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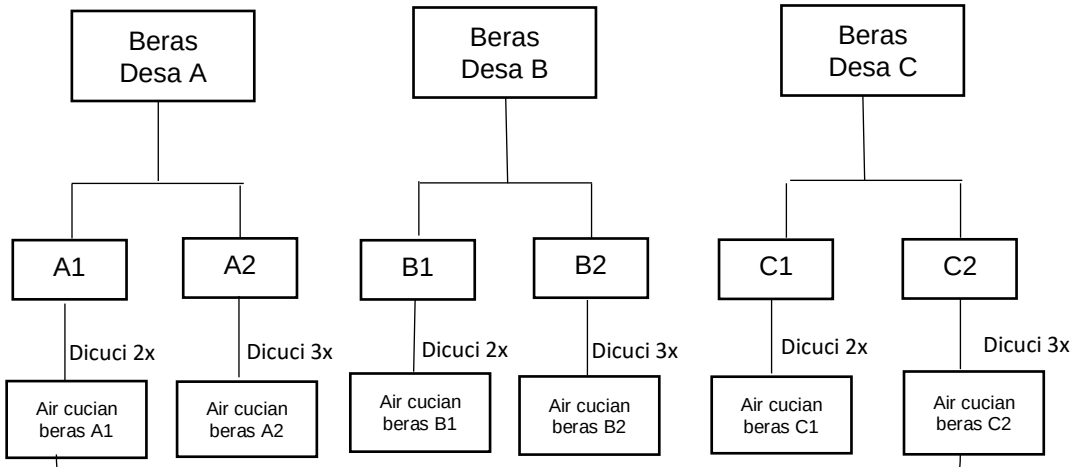
Lampiran 1

Skema kerja penyiapan larutan uji

a. Uji pestisida pada air cucian beras dengan menggunakan metode GC/MS



- Sampel air cucian beras sebanyak 10 mL tambahkan 15 ml asetonitril, kocok kuat selama 1 menit dan di inkubasi pada suhu -18°C selama 15 menit
- Tambahkan 6 g $MgSO_4$ dan 1 g natrium asetat anhidrat, kocok dengan tangan selama 1 menit kemudian sentrifugasi selama 5 menit

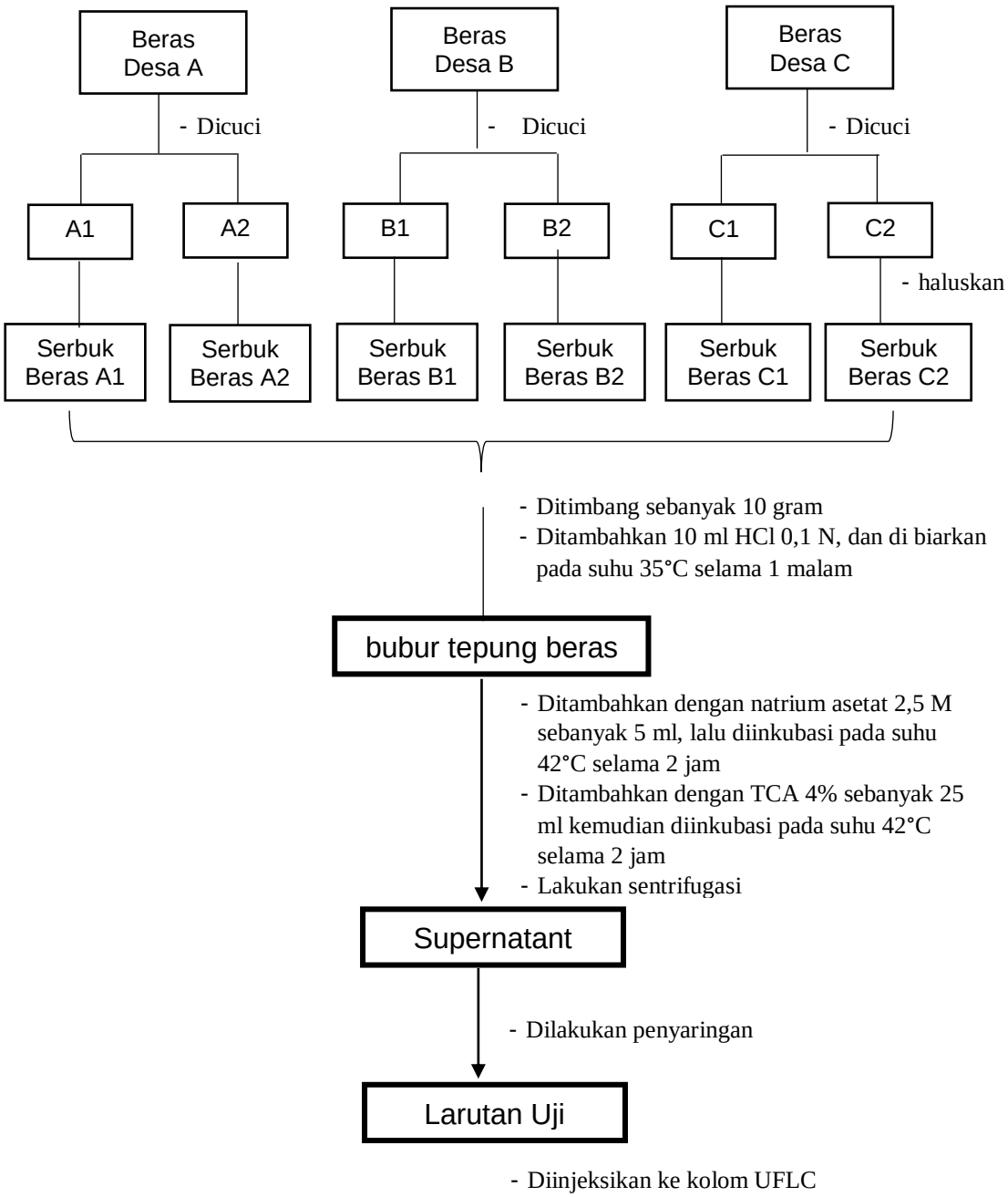
Fase organik

- Ambil 8 mL supernatan, masukkan ke dalam tabung sentrifuge yang berisi 150 mg $MgSO_4$, 50 mg PSA dan 50 mg C_{18}, kocok selama 1-2 menit
- Sentrifuge kembali selama 2 menit dengan kecepatan 4400 rpm

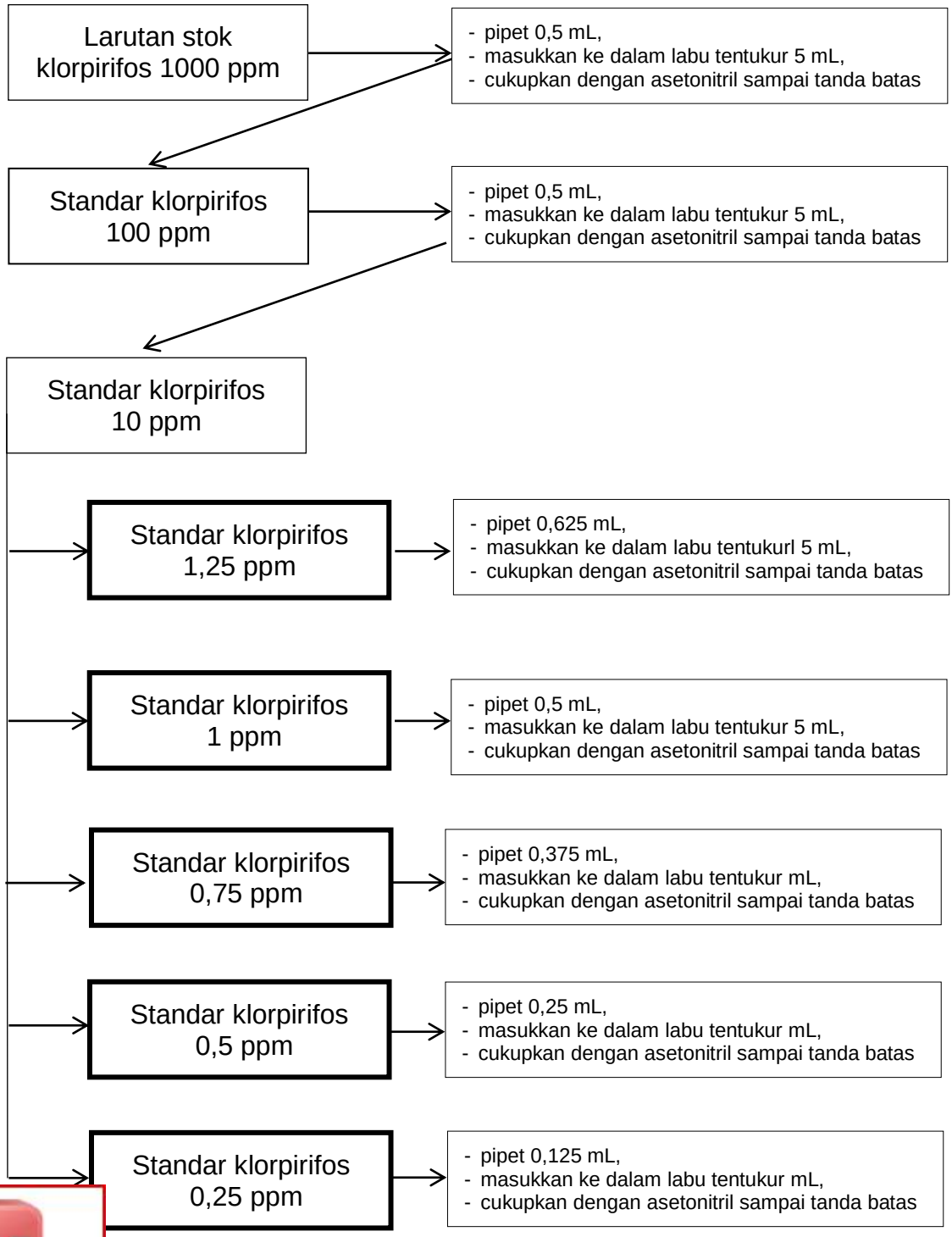
Larutan uji

- Ambil 1,5 mL supernatan , masukkan ke dalam vial untuk dianalisis menggunakan GC/MS

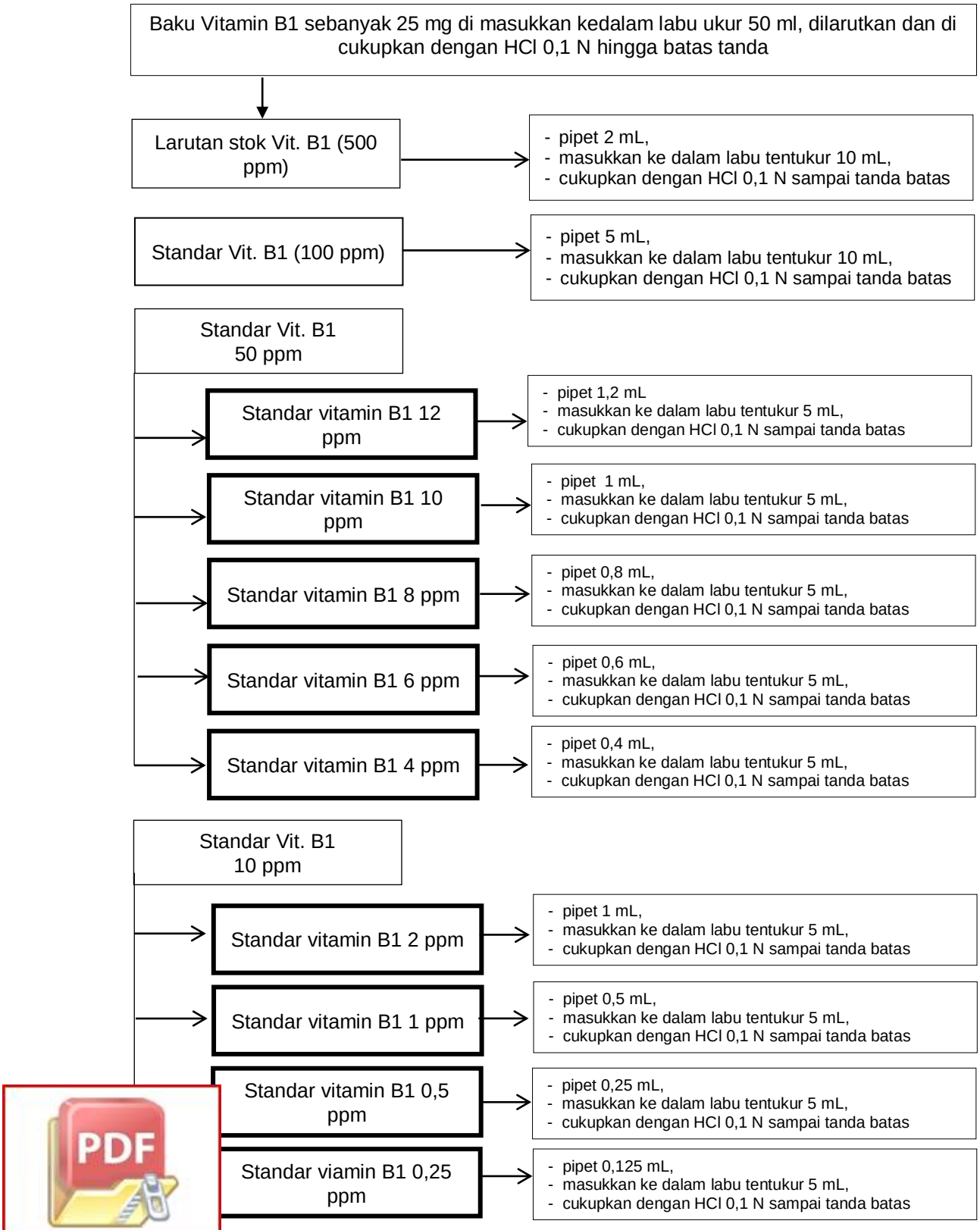
b. Vitamin B1 pada beras dengan menggunakan metode UFLC



Pembuatan larutan standar Klorpirifos



Pembuatan larutan standar Vitamin B1



Lampiran 2

Perhitungan pengenceran larutan standar

a. Standar Klorpirifos

Larutan stok klorpirifos 1000 ppm

- Untuk 100 ppm = $\frac{100\ mg/L \times 5\ mL}{1000\ mg/L}$ = 0,5 mL
- Untuk 10 ppm = $\frac{10\ mg/L \times 5\ mL}{100\ mg/L}$ = 0,5 mL
- Untuk 1,25 ppm = $\frac{1{,}25\ mg/L \times 5\ mL}{10\ mg/L}$ = 0,625 mL
- Untuk 1 ppm = $\frac{1\ mg \times 5\ mL}{10\ mg/L}$ = 0,5 mL
- Untuk 0,75 ppm = $\frac{0.75\ mg\ x\ 5\ mL}{10\ mg/L}$ = 0,375 mL
- Untuk 0,5 ppm = $\frac{0.5\ mg \times 5\ mL}{10\ mg/L}$ = 0,25 mL
- Untuk 0,25 ppm = $\frac{0.25\ mg\ x\ 5\ mL}{10\ mg/L}$ = 0,125 mL

b. Standar Vitamin B1

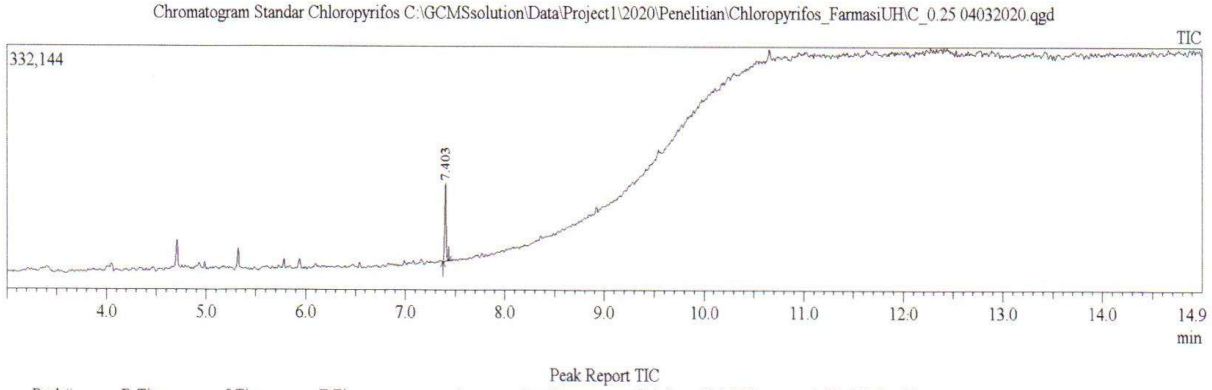
Larutan stok vitamin B1 500 ppm

- Untuk 100 ppm = $\frac{100\ mg/L \times 10\ mL}{500\ mg/L}$ = 2 mL
- Untuk 50 ppm = $\frac{50\ mg/L \times 10\ mL}{100\ mg/L}$ = 5 mL
- Untuk 12 ppm = $\frac{12\ mg/L \times 5\ mL}{50\ mg/L}$ = 0,5 mL
- Untuk 10 ppm = $\frac{10\ mg/L \times 5\ mL}{50\ mg/L}$ = 1 mL
- Untuk 8 ppm = $\frac{8\ mg/L \times 5\ mL}{50\ mg/L}$ = 0,8 mL
- Untuk 6 ppm = $\frac{6\ mg/L \times 5\ mL}{50\ mg/L}$ =0,6 mL
- Untuk 4 ppm = $\frac{4\ mg/L \times 5\ mL}{50\ mg/L}$ = 0,4 mL
- Untuk 2 ppm = $\frac{2\ mg/L \times 5\ mL}{10\ mg/L}$ = 1 mL
- Untuk 1 ppm = $\frac{1\ mg/L \times 5\ mL}{10\ mg/L}$ = 0,5 mL
- Untuk 0,5 ppm = $\frac{0{,}5\ mg/L \times 5\ mL}{10\ mg/L}$ = 0,25 mL
- Untuk 0,25 ppm = $\frac{0{,}25\ mg/L \times 5\ mL}{50\ mg/L}$ = 0,125 mL

Lampiran 3

Kromatogram Standar Klorpirifos dan Sampel

1. Deret Standar 0.25 ppm

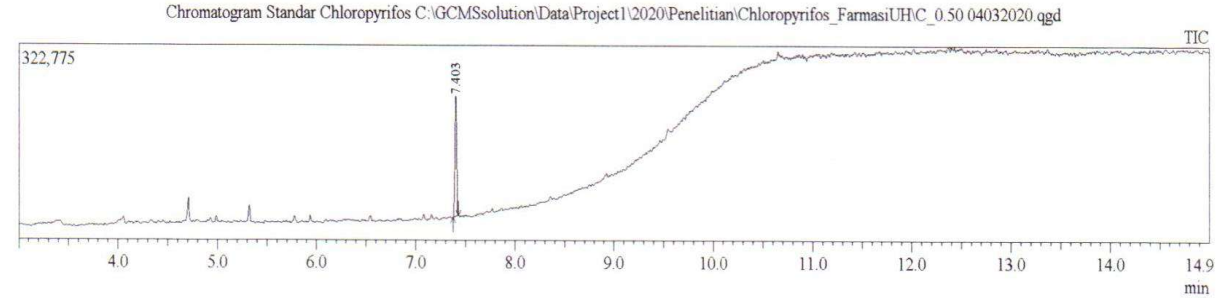


Peak Report TIC

Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	7.403	7.380	7.435	116902	100.00	107543	100.00	1.09		Chlorpyrifos
				116902	100.00	107543	100.00			

Gambar 7. Kromatogram standar klorpirifos 0,25 ppm

2. Deret Standar 0.50 ppm

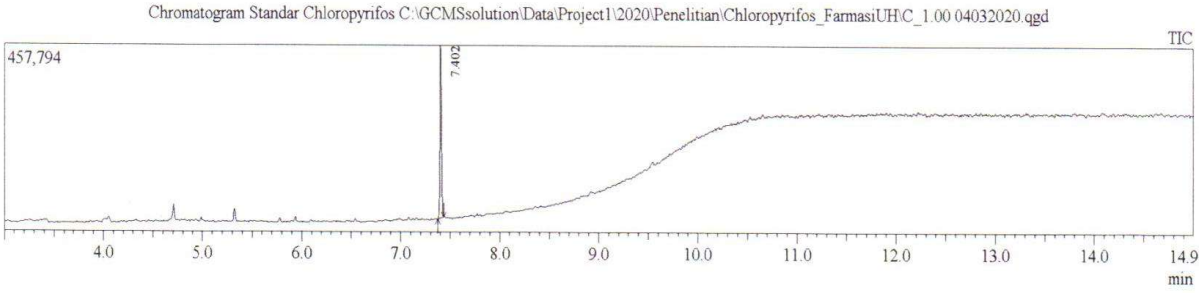


Peak Report TIC

Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	7.403	7.375	7.430	217371	100.00	199117	100.00	1.09		Chlorpyrifos
				217371	100.00	199117	100.00			

Gambar 8. Kromatogram standar klorpirifos 0,5 ppm

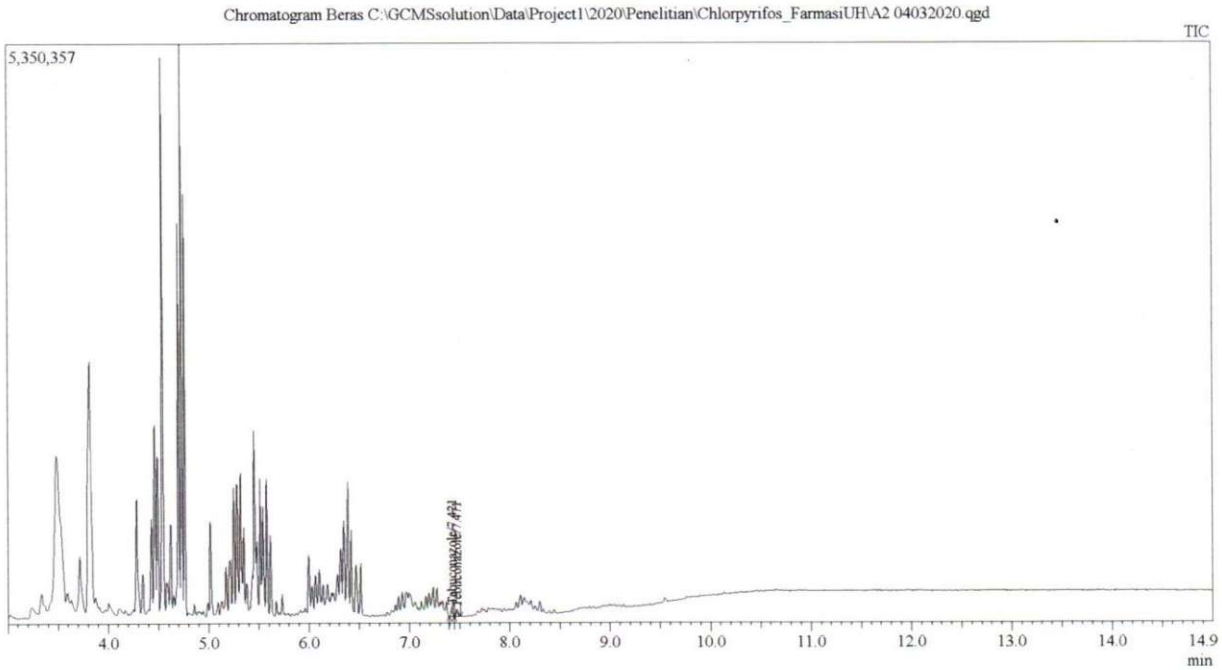
4. Deret Standar 1.00



Peak Report TIC

Peak#	R.Time	I.Time	F.Time	Area	Area%	Height	Height%	A/H	Mark	Name
1	7.402	7.375	7.430	458979	100.00	422310	100.00	1.09		Chlorpyrifos
				458979	100.00	422310	100.00			

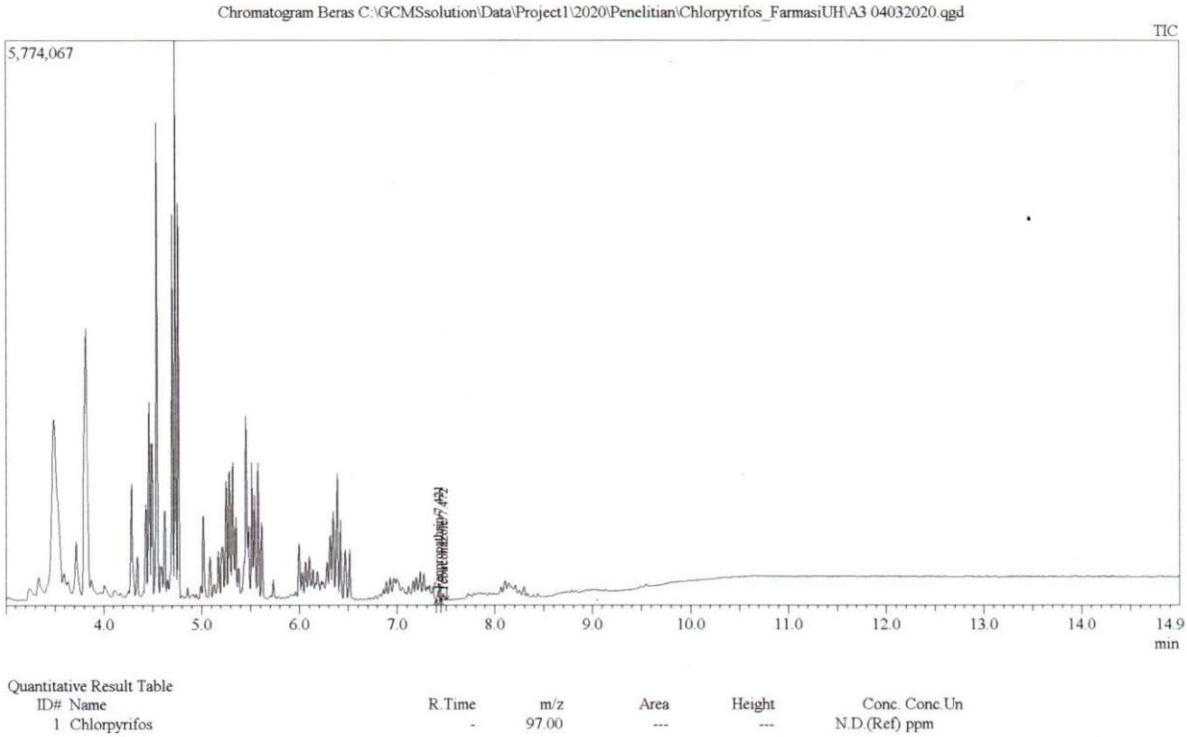
Gambar 9. Kromatogram standar klorpirifos 1 ppm



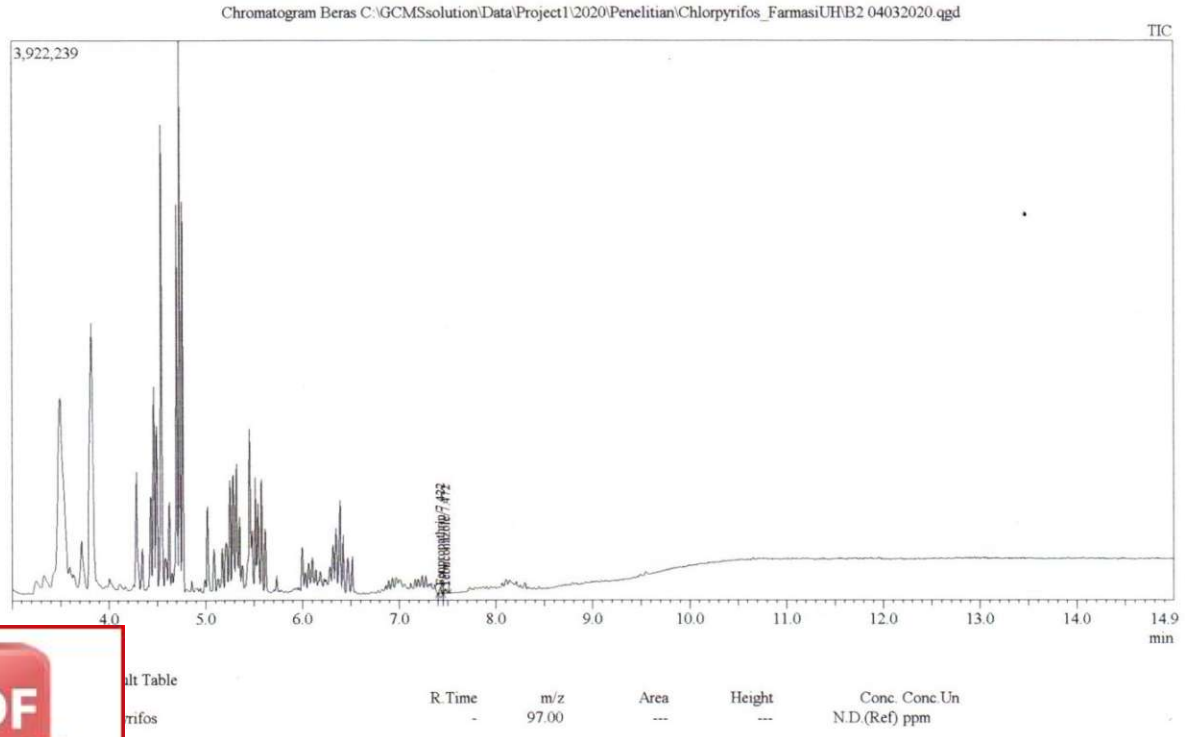
Quantitative Result Table

ID#	Name	R.Time	m/z	Area	Height	Conc.	Conc.Un
1	Chlorpyrifos	-	97.00	---	---	N.D.	(Ref) ppm

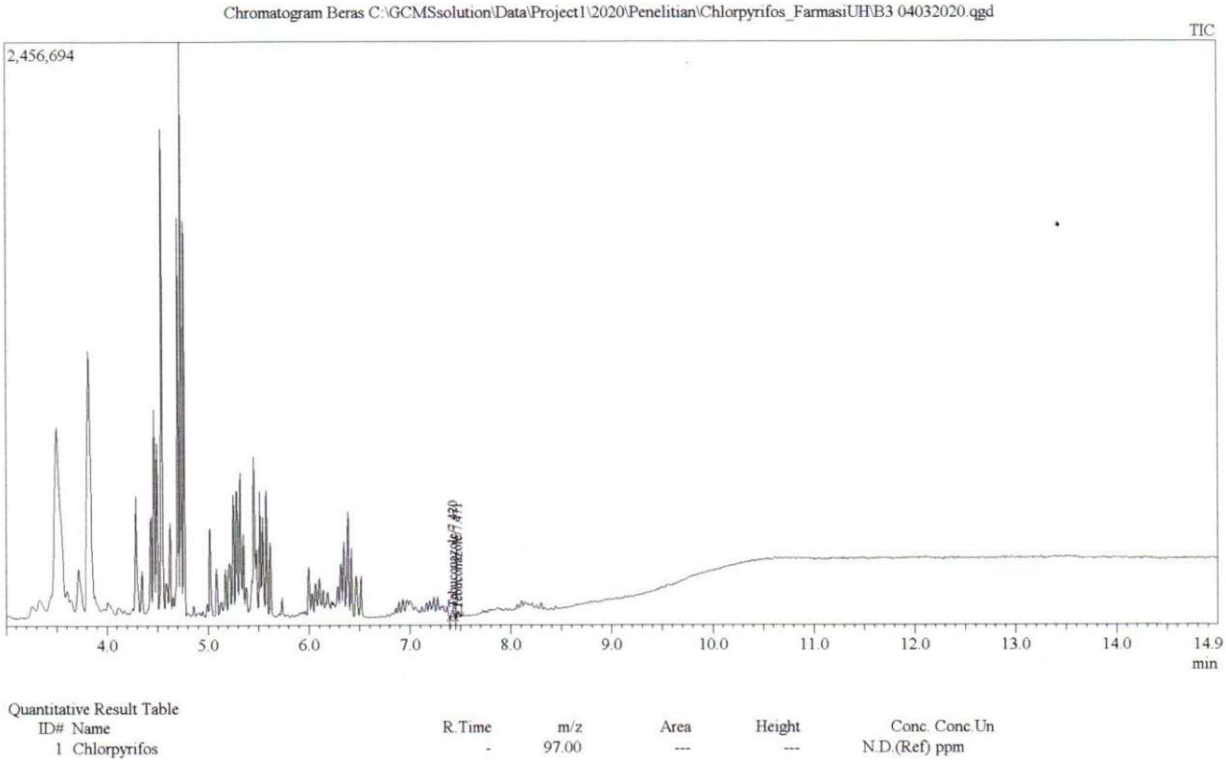
Gambar 10. Kromatogram sampel A pencucian 2x



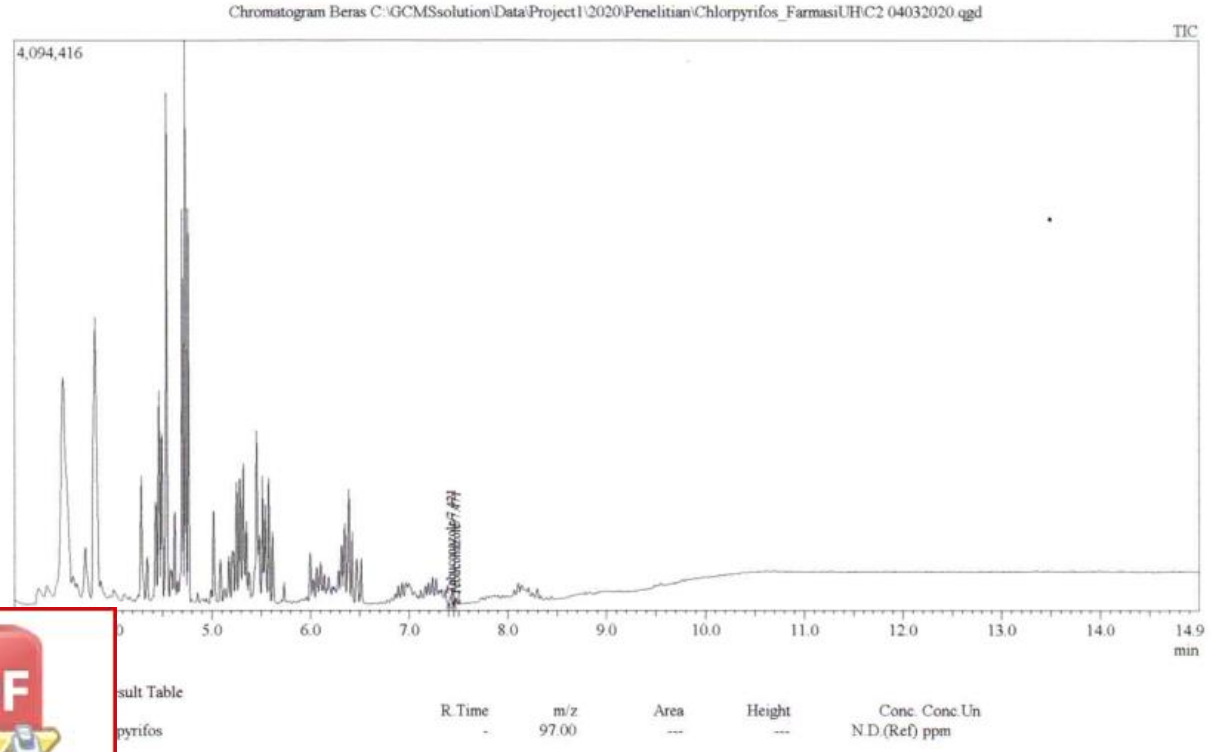
Gambar 11. Kromatogram sampel A pencucian 3x



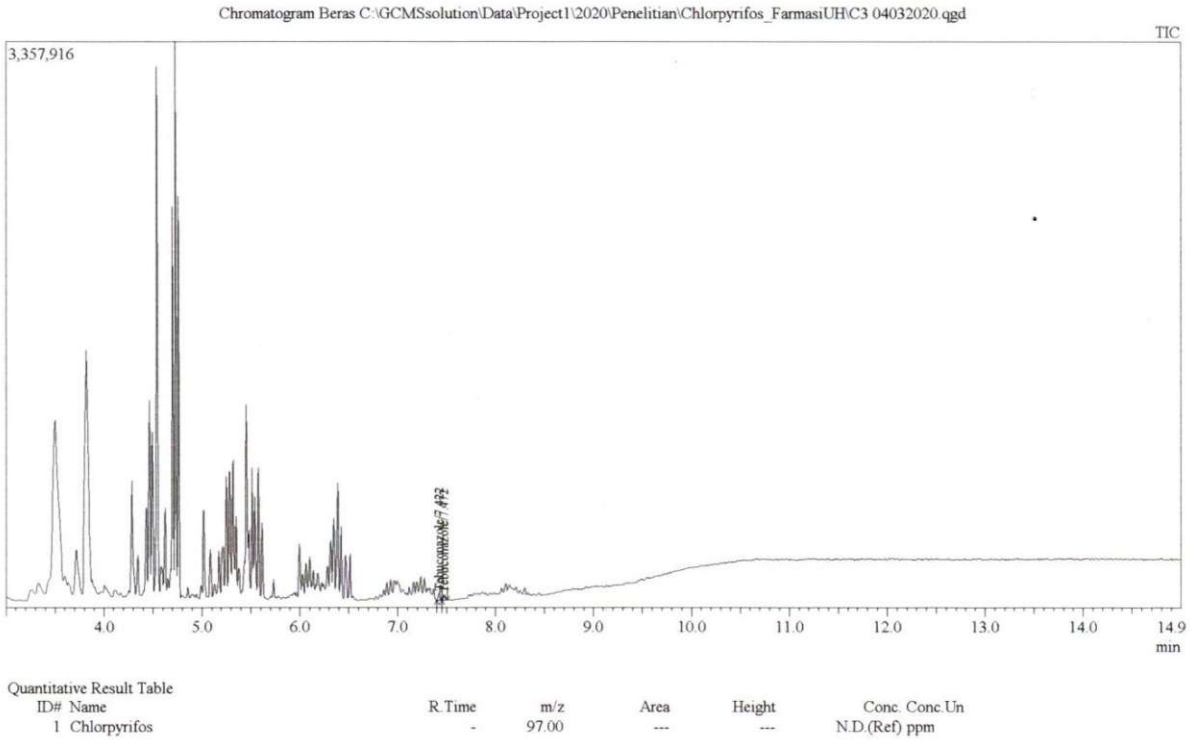
Gambar 12. Kromatogram sampel B pencucian 2x



Gambar 13. Kromatogram sampel B pencucian 3x



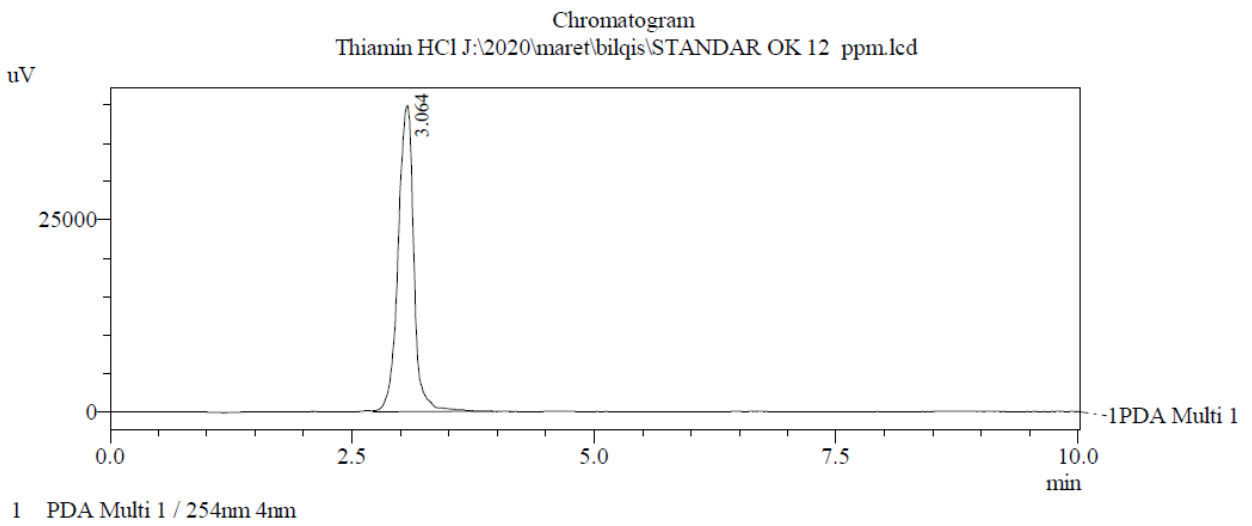
Gambar 14. Kromatogram sampel C pencucian 2x



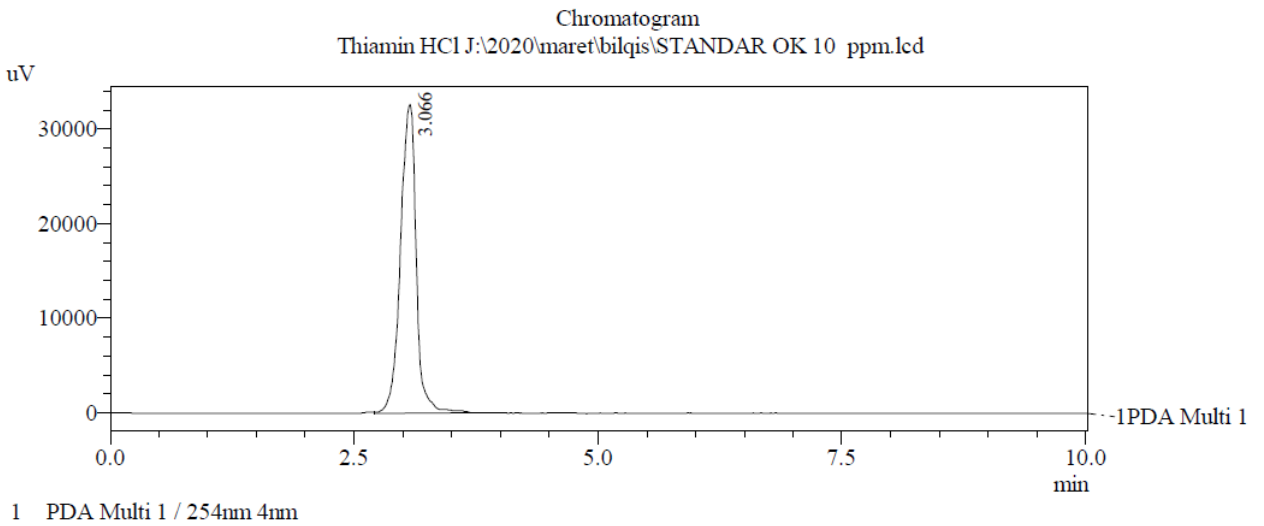
Gambar 15. Kromatogram sampel C pencucian 3x

Lampiran 4

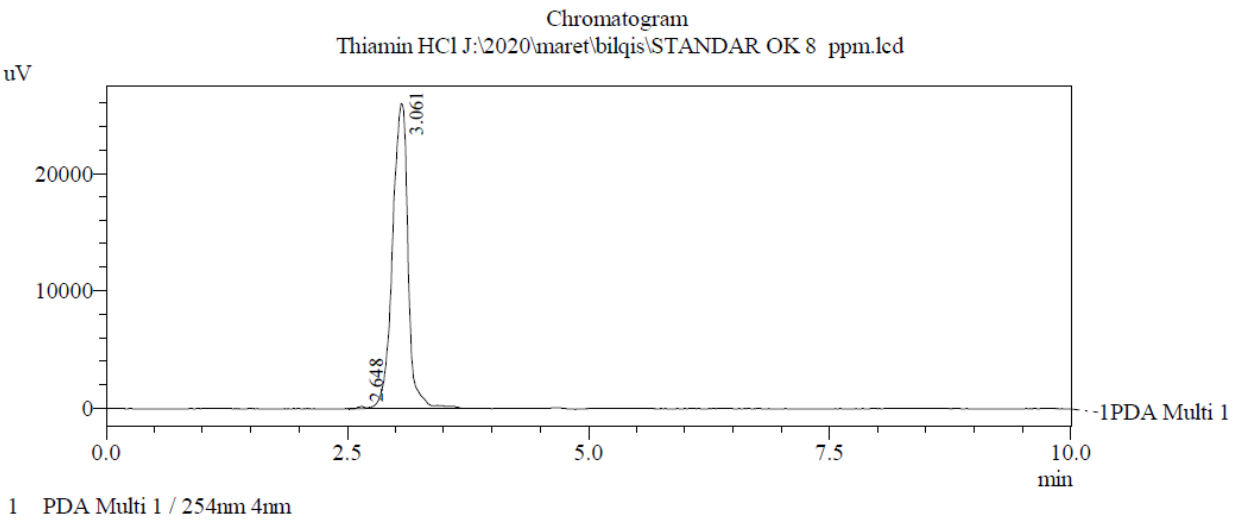
Kromatogram Standar Vitamin B1 (Thiamin HCl)



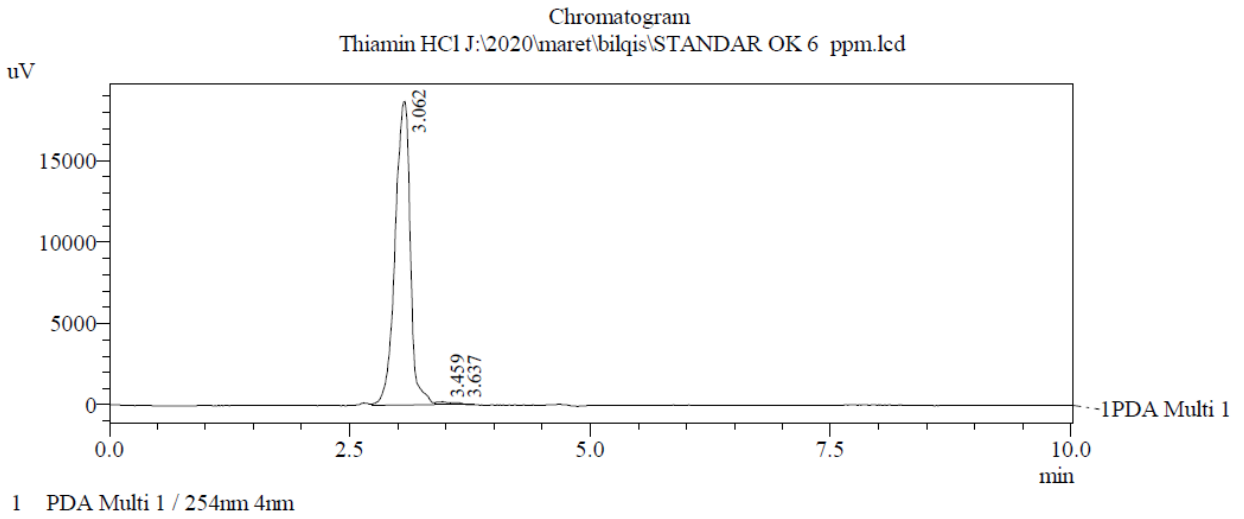
Gambar 16. Kromatogram baku vitamin B1 12 ppm



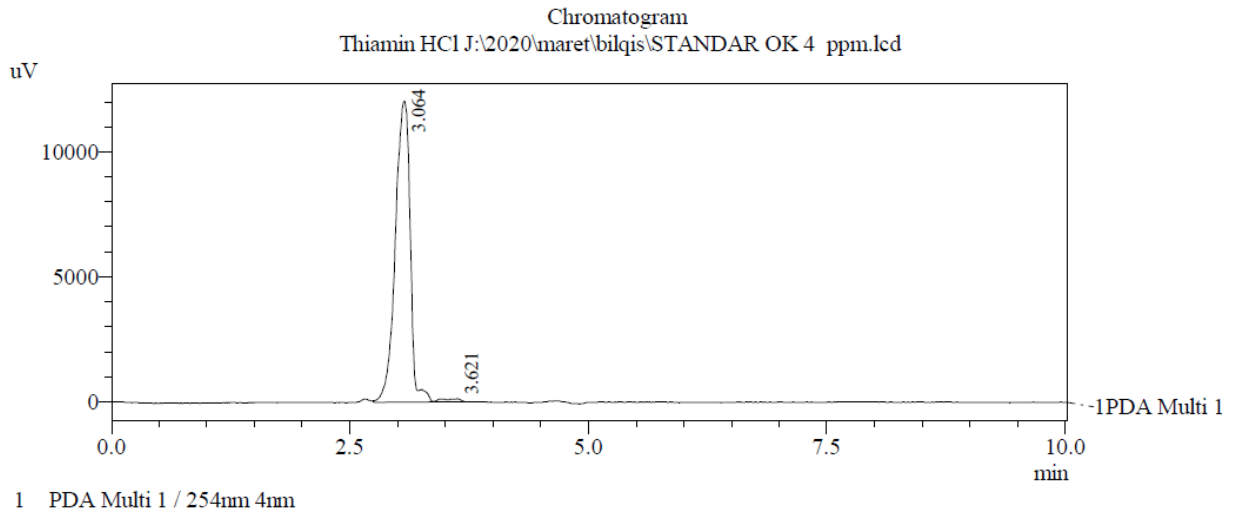
Gambar 17. Kromatogram baku vitamin B1 10 ppm



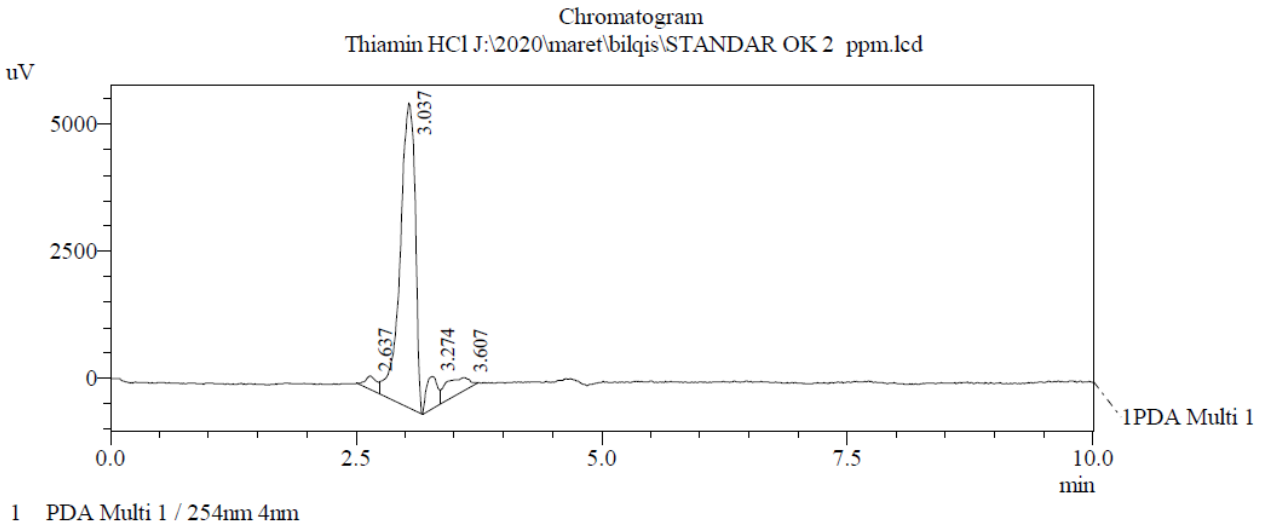
Gambar 17. Kromatogram baku vitamin B1 8 ppm



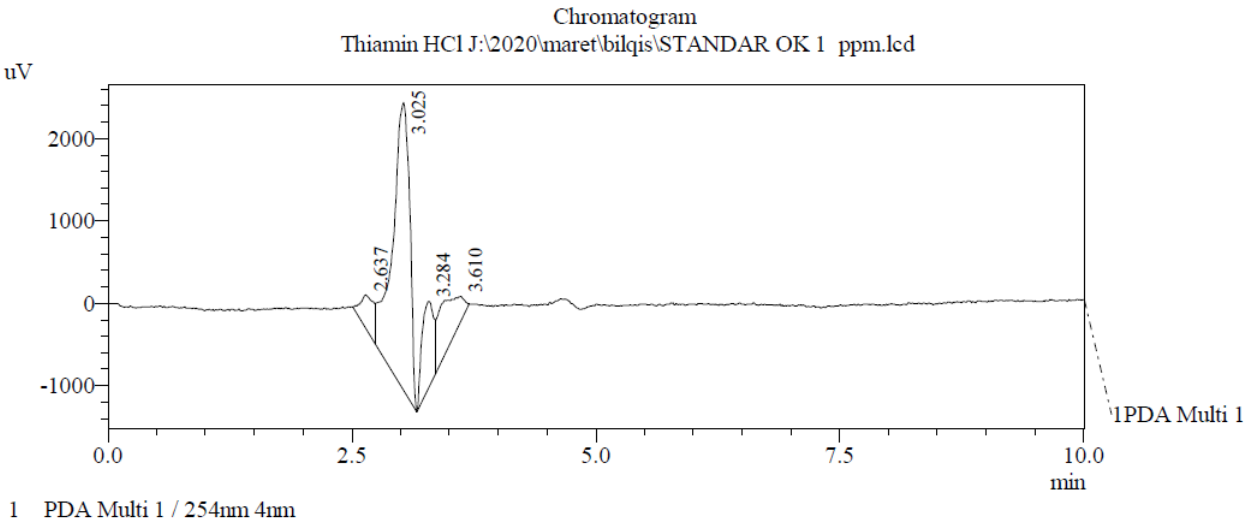
Gambar 18. Kromatogram baku vitamin B1 6 ppm



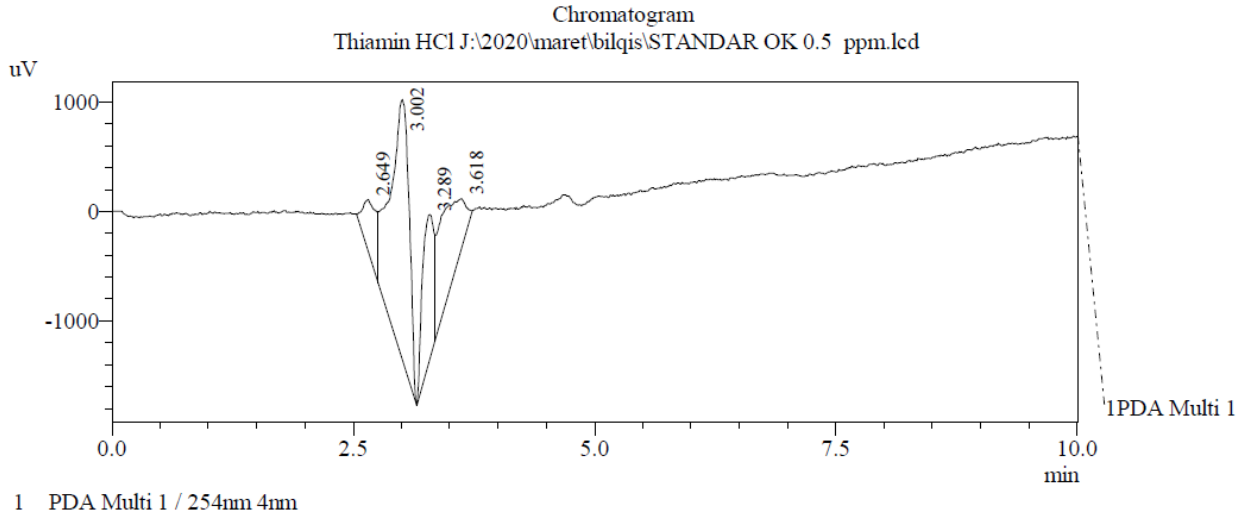
Gambar 19. Kromatogram baku vitamin B1 4 ppm



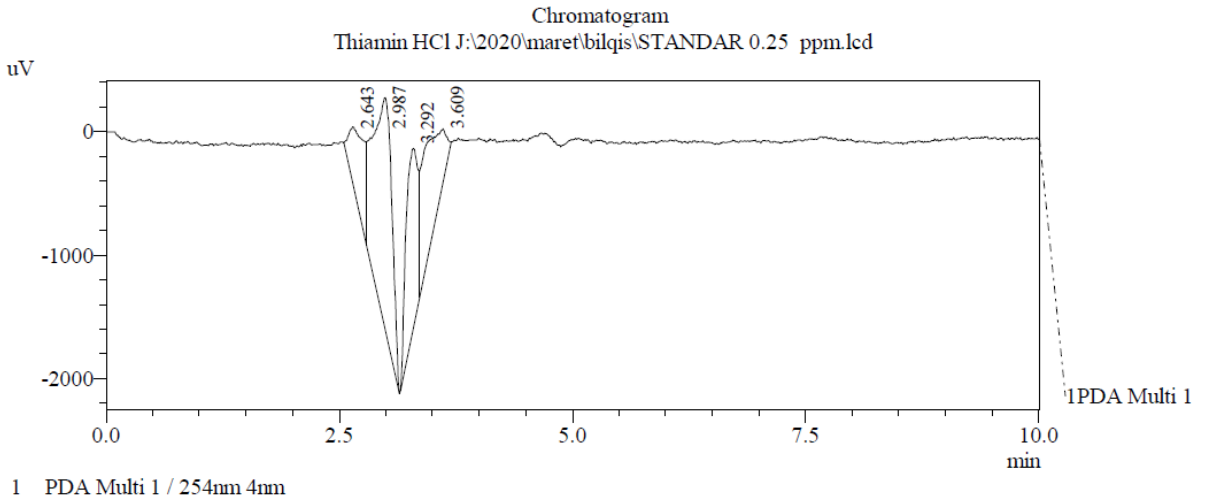
Gambar 20. Kromatogram baku vitamin B1 2 ppm



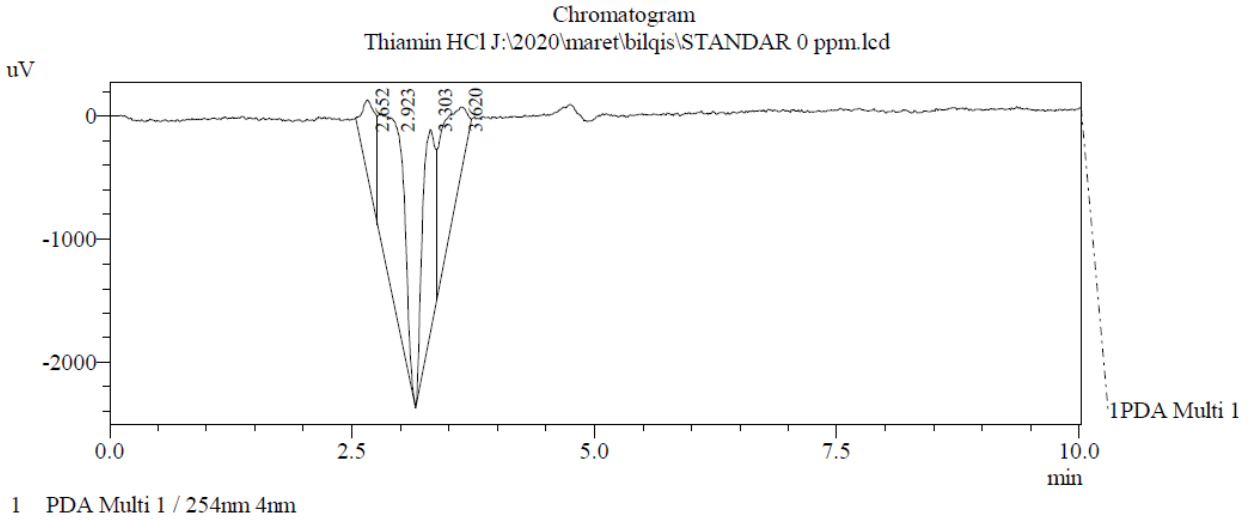
Gambar 21. Kromatogram baku vitamin B1 1 ppm



Gambar 22. Kromatogram baku vitamin B1 0,5 ppm



Gambar 23. Kromatogram baku vitamin B1 0,25 ppm



Gambar 24. Kromatogram baku vitamin B1 0 ppm

Lampiran 5

Perhitungan kadar Vitamin B1 pada sampel

Penentuan Kadar Sampel

Persamaan garis lurus: y= 36809x – 3554,8

Konsentrasi sampel 5.000 ng/µL

Rumus penentuan kadar: $\frac{KOnsentrasi\ Vitamin\ B1\ \left(\frac{ng}{\mu L}\right)}{Konsentrasi\ sampel\ \left(\frac{ng}{\mu L}\right)}\ x\ 100\%$

Perhitungan Persen Kadar Sampel Beras A dicuci 2x (A 2x)

X sampel A2x (1) = $\frac{23483+3554,8}{36809} = 0,734543$ ng/20µL= 0,03672 ng/µL

Kadar XA2x (1) = $\frac{0,03672}{5000} X\ 100\% = 0,0007344\% = 0,0007344$ g/100 g

= 0, 734 mg/100 g

X sampel A2x (2) = $\frac{20898+3554,8}{36809} = 0,664315$ ng/20µL= 0,033215 ng/µL

Kadar XA2x (2) = $\frac{0,033215}{5000} X\ 100\% = 0,0006643\% = 0,0006643$ g/100 g

= 0, 664 mg/100 g

X sampel A2x (3) = $\frac{24484+3554,8}{36809} = 0,761737$ ng/20µL= 0,038086 ng/µL

Kadar XA2x (3) = $\frac{0,038086}{5000} X\ 100\% = 0,000761\% = 0,000761$ g/100 g

= 0, 761 mg/100 g

Sampel	AUC Sebenarnya	Kadar (mg/100g)	Rata-rata (mg/100g)
Beras A dicuci 2x	23483	0,734	0,719± 0,050
	20898	0,664	
	24484	0,761	

Perhitungan Persen Kadar Sampel Beras B dicuci 2x (B 2x)

X sampel B2x (1) = $\frac{24956+3554,8}{36809}$ = 0,7745 ng/20µL= 0,03872 ng/µL

Kadar XA2x (1) = $\frac{0,03872}{5000} X\ 100\% = 0,000774\% = 0,000774$ g/100 g

= 0, 774 mg/100 g

X sampel A2x (2) = $\frac{24630+3554,8}{36809}$ = 0,7657 ng/20µL= 0,03828 ng/µL

Kadar XA2x (2) = $\frac{0,03828}{5000} X\ 100\% = 0,0007657\% = 0,0007657$ g/100 g

= 0, 765 mg/100 g

X sampel A2x (3) = $\frac{26124+3554,8}{36809}$ = 0,80629 ng/20µL= 0,04031 ng/µL

Kadar XA2x (3) = $\frac{0,04031}{5000} X\ 100\% = 0,000806\% = 0,000806$ g/100 g

= 0, 806 mg/100 g

Sampel	AUC Sebenarnya	Kadar (mg/100g)	Rata-rata (mg/100g)
Beras B dicuci 2x	24956	0,774	0,781± 0,021
	24630	0,765	
	26124	0,806	

Perhitungan Persen Kadar Sampel Beras C dicuci 2x (C 2x)

X sampel C2x (1) = $\frac{24081+3554,8}{36809}$ = 0,75078 ng/20µL= 0,03753 ng/µL

C2x (1) = $\frac{0,03753}{5000} X\ 100\% = 0,0007507\% = 0,0007507$ g/100 g

= 0, 750 mg/100 g

X sampel C2x (2) = $\frac{23784+3554,8}{36809}$ = 0,742720 ng/20µL= 0,037136 ng/µL

Kadar XC2x (2) = $\frac{0,037136}{5000} X\ 100\% = 0,0007427\% = 0,0007427$ g/100 g

= 0, 742 mg/100 g

X sampel C2x (3) = $\frac{23489+3554,8}{36809}$ = 0,734706 ng/20µL= 0,036735 ng/µL

Kadar XC2x (3) = $\frac{0,036735}{5000} X\ 100\% = 0,000734\% = 0,000734$ g/100 g

= 0, 734 mg/100 g

Sampel	AUC Sebenarnya	Kadar (mg/100g)	Rata-rata (mg/100g)
Beras C dicuci 2x	24081	0,750	0,742± 0,008
	23784	0,742	
	23489	0,734	

Perhitungan Persen Kadar Sampel Beras A dicuci 3x (A 3x)

X sampel A3x (1) = $\frac{22823+3554,8}{36809}$ = 0,716612 ng/20µL= 0,03583 ng/µL

Kadar XA3x (1) = $\frac{0,03585}{5000} X\ 100\% = 0,0007166\% = 0,0007166$ g/100 g

= 0, 716 mg/100 g

X sampel A3x (2) = $\frac{23620+3554,8}{36809}$ = 0,738265 ng/20µL= 0,036913 ng/µL

Kadar XA3x (2) = $\frac{0,036913}{5000} X\ 100\% = 0,0007382\% = 0,0007382$ g/100 g

= 0, 738 mg/100 g

el A3x (3) = $\frac{27024+3554,8}{36809}$ = 0,8307424 ng/20µL= 0,04153 ng/µL

Kadar XA3x (3) $= \frac{0{,}04153}{5000} X\ 100\% = 0{,}0008307\% = 0{,}0008307$ g/100 g

= 0, 830 mg/100 g

Sampel	AUC Sebenarnya	Kadar (mg/100g)	Rata-rata (mg/100g)
Beras A dicuci 3x	22823	0,716	0,761± 0,060
	23630	0,738	
	27024	0,830	

Perhitungan Persen Kadar Sampel Beras B dicuci 3x (B 3x)

X sampel B3x (1) $= \frac{12933+3554{,}8}{36809} = 0{,}447928$ ng/20µL= 0,0223 ng/µL

Kadar XB3x (1) $= \frac{0{,}0223}{5000} X\ 100\% = 0{,}0004479\% = 0{,}0004479$ g/100 g

= 0, 447 mg/100 g

X sampel B3x (2) $= \frac{23990+3554{,}8}{36809} = 0{,}74831$ ng/20µL= 0,037415 ng/µL

Kadar XB3x (2) $= \frac{0{,}037415}{5000} X\ 100\% = 0{,}0007483\% = 0{,}0007483$ g/100 g

= 0, 748 mg/100 g

X sampel B3x (3) $= \frac{23654+3554{,}8}{36809} = 0{,}739188$ ng/20µL= 0,03695 ng/µL

Kadar XB3x (3) $= \frac{0{,}03695}{5000} X\ 100\% = 0{,}0007391\% = 0{,}0007391$ g/100 g

= 0, 739 mg/100 g

Sampel	AUC Sebenarnya	Kadar (mg/100g)	Rata-rata (mg/100g)
Beras B dicuci 3x	12933	0,447	0,644± 0,171
	23990	0,748	
	23654	0,739	

Perhitungan Persen Kadar Sampel Beras C dicuci 3x (C 3x)

X sampel C3x (1) $= \frac{22596+3554,8}{36809} = 0,71044$ ng/20µL= 0,0355 ng/µL

Kadar XC3x (1) $= \frac{0,0355}{5000} X\ 100\% = 0,00071044\% = 0,000\ 71044$g/100 g

= 0, 710 mg/100 g

X sampel C3x (2) $= \frac{25059+3554,8}{36809} = 0,77735$ ng/20µL= 0,03886 ng/µL

Kadar XC3x (2) $= \frac{0,03886}{5000} X\ 100\% = 0,0007773\% = 0,0007773$ g/100 g

= 0, 777 mg/100 g

X sampel C3x (3) $= \frac{26366+3554,8}{36809} = 0,81286$ ng/20µL= 0,0406 ng/µL

Kadar XC3x (3) $= \frac{0,0406}{5000} X\ 100\% = 0,000812\% = 0,000812$ g/100 g

= 0, 812 mg/100 g

Sampel	AUC Sebenarnya	Kadar (mg/100g)	Rata-rata (mg/100g)
Beras C dicuci 3x	22596	0,710	0,766± 0,051
	25059	0,777	
	26366	0,812	

Perhitungan Persen Kadar Sampel Beras Tidak dicuci

X sampel tidak dicuci= $\frac{26102+3554,8}{36809} = 0,805694$ ng/20µL= 0,040284 ng/µL

Kadar beras tidak dicuci (1)= $\frac{0,040284}{5000} X\ 100\% = 0,000805\% = 0,0008065$ g/100 g

= 0, 805 mg/100 g

X sampel tidak dicuci (2) $= \frac{26356+3554,8}{36809} = 0,81259$ ng/20µL= 0,046629 ng/µL

Kadar beras tidak dicuci (2)= $\frac{0,046629}{5000} X\ 100\% = 0,000812\% = 0,000812$ g/100g

= 0, 812 mg/100 g

X sampel tidak dicuci (3) $= \frac{27815+3554,8}{36809} = 0,852231$ ng/20µL= 0,04261 ng/µL

Kadar beras tidak dicuci (3)= $\frac{0,04261}{5000} X\ 100\% = 0,000852\% = 0,000852$ g/100 g

= 0, 852 mg/100 g

Sampel	AUC Sebenarnya	Kadar (mg/100g)	Rata-rata (mg/100g)
Beras Tidak Dicuci	26102	0,805	0,823± 0,025
	26356	0,812	
	27815	0,853	

Lampiran 6

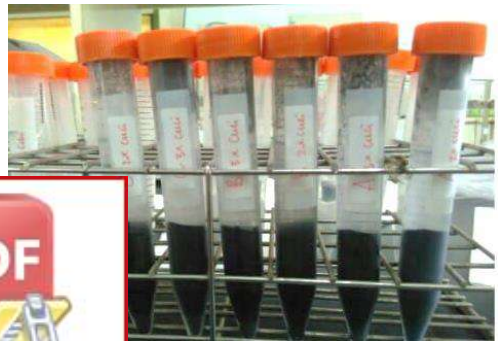
Dokumentasi penelitian

Gambar 27. Penimbangan sampel beras

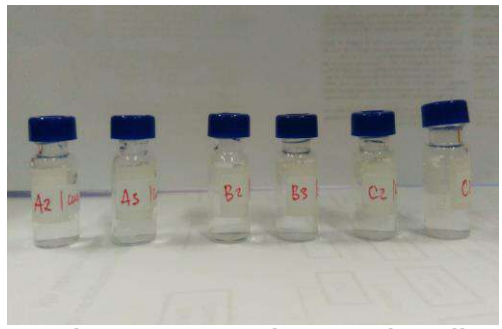
Gambar 28. Pencucian sampel beras

Gambar 29. Proses pengocokan pada saat ekstraksi

Gambar 30. Penambahan serbuk QuEChERS

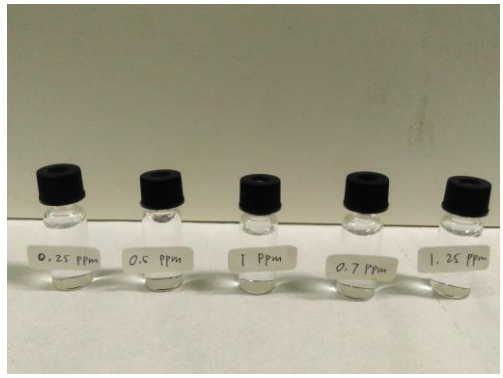


31. Ekstraksi sampel penambahan serbuk RS



Gambar 32. Sampel yang siap di analisis pada GC/MS

Gambar 33. Pembuatan larutan baku klorpirifos



Gambar 34. Standar baku klorpirifos yang siap analisis

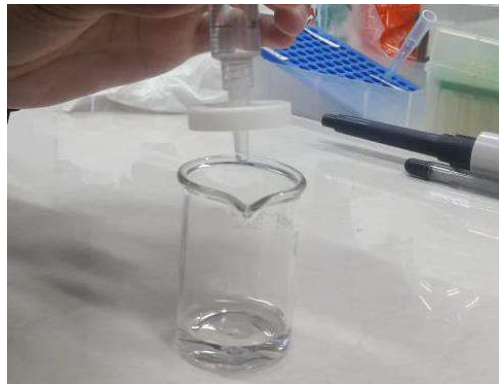
Gambar 35. Penempatan vial sampel pada saat akan diinjeksikan pada GC/MS

Gambar 36. Alat GC/MS

37. Proses pengadukan encucian beras analisis 1

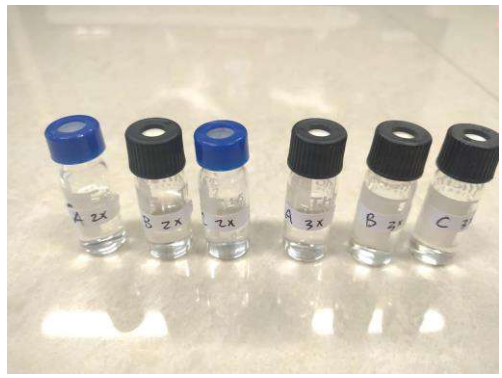
Gambar 38. Proses ekstraksi dan inkubasi

Gambar 39. Proses sentrifugasi sampel hasil ekstraksi



Gambar 40. Penyaringan sampel dengan filter membrane 0,45 μm.

Gambar 41. Pengenceran sampel yang akan dianalisis



Gambar 42. Sampel siap diinjeksikan pada UFLC

43. Buffer Asetat sebagai a analisis vitamin B1

Gambar 44. Instrumen UFLC