

# **TEMBOTRIONE**

## **Collaborative Study**

Small scale collaborative study for the  
Determination of Tembotrione in Tembotrione TC, SC and  
OD by HPLC

Report to CIPAC by CHIPAC

Method Developed by  
Jiangxi Tianyu Chemical Co., Ltd

May 2023

## 1. Participants

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

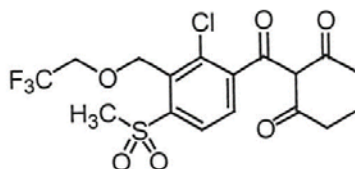
Index	Contact	Lab	Address
Lab 1	Shenglin Li	Jiangxi Tianyu Chemical Co., Ltd	Yanhua Road, Xingan Salt Chemical Industrial Park, Xingan County, Jiangxi Province, P. R. China
Lab 2	Mirror Chen	GreenTech Laboratory Co., Ltd.	Building 2, No. 650, Shunqing Road, Songjiang, Shanghai, China
Lab 3	Yan Liu	National Chemical Low Carbon Science and Technology Co., Ltd	No.3 Kangpu Road, Zhoushi Town, Kunshan City, Jiangsu Province, China
Lab 4	Haiyan Jiang	Pilarcise Laboratory Co., Ltd.	Building .1500 Hang-Tang Road, Feng Xian District, Shanghai, CHINA

## 2. Active Ingredient: General Information

ISO common name: Tembotrione

CAS No.: 335104-84-2

Structure:



Molecular mass: 440.8

Empirical formula: C<sub>17</sub>H<sub>16</sub>ClF<sub>3</sub>O<sub>6</sub>S

Activity: herbicide

## 3. Samples

Six test samples and tembotrione analytical standard were sent to the participants:

- (1) Tembotrione tech. sample (TC1)
- (2) Tembotrione tech. sample (TC2)
- (3) Tembotrione tech. sample (SC1)
- (4) Tembotrione tech. sample (SC2)
- (5) Tembotrione tech. sample (OD1)
- (6) Tembotrione tech. sample (OD2)

(7) Tembotrione, reference standard (purity 986 g/kg)

All participants sent back their results in time.

## 4. Method

### 4.1 Scope

The determination of tembotrione active ingredient content was assayed in technical material (TC), SC and OD formulation.

### 4.2 Outline of method

Tembotrione is determined by high performance liquid chromatography on a reversed phase column (C18) with UV detection at 284 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. (°C)	Wavelength (nm)	Injection Volume (µL)	Mobile phase (v/v)
1	SHIMADZ ULC-20AT	ZORBAX SB-C <sub>18</sub> (250×4.6 mm, 5 µm)	1.0	30	284	5	acetonitrile: phosphoric acid aqueous solution, 60:40
2	SHIMADZ ULC-2050 C 3D	ZORBAX SB-C <sub>18</sub> (250×4.6 mm, 5 µm)	1.0	30	284	5	acetonitrile: phosphoric acid aqueous solution, 60:40
3	Waters 2695	Waters Xbridge C <sub>18</sub> (250×4.6 mm, 5 µm)	1.0	30	284	5	acetonitrile: phosphoric acid aqueous solution, 60:40
4	Agilent 1260	Agilent Eclipse Plus C <sub>18</sub> (250×4.6 mm, 5 µm)	1.0	30	284	5	acetonitrile: phosphoric acid aqueous solution, 60:40

## 6. Deviations and Remarks

There is no deviation in the experiments between the four laboratories, and no remarks need to be made.

## **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 tembotrione**

The statistical evaluation of the data was accomplished 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 1-12 and Figures 1-6, respectively, the full set of all laboratories (4 participants) are reported. A comparison of the  $RSD_R$  of this collaborative study with the unmodified Horwitz equation showed that the reproducibility relative standard deviation ( $RSD_R$ ) is lower the Horwitz value for all the products (see in Table 7-12). The Horwitz Ratio (HorRat) for each sample was found lower than 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 tembotrione contents in TC, SC and OD. CHIPAC propose that a full scale collaborative trial might be conducted.

**Table 1 Results of analysis of tembotrione content in the TC1**

	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi <sup>2</sup>	Standard Deviation Si	Si <sup>2</sup>
	1	2	1	2				
Lab 1	963.3	964.0	963.3	960.3	962.73	926839.4256	1.6500	2.7225
Lab 2	955.4	954.7	955.0	953.4	954.63	911308.8906	0.8655	0.7492
Lab 3	959.5	962.6	959.4	961.6	960.78	923088.6006	1.5840	2.5092
Lab 4	962.9	963.2	963.0	963.6	963.18	927706.0806	0.3096	0.0958

**Table 2 Results of analysis of tembotrione content in the TC2**

	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi <sup>2</sup>	Standard Deviation Si	Si <sup>2</sup>
	1	2	1	2				
Lab 1	952.9	952.3	954.4	952.7	953.08	908351.9556	0.9179	0.8425
Lab 2	957.7	961.5	961.3	962.4	960.73	922992.5256	2.0726	4.2958
Lab 3	953.8	954.6	955.6	954.2	954.55	911165.7025	0.7724	0.5967
Lab 4	966.1	967.2	967.8	967.1	967.05	935185.7025	0.7047	0.4967

**Table 3 Results of analysis of tembotrione content in the SC1**

	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi <sup>2</sup>	Standard Deviation Si	Si <sup>2</sup>
	1	2	1	2				
Lab 1	345.7	345.5	344.1	345.3	345.15	119128.5225	0.7188	0.5167
Lab 2	343.9	343.2	343.3	343.6	343.50	117992.2500	0.3162	0.1000
Lab 3	347.4	347.4	350.7	347.2	348.18	121225.8306	1.6860	2.8425
Lab 4	349.3	349.2	349.2	348.8	349.13	121888.2656	0.2217	0.0492

**Table 4 Results of analysis of tembotrione content in the SC2**

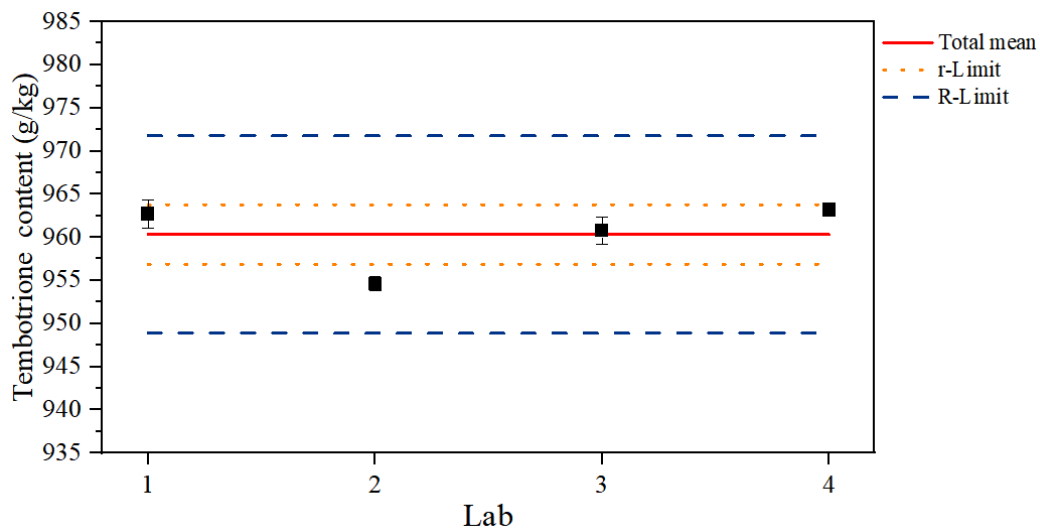
	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi <sup>2</sup>	Standard Deviation Si	Si <sup>2</sup>
	1	2	1	2				
Lab 1	344.7	345.0	343.6	344.8	344.53	118697.4756	0.6292	0.3958
Lab 2	343.1	345.1	342.6	345.1	343.98	118318.8006	1.3150	1.7292
Lab 3	355.8	354.5	346.1	345.8	350.55	122885.3025	5.3395	28.5100
Lab 4	349.1	349.4	348.4	348.7	348.90	121731.21	0.4397	0.1933

**Table 5 Results of analysis of tembotrione content in the OD1**

	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi <sup>2</sup>	Standard Deviation Si	Si <sup>2</sup>
	1	2	1	2				
Lab 1	85.4	84.7	85.3	85.5	85.23	7263.3006	0.3594	0.1292
Lab 2	83.9	84.6	84.5	84.5	84.38	7119.1406	0.3202	0.1025
Lab 3	84.5	85.0	84.7	84.4	84.65	7165.6225	0.2646	0.0700
Lab 4	87.5	87.5	87.5	87.5	87.50	7656.2500	0.0000	0.0000

**Table 6 Results of analysis of tembotrione content in the OD2**

	Day1(g/kg)		Day2(g/kg)		Average Yi	Yi <sup>2</sup>	Standard Deviation Si	Si <sup>2</sup>
	1	2	1	2				
Lab 1	85.4	85.8	85.0	85.6	85.45	7301.7025	0.3416	0.1167
Lab 2	83.8	84.8	84.3	83.6	84.13	7077.0156	0.5377	0.2892
Lab 3	84.4	84.5	84.1	84.5	84.38	7119.1406	0.1893	0.0358
Lab 4	87.3	87.2	87.3	87.1	87.23	7608.2006	0.0957	0.0092

**Figure 1. Graphical presentation of TC1 data**

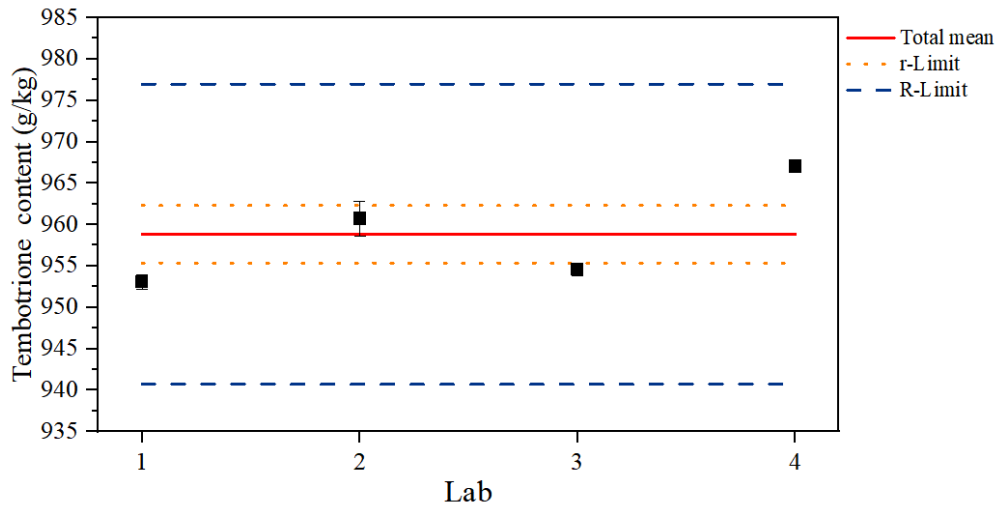


Figure 2. Graphical presentation of TC2 data

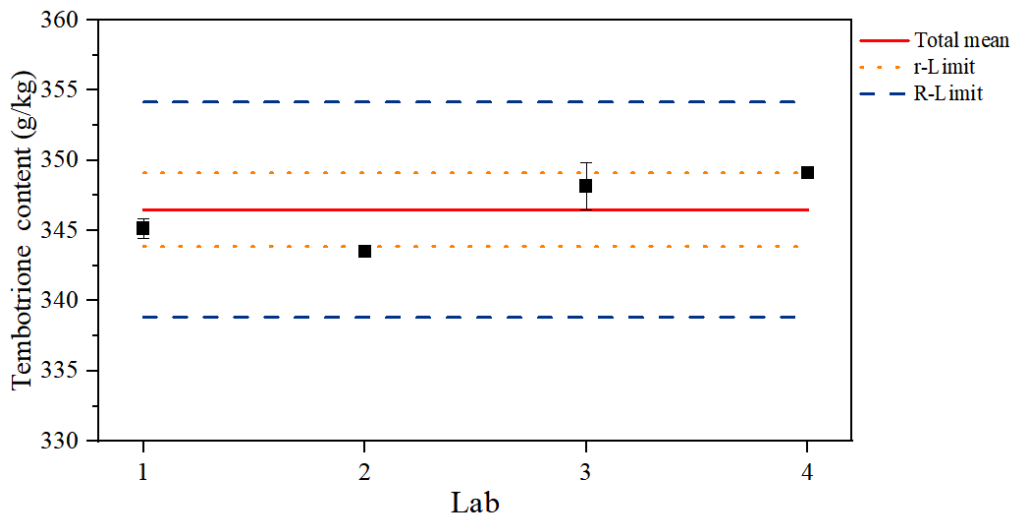


Figure 3. Graphical presentation of SC1 data

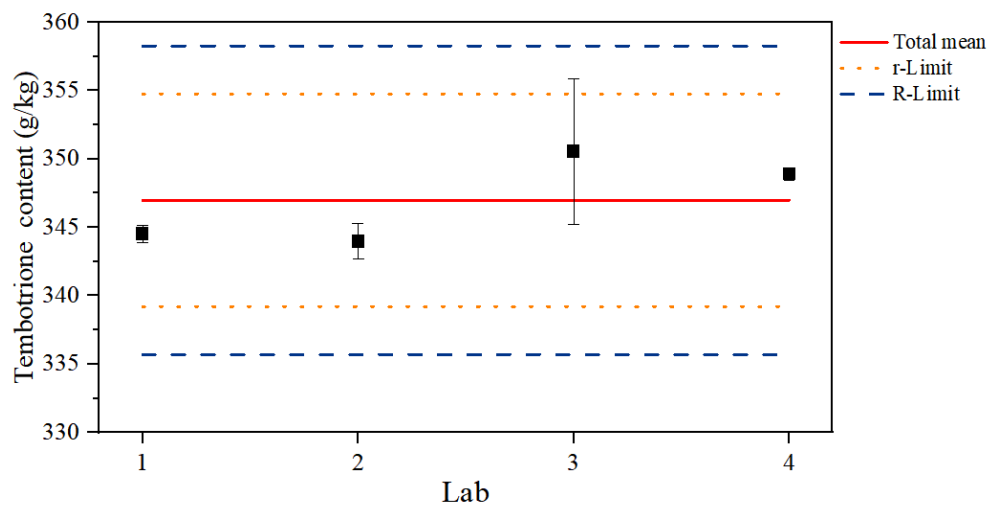


Figure 4. Graphical presentation of SC2 data

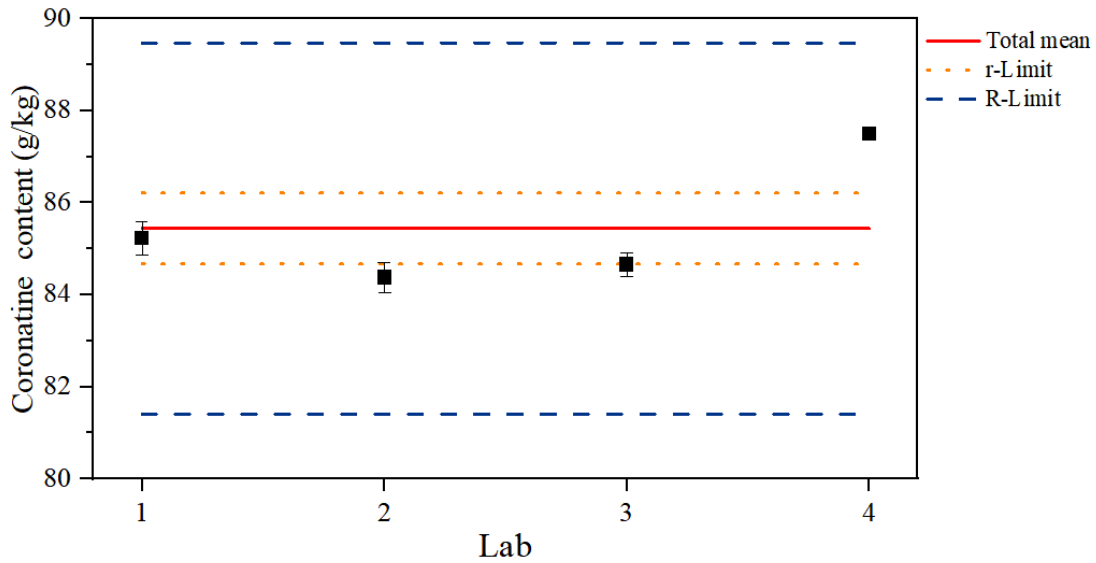


Figure 5. Graphical presentation of OD1 data

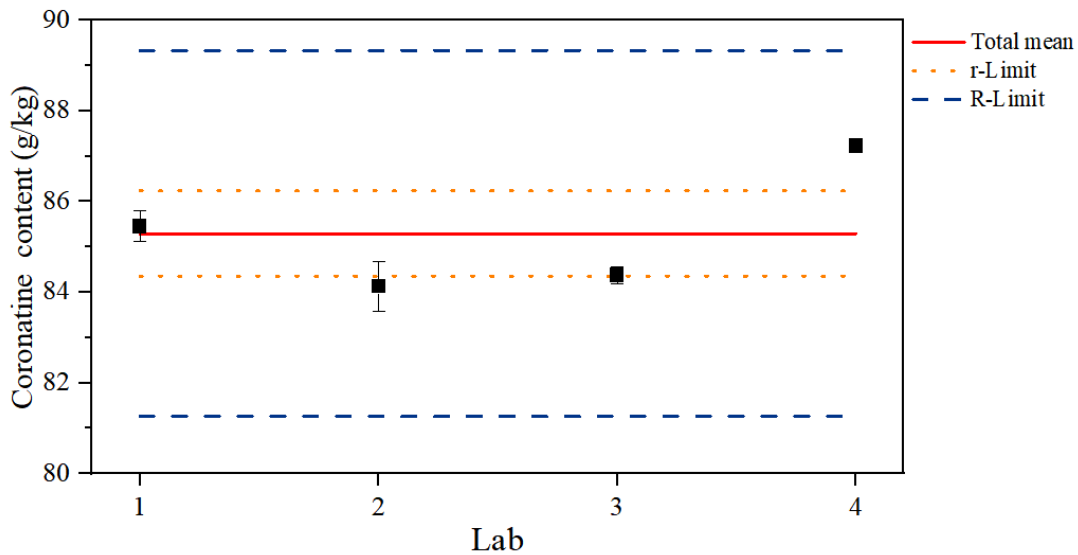


Figure 6. Graphical presentation of OD2 data

Table 7 Statistics of the results of TC1

$S_1 = \text{Sum } Y_i$	3841.3000		
$S_2 = \text{Sum } Y_i^2$	3688942.9975		
$S_3 = \text{Sum } S_i^2$	6.0767		
No. Lab P	4		
No. Determination n	4		
Average $Y = S_1/P$	960.3250		
$S_r^2 = S_3/P$	1.5192	$S_r$	1.2325
$S_L^2 = [(P \cdot S_2 - S_1^2)/P(P-1)] - S_r^2/n$	15.1452	$S_L$	3.8917
$S_R^2 = S_r^2 + S_L^2$	16.6644	$S_R$	4.0822
Repeatability $r = 2.8 \cdot S_r$	3.4511		



Reproducibility $R=2.8 \cdot S_R$	11.4302
Relative Standard Deviation of Repeatability $RSD_r=S_r \cdot 100/Y$	0.1283
Relative Standard Deviation of Reproducibility $RSD_R=S_R \cdot 100/Y$	0.43
Horwitz $RSD_R(\text{Hor})=2^{1-0.5 \cdot \log(Y/1000)}$	2.01
HorRat	0.21

**Table 8 Statistics of the results of TC2**

$S_1=\text{Sum } Y_i$	3835.4000		
$S_2=\text{Sum } Y_i^2$	3677695.8863		
$S_3=\text{Sum } S_i^2$	6.2317		
No. Lab P	4		
No. Determination n	4		
Average $Y=S_1/P$	958.8500		
$S_r^2=S_3/P$	1.5579	$S_r$	1.2482
$S_L^2=[(P \cdot S_2 - S_1^2)/P(P-1)] - S_r^2/n$	40.4759	$S_L$	6.3621
$S_R^2=S_r^2+S_L^2$	42.0339	$S_R$	6.4834
Repeatability $r=2.8 \cdot S_r$	3.4949		
Reproducibility $R=2.8 \cdot S_R$	18.1534		
Relative Standard Deviation of Repeatability $RSD_r=S_r \cdot 100/Y$	0.1302		
Relative Standard Deviation of Reproducibility $RSD_R=S_R \cdot 100/Y$	0.68		
Horwitz $RSD_R(\text{Hor})=2^{1-0.5 \cdot \log(Y/1000)}$	2.01		
HorRat	0.34		

**Table 9 Statistics of the results of SC1**

$S_1=\text{Sum } Y_i$	1385.9500		
$S_2=\text{Sum } Y_i^2$	480234.8688		
$S_3=\text{Sum } S_i^2$	3.5083		
No. Lab P	4		
No. Determination n	4		
Average $Y=S_1/P$	346.4875		
$S_r^2=S_3/P$	0.8771	$S_r$	0.9365
$S_L^2=[(P \cdot S_2 - S_1^2)/P(P-1)] - S_r^2/n$	6.6201	$S_L$	2.5730
$S_R^2=S_r^2+S_L^2$	7.4972	$S_R$	2.7381

Repeatability $r=2.8*S_r$	2.6223
Reproducibility $R=2.8*S_R$	7.6667
Relative Standard Deviation of Repeatability $RSD_r=S_r*100/Y$	0.2703
Relative Standard Deviation of Reproducibility $RSD_R=S_R*100/Y$	0.79
Horwitz $RSD_R(Hor)=2^{1-0.5*\log(Y/1000)}$	2.35
HorRat	0.34

**Table 10 Statistics of the results of SC2**

$S_1=Sum Y_i$	1387.9500		
$S_2=Sum Y_i^2$	481632.7888		
$S_3=Sum S_i^2$	30.8283		
No. Lab P	4		
No. Determination n	4		
Average $Y=S_1/P$	346.9875		
$S_r^2=S_3/P$	7.7071	$S_r$	2.7762
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n$	8.5693	$S_L$	2.9273
$S_R^2=S_r^2+S_L^2$	16.2764	$S_R$	4.0344
Repeatability $r=2.8*S_r$	7.7733		
Reproducibility $R=2.8*S_R$	11.2963		
Relative Standard Deviation of Repeatability $RSD_r=S_r*100/Y$	0.8001		
Relative Standard Deviation of Reproducibility $RSD_R=S_R*100/Y$	1.16		
Horwitz $RSD_R(Hor)=2^{1-0.5*\log(Y/1000)}$	2.35		
HorRat	0.50		

**Table 11 Statistics of the results of OD1**

$S_1=Sum Y_i$	341.7500
$S_2=Sum Y_i^2$	29204.3138
$S_3=Sum S_i^2$	0.3017

No. Lab P	4		
No. Determination n	4		
Average $Y=S_1/P$	85.4375		
$S_r^2=S_3/P$	0.0754	$S_r$	0.2746
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n$	1.9972	$S_L$	1.4132
$S_R^2=S_r^2+S_L^2$	2.0726	$S_R$	1.4397
Repeatability $r=2.8*S_r$	0.7689		
Reproducibility $R=2.8*S_R$	4.0310		
Relative Standard Deviation of Repeatability $RSD_r=S_r*100/Y$	0.3214		
Relative Standard Deviation of Reproducibility $RSD_R=S_R*100/Y$	1.69		
Horwitz $RSD_R(Hor)=2^{1-0.5*\log(Y/1000)}$	2.90		
HorRat	0.58		

Table 12 Statistics of the results of OD2

$S_1=Sum Y_i$	341.1750		
$S_2=Sum Y_i^2$	29106.0594		
$S_3=Sum S_i^2$	0.4508		
No. Lab P	4		
No. Determination n	4		
Average $Y=S_1/P$	85.2938		
$S_r^2=S_3/P$	0.1127	$S_r$	0.3357
$S_L^2=[(P*S_2-S_1^2)/P(P-1)]-S_r^2/n$	1.9599	$S_L$	1.4000
$S_R^2=S_r^2+S_L^2$	2.0726	$S_R$	1.4397
Repeatability $r=2.8*S_r$	0.9400		
Reproducibility $R=2.8*S_R$	4.0310		
Relative Standard Deviation of Repeatability $RSD_r=S_r*100/Y$	0.3936		
Relative Standard Deviation of Reproducibility $RSD_R=S_R*100/Y$	1.69		
Horwitz $RSD_R(Hor)=2^{1-0.5*\log(Y/1000)}$	2.90		

HorRat	0.58
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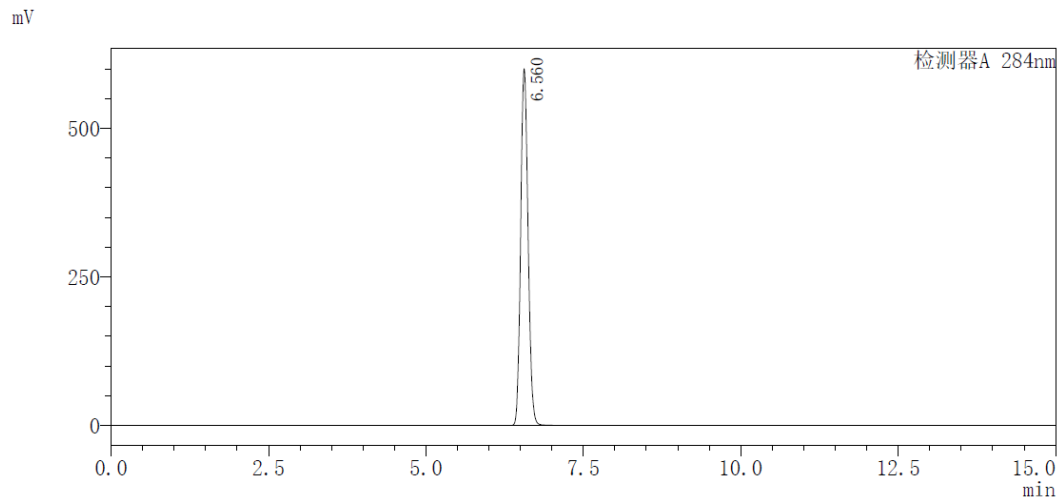


Figure 7. HPLC chromatogram of tembotrione standard (Lab 1)

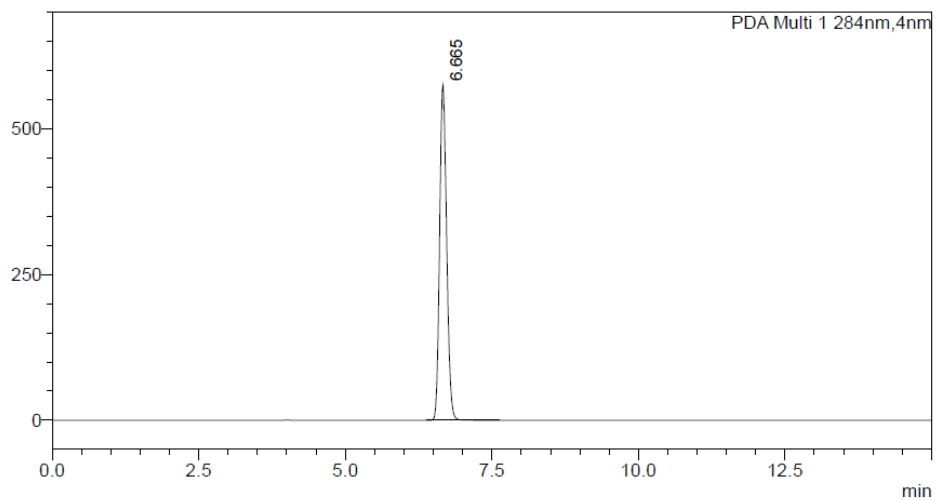


Figure 8. HPLC chromatogram of tembotrione standard (Lab 2)

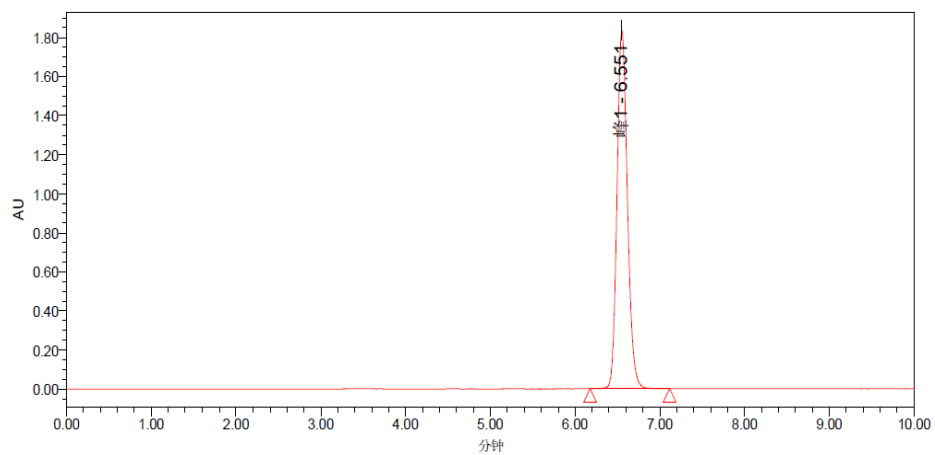


Figure 9. HPLC chromatogram of tembotrione standard (Lab 3)

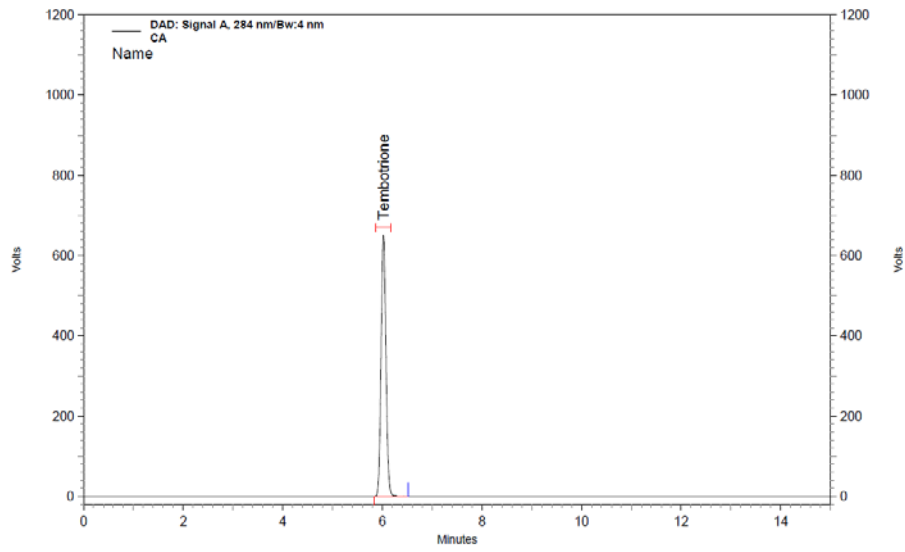


Figure 10. HPLC chromatogram of tembotrione standard (Lab 4)

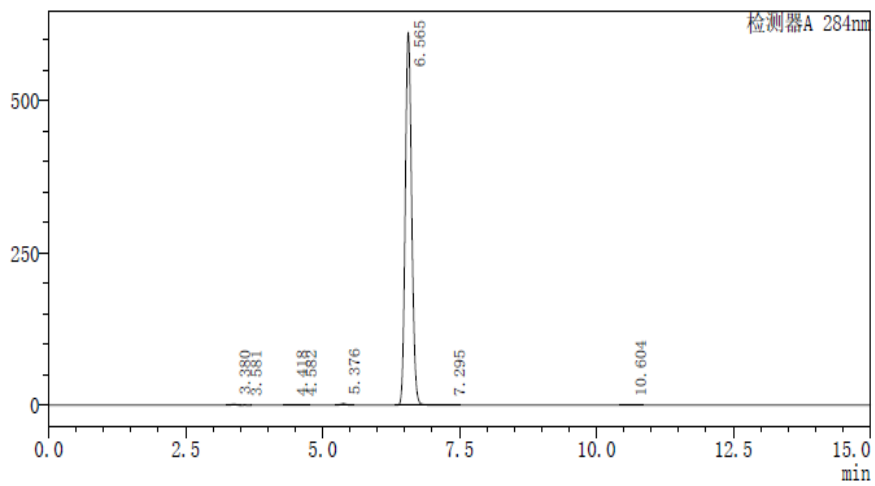


Figure 11. HPLC chromatogram of tembotrione TC (Lab 1)

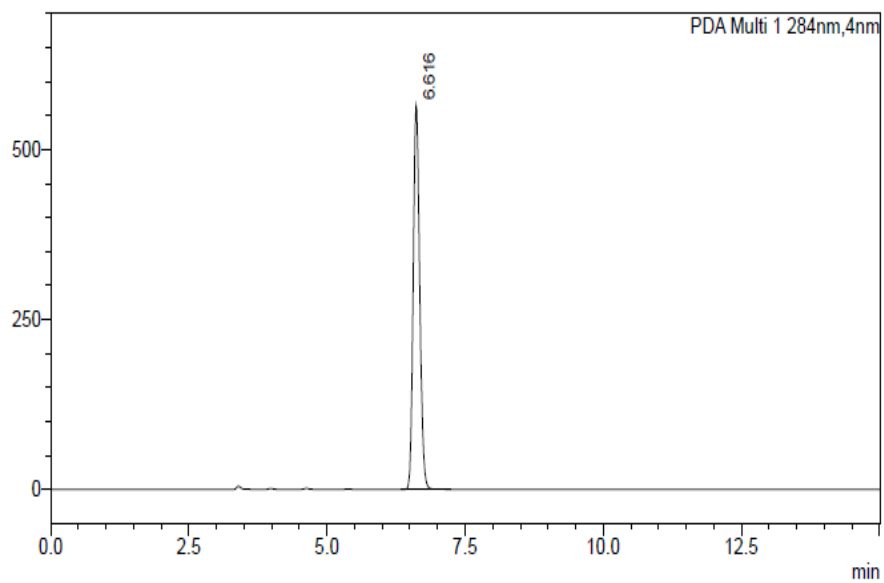


Figure 12. HPLC chromatogram of tembotrione TC (Lab 2)

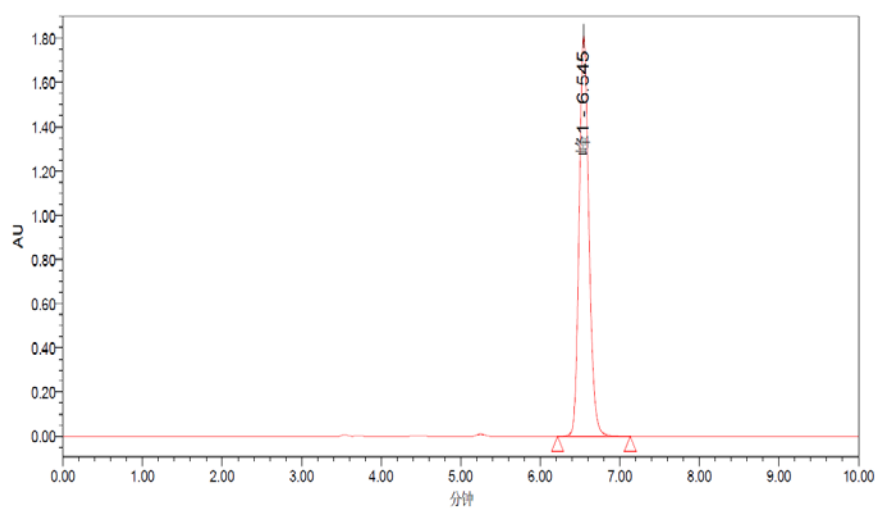


Figure 13. HPLC chromatogram of tembotrione TC (Lab 3)

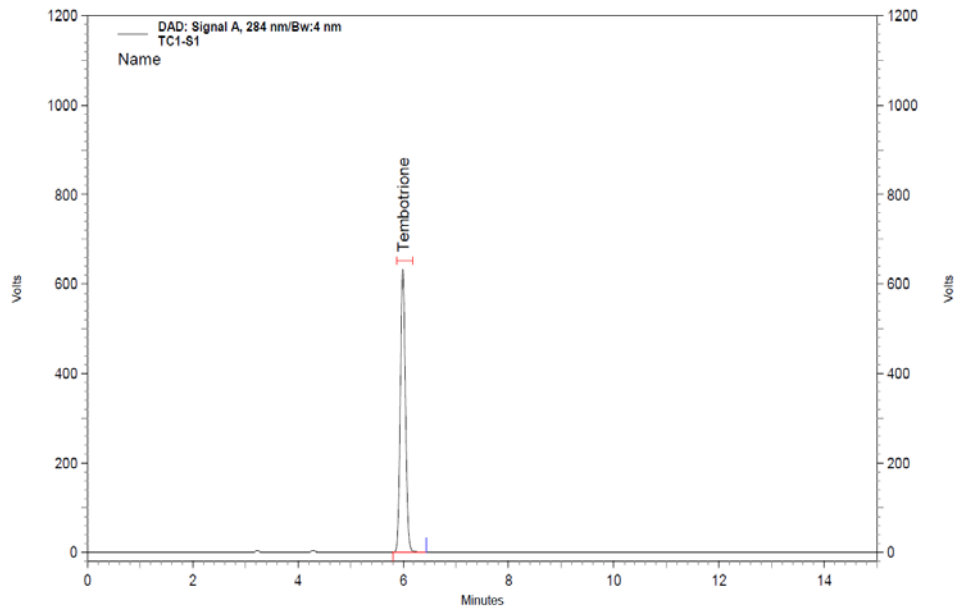


Figure 14. HPLC chromatogram of tembotrione TC (Lab 4)

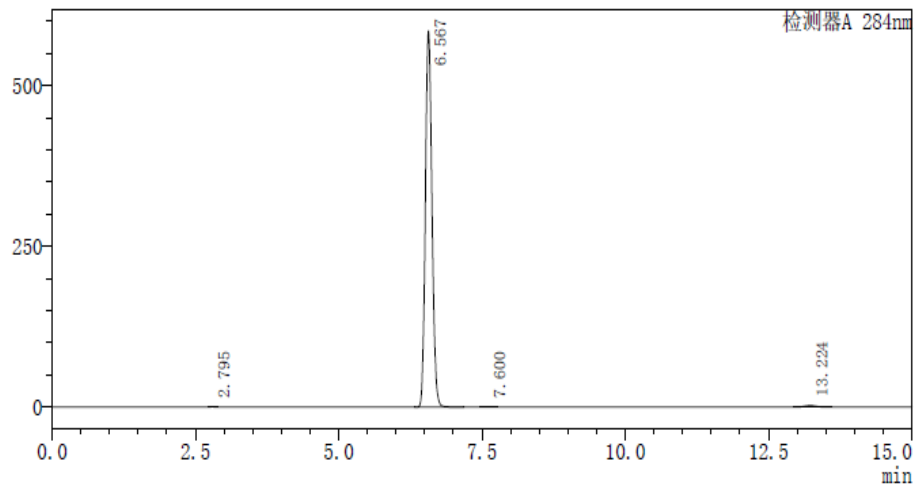
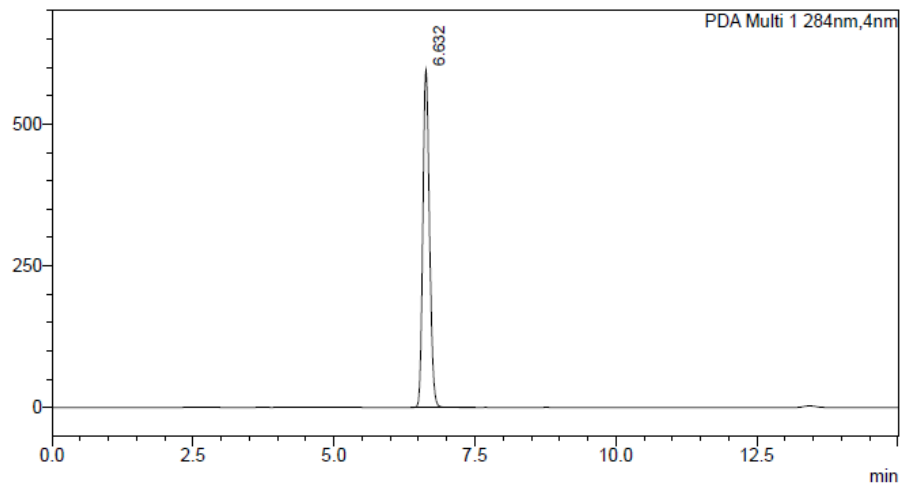
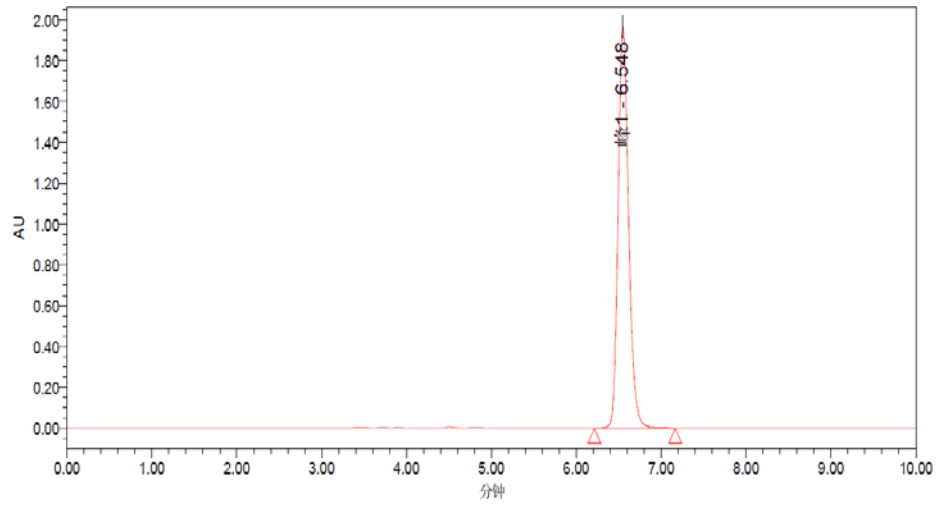
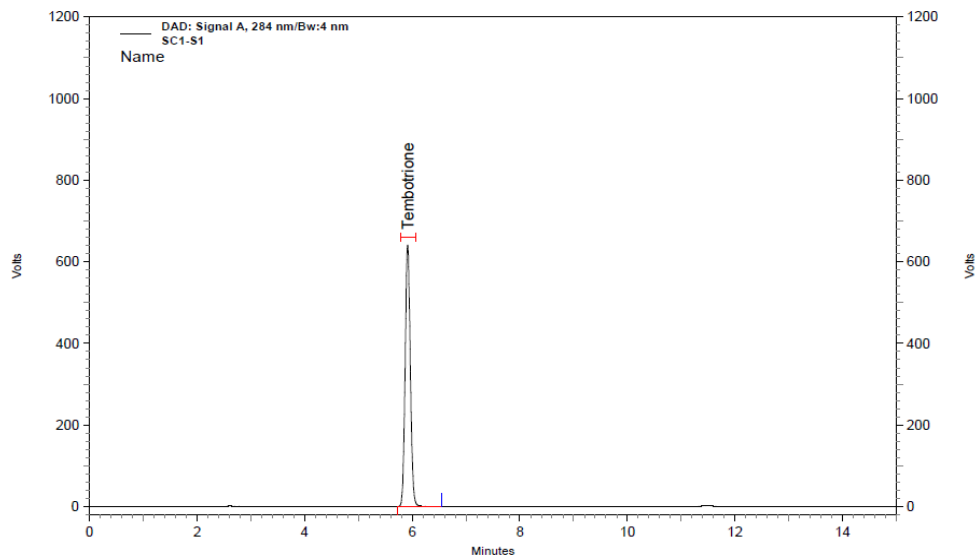


Figure 15. HPLC chromatogram of tembotrione SC (Lab 1)



**Figure 16. HPLC chromatogram of tembotrione SC (Lab 2)****Figure 17. HPLC chromatogram of tembotrione SC (Lab 3)****Figure 18. HPLC chromatogram of tembotrione SC (Lab 4)**



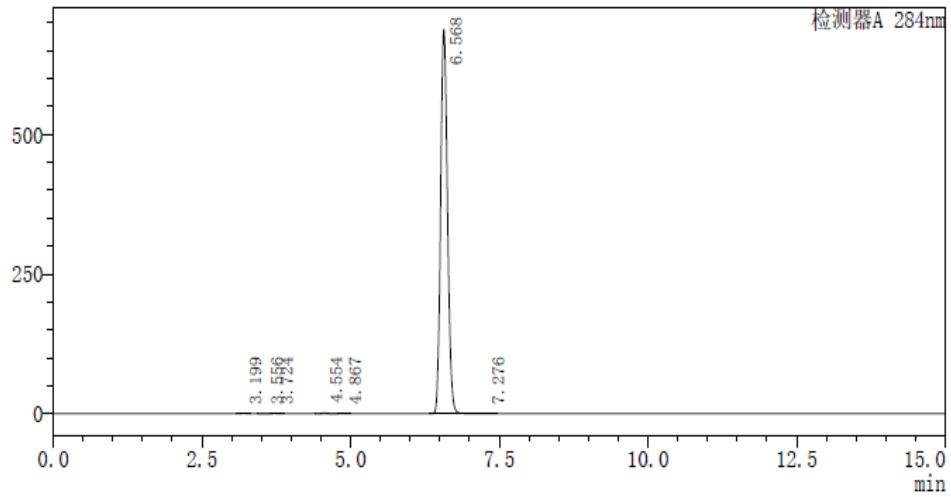


Figure 19. HPLC chromatogram of tembotrione OD (Lab 1)

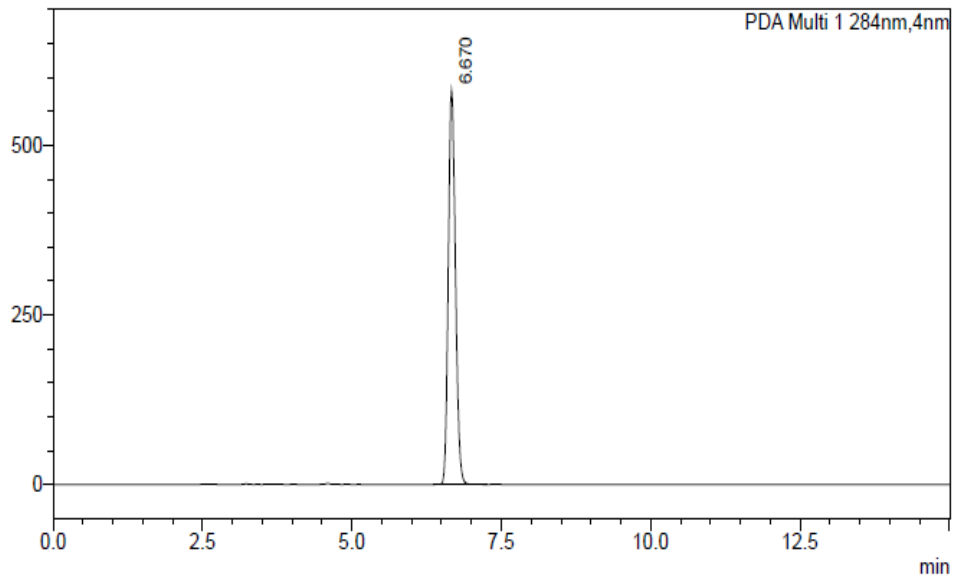


Figure 20. HPLC chromatogram of tembotrione OD (Lab 2)

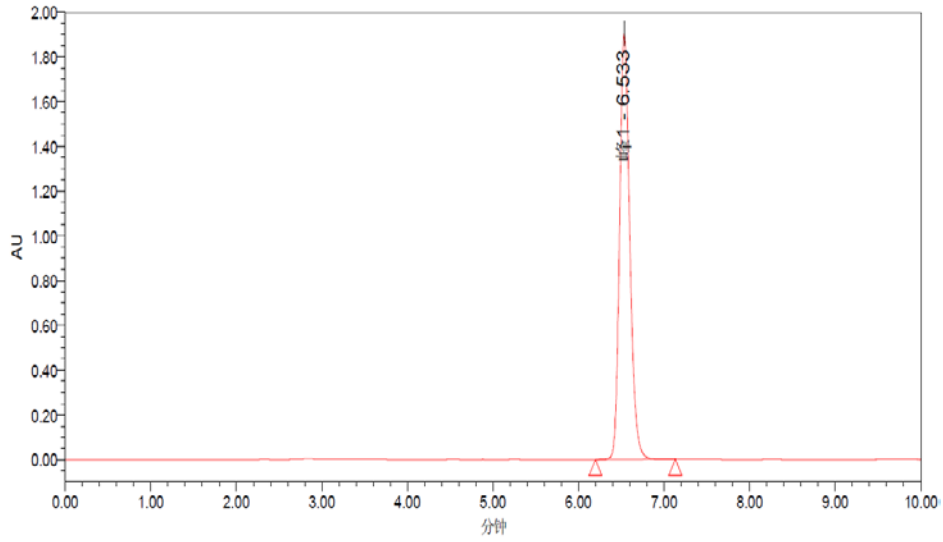


Figure 21. HPLC chromatogram of tembotrione OD (Lab 3)

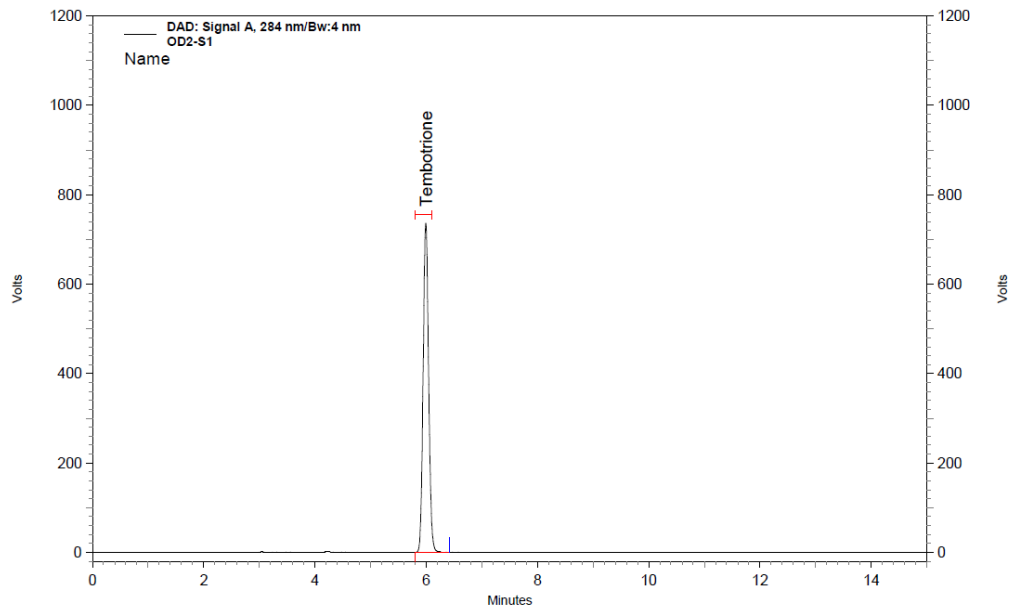


Figure 22. HPLC chromatogram of tembotrione OD (Lab 4)