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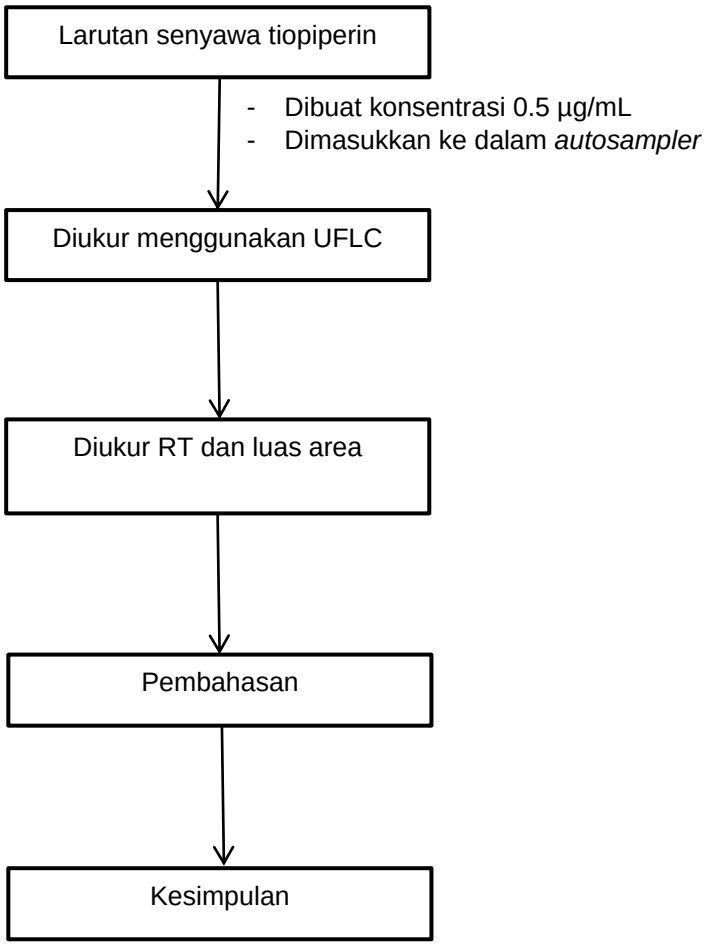
LAMPIRAN

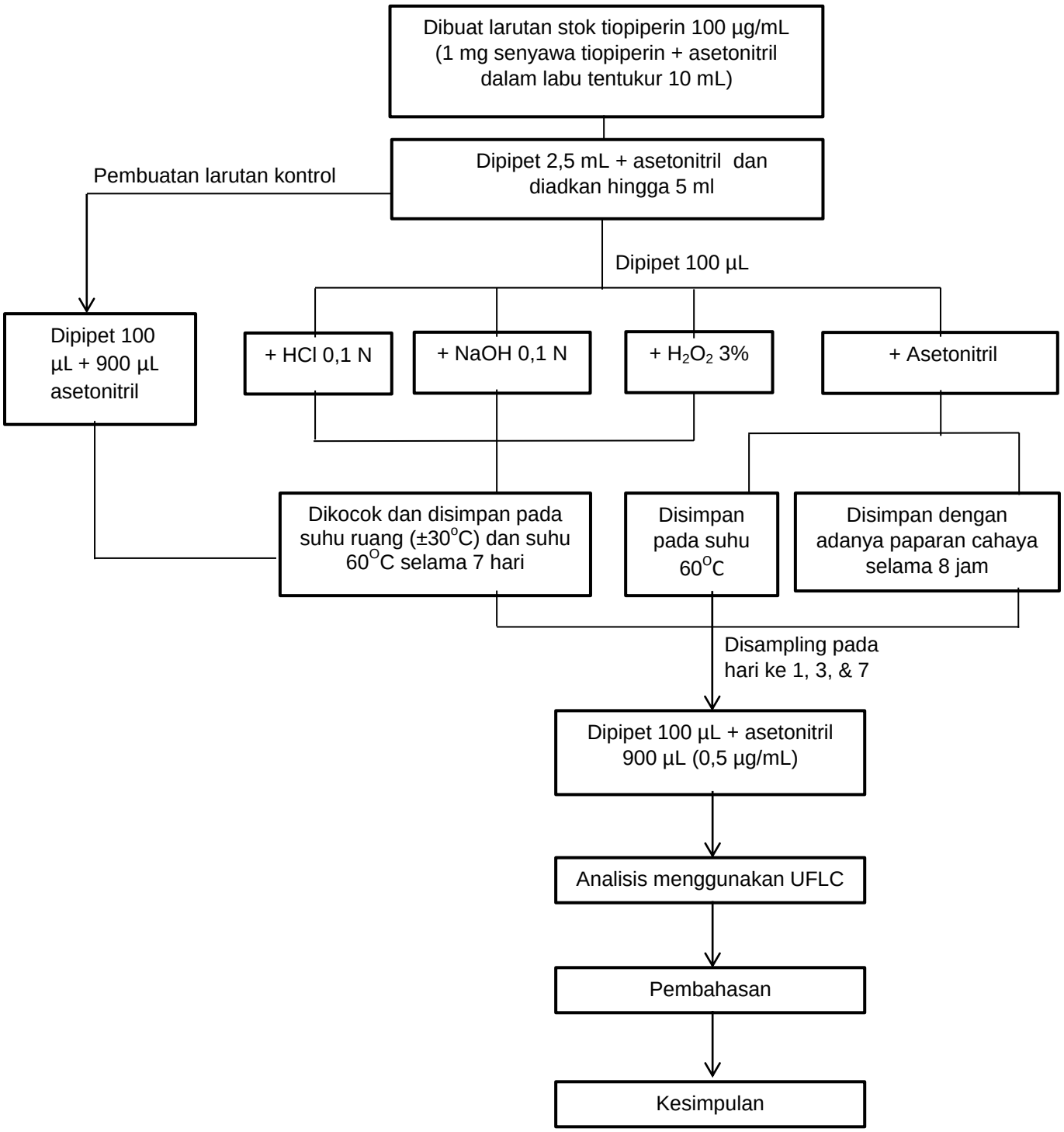
Lampiran 1

Skema Kerja (Uji kesesuaian sistem)



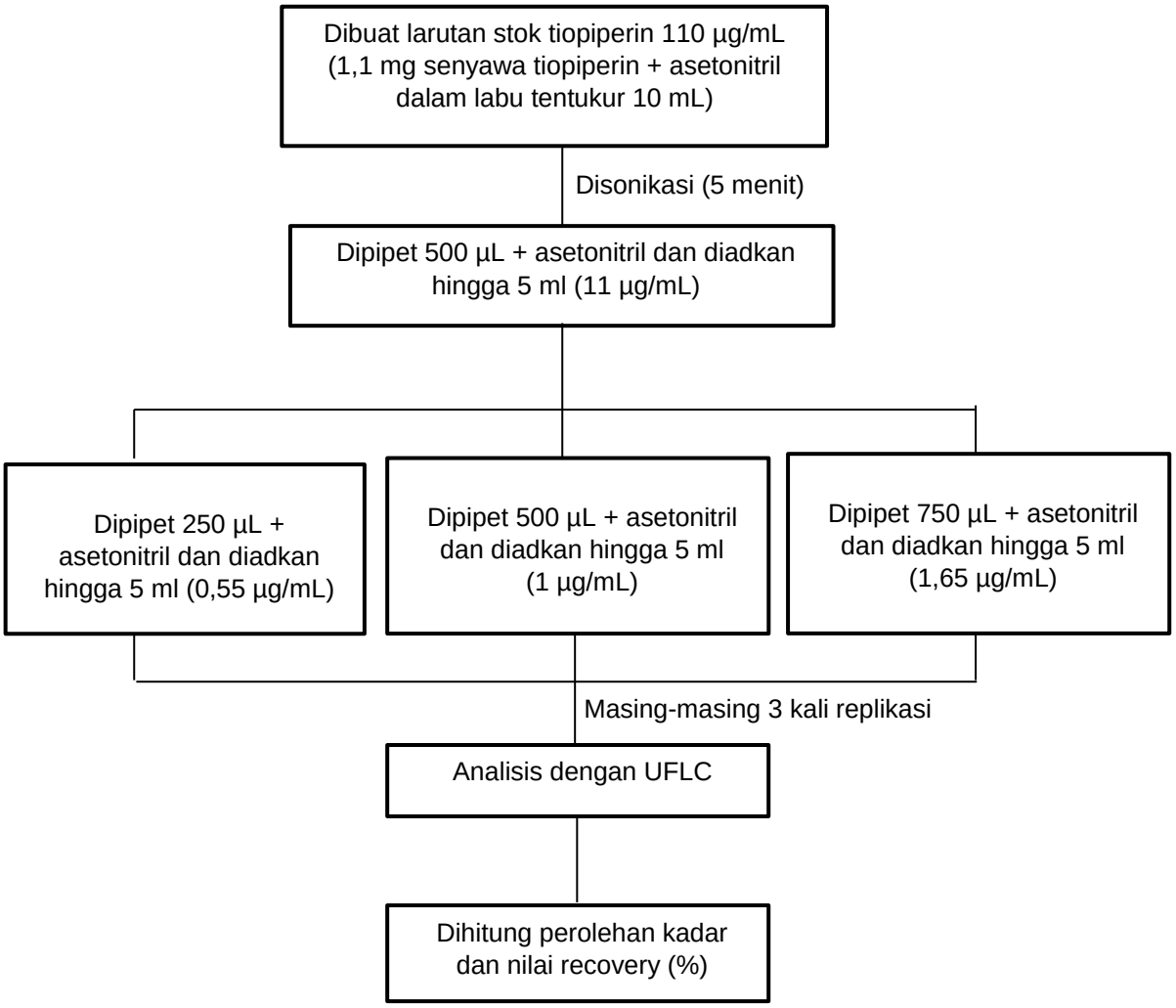
Lampiran 2

(FDS)

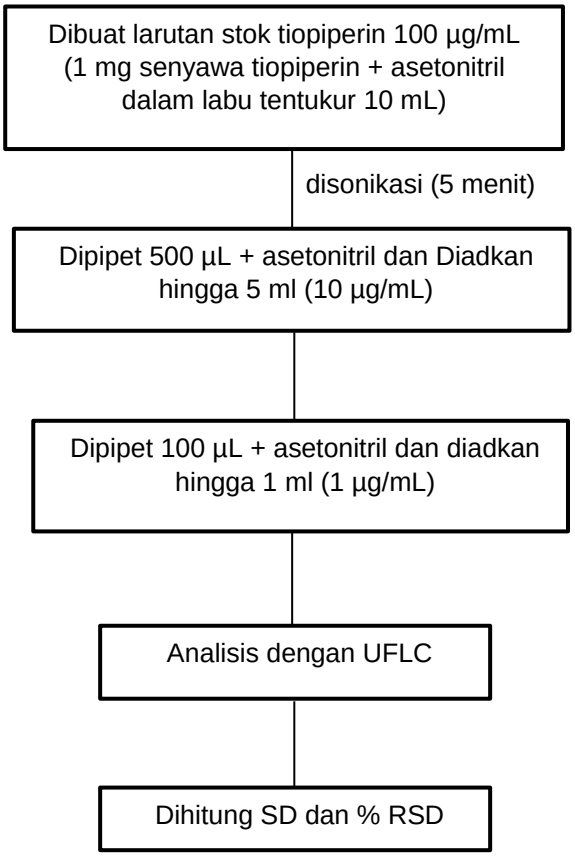


Lampiran 3

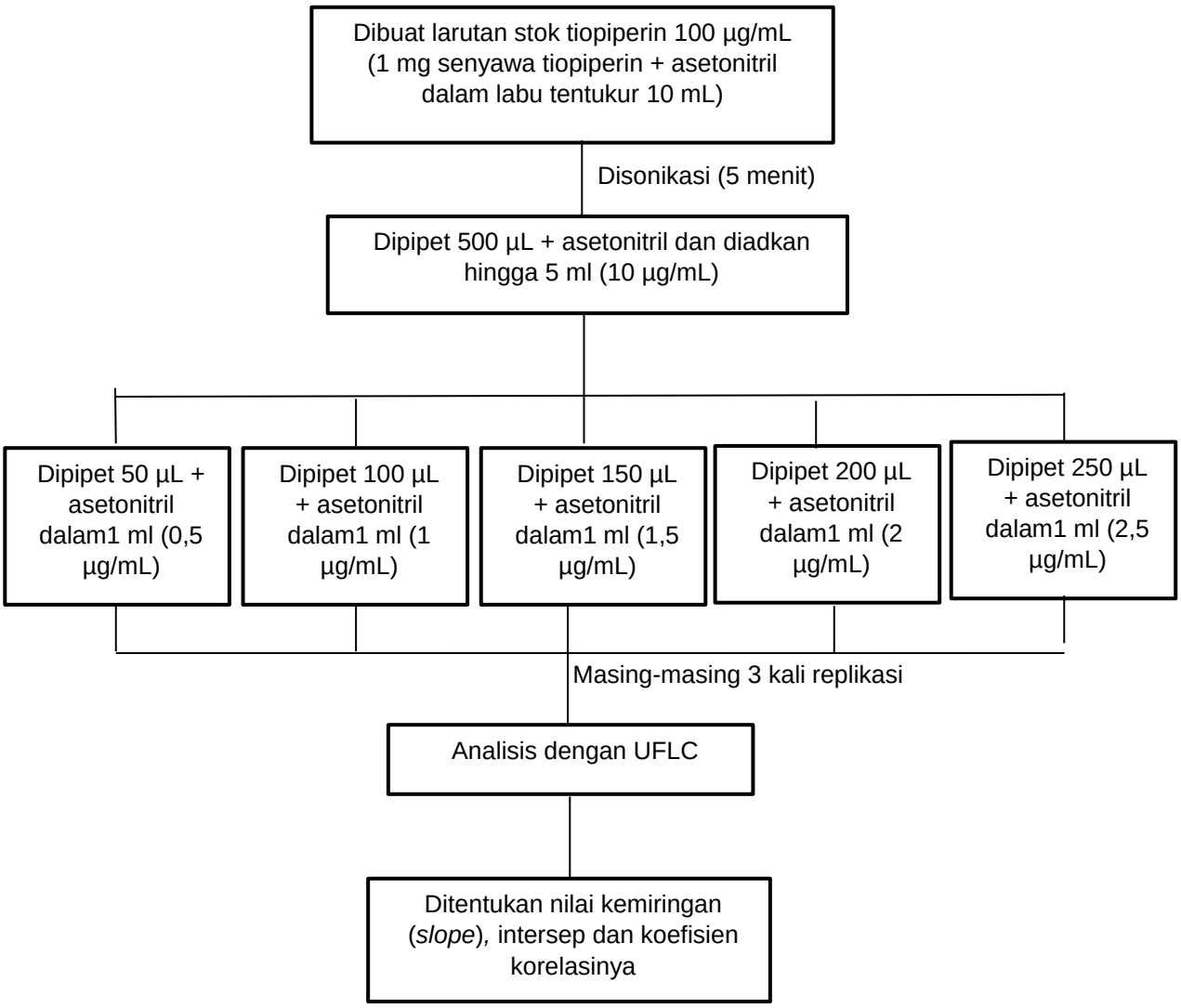
1. Validasi Metode (Akurasi)



2. Presisi


Dibuat larutan stok tiopiperin 100 µg/mL
(1 mg senyawa tiopiperin + asetonitril
dalam labu tentukur 10 mL)
disonikasi (5 menit)
Dipipet 500 µL + asetonitril dan Diadkan
hingga 5 ml (10 µg/mL)
Dipipet 100 µL + asetonitril dan diadkan
hingga 1 ml (1 µg/mL)
Analisis dengan UFLC
Dihitung SD dan % RSD

3. Linearitas



Lampiran 4

Perhitungan

1. Untuk pembuatan pereaksi asam (HCl 0,1 N), basa (NaOH 0,1 N) dan hidrogen peroksida (H_2O_2 3%), maka perhitungannya adalah

   a) Larutan HCl 0,1 N

   Konsentrasi 37%

   Berat jenis = 1,19 g/mL

   Berat molekul = 36,5 g/mol

   N = ((10 x % x bj) x valensi)/BM

   = ((10 x 37% x 1,19 g/ml)x1)/36,5

   = 12,06 N

   Dik : M_1 = 12,06 N Dit : V_1 = …….?

   M_2 = 0,1 M

   V_2 = 50 mL

   Maka :

   M_1 x V_1 = M_2 x V_2

   12,06 N x V_1 = 0,1 N x 50 mL

   V_1 = 0.4 mL

   b) Larutan NaOH 0,1 N

   Dik : Bobot mol NaOH = 40 g/mol Dit : Massa = …….?

   Volume = 50 mL

   M = 0,1 N

   Maka :

$$M: \frac{massa\ NaOH\ (gram)}{Bobot\ molekul\ NaOH\ (\frac{g}{mol})}\ x\ \frac{1000}{volume\ (mL)}$$

$$0{,}1\ M = \frac{massa}{40\ g/mol}\ x \frac{1000}{50\ mL}$$

$$0{,}1\ M = \frac{massa}{40\ g/mol}\ x\ 20/mL$$

$$massa = \frac{0{,}1\ M\ x\ 40\frac{g}{mol}}{20/ml}$$

$$\text{massa } = 0{,}2 \text{ gram } (200 \text{ mg})$$

c) Larutan H_2O_2 3%

Dik : M_1= 0,3 M Dit : V_1 =.....?

M_2 = 0,03 M

V_2 = 50 mL

Maka :

M_1 x V_1 = M_2 x V_2

0,3 M x V_1 = 0,03 M x 50 mL

V_1 = 1,5 Mml/0,35 M

V_1 = 5 mL

2. Untuk pembuatan larutan stok dalam FDS dan konsentrasi uji degradasi, maka perhitungannya sebagai berikut :

a) Konsentrasi larutan stok 100 µg/mL

Dik : Massa sampel = 1 mg

Volume pelarut = 10 mL

Maka :

$$\text{konsentrasi} = \frac{\text{Massa Sampel}}{\text{Volume Pelarut}}$$

$$= \frac{1 \text{ mg}}{10 \text{ mL}}$$

= 0,1 x 1000 µg/mL

= 100 µg/mL

b) Konsentrasi larutan stok 50 µg/mL

Dik : M_1 = 100 µg/mL Dit : V_1 =.....?

M_2 = 50 µg/mL

V_2 = 5 mL

Maka :

M_1 x V_1 = M_2 x V_2

100 µg/mL x V_1 = 50 µg/mL x 5 mL

V_1 = 2,5 mL

c) Konsentrasi larutan uji degradasi

Larutan 5 µg/mL

Dik : M_1= 50 µg/mL Dit : V_1 =.....?

M_2 = 5 µg/mL

V_2 = 1 mL

Maka :

M_1 x V_1 = M_2 x V_2

50 µg/mL x V_1 = 5 µg/mL x 1 mL

V_1 = 5 / 50

V_1 = 0,1 mL = 100 µL

Larutan 0.5 µg/mL

Dik : M_1= 5 µg/mL Dit : V_1 =.....?

M_2 = 0,5 µg/mL

V_2 = 1 mL

Maka :

M_1 x V_1 = M_2 x V_2

5 µg/mL x V_1 = 0,5 µg/mL x 1 mL

V_1 = 5 / 0,5

V_1 = 0,1 mL = 100 µL

3. Untuk pembuatan larutan dalam validasi metode UFLC, maka perhitungannya sebagai berikut :

   Akurasi

   (a) Konsentrasi larutan stok 110 µg/mL

   Dik : Massa sampel = 1,1 mg
   Volume pelarut = 10 mL

   Maka :

$$\text{konsentrasi} = \frac{\text{Massa Sampel}}{\text{Volume Pelarut}}$$

$$= \frac{1{,}1\ \text{mg}}{10\ \text{mL}}$$

   = 0,11 x 1000 µg/mL

   = 110 µg/mL

   (b) Konsentrasi larutan stok 10 µg/mL

   Dik : M_1 = 110 µg/mL Dit : V_1 =.....?
   M_2 = 11 µg/mL
   V_2 = 5 mL

   Maka :

   M_1 x V_1 = M_2 x V_2
   110 µg/mL x V_1 = 11 µg/mL x 5 mL
   V_1 = 0,5 mL = 500 µL

   Larutan 0.5 µg/mL

   Dik : M_1 = 10 µg/mL Dit : V_1 =.....?
   M_2 = 0,55 µg/mL
   V_2 = 5 mL

   Maka :

   M_1 x V_1 = M_2 x V_2
   11 µg/mL x V_1 = 0,55 µg/mL x 5 mL

V_1 = 0,25 mL = 250 µL

Larutan 1 µg/mL

Dik : M_1 = 11 µg/mL Dit : V_1 =.....?

M_2 = 1 µg/mL

V_2 = 5 mL

Maka :

M_1 x V_1 = M_2 x V_2

10 µg/mL x V_1 = 1 µg/mL x 5 mL

V_1 = 0,45 mL = 450 µL

Larutan 1,65 µg/mL

Dik : M_1 = 10 µg/mL Dit : V_1 =.....?

M_2 = 1,65 µg/mL

V_2 = 5 mL

Maka :

M_1 x V_1 = M_2 x V_2

10 µg/mL x V_1 = 1,65 µg/mL x 5 mL

V_1 = 0,75 mL = 750 µL

Presisi dan Linearitas

a. Konsentrasi larutan stok 100 µg/mL

Dik : Massa sampel = 1 mg

Volume pelarut = 10 mL

Maka :

$$\text{konsentrasi} = \frac{\text{Massa Sampel}}{\text{Volume Pelarut}}$$

$$= \frac{1\ \text{mg}}{10\ \text{mL}}$$

= 0,1 x 1000 µg/mL

= 100 µg/mL

b. Konsentrasi larutan stok 10 µg/mL

Dik : M_1 = 100 µg/mL Dit : V_1 =.....?

M_2 = 10 µg/mL

V_2 = 5 mL

Maka :

M_1 x V_1 = M_2 x V_2

100 µg/mL x V_1 = 10 µg/mL x 5 mL

V_1 = 0,5 mL

Larutan 0,5 µg/mL

Dik : M_1 = 10 µg/mL Dit : V_1 =.....?

M_2 = 0,5 µg/mL

V_2 = 1 mL

Maka :

M_1 x V_1 = M_2 x V_2

10 µg/mL x V_1 = 0,5 µg/mL x 1 mL

V_1 = 0,05 mL = 50 µL

Larutan 1 µg/mL

Dik : M_1 = 10 µg/mL Dit : V_1 =.....?

M_2 = 1 µg/mL

V_2 = 1 mL

Maka :

M_1 x V_1 = M_2 x V_2

10 µg/mL x V_1 = 1 µg/mL x 1 mL

V_1 = 0,1 mL = 100 µL

Larutan 1,5 µg/mL

Dik : M_1 = 10 µg/mL Dit : V_1 =.....?

M_2 = 1,5 µg/mL

V_2 = 1 mL

Maka :

M_1 x V_1 = M_2 x V_2

10 µg/mL x V_1 = 1,5 µg/mL x 1 mL

V_1 = 0,15 mL = 150 µL

Larutan 2 µg/mL

Dik : M_1 = 10 µg/mL Dit : V_1 =.....?

M_2 = 2 µg/mL

V_2 = 1 mL

Maka :

M_1 x V_1 = M_2 x V_2

10 µg/mL x V_1 = 2 µg/mL x 1 mL

V_1 = 0,2 mL = 200 µL

Larutan 2,5 µg/mL

Dik : M_1 = 10 µg/mL Dit : V_1 =.....?

M_2 = 2,5 µg/mL

V_2 = 1 mL

Maka :

M_1 x V_1 = M_2 x V_2

10 µg/mL x V_1 = 2,5 µg/mL x 1 mL

V_1 = 0,25 mL = 250 µL

Lampiran 5

(Data hasil uji kesesuaian sistem)

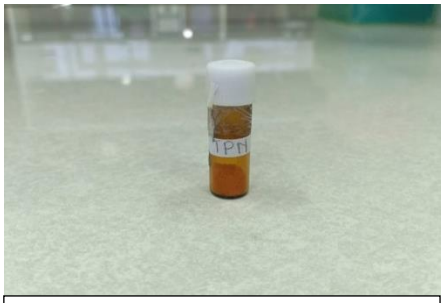
Tabel 15. Analisis hasil uji kesesuaian sistem

Senyawa	TR (menit)	AUC	*Tailing factor*	N
Piperin	3,904	17026	1	6096,5
Tiopiperin	16,668	15118	1	17758,6

Lampiran 6

(Dokumentasi penelitian)

Gambar 16. Senyawa piperin



Gambar 17. Senyawa tiopiperin

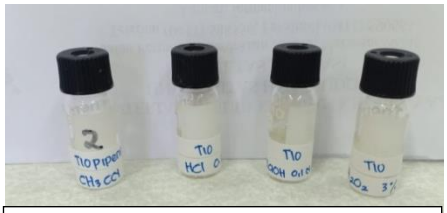
Gambar 18. Pereaksi NaOH 0,1 N, HCl 0,1 N dan H_2O_2 3%

Gambar 19. Penimbangan senyawa tiopiperin (1 mg)

Gambar 20. Larutan senyawa tiopiperin 100 µg/mL

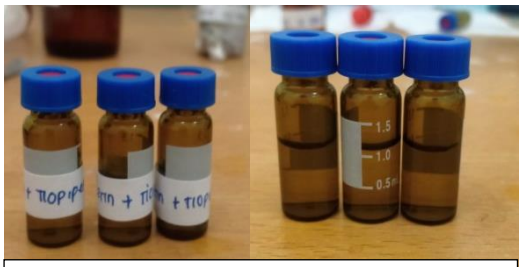
Gambar 21. Larutan sampel konsentrasi 50 µg/mL

Gambar 22. Proses sonikasi sampel uji, menggunakan alat sonikator



Gambar 23. Larutan uji yang disimpan pada suhu ruang (±30^{o}C)

Gambar 24. Larutan uji yang disimpan pada suhu 60^{o}C



Gambar 28. Larutan uji presisi

Gambar 25. Penimbangan senyawa tiopiperin (1 mg) pada validasi metode UFLC

Gambar 29. Pengukuran larutan uji dengan alat UFLC

Gambar 26. Larutan stok 100 µg/mL pada validasi metode

Gambar 30. Alat thermohygrometer

Gambar 27. Larutan uji akurasi dengan 3 konsentrasi berbeda