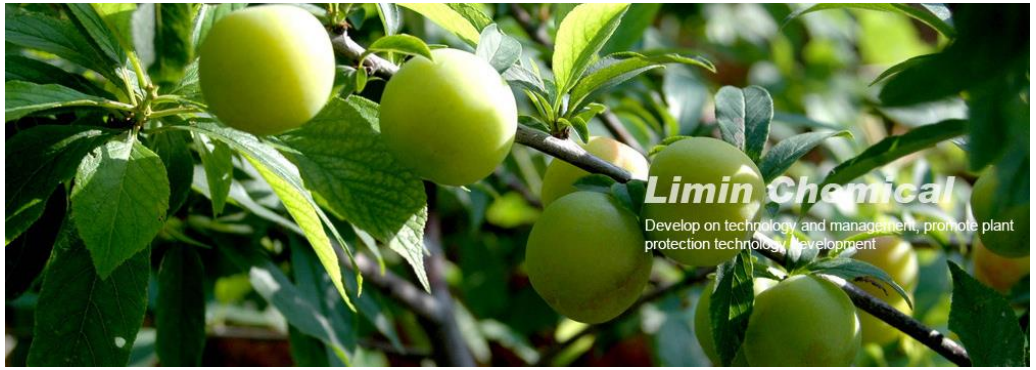
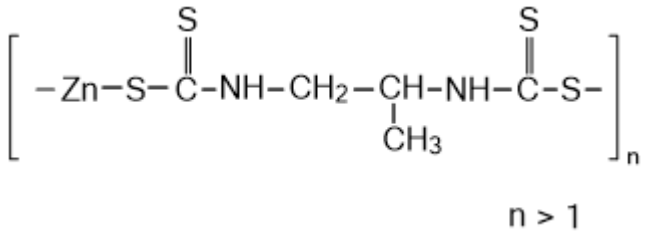
Limin Chemical Co., Ltd

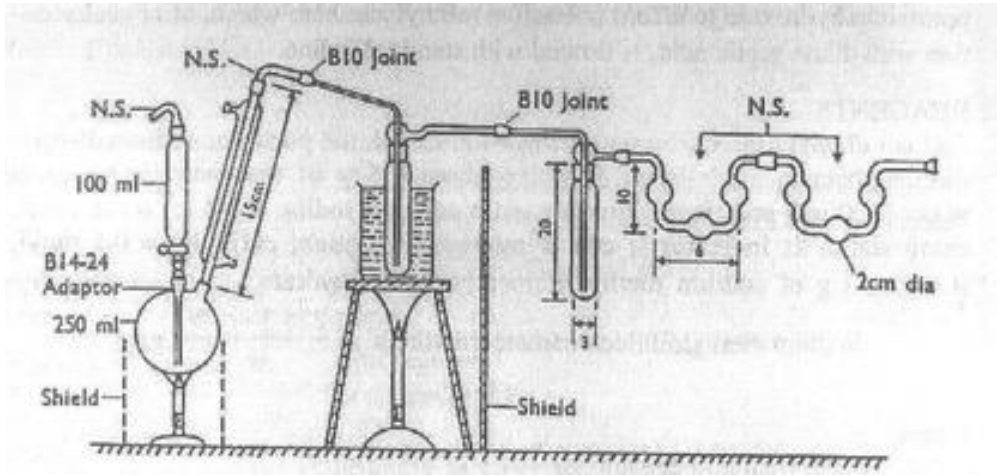


PROPINEB – Small-Scale CIPAC Collaborative Trial

General information of propineb

ISO common name	**propineb**
Structure	 n > 1
Molecular formula	$(C_5H_8N_2S_4Zn)x$
Relative molecular mass	289.8g/mol
CAS No	12071-83-9
Solubility	Insoluble in water and most organic solvents
Description	White powder
Stability	Decomposed under acid conditions
Formulation Type	Wettable powders, water dispersible granules, suspension concentrates and dustable powders
Activity	Fungicide

Background of the study

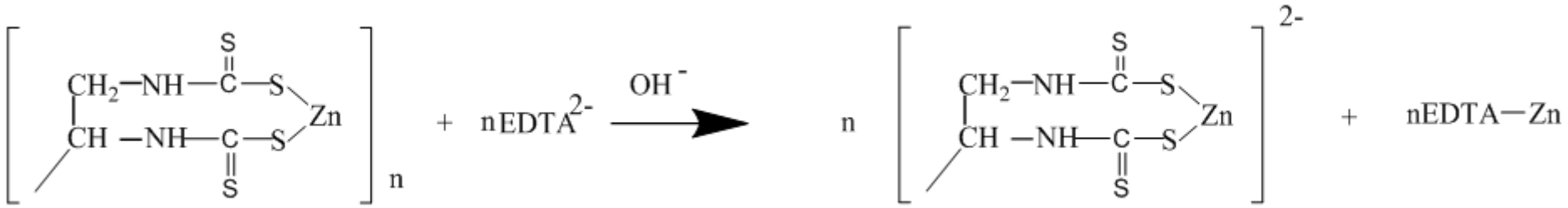


- Time-consuming
- Use hazardous reagents
- Release toxic substance

VS

- Easy operation
- Faster
- Environmental friendly

Background of the study



Participants

Participant	Contact
Zhejiang KBchem Testing Co., Ltd.	Haisheng Miao
Jiangsu Agricultural Product Quality Inspection and Testing Center	Tang Huimin
Pesticides Test Laboratory of Shenyang Research	Mei Baogui
Nutrichem Laboratory Co., Ltd	Jing Gao
Limin Chemical Co., Ltd.	Xu Mei

Analytical Method

✓ Recommended Conditions	
Equipment	HPLC
Column	Agilent Extent C18 (4.6×150mm, 5μm)
Flow rate	1.0 ml/min
Column temperature	20℃
Wavelength of detector	280nm
Injection volume	5μl
Environment temperature	20±2℃
Mobile phase	Solution A- Methaol =65:35 (V/V)
Run time	13 min

✓ Samples	
Propineb Technical	TC-1
	TC-2
Propineb WP	WP-1
	WP-2
	WP-3
Reference standard	purity 90.0% (w/w) calibrated by commercial standard(Dr.Ehrenstorfer, Germany) using CIPAC method.

✓Protocol
The samples were analyzed on two different days with duplicate injections of two weighing per sample. Test and reference solutions were prepared freshly on each day.
Test and reference solutions were prepared fresh on each day.
The four injections of each test solution were bracketed by single injections of the calibration solution. The average response factor, used to calculate the amount of propineb in the test solution, was calculated using the injection before and after the test injections.

Analytical Method

Praperation of Solutions

Solution A	Weigh 3.72g EDTA, 1.42g disodium hydrogen phosphate, and 3.39g tetrabutylammonium hydrogen sulfate, dissolve them in 1000 mL water, and use saturated sodium hydroxide solution to prepare solution of pH 10.0. After sonication, filter the solution with 0.22μm membrane for future use.
Solution B	Weigh 7.44 g EDTA, 1.42 g disodium hydrogen phosphate and 3 g sodium sulfite, dissolve them in 1000 mL water, and use saturated sodium hydroxide solution to prepare solution of pH 11.0. After sonication, let stand for 30 min for future use.
Calibration solution	Weigh Propineb standard containing around 40mg (accurate to 0.2mg) pure Propineb accurately, place the sample into a 100 mL volumetric flask, add 85 mL solution B, and use ultrasound around 5 min to dissolve. Restore to room temperature and fill to the volume with the solution B and mix thoroughly. Transfer 5 mL into 50 mL volumetric flask accurately, dilute to the mark with solution B and mix thoroughly, and filter it with 0.22μm membrane for future use.
Test item solution	Weigh the test item containing around 40mg (accurate to 0.2mg) pure Propineb accurately, place the sample into a 100 mL volumetric flask, add 85 mL buffer solution B, and using ultrasound around 5 min to dissolve. Restore to room temperature and fill to the volume with the solution B and mix thoroughly. Transfer 5 mL into a 50 mL volumetric flask accurately, dilute to the mark with solution B and mix thoroughly, and filter it with 0.22μm membrane for future use.
Determination	Inject 5μL of the calibration solution C1 and repeat the injections until retention times and peak areas deviate by less than ± 0.5% from the mean for 3 successive injections. Then carry out determination according to the sequence of calibration solution, test item solution, test item solution and calibration solution.

Data Evaluation

- 5 laboratories' data was included within the statistical assessment
- The statistical evaluations of the data were carried out according to ISO 5725.
- Use Grubbs test and Cochran's test to identify outliers.

Summary of Statistical Results

Sample	Lab	Day 1		Day 2		Mean (g/kg)	Std. Dev.
		1	2	1	2		
TC-1	1	896.6	893.6	895.9	893.1	894.8	1.72
	2	891.2	896.2	892.7	897.4	894.4	2.91
	3	901.3	901.5	897.1	901.1	900.2	2.11
	4	899.3	902.0	903.0	899.4	900.9	1.85
	5	896.2	903.4	902.8	900.9	900.8	3.28
TC-2	1	892.4	894.6	884.8	889.4	890.3	4.21
	2	890.6	896.8	890.1	895.8	893.3	3.46
	3	901.8	898.7	894.4	895.5	897.6	3.33
	4	901.2	902.8	904.4	901.2	902.4	1.53
	5	897.8	898.8	898.1	903.5	899.6	2.66

- There was no outliers according to Cochran test and Grubbs test.

Sample	Lab	Day 1		Day 2		Mean (g/kg)	Std. Dev.
		1	2	1	2		
WP-1	1	693.8	698.3	698.9	700.3	697.8	2.79
	2	706.7	707.2	698.1	703.2	703.8	4.19
	3	709.8	702.9	712.7	702.1	706.9	5.20
	4	711.9	710.2	709.2	708.6	710.0	1.47
	5	706.2	708.6	705.8	702.6	705.8	2.46
WP-2	1	696.3	695.6	702.2	701.2	698.8	3.37
	2	704.8	706.9	695.7	700.0	701.9	5.05
	3	711.5	712.3	713.4	714.3	712.9	1.23
	4	709.2	711.4	709.3	705.7	708.9	2.35
	5	703.1	707.5	708.5	704.0	705.8	2.63
WP-3	1	692.2	694.0	701.4	691.7	694.8	4.51
	2	706.4	705.7	699.7	699.6	702.9	3.72
	3	711.0	710.6	708.4	711.9	710.5	1.49
	4	708.3	710.8	708.3	709.7	709.3	1.19
	5	705.5	702.7	705.2	707.3	705.2	1.88

Summary of Statistical Results

Fig.1: TC-1

Fig.2: TC-2

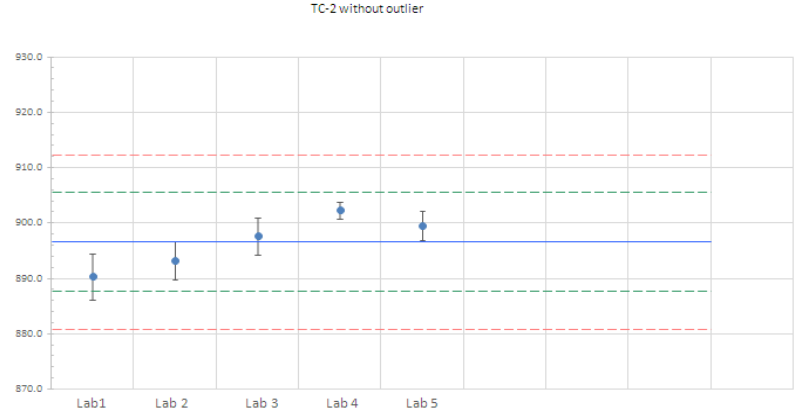


Fig.3: WP-1

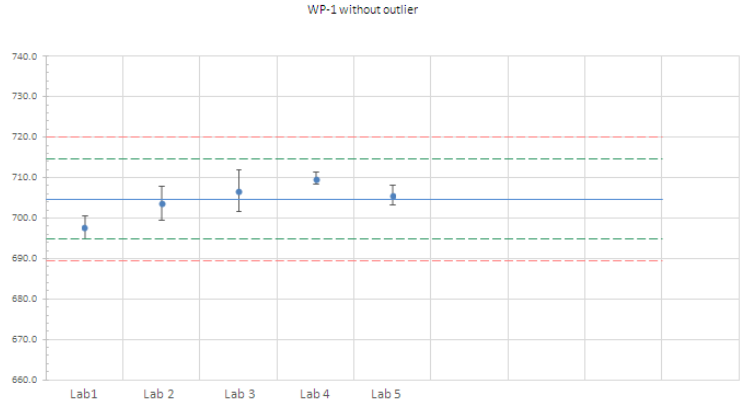


Fig.4: WP-2

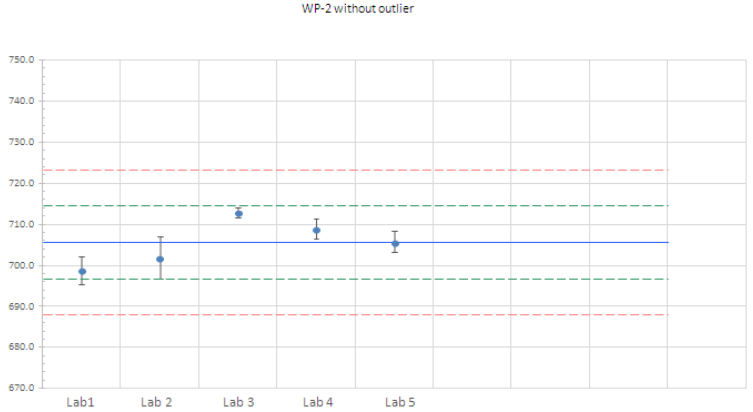
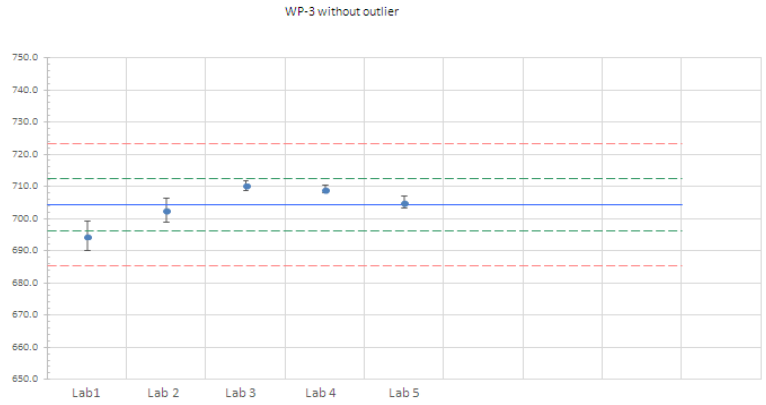


Fig.5: WP-3



Summary of the results of all laboratories

	TC-1	TC-2	WP-1	WP-2	WP-3
Xm	898.2	896.6	704.9	705.7	704.5
L	5	5	5	5	5
Sr	2.45	3.17	3.48	3.19	2.88
SL	3.11	4.57	4.18	5.32	6.06
SR	3.96	5.56	5.44	6.21	6.71
r	6.94	8.96	9.85	9.03	8.15
R	11.10	15.74	15.39	17.56	18.99
RSDr	0.27	0.35	0.49	0.45	0.41
RSDR	0.44	0.62	0.77	0.88	0.95
RSDR (Hor)	2.03	2.03	2.11	2.11	2.11

Xm	=	Overall sample mean, in unit of g/kg
L	=	Number of laboratories
Sr	=	Repeatability standard deviation
S_L	=	“pure” between laboratory standard deviation
S_R	=	reproducibility standard deviation
r	=	Repeatability limit
R	=	Reproducibility limit
RSDr	=	relative repeatability standard deviation
RSDR	=	relative reproducibility standard deviation
RSDR(Hor)	=	relative reproducibility standard deviation (Horwitz equation)

Conclusions

- The results showed that HorRat value is 0.22，0.30，0.36，0.42 and 0.45 for TC-1, TC-2, WP-1, WP-2 and WP-3 respectively. The modified method could determine the content accurately.
- Limin Chemical Co. Ltd. consider this method to be suitable for the intended purpose. New HPLC method which is more environmental, more efficient.

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Many thanks to the participants of collaborative trials and CIPAC

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Q&A

Thanks!