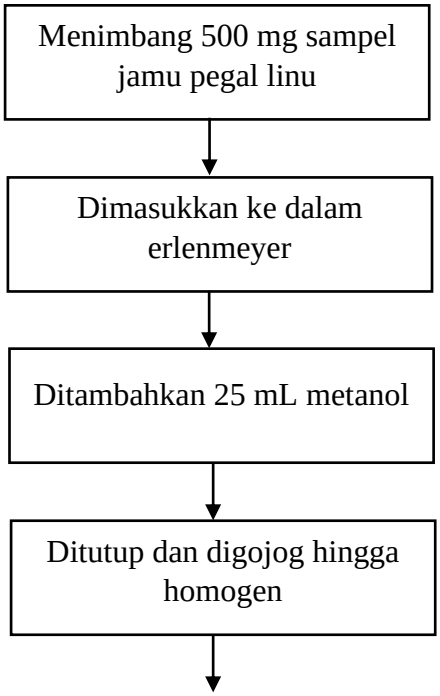
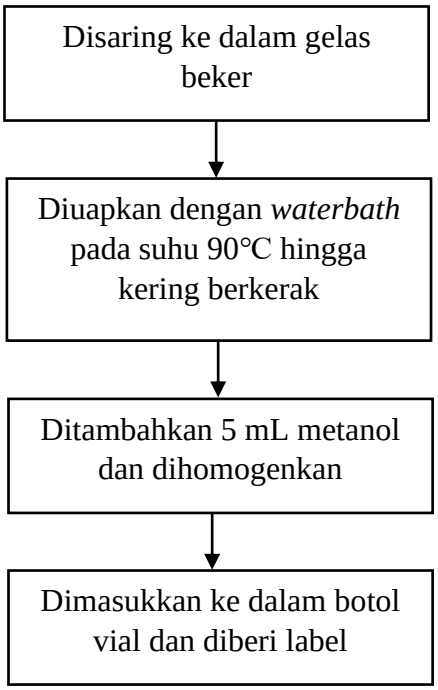
LAMPIRAN

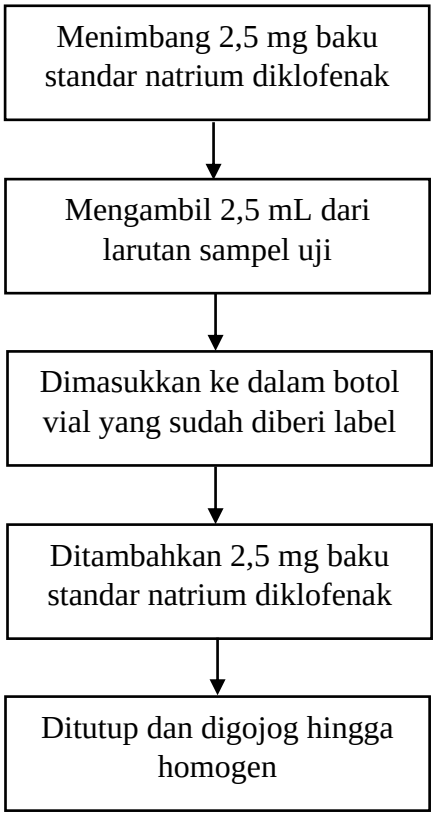
Lampiran 1. Diagram Alir Pembuatan Larutan Baku Standar Natrium Diklofenak 1000 ppm

Lampiran 2. Diagram Alir Pembuatan Larutan Sampel Uji

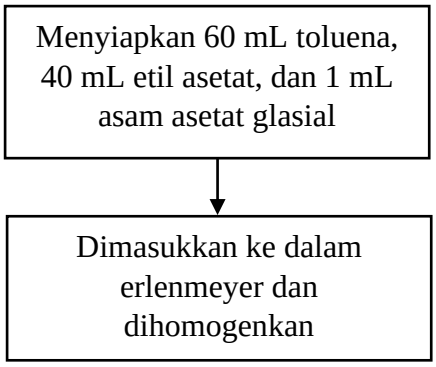




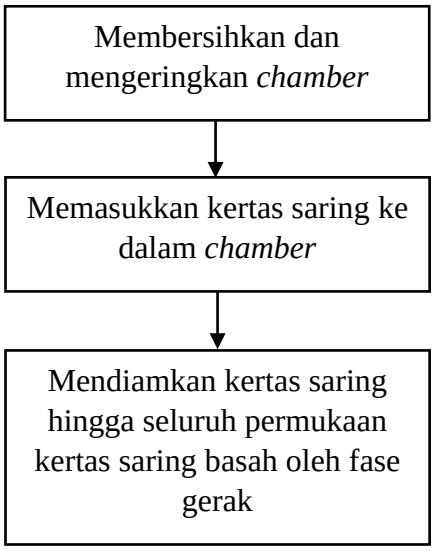
Lampiran 3. Diagram Alir Pembuatan Larutan *Spiked* Sampel



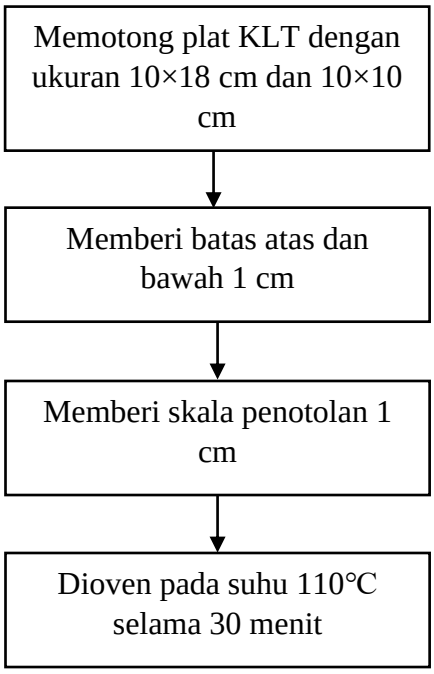
Lampiran 4. Diagram Alir Persiapan Fase Gerak



Lampiran 5. Diagram Alir Penjenuhan Fase Gerak dengan Kertas Saring



Lampiran 6. Diagram Alir Persiapan Fase Diam Plat KLT Silika Gel 60 F_{254}



Lampiran 7. Diagram Alir Pengujian dengan Kromatografi Lapis Tipis (KLT)

Lampiran 8. Perhitungan Konsentrasi Larutan Baku Standar Natrium Diklofenak 1000 ppm

Diketahui: Berat baku standar natrium diklofenak = 10 mg
Volume metanol = 10 mL

Ditanyakan: Berapakah konsentrasi dari larutan baku standar natrium diklofenak yang akan digunakan?

Jawab: ppm = $\frac{\text{mg}}{\text{L}} = \frac{10\text{ mg}}{0{,}01\text{ L}}$ = 1000 mg/L = 1000 ppm

Lampiran 9. Perhitungan Perbandingan Fase Gerak

Volume fase gerak yang akan digunakan melalui perbandingan dari toluena : etil asetat : asam asetat glasial (60:40:1 = 101 mL)

Volume yang dibutuhkan toluena: $\frac{60}{101} \times 101 = 60$ mL

Volume yang dibutuhkan etil asetat: $\frac{40}{101} \times 101 = 40$ mL

Volume yang dibutuhkan asam asetat glasial: $\frac{1}{101} \times 101 = 1$ mL

Lampiran 10. Perhitungan Konsentrasi Larutan *Spiked* Sampel

Diketahui: Berat baku standar natrium diklofenak = 2,5 mg
Volume larutan sampel uji yang diambil = 2,5 mL

Ditanyakan: Berapakah konsentrasi dari larutan *spiked* sampel yang akan digunakan?

Jawab: ppm = $\frac{\text{mg}}{\text{L}} = \frac{2{,}5\text{ mg}}{0{,}0025\text{ L}} = 1000\text{ mg/L} = 1000\text{ ppm}$

Lampiran 11. Perhitungan Nilai *Rf* Baku Standar Natrium Diklofenak

Nilai *Rf* = $\frac{\text{Jarak yang ditempuh zat}}{\text{Jarak yang ditempuh pelarut}}$

- Baku standar natrium diklofenak pada plat KLT Pertama:
  $\frac{5\ cm}{8\ cm} = 0{,}625$ = 0,63
- Baku standar natrium diklofenak pada plat KLT Kedua:
  $\frac{5{,}3\ cm}{8\ cm} = 0{,}6625$ = 0,66

Lampiran 12. Perhitungan Nilai *Rf* pada Setiap Bercak Noda

Nilai *Rf* = $\frac{\text{Jarak yang ditempuh zat}}{\text{Jarak yang ditempuh pelarut}}$

1. Sampel A
   - *Spiked* sampel A:
     Noda 1: $\frac{2{,}2\ cm}{8\ cm} = 0{,}275$ = 0,28
     Noda 2: $\frac{4\ cm}{8\ cm} = 0{,}5$
     Noda 3: $\frac{4{,}8\ cm}{8\ cm} = 0{,}6$
   - Replikasi 1:
     Noda 1: $\frac{2{,}2\ cm}{8\ cm} = 0{,}275$ = 0,28
     Noda 2: $\frac{4\ cm}{8\ cm} = 0{,}5$
   - Replikasi 2:
     Noda 1: $\frac{2{,}3\ cm}{8\ cm} = 0{,}2875$ = 0,29

Noda 2: $\frac{4\ cm}{8\ cm} = 0{,}5$

- Replikasi 3

  Noda 1: $\frac{2{,}3\ cm}{8\ cm} = 0{,}2875 = 0{,}29$

  Noda 2: $\frac{4\ cm}{8\ cm} = 0{,}5$

2. Sampel B
   - *Spiked* sampel B:

     Noda 1: $\frac{1{,}1\ cm}{8\ cm} = 0{,}1375 = 0{,}14$

     Noda 2: $\frac{2{,}2\ cm}{8\ cm} = 0{,}275 = 0{,}28$

     Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$

     Noda 4: $\frac{4{,}7\ cm}{8\ cm} = 0{,}5875 = 0{,}59$

     Noda 5: $\frac{7{,}5\ cm}{8\ cm} = 0{,}9375 = 0{,}94$
   - Replikasi 1:

     Noda 1: $\frac{1{,}2\ cm}{8\ cm} = 0{,}15$

     Noda 2: $\frac{2{,}2\ cm}{8\ cm} = 0{,}275 = 0{,}28$

     Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$

     Noda 4: $\frac{7{,}5\ cm}{8\ cm} = 0{,}9375 = 0{,}94$
   - Replikasi 2:

     Noda 1: $\frac{1{,}1\ cm}{8\ cm} = 0{,}1375 = 0{,}14$

     Noda 2: $\frac{2{,}2\ cm}{8\ cm} = 0{,}275 = 0{,}28$

     Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$

     Noda 4: $\frac{7{,}5\ cm}{8\ cm} = 0{,}9375 = 0{,}94$
   - Replikasi 3:

     Noda 1: $\frac{1{,}1\ cm}{8\ cm} = 0{,}1375 = 0{,}14$

     Noda 2: $\frac{2{,}2\ cm}{8\ cm} = 0{,}275 = 0{,}28$

     Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$

     Noda 4: $\frac{7{,}5\ cm}{8\ cm} = 0{,}9375 = 0{,}94$
3. Sampel C
   - *Spiked* sampel C:

     Noda 1: $\frac{1{,}1\ cm}{8\ cm} = 0{,}1375 = 0{,}14$

     Noda 2: $\frac{2{,}2\ cm}{8\ cm} = 0{,}275 = 0{,}28$

     Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$

     Noda 4: $\frac{4{,}7\ cm}{8\ cm} = 0{,}5875 = 0{,}59$

     Noda 5: $\frac{7{,}4\ cm}{8\ cm} = 0{,}925 = 0{,}93$
   - Replikasi 1:

     Noda 1: $\frac{1\ cm}{8\ cm} = 0{,}125 = 0{,}13$

Noda 2: $\frac{2,1\ cm}{8\ cm} = 0,2625 = 0,26$
Noda 3: $\frac{4,1\ cm}{8\ cm} = 0,5125 = 0,51$
Noda 4: $\frac{4,7\ cm}{8\ cm} = 0,5875 = 0,59$
Noda 5: $\frac{7,4\ cm}{8\ cm} = 0,925 = 0,93$

- Replikasi 2:
  Noda 1: $\frac{1\ cm}{8\ cm} = 0,125 = 0,13$
  Noda 2: $\frac{2,2\ cm}{8\ cm} = 0,275 = 0,28$
  Noda 3: $\frac{4,1\ cm}{8\ cm} = 0,5125 = 0,51$
  Noda 4: $\frac{4,8\ cm}{8\ cm} = 0,6$
  Noda 5: $\frac{7,4\ cm}{8\ cm} = 0,925 = 0,93$
- Replikasi 3:
  Noda 1: $\frac{1\ cm}{8\ cm} = 0,125 = 0,13$
  Noda 2: $\frac{2,2\ cm}{8\ cm} = 0,275 = 0,28$
  Noda 3: $\frac{4,1\ cm}{8\ cm} = 0,5125 = 0,51$
  Noda 4: $\frac{4,8\ cm}{8\ cm} = 0,6$
  Noda 5: $\frac{7,4\ cm}{8\ cm} = 0,925 = 0,93$

4. Sampel D
   - *Spiked* sampel D:
     Noda 1: $\frac{1,1\ cm}{8\ cm} = 0,1375 = 0,14$
     Noda 2: $\frac{2,1\ cm}{8\ cm} = 0,2625 = 0,26$
     Noda 3: $\frac{4,2\ cm}{8\ cm} = 0,525 = 0,53$
     Noda 4: $\frac{4,8\ cm}{8\ cm} = 0,6$
     Noda 5: $\frac{7,4\ cm}{8\ cm} = 0,925 = 0,93$
   - Replikasi 1:
     Noda 1: $\frac{1,1\ cm}{8\ cm} = 0,1375 = 0,14$
     Noda 2: $\frac{2,1\ cm}{8\ cm} = 0,2625 = 0,26$
     Noda 3: $\frac{4,2\ cm}{8\ cm} = 0,525 = 0,53$
     Noda 4: $\frac{4,8\ cm}{8\ cm} = 0,6$
     Noda 5: $\frac{7,5\ cm}{8\ cm} = 0,9375 = 0,94$
   - Replikasi 2:
     Noda 1: $\frac{1,1\ cm}{8\ cm} = 0,1375 = 0,14$
     Noda 2: $\frac{1,1\ cm}{8\ cm} = 0,275 = 0,28$
     Noda 3: $\frac{4,2\ cm}{8\ cm} = 0,525 = 0,53$
     Noda 4: $\frac{4,8\ cm}{8\ cm} = 0,6$
     Noda 5: $\frac{7,5\ cm}{8\ cm} = 0,9375 = 0,94$

- Replikasi 3:
  Noda 1: $\frac{1\ cm}{8\ cm} = 0{,}125 = 0{,}13$
  Noda 2: $\frac{2{,}2\ cm}{8\ cm} = 0{,}275 = 0{,}28$
  Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$
  Noda 4: $\frac{4{,}8\ cm}{8\ cm} = 0{,}6$
  Noda 5: $\frac{7{,}5\ cm}{8\ cm} = 0{,}9375 = 0{,}94$

5. Sampel E
   - *Spiked* sampel E:
     Noda 1: $\frac{2{,}4\ cm}{8\ cm} = 0{,}3$
     Noda 2: $\frac{4{,}2\ cm}{8\ cm} = 0{,}525 = 0{,}53$
     Noda 3: $\frac{5\ cm}{8\ cm} = 0{,}625 = 0{,}63$
     Noda 4: $\frac{7{,}7\ cm}{8\ cm} = 0{,}9625 = 0{,}96$
   - Replikasi 1:
     Noda 1: $\frac{2{,}4\ cm}{8\ cm} = 0{,}3$
     Noda 2: $\frac{4{,}2\ cm}{8\ cm} = 0{,}525 = 0{,}53$
     Noda 3: $\frac{7{,}6\ cm}{8\ cm} = 0{,}95$
   - Replikasi 2:
     Noda 1: $\frac{2{,}3\ cm}{8\ cm} = 0{,}2875 = 0{,}29$
     Noda 2: $\frac{4{,}2\ cm}{8\ cm} = 0{,}525 = 0{,}53$
     Noda 3: $\frac{7{,}6\ cm}{8\ cm} = 0{,}95$
   - Replikasi 3:
     Noda 1: $\frac{2{,}3\ cm}{8\ cm} = 0{,}2875 = 0{,}29$
     Noda 2: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$
     Noda 3: $\frac{7{,}6\ cm}{8\ cm} = 0{,}95$

6. Sampel F
   - *Spiked* sampel F:
     Noda 1: $\frac{1{,}1\ cm}{8\ cm} = 0{,}1375 = 0{,}14$
     Noda 2: $\frac{2{,}3\ cm}{8\ cm} = 0{,}2875 = 0{,}29$
     Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$
     Noda 4: $\frac{4{,}8\ cm}{8\ cm} = 0{,}6$
     Noda 5: $\frac{6{,}9\ cm}{8\ cm} = 0{,}8625 = 0{,}86$
     Noda 6: $\frac{7{,}6\ cm}{8\ cm} = 0{,}95$
   - Replikasi 1:
     Noda 1: $\frac{1{,}1\ cm}{8\ cm} = 0{,}1375 = 0{,}14$
     Noda 2: $\frac{2{,}3\ cm}{8\ cm} = 0{,}2875 = 0{,}29$

Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$

Noda 4: $\frac{6{,}9\ cm}{8\ cm} = 0{,}8625 = 0{,}86$

Noda 5: $\frac{7{,}5\ cm}{8\ cm} = 0{,}9375 = 0{,}94$

- Replikasi 2:

  Noda 1: $\frac{1{,}1\ cm}{8\ cm} = 0{,}1375 = 0{,}14$

  Noda 2: $\frac{2{,}3\ cm}{8\ cm} = 0{,}2875 = 0{,}29$

  Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$

  Noda 4: $\frac{6{,}9\ cm}{8\ cm} = 0{,}8625 = 0{,}86$

  Noda 5: $\frac{7{,}5\ cm}{8\ cm} = 0{,}9375\ 0{,}94$

- Replikasi 3:

  Noda 1: $\frac{1{,}1\ cm}{8\ cm} = 0{,}1375 = 0{,}14$

  Noda 2: $\frac{2{,}3\ cm}{8\ cm} = 0{,}2875 = 0{,}29$

  Noda 3: $\frac{4{,}1\ cm}{8\ cm} = 0{,}5125 = 0{,}51$

  Noda 4: $\frac{6{,}9\ cm}{8\ cm} = 0{,}8625 = 0{,}86$

  Noda 5: $\frac{7{,}4\ cm}{8\ cm} = 0{,}925 = 0{,}93$

Lampiran 13. Perhitungan Rata-Rata Nilai *Rf* pada Sampel

$$\text{Rata} - \text{rata nilai Rf} = \frac{\text{Penjumlahan nilai Rf dari replikasi}}{\text{Banyaknya replikasi}}$$

1. Sampel A

   Nilai *Rf* pada noda 2 replikasi 1, replikasi 2, dan replikasi 3:

   $$\frac{0{,}5+0{,}5+0{,}5}{3} = 0{,}5$$

2. Sampel B

   Nilai *Rf* pada noda 3 replikasi 1, replikasi 2, dan replikasi 3:

   $$\frac{0{,}51+0{,}51+0{,}51}{3} = 0{,}5125 = 0{,}51$$

3. Sampel C

   Nilai *Rf* pada noda 4 replikasi 1, replikasi 2, dan replikasi 3:

   $$\frac{0{,}59+0{,}6+0{,}6}{3} = 0{,}5966666667 = 0{,}60$$

4. Sampel D

   Nilai *Rf* pada noda 4 replikasi 1, replikasi 2, dan replikasi 3:

   $$\frac{0{,}6+0{,}6+0{,}6}{3} = 0{,}6$$

5. Sampel E

   Nilai *Rf* pada noda 2 replikasi 1, replikasi 2, dan replikasi 3:

   $$\frac{0{,}53+0{,}53+0{,}51}{3} = 0{,}5266666667 = 0{,}53$$

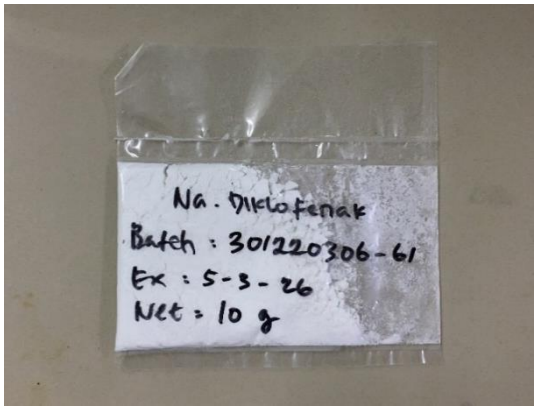
6. Sampel F

   Nilai *Rf* pada noda 3 replikasi 1, replikasi 2, dan replikasi 3:

   $$\frac{0{,}51+0{,}51+0{,}51}{3} = 0{,}5125 = 0{,}51$$

Lampiran 14. Dokumentasi Penelitian

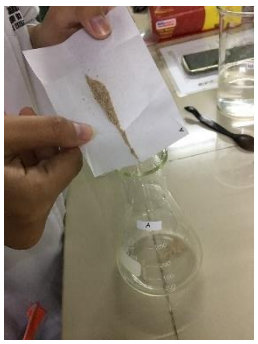
Sampel jamu pegal linu



Baku standar natrium diklofenak

Penimbangan 500 mg tiap sampel sebagai larutan uji sampel

Penimbangan 10 mg baku standar natrium diklofenak sebagai larutan baku standar

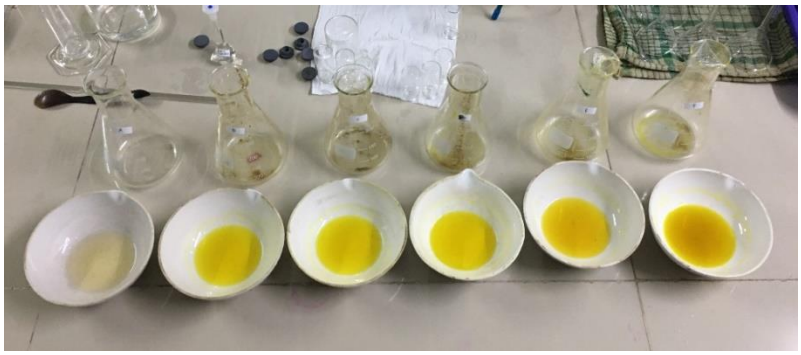


Penuangan masing-masing sampel ke dalam erlenmeyer

Penambahan 25 mL metanol ke dalam masing-masing sampel

Hasil dari penambahan 25 mL metanol ke dalam tiap sampel jamu pegal linu

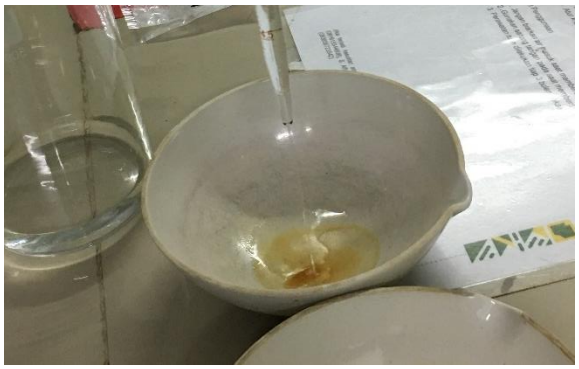
Penyaringan masing-masing sampel jamu pegal linu



Hasil dari penyaringan tiap sampel jamu pegal linu

Penguapan sampel jamu pegal linu di atas *waterbath*

Hasil penguapan sampel jamu pegal linu dengan *waterbath*

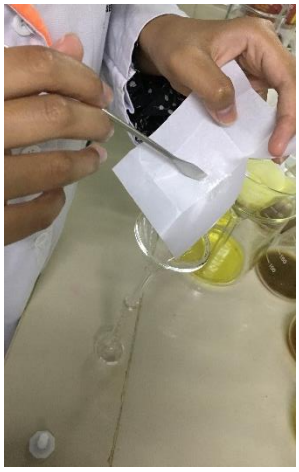


Penambahan 5 mL metanol dalam sampel jamu pegal linu setelah penguapan

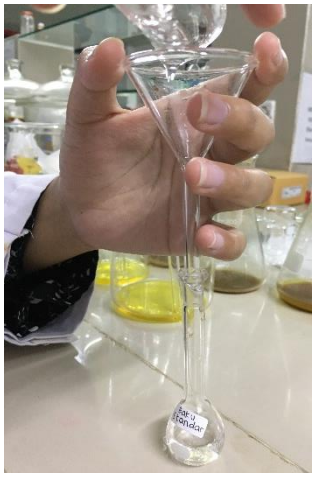
Proses menghomogenkan sampel jamu pegal linu setelah ditambahkan dengan 5 mL metanol

Penuangan sampel jamu pegal linu ke dalam botol vial

Hasil larutan sampel uji

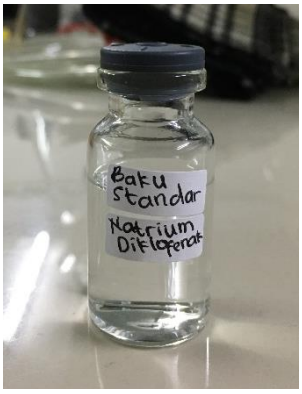


Penuangan 10 mg baku standar natrium diklofenak ke dalam labu ukur 10 mL



Penambahan 10 mL metanol ke dalam labu ukur 10 mL

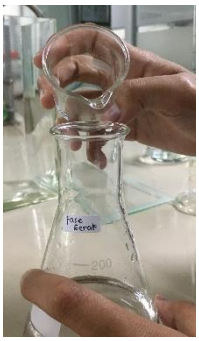
Penuangan larutan baku standar natrium diklofenak ke dalam botol vial



Larutan baku standar natrium diklofenak 1000 ppm

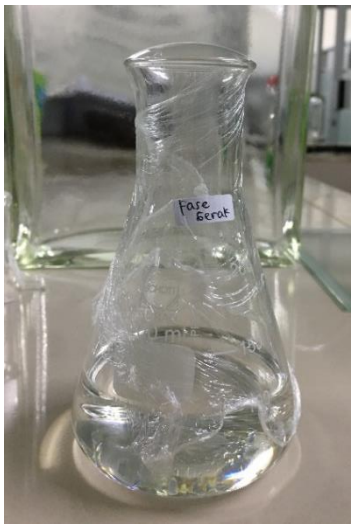
Toluena, etil asetat, dan asam asetat glasial





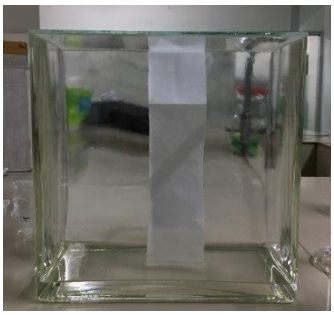
Penuangan 60 mL toluena, 40 mL etil asetat, dan 1 mL asam asetat glasial ke dalam erlenmeyer sebagai fase gerak

Proses menghomogenkan fase gerak



Hasil fase gerak

Menuangkan fase gerak ke dalam *chamber*



Proses penjenuhan *chamber* fase gerak dengan kertas saring

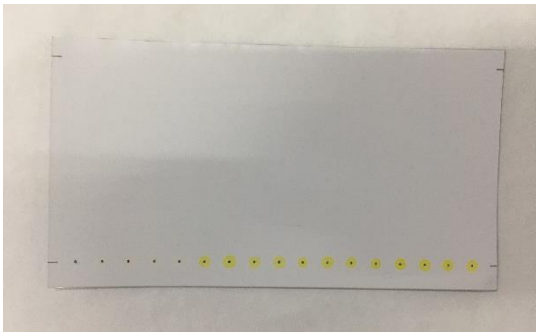
Penimbangan 2,5 mg baku standar natrium diklofenak untuk larutan *spiked* sampel

Mengambil 2,5 mL larutan sampel uji untuk digunakan sebagai larutan *spiked* sampel

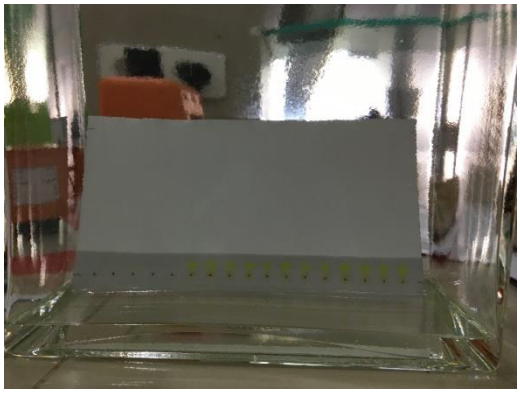
Penambahan 2,5 mg baku standar natrium diklofenak ke dalam larutan yang akan menjadi larutan *spiked* sampel

Hasil larutan *spiked* sampel

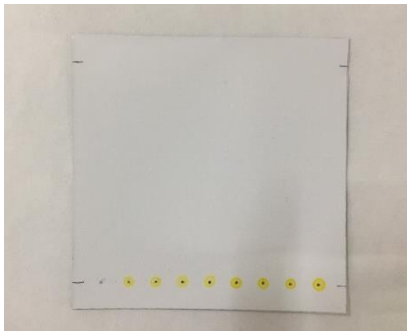
Larutan sampel uji



Penotolan pada plat KLT pertama

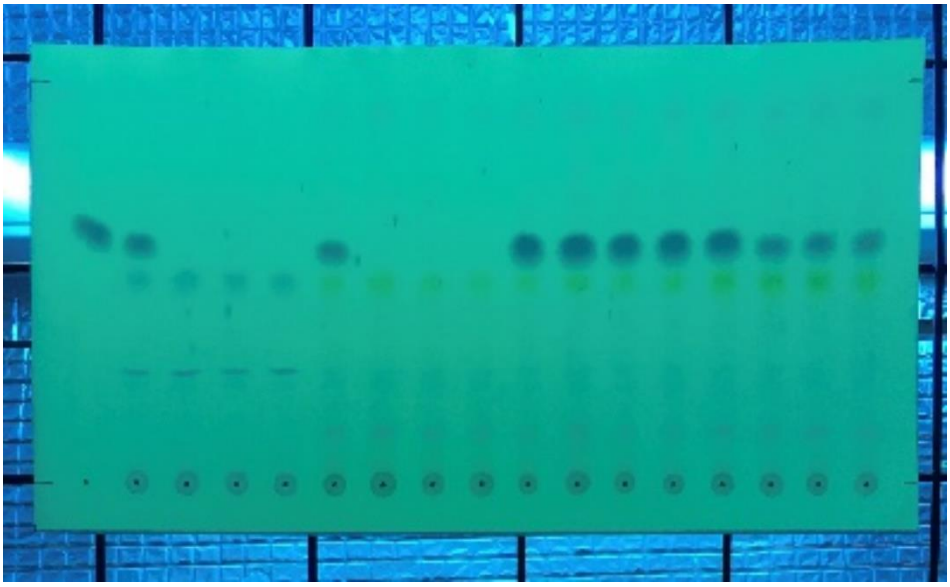


Proses elusi plat KLT pertama

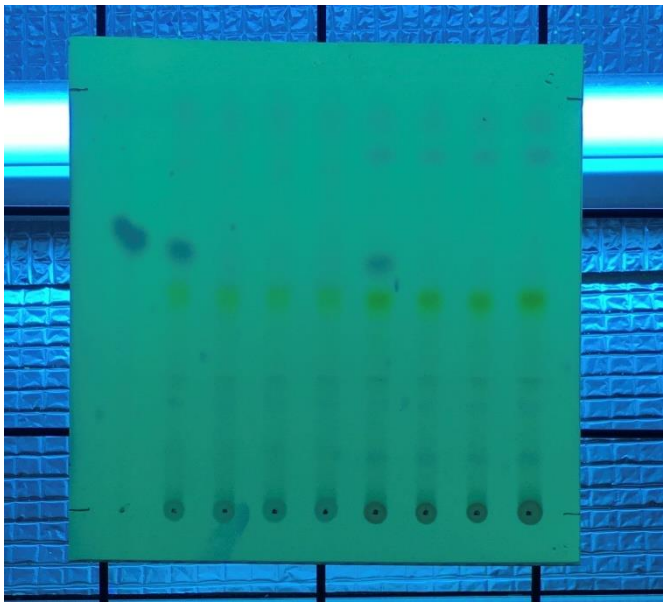


Penotolan pada plat KLT kedua

Proses elusi plat KLT kedua



Hasil pada plat KLT pertama



Hasil pada plat KLT kedua

Lampiran 15. ***Certificate Of Analysis*** **(Natrium Diklofenak)**

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HENAN DONGTAI PHARM CO.,LTD.

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CERTIFICATE OF ANALYSIS

Product: Diclofenac Sodium (bromine free)		According to: USP2021
Batch No.: 301201122-51	Mfg. Date:Nov.22,2020	Retest Date: Nov.21,2024
Quantity:1000kg	Packing: 25kg/fibre drum	Release Date: Jul.27,2021

ITEMS	SPECIFICATION	RESULTS
Characters	White to off-white crystalline powder,	White crystalline powder
Melting point	About 284℃ ,with decomposition	Conform
Identification	A.IR	Conform
	B.HPLC	Conform
	C. Test of sodium salt	Conform
Appearance of solution	5.0% of methanol solution	Clear
	440nm, NMT 0.05	0.009
Clarity of solution	Equal to methanol	Conform
PH	7.0~8.5	7.34
Related substances	Diclofenac impurity A:NMT0.2%	0.005%
	Any individual impurity:NMT0.2%	0.057%
	Total impurities:NMT0.5%	0.083%
Loss on drying	NMT0.5% at 105℃, 3	0.12%
Assay	99.0%~101.0%	99.7%
Conclusion: It accords with USP2021 Be up to the Standard.		
Reported by:Liu Wenhua		Approved by: Cheng Guojun