ABAMECTIN

Collaborative Study

Small scale collaborative study for the

Determination of Abamectin in TC by HPLC

Report to CIPAC by

Chinese Pesticide Analytical Committee（CHIPAC）

Method Developed by

Hebei Xingbai Agricultural Technology Co., Ltd.

May 2024

1. Participants

Small Scale Collaborative Study for the determination of abamectin TC by HPLC was organized by CHIPAC, and participated by 4 laboratories. All of the laboratories provided their results, which are presented in the following sections.

Index	Contact	Lab
Lab 1	Xianwei Liu	Hebei Xingbai Agrochem Group Co., Ltd.
Lab 2	Hongxia Li	Nutrichem Laboratory Co., Ltd
Lab 3	Mingwei Zhu	Nutrichem (Shandong) Laboratory Co., Ltd
Lab 4	Cong Yan	Jiangsu Rotam Chemistry Co., Ltd.

2. Active Ingredient: General Information

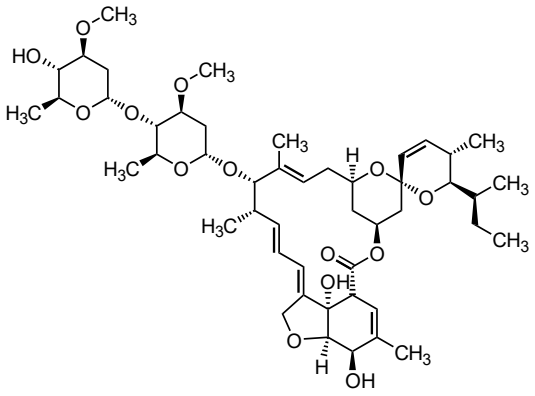
ISO common name: Abamectin

CAS No.: Abamectin: 71751-41-2

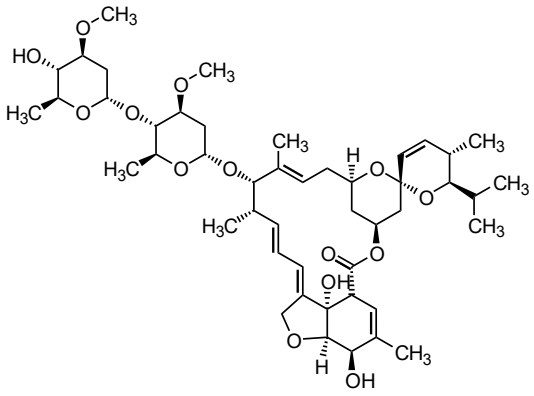
Avermectin B_{1a}: 65195-55-3

Avermectin B_{1b}: 65195-56-4

Structure:



avermectin B_{1a}



avermectin B_{1b}

Molecular mass: 873.1(Avermectin B_{1a}); 859.1 (Avermectin B_{1b})

Empirical formula: $C_{48}H_{72}O_{14}$ (Avermectin B_{1a}); $C_{47}H_{70}O_{14}$ (Avermectin B_{1b})

3. Samples

Three test samples and abamectin analytical standard were sent to the participants:

(1) Abamectin tech. sample (TC-1)

(2) Abamectin tech. sample (TC-2)

(3) Abamectin tech. sample (TC-3)
(4) Abamectin, reference standard

All participants sent back their results in time.

4. Method

4.1 Scope

The determination of abamectin active ingredient content was assayed in technical material (TC).

4.2 Outline of method

Abamectin is determined by high performance liquid chromatography on a reversed phase column (C_{18}) with UV detection at 245 nm and external standardization.

4.3 Procedure for the collaborative trial

The samples were analyzed on two different days, each day involving duplicate injections of duplicate weights. Both test and reference solutions were freshly prepared on each day.

5. Analytical conditions

Lab	Instrument	Column	Flow Rate (mL/min)	Column Temp. (℃)	Wavelength (nm)	Injection Volume (μL)	Mobile phase (v/v)	Remark
1	ThermoFisher UltiMate 3000	SHIMADZU VP-ODS-C_{18} (250×4.6 mm, 5 μm)	0.8	30	245	5	methanol: acetonitrile: water, 55:30:15	
2	Agilent 1100	Agilent ZORBAX RX-C_{18} (250×4.6 mm, 5 μm)	0.8	30	245	5	methanol: acetonitrile: water, 55:30:15	
3	Agilent 1200	Venusil BP C_{18} (250×4.6 mm, 5 μm)	0.8	30	245	5	methanol: acetonitrile: water, 55:30:15	
4	SHIMADZU LCMS2020; Agilent 1260	Agilent TC-C_{18} (250×4.6 mm, 5 μm)	0.8	30	245	5	methanol: acetonitrile: water, 55:30:15	Different instrument were used on the first and second day

6. Deviations and Remarks

No deviations.

7. Evaluation and Discussion

7.1 Evaluation of the Quality of Data and Chromatograms

The data obtained from each laboratory was visually reviewed and no significant deviation regarding the chromatography which might affect the analysis results was founded.

Therefore, all data sets were included within the statistical assessment. The report below contains statistical evaluations with the full set of 4 participating laboratories.

7.2 Determination of abamectin

The statistical evaluation of the data was done following the “Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods”, according to DIN ISO 5725. The testing for outliers/stragglers of the laboratory mean values were not performed.

In Tables 1-7 and Figures 1-11, respectively, the full set of all laboratories are reported. The between lab experimental Relative Reproducibility Standard Deviation (RSD_R) is below the Horwitz value for all samples (see in Table 5-7), and the Horwitz Ratio (HorRat) was found within the desired range (0.3-1.0). Due to the universal applicability of the method, this collaborative trial is acceptable.

8. Conclusions

From the results shown above, the method can be considered applicable for the determination of abamectin contents in TC. CHIPAC propose that a full scale collaborative trial might be conducted.

Table 1 Results of analysis of abamectin (B_{1b}+B_{1a}) content in the TC1

	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi^2	Standard Deviation S_i	Si^2
	1	2	1	2				
Lab 1	962.2	967.0	965.0	963.3	964.4	930019.1	2.095	4.389
Lab 2	952.7	955.9	954.0	958.7	955.3	912645.9	2.606	6.789
Lab 3	961.6	956.6	960.9	964.0	960.8	923088.6	3.084	9.509
Lab 4	950.8	950.0	947.6	948.4	949.2	900980.6	1.461	2.133

Table 2 Results of analysis of abamectin (B_{1b}+B_{1a}) content in the TC2

	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi^2	Standard Deviation S_i	Si^2
	1	2	1	2				
Lab 1	964.2	961.8	966.9	961.8	963.7	928669.5	2.430	5.903
Lab 2	953.9	958.7	963.3	956.1	958.0	917764.0	4.041	16.33
Lab 3	960.4	956.3	959.5	962.2	959.6	920832.2	2.470	6.100
Lab 4	944.5	936.5	945.2	942.2	942.1	887552.4	3.947	15.58

Table 3 Results of analysis of abamectin (B_{1b}+B_{1a}) content in the TC3

	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi^2	Standard Deviation S_i	Si^2
	1	2	1	2				
Lab 1	962.8	962.3	960.0	959.0	961.0	923569.1	1.819	3.309
Lab 2	956.8	952.4	954.8	962.2	956.6	914987.9	4.174	17.42
Lab 3	960.8	963.7	965.3	961.3	962.8	926935.7	2.106	4.436
Lab 4	951.6	953.1	950.9	950.4	951.5	905352.3	1.175	1.380

Table 4 Ratio of B_{1a} to B_{1b} of avermectin in the TC1, TC2 and TC3

	TC1				TC2				TC3			
	Day1		Day2		Day1		Day2		Day1		Day2	
	1	2	1	2	1	2	1	2	1	2	1	2
Lab 1	125.2	126.4	124.2	128.1	127.3	162.0	124.7	122.3	127.0	127.4	123.5	125.0
Lab 2	137.9	145.6	142.7	145.1	137.3	145.1	136.6	139.8	138.3	148.9	138.1	146.4
Lab 3	118.3	120.5	122.1	120.7	115.1	117.3	122.0	115.7	122.2	121.1	122.2	118.9
Lab 4	111.5	110.4	103.3	100.7	107.0	108.4	98.8	99.0	103.9	105.4	95.0	95.1

Table 5 Statistics of the results of TC1

S_1=Sum Yi	3829.7		
S_2=Sum Yi^2	3666734.2		
S_3=Sum Si^2	22.82		
No. Lab P	4		
No.Determination n	4		
Average Y=S_1/P	957.4		
Sr^2=S_3/P	5.705	S_r	2.389
S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n	42.44	S_L	6.514
S_R^2=S_r^2+S_L^2	48.14	S_R	6.938
Repeatibility r=2.8*S_r	6.688		
Reporducibility R=2.8*S_R	19.43		
Relative Standard Deviation of Repeatability RSD_r=S_r*100/Y	0.2495		
Relative Standard Deviation of Reporducibility RSD_R=S_R*100/Y	0.7247		
Horwitz RSD_R(Hor)=2^[1-0.5*log(Y/1000)]	2.013		
HorRat	0.4		

Table 6 Statistics of the results of TC2

S_1=Sum Yi	3823.4		
S_2=Sum Yi^2	3654818.1		
S_3=Sum Si^2	43.92		
No. Lab P	4		
No.Determination n	4		
Average Y=S_1/P	955.8		
Sr^2=S_3/P	10.98	S_r	3.313
S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n	86.91	S_L	9.323
S_R^2=S_r^2+S_L^2	97.89	S_R	9.894
Repeatibility r=2.8*S_r	9.278		
Reporducibility R=2.8*S_R	27.70		
Relative Standard Deviation of Repeatability RSD_r=S_r*100/Y	0.3467		
Relative Standard Deviation of Reporducibility RSD_R=S_R*100/Y	1.035		
Horwitz RSD_R(Hor)=2^[1-0.5*log(Y/1000)]	2.014		
HorRat	0.5		

Table 7 Statistics of the results of TC3

S_1=Sum Yi	3831.9		
S_2=Sum Yi^2	3670844.9		
S_3=Sum Si^2	26.55		
No. Lab P	4		
No.Determination n	4		
Average Y=S_1/P	958.0		
Sr^2=S_3/P	6.637	S_r	2.576
S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n	23.77	S_L	4.876
S_R^2=S_r^2+S_L^2	30.41	S_R	5.515
Repeatibility r=2.8*S_r	7.214		
Reporducibility R=2.8*S_R	15.44		
Relative Standard Deviation of Repeatability RSD_r=S_r*100/Y	0.2689		
Relative Standard Deviation of Reporducibility RSD_R=S_R*100/Y	0.5757		
Horwitz RSD_R(Hor)=2^[1-0.5*log(Y/1000)]	2.013		
HorRat	0.3		

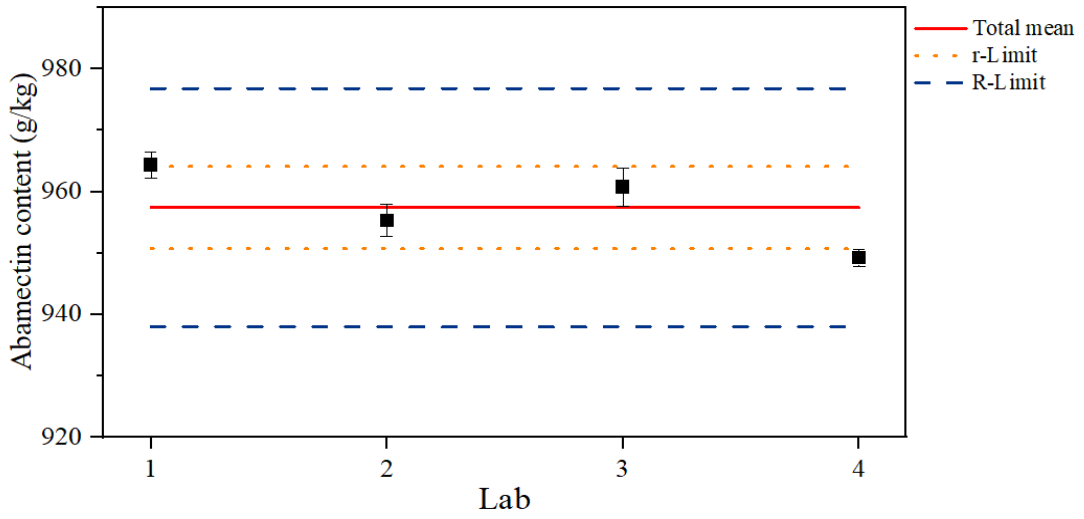


Figure 1. Graphical presentation of TC1 data

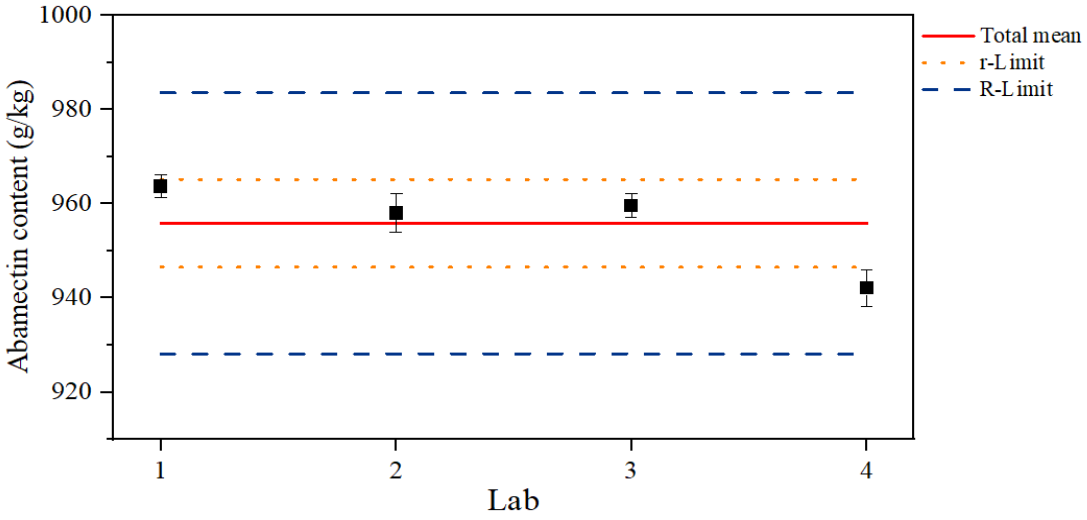


Figure 2. Graphical presentation of TC2 data

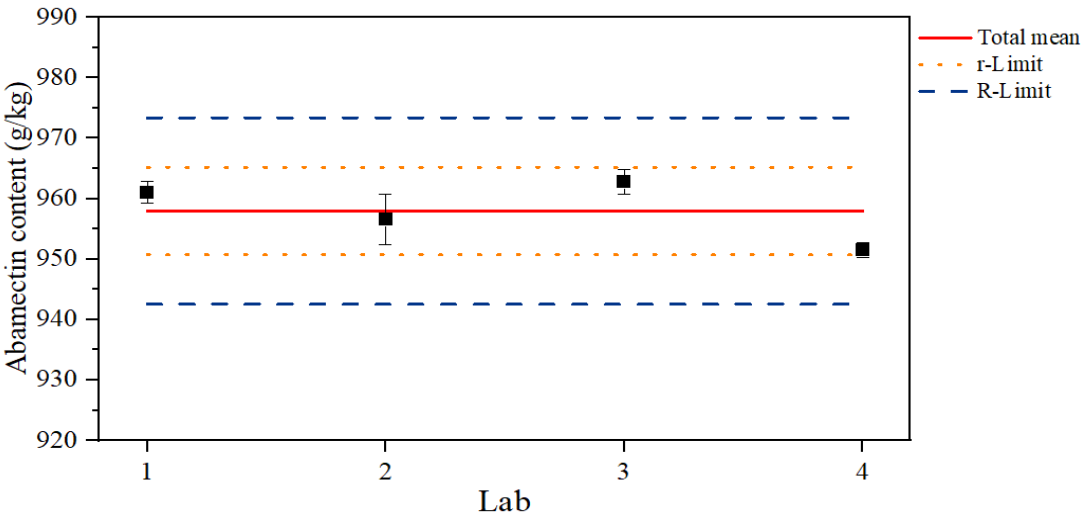


Figure 3. Graphical presentation of TC3 data

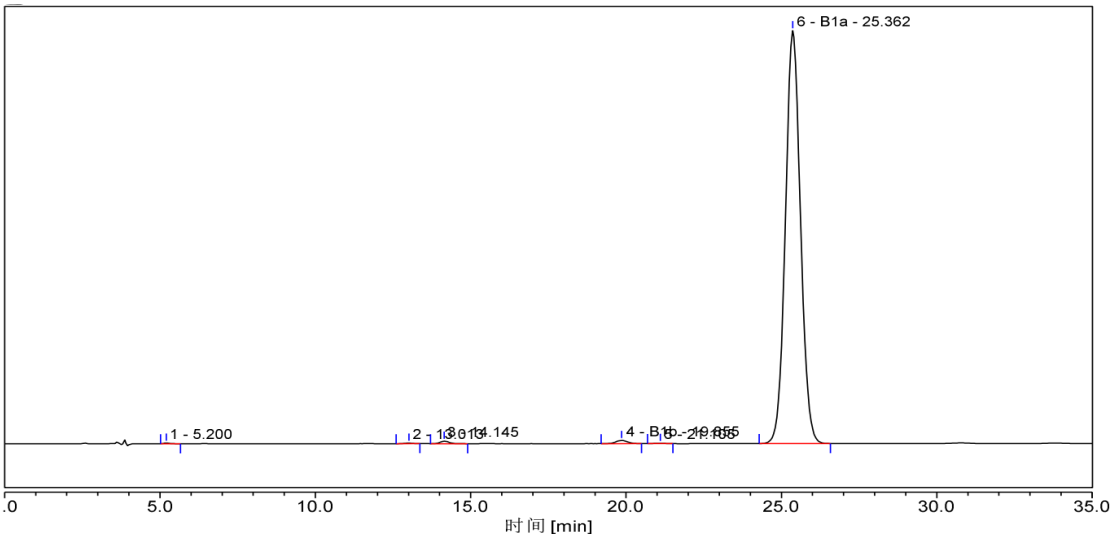


Figure 4. HPLC chromatogram of abamectin standard (Lab 1)

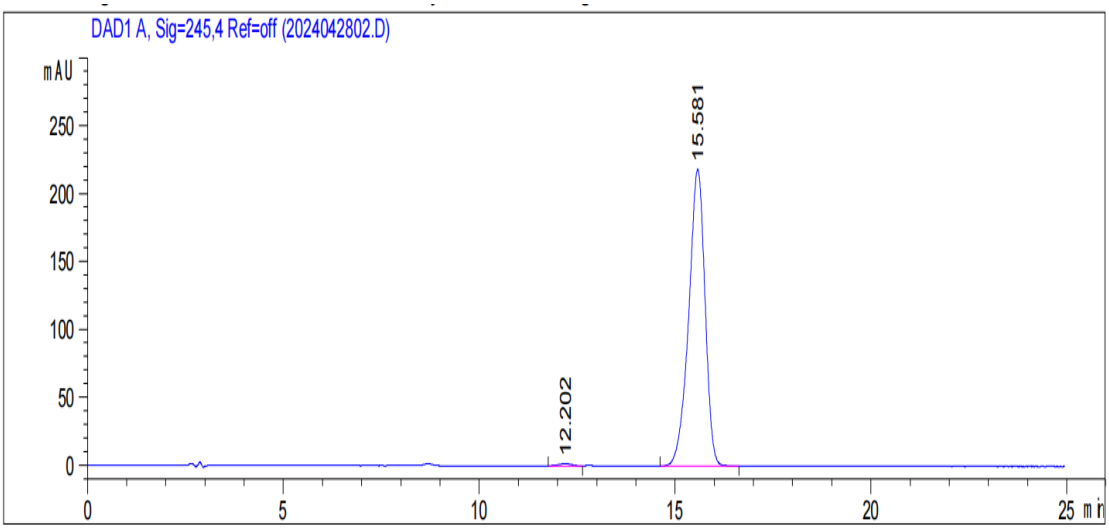


Figure 5. HPLC chromatogram of abamectin standard (Lab 2)

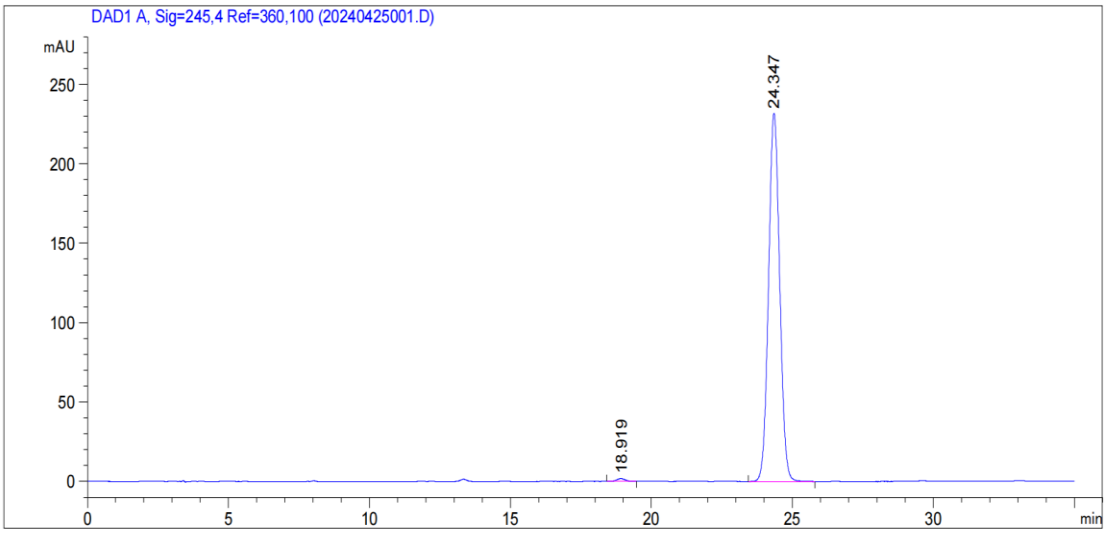


Figure 6. HPLC chromatogram of abamectin standard (Lab 3)

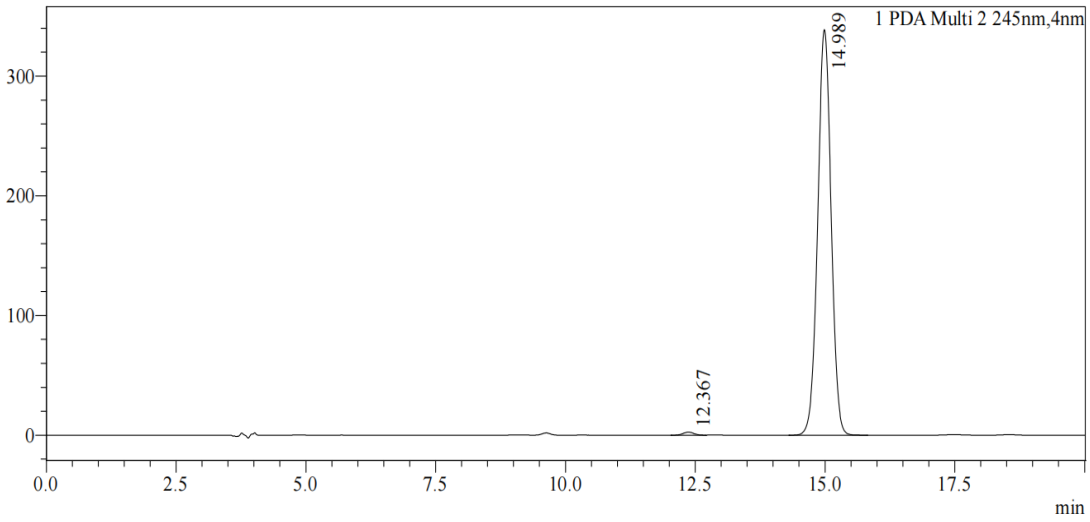


Figure 7. HPLC chromatogram of abamectin standard (Lab 4)

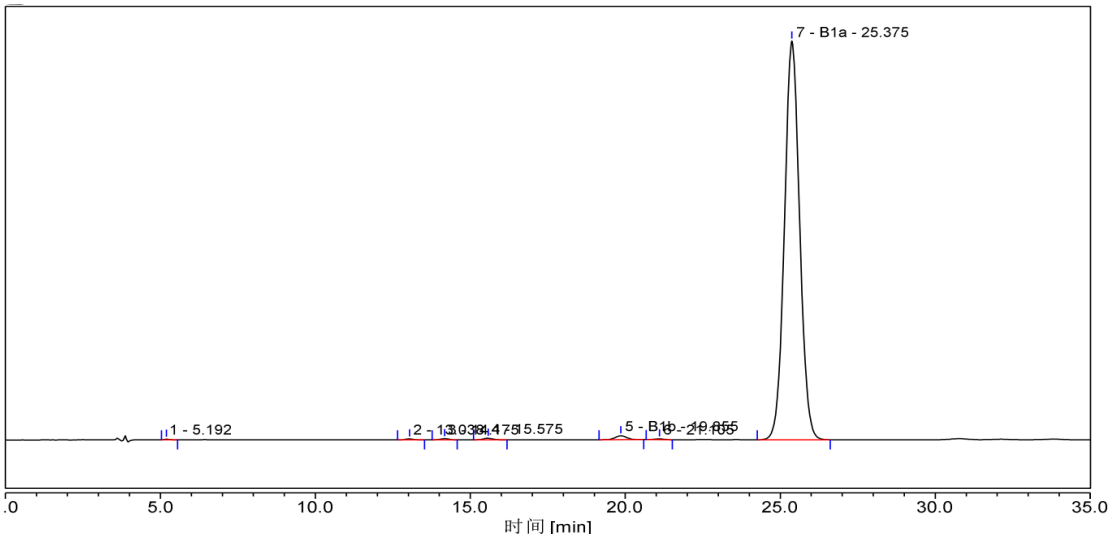


Figure 8. HPLC chromatogram of abamectin TC (Lab 1)

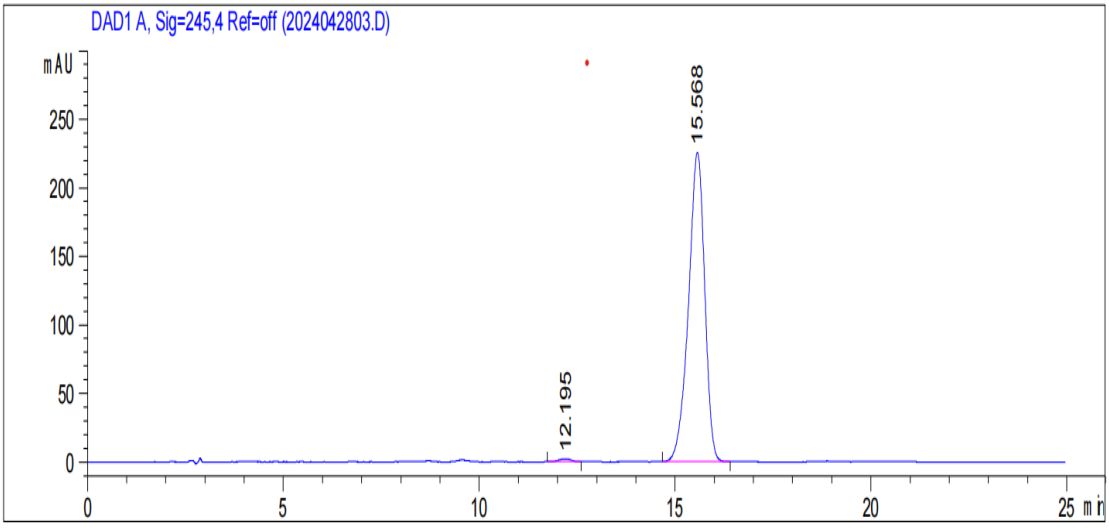


Figure 9. HPLC chromatogram of abamectin TC (Lab 2)

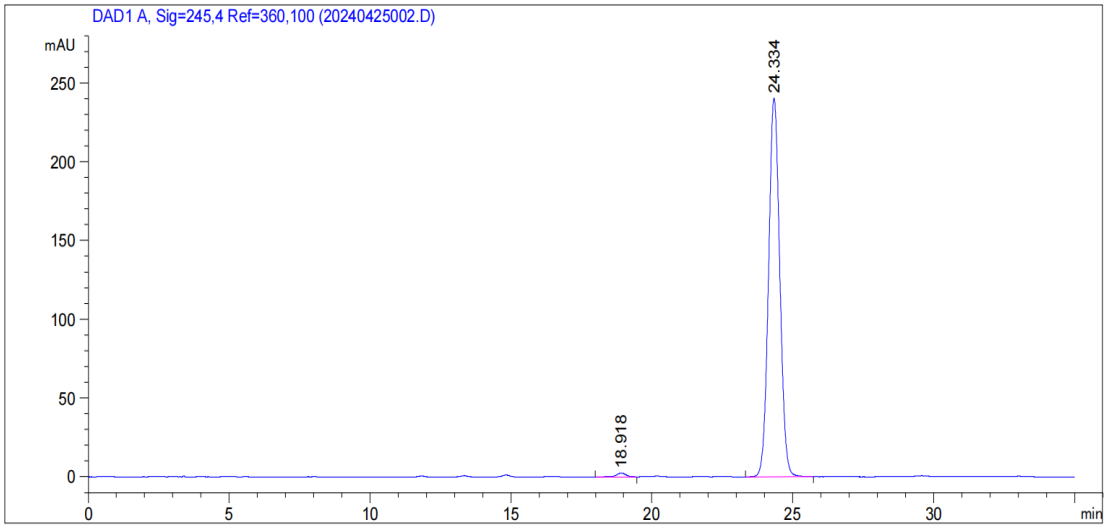


Figure 10. HPLC chromatogram of abamectin TC (Lab 3)

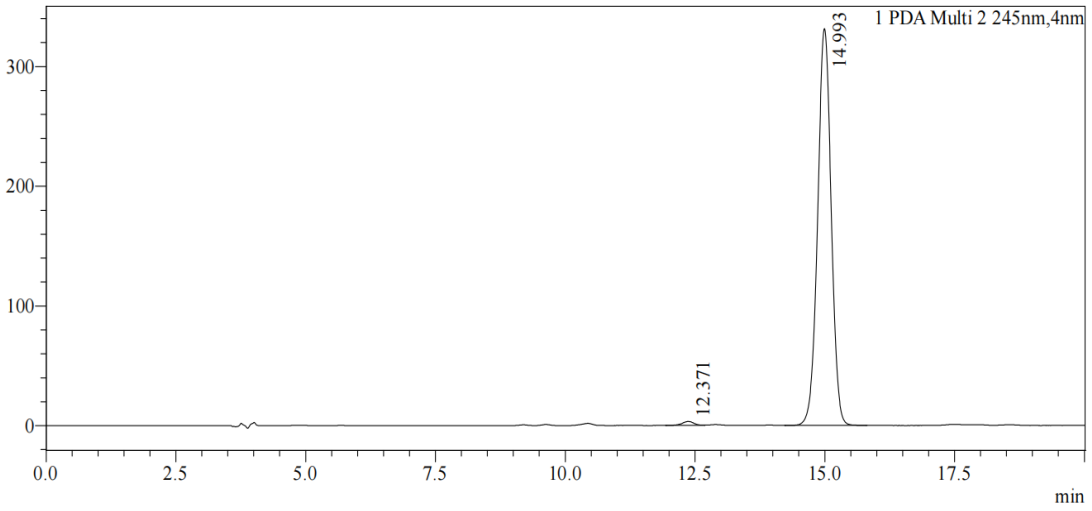


Figure 11. HPLC chromatogram of abamectin TC (Lab 4)