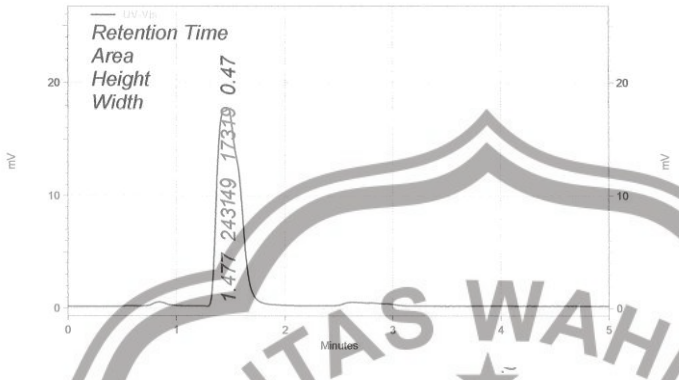
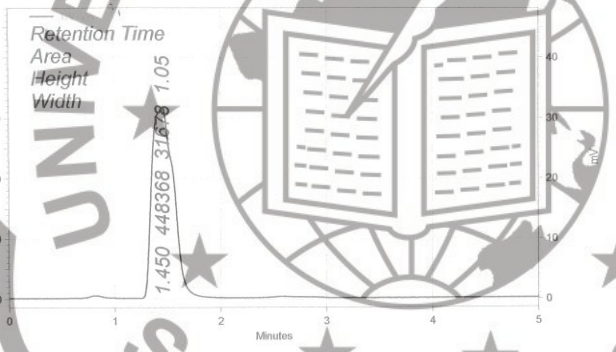
LAMPIRAN

Lampiran 1. Contoh Kromatogram Kurva Baku Prednison

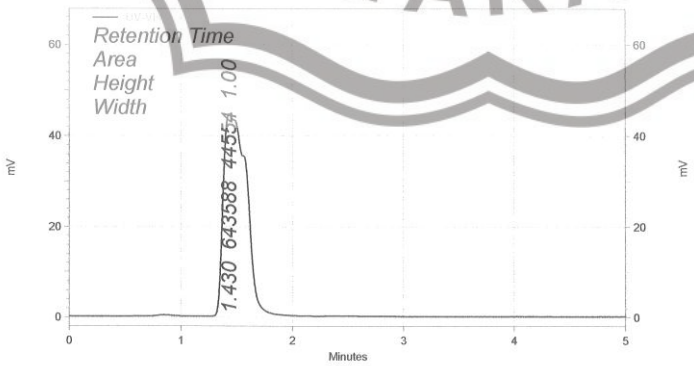
a. Larutan Baku Prednison 4 µg/mL



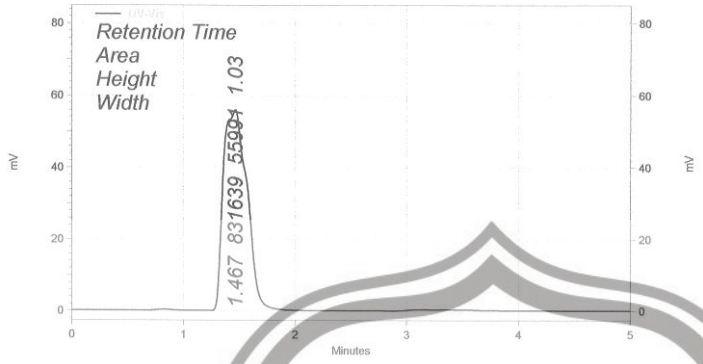
b. Larutan Baku Prednison 8 µg/mL



c. Larutan Baku Prednison 12 µg/mL

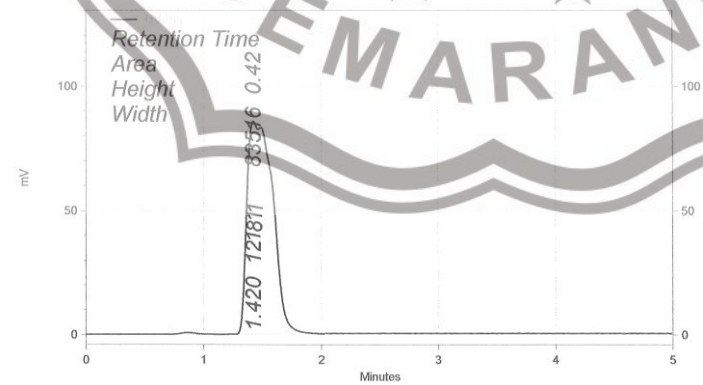


d. Larutan Baku Prednison 16 µg/mL



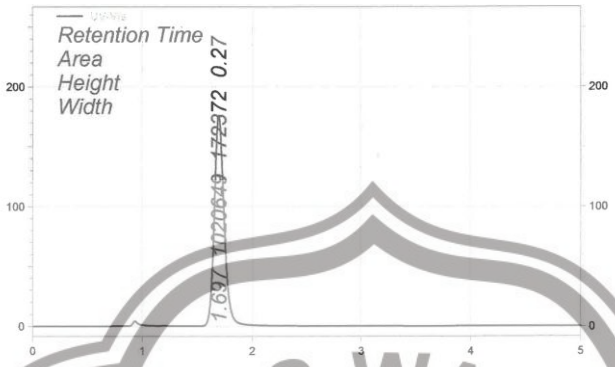
e. Larutan Baku Prednison 20 µg/mL

f. Larutan Baku Prednison 24 µg/mL

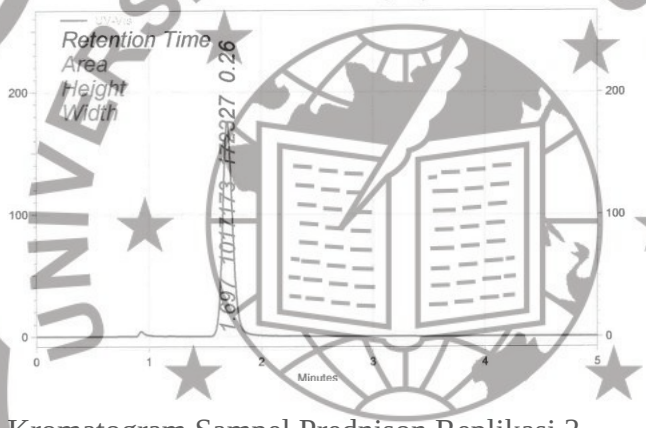


Lampiran 2. Contoh Kromatogram Sampel Prednison Replikasi 6 kali

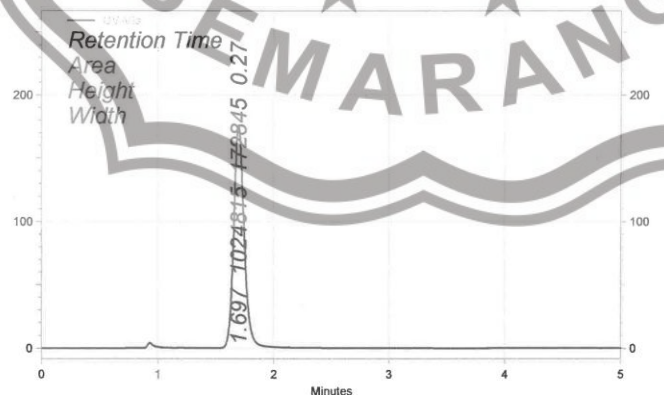
a. Kromatogram Sampel Prednison Replikasi 1



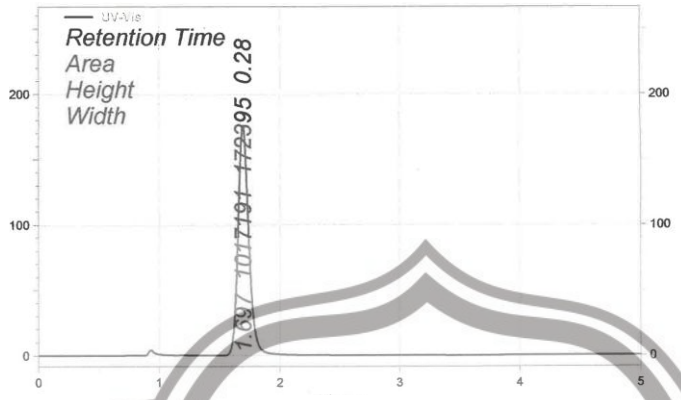
b. Kromatogram Sampel Prednison Replikasi 2



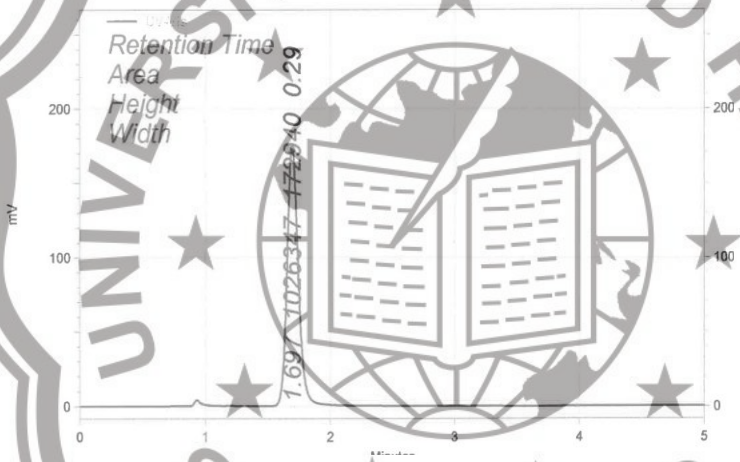
c. Kromatogram Sampel Prednison Replikasi 3



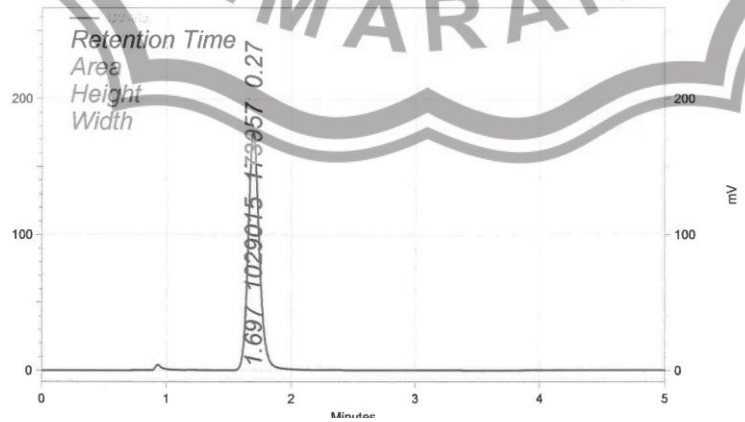
d. Kromatogram Sampel Prednison Replikasi 4



e. Kromatogram Sampel Prednison Replikasi 5



f. Kromatogram Sampel Prednison Replikasi 6



Lampiran 3. Contoh Perhitungan Perolehan Kembali Prednison dengan Metode Standard Addition Method

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 Prednison= 433481

      2) Kadar Prednison berdasarkan persamaan garis Y = 48619,26x+ 56336,13 adalah 8,091 µg/mL

   b. Konsentrasi bahan baku yang ditambahkan (C)

      1) Luas puncak Prednison= 247755

      3) Kadar prednison berdasarkan persamaan garis Y= 48619,26x + 56336,13 adalah 4,306 µg/mL

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

      1) Luas puncak total analit 1 = 645678

         Luas puncak total analit 2 = 644814

         Luas puncak total analit 3 = 64490

      2) Berdasarkan persamaan garis Y = 48619,26x+56336,13 maka :

         Kadar total analit 1 = 12,416 µg/mL

         Kadar total analit 2 = 12,398 µg/mL

         Kadar total analit 3 = 12,402 µg/mL

2. Perhitungan perolehan kembali

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

a. Analit 1

% perolehan kembali = $\frac{12,416-8,091}{4,306}$ x 100% = 100,441 %

b. Analit 2

% perolehan kembali = $\frac{12,398-8,091}{4,306}$ x 100% = 100,023 %

c. Analit 3

% perolehan kembali = $\frac{12,402-8,091}{4,306}$ x 100% = 100,116 %

Lampiran 4. Perhitungan LOD dan LOQ Prednison

X	Xi^2	Xi-$\bar{X}$	$(Xi-\bar{X})^2$	Yi	Yc	(Yi-Yc)	$(Yi-Yc)^2$
4	16	-10	100	246149	232756	13392,9	179370574
8	64	-6	36	448368	429016	19351,5	374481713
12	144	-2	4	643588	625277	18311,1	335297482
16	256	2	4	831639	821537	10101,7	102044949
20	400	6	36	1033480	1017798	15682,3	245935474
24	576	10	100	1218811	1214058	4752,93	22590344
14	1456		280				1259720536

Dari persamaan Y = 48619,26x + 56336,13 maka Yc dapat dihitung :

1. Y = 48619,26x + 56336,13

   Y = 48619,26 (4) + 56336,13

   Y = 250813,2

2. Y = 48619,26x + 56336,13

3. Y = 48619,26 (8) + 56336,13

Y = 445290,21

4. Y = 48619,26x + 56336,13
5. Y = 48619,26 (12) + 56336,13

   Y = 639767,25
6. Y = 48619,26x + 56336,13

   Y = 48619,26 (16) + 56336,13

   Y = 834244,29
7. Y = 48619,26x + 56336,13

   Y = 48619,26 (20) + 56336,13

   Y = 1028721,3
8. Y = 48619,26x + 56336,13

   Y = 48619,26 (24) + 56336,13

   Y = 1223198,4
9. Persamaan kurva baku : Y = 48619,26x + 56336,13 (r = 0,999877)

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

$= (1259720536/4)^{1/2}$

$= 17746,3$

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

$= 17746,3 \times \sqrt{\frac{1456}{6 \times 280}}$

$= 17746,3 \times 0,930949$

= 16520,900

Perhitungan 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 = 56336,13 + 3 (16520,900)

= 56336,13 + 49562,7

= 105898,83

Maka nilai LOD :

Y = 48619,26x + 56336,13

105898,83 = 48619,26x + 56336,13

X = 1,020 µg/mL

Perhitungan LOQ :

Dihitung berdasarkan rumus $Y = Y_B + 10\ S_B$

Y = 56336,13 + 10 (16520,900)

= 56336,13 + 165209

= 157428,9

Maka nilai LOQ :

Y = 48619,26x + 56336,13

221545,2 = 48619,26x + 56336,13

X = 3,398 µg/mL

Lampiran 5. Contoh Perhitungan Kadar Prednison dalam Sediaan Tablet

Persamaan regresi linier kurva baku adalah

Y = BX + A

Y = 48619,26x + 56336,13

Replikasi 1

Y = 9566,71x + 62336,33

1020649 = 48619,26x – 56336,13

X = 19,834 µg/mL

Sehingga kadar prednison dalam 200 mg pada sediaan tablet adalah

X = 200,581 µg/mL x 10 ml

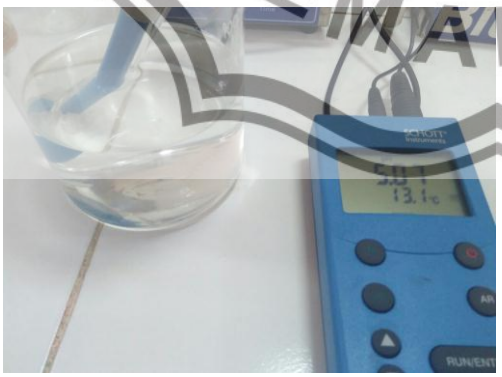
= 2005,81 µg/mL / 1000

= 5,015 mg

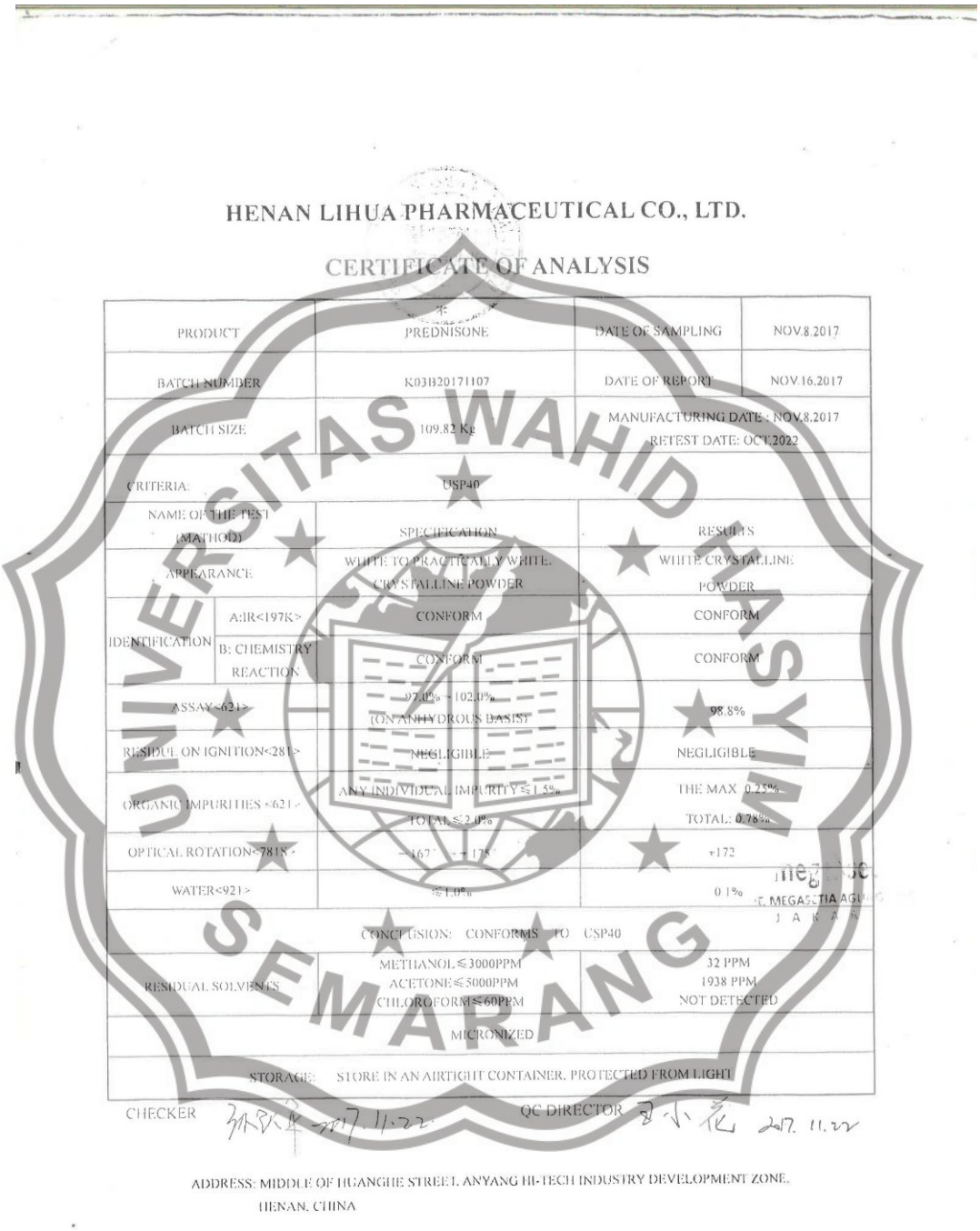
% Kadar prenison dalam sediaan tablet

$= \frac{5,015\ mg}{5mg} \times 100\% = 100,3\ \%$

Lampiran 6. Dokumentasi Penelitian



Lampiran 7. Certificate Of Analysis Prednison



HENAN LIHUA PHARMACEUTICAL CO., LTD.

CERTIFICATE OF ANALYSIS

PRODUCT	PREDNISONE	DATE OF SAMPLING	NOV.8.2017
BATCH NUMBER	K03B20171107	DATE OF REPORT	NOV.16.2017
BATCH SIZE	109.82 Kg	MANUFACTURING DATE : NOV.8.2017 RETEST DATE: OCT.2022	
CRITERIA:	USP40		
NAME OF THE TEST (METHOD)		SPECIFICATION	RESULTS
APPEARANCE		WHITE TO PRACTICALLY WHITE, CRYSTALLINE POWDER	WHITE CRYSTALLINE POWDER
IDENTIFICATION	A:IR<197K>	CONFORM	CONFORM
	B: CHEMISTRY REACTION	CONFORM	CONFORM
ASSAY<621>		97.0% ~ 102.0% (ON ANHYDROUS BASIS)	98.8%
RESIDUL ON IGNITION<281>		NEGLIGIBLE	NEGLIGIBLE
ORGANIC IMPURITIES <621>		ANY INDIVIDUAL IMPURITY ≤ 1.5% TOTAL ≤ 2.0%	THE MAX 0.25% TOTAL: 0.78%
OPTICAL ROTATION<781S>		+167° ~ +178°	+172
WATER<921>		≤ 1.0%	0.1%
CONCLUSION: CONFORMS TO USP40			
RESIDUAL SOLVENTS		METHANOL ≤ 3000PPM ACETONE ≤ 5000PPM CHLOROFORM ≤ 60PPM	32 PPM 1938 PPM NOT DETECTED
MICRONIZED			
STORAGE: STORE IN AN AIRTIGHT CONTAINER, PROTECTED FROM LIGHT			

CHECKER 2017.11.22 QC DIRECTOR 2017.11.22

ADDRESS: MIDDLE OF HUANGHE STREET, ANYANG HI-TECH INDUSTRY DEVELOPMENT ZONE, HENAN, CHINA

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

SURAT KETERANGAN
No.07/Lab. Kimia Farmasi/ C.05/UWH/I/ 2019

Assalamu'alaikum Wr. Wb.

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

Nama : Elsa Agnes Belinda
NIM : 145010160
Fak/ Univ/ Sekolah : Farmasi / Universitas Wahid Hasyim Semarang

Telah melakukan Penelitian Penetapan Kadar Prednison menggunakan HPLC di Laboratorium Kimia Analisa, Fakultas Farmasi Universitas Wahid Hasyim Semarang, dengan judul penelitian :

" Validasi Metode Penetapan Kadar Prednison dengan Menggunakan Kromatografi Cair Kinerja Tinggi dan Aplikasinya Pada Sediaan Tablet "

Demikian surat keterangan ini dibuat untuk dipergunakan semestinya.

Wassalamu'alaikum Wr. Wb.

Semarang, Januari 2019
Ka.Bag Kimia Farmasi

Maria Ulfah, M.Sc, Apt