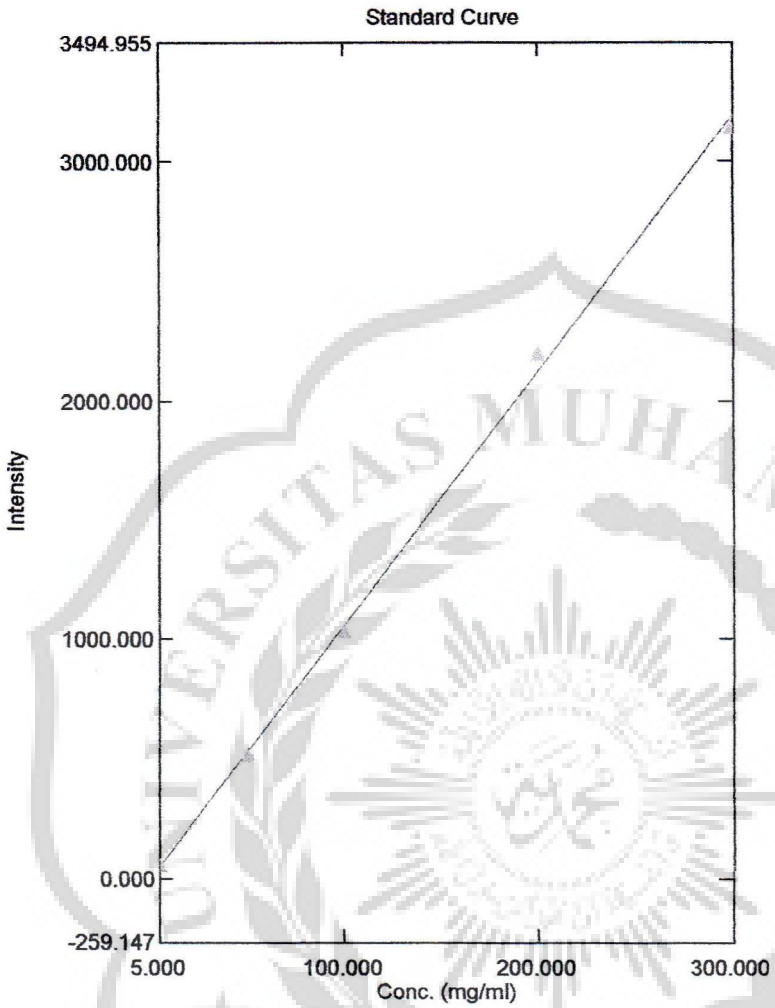
[Calibration Curve]
Column for Cal. Curve: EX310.0_EM427.0
Cal. Curve Type: Multi Point
Cal. Curve Unit: mg/ml
Selected Wavelength: EX310.0_EM427.0
Calibration Equation: Int = K1*(Conc) + K0
Zero Interception: No

	Sample Name	Type	Conc	EX310.0_EM427.0
1	pH 8	Standard	100.000	1339.295
2	pH 9	Standard	100.000	1356.281
3	pH 10	Standard	100.000	1387.229
4	pH 11	Standard	100.000	1336.637
5	pH 12	Standard	100.000	1326.775

Standard Table Report

Print Date: 30/06/2018 11:33:52 AM

File Name: D:\IKA FAJAR RAKHMAWATI 1408010109\KURVA BAKU\KURVA BAKU (26



y = 10.6154 x + 0.617400
r2 = 0.99870

Date/Time: 08/05/2018 01:13:19 PM
Analyst:
Comment:

[Instrument Parameter]

EX Bandwidth:	5.0 nm
EM Bandwidth:	5.0 nm
Sensitivity:	Low
Accumulation Time:	10 ms

[Wavelengths]

Column Name1:	EX310.0_EM427.0
EX Wavelength:	310.0 nm
EM Wavelength:	427.0 nm

[Calibration Curve]

Column for Cal. Curve:	EX310.0_EM427.0
Cal. Curve Type:	Multi Point
Cal. Curve Unit:	mg/ml
Selected Wavelength:	EX310.0_EM427.0
Calibration Equation:	Int = K1*(Conc) + K0
Zero Interception:	No

	Sample Name	Type	Conc	EX310.0_EM427.0
1	kuba 1	Standard	5.000	56.871
2	kuba 2	Standard	50.000	517.179
3	kuba 3	Standard	100.000	1037.032
4	kuba 4	Standard	200.000	2200.110
5	kuba 5	Standard	300.000	3144.998

Sample Table Report

Print Date: 30/06/2018 11:35:32 AM

File Name: D:\IKA FAJAR RAKHMAWATI 1408010109\KURVA BAKU (26 APRIL).fqqf

Date/Time: 20/05/2018 02:19:45 PM
Analyst:
Comment:

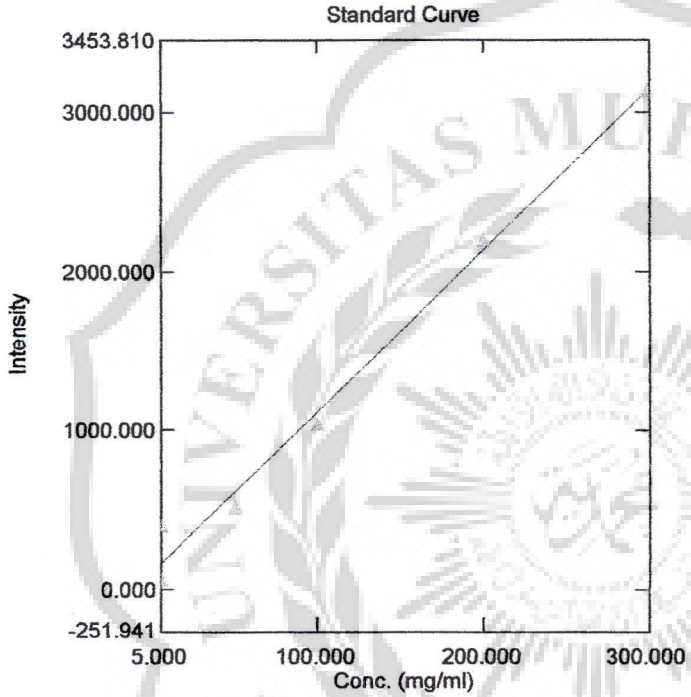
[Instrument Parameter]
EX Bandwidth: 5.0 nm
EM Bandwidth: 5.0 nm
Sensitivity: Low
Accumulation Time: 10 ms

[Wavelengths]
Column Name1: EX310.0_EM427.0
EX Wavelength: 310.0 nm
EM Wavelength: 427.0 nm

[Calibration Curve]
Column for Cal. Curve: EX310.0_EM427.0
Cal. Curve Type: Multi Point
Cal. Curve Unit: mg/ml
Selected Wavelength: EX310.0_EM427.0
Calibration Equation: Int = K1*(Conc) + K0
Zero Interception: No

[Standard]
Data Acquired by: Instrument
Repeat Count: 1
Delay sample read: No

[Sample]
Data Acquired by: Instrument
Repeat Count: 1
Delay sample read: No



y = 10.0957 x + 115.488
r2 = 0.98763

	Sample Name	Type	Conc	EX310.0_EM427.0
1	presisi 1	Unknown	97.447	1035.057
2	presisi 2	Unknown	97.073	1031.087
3	presisi 3	Unknown	97.552	1036.174
4	presisi 4	Unknown	97.486	1035.470
5	presisi 5	Unknown	97.639	1037.100
6	presisi 6	Unknown	97.292	1033.412
7	akurasi non baku	Unknown	51.722	549.671
8	akurasi non baku	Unknown	49.058	521.390
9	akurasi non baku	Unknown	49.321	524.183
10	akurasi baku 2	Unknown	100.077	1062.979

Sample Table Report

Print Date: 30/06/2018 11:35:32 AM

File Name: D:\IKA FAJAR RAKHMAWATI 1408010109\KURVA BAKU (26 APRIL).fqqf

Date/Time: 20/05/2018 02:19:45 PM
Analyst:
Comment:

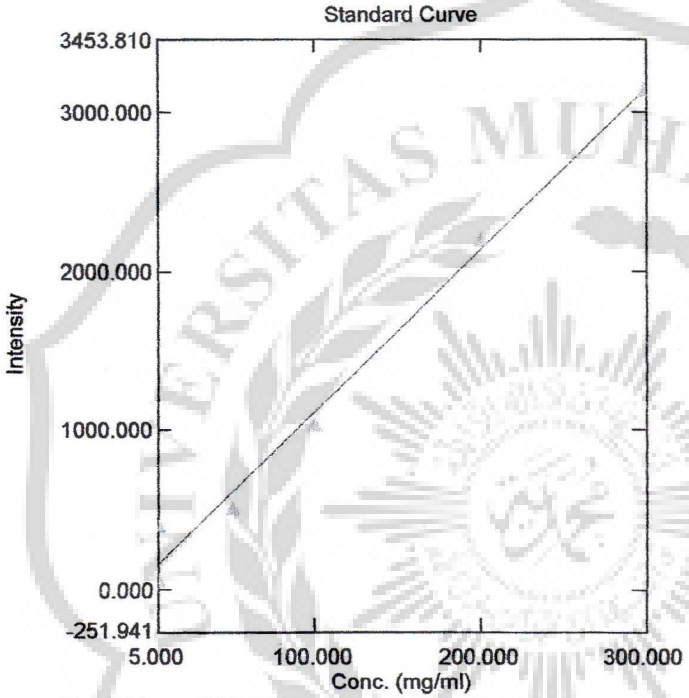
[Instrument Parameter]
EX Bandwidth: 5.0 nm
EM Bandwidth: 5.0 nm
Sensitivity: Low
Accumulation Time: 10 ms

[Wavelengths]
Column Name1: EX310.0_EM427.0
EX Wavelength: 310.0 nm
EM Wavelength: 427.0 nm

[Calibration Curve]
Column for Cal. Curve: EX310.0_EM427.0
Cal. Curve Type: Multi Point
Cal. Curve Unit: mg/ml
Selected Wavelength: EX310.0_EM427.0
Calibration Equation: Int = K1*(Conc) + K0
Zero Interception: No

[Standard]
Data Acquired by: Instrument
Repeat Count: 1
Delay sample read: No

[Sample]
Data Acquired by: Instrument
Repeat Count: 1
Delay sample read: No



y = 10.0957 x + 115.488
r2 = 0.98763

	Sample Name	Type	Conc	EX310.0_EM427.0
11	akurasi baku 2	Unknown	100.216	1064.450
12	akurasi baku 2	Unknown	100.076	1062.964
13	akurasi baku 3	Unknown	150.632	1599.641
14	akurasi baku 3	Unknown	144.643	1536.065
15	akurasi baku 3	Unknown	156.796	1665.076
16	akurasi baku 1	Unknown	74.152	787.773
17	akurasi baku 1	Unknown	75.780	805.052
18	akurasi baku 1	Unknown	75.386	800.871