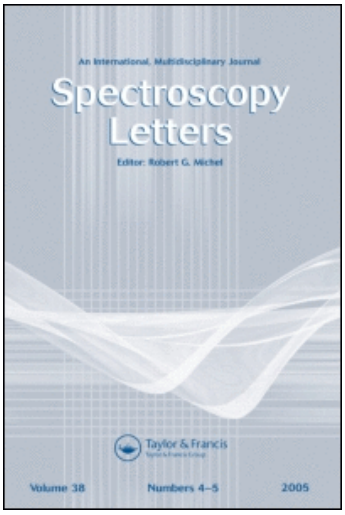

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UV-Spectroscopic Determination of Mixtures of Tetramethrin and Piperonyl Butoxide in Pharmaceutical Formulations

M. Sharaf El-din[a]; A. El-brashy[a]
[a] Department of Analytical Chemistry, Faculty of Pharmacy, Mansoura University, Mansoura, Egypt

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UV-SPECTROSCOPIC DETERMINATION OF MIXTURES OF TETRAMETHRIN AND PIPERONYL BUTOXIDE IN PHARMACEUTICAL FORMULATIONS

M. Sharaf El-Din and A. El-Brashy

Department of Analytical Chemistry, Faculty of Pharmacy, Mansoura University, Mansoura, Egypt.

ABSTRACT: A simple rapid and accurate UV-spectroscopic method have been developed for determination of tetramethrin in presence of piperonyl butoxide, which are present in Licid^R, a newly introduced pharmaceutical drug in Egypt used as antilice, and antiscopis. The method is applicable in the concentration range of 1-12 $\mu g.ml^{-1}$ and 1-50 $\mu g.ml^{-1}$ for tetramethrin and piperonyl-butoxide respectively. Average recoveries for tetramethrin and piperonyl butoxide in Licid^R spray and Licid^R lotion are 98.14 ± 0.3 - 103.88 ± 1.88 (Tetra), 103.78 ± 1.23 (piperonyl) and 100.62 ± 0.89 - 100.94 ± 1.10 (Tetra), 102.38 ± 1.02 (piperonyl) for the spray and lotion, respectively.

INTRODUCTION:

Synthetic pyrethroids have been proved recently to be broad spectrum insecticide. They are usually used in conjunction with a synergist such as piperonyl butoxide which increase its biological activity. In Egypt, Licid^R is a

newly introduced drug containing pyrethroid; tetramethrin, used against lice infestation and in the treatment of scopis, its efficiency as a caricide have been also proved (1). GLC was used for determination of pyrethroids in technical preparations and aerosol formulation (2-4). Densitometric method have also been reported (5).

This paper describes a simple, rapid and sensitive UV-spectroscopic method for the determination of tetramethrin and piperonyl butoxide in a mixture, the active substances of Licid^R, in pure form and pharmaceutical formulations.

EXPERIMENTAL:

Apparatus: Berkin Elmer 550 S Spectrophotometer.

Materials: Tetramethrin (Neo-Pynamin), piperonyl butoxide and Licid^R, spray and lotion were provided from Misr Co. for Pharm. Ind. Mataria, Cairo (each 100 gm contain 0.6 g tetramethrin and 2.4 g piperonyl butoxide. n-Hexane used as a solvent (BDH).

Procedure:

Prepare stock solution containing 1.0 mg.ml^{-1} of each tetramethrin and piperonyl butoxide in n-hexane. Diluted solutions are prepared by diluting these solutions, with n-hexane.

TABLE (1): Collective data of mixtures of tetramethrin and piperonyl butoxide.

Ratio Tetra/Piperonyl	Concentration Range, ($\mu g.ml^{-1}$)		Regression equation $A = a + bc$*
	Tetra	Piperonyl	
1:0.0	2-10	-	A = 0.00480 + 0.08960 C
0.0:1	-	5-50	A = 0.00427 + 0.01495 C
1:1	2-8	2-8	A = -0.01826 + 0.05439 C
1:2	2-8	4-16	A = 0.01390 + 0.03797 C
1:3	1.20- 4.80	3.60-14.40	A = -0.01150 + 0.03271 C
1:4	0.80- 8.00	3.20-32.00	A = -0.00327 + 0.02117 C
2:1	2.80-10.80	1.40- 5.40	A = 0.02016 + 0.05750 C
3:1	2.40-11.25	0.80- 3.75	A = 0.02730 + 0.06357 C
4:1	3.84-11.52	0.96- 2.88	A = -0.02149 + 0.07171 C

* Where:

A = Absorbance at λ_{max}; 225 nm for tetramethrin and Total in the mixture and 285 nm for piperonyl butoxide.
a = Intercept.
b = Slope.
c = Concentration in $\mu g.ml^{-1}$.

Mixtures are prepared by mixing tetramethrin and piperonyl butoxide in different ratios using n-hexane.

Calibration Curve:

Pipet an aliquot volume containing tetramethrin or piperonyl butoxide or mixture according to the amounts given in Table (1) into a 10 ml volumetric flask, complete to the volume with n-hexane and measure the absorbance at 285 nm for piperonyl butoxide and at 225 nm for tetramethrin alone or mixtures containing both tetramethrin and piperonyl butoxide in different ratios. Plot the absorbance versus concentration using n-hexane as a blank.

Procedure for pharmaceutical formulations:

A. Spray: The propellent is allowed firstly to release slowly and causionally, the concentrate is heated lightly at 40°C to release the last traces of the propellent. The concentrate is treated then as if it is lotion solution.

B. Lotion: Pipet an aliquot volume of the lotion solution or concentrate obtained from the spray equivalent to 50 mg of the total tetramethrin and piperonyl butoxide in a 100 ml volumetric flask, complete to volume with n-hexane, dilute 10 ml of this solution to 100 ml in another volumetric flask with n-hexane. Pipet aliquots containing 67.5-360 µg in 10 ml volumetric flasks, complete to volume with n-hexane and measure the absorbance by either method I or method II.

Method I:

Measure the absorbance at 285 nm for piperonyl butoxide and at 225 nm for the total tetramethrin and piperonyl butoxide against n-hexane. Calculate the amounts of each piperonyl butoxide and the total amounts from the previous calibration curves; tetramethrin is determined by difference.

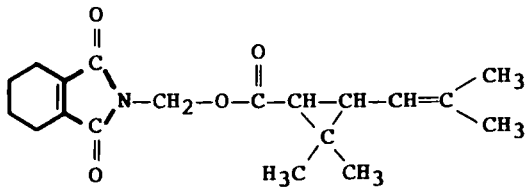
Method II:

Measure the absorbance at 285 nm for piperonyl butoxide (against n-hexane) and at 225 nm against the

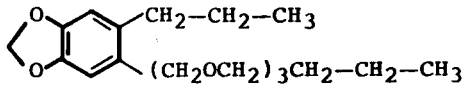
same amount of piperonyl butoxide in the mixture for tetramethrin only. The amounts of each two constituents are calculated from the previous caliberation curves (Fig. 2).

RESULTS AND DISCUSSION:

The newly introduced insecticidal drug, Licid^R, contains the synthetic pyrethroid, tetramethrin, N-(hydroxymethyl) cyclohexene 1,2-dicarboxymide 2,2-dimethyl-3 - 2-methylpropenyl cyclopropane, synergisted by piperonyl butoxide which enhances the efficiency of treatmethrin to obtain a prolong insecticidal action. These two constituents are present in Licid^R in the ratio of 1:4, tetramethrin : piperonyl butoxide.



Tetramethrin



Piperonyl butoxide

The usual method for the analysis of such preparation depends on GLC method. It was advantagiously to improve

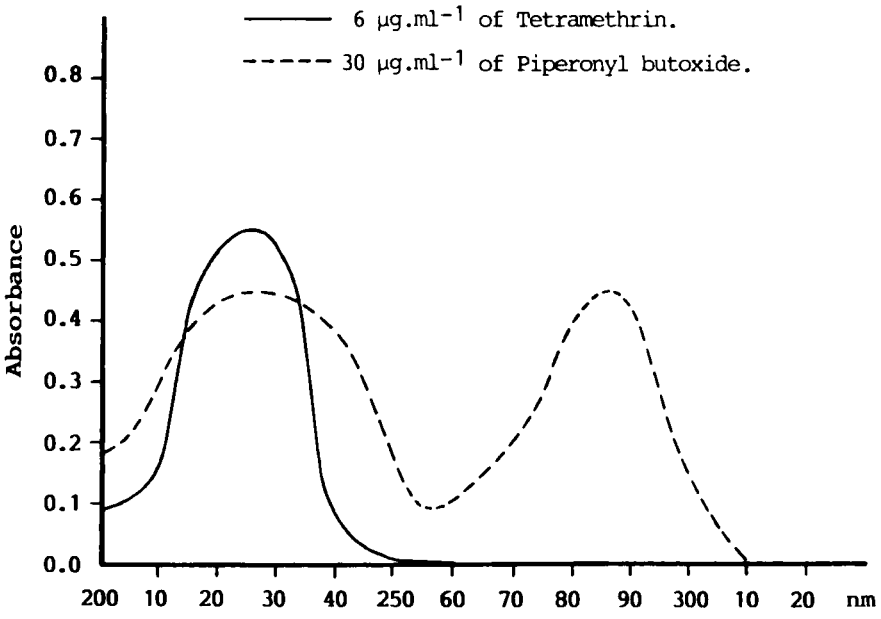


FIGURE (1): Absorption spectra of tetramethrin and piperonyl butoxide in n-hexane.

a simple method for its analysis. The UV-spectrum of tetramethrin shows a maximum absorbance at 225 nm while that of piperonyl butoxide shows two maxima at 285 and 230 nm (Fig. 1) with molar absorptivities 3×10^4 and 5.2×10^3 for the two compounds respectively. Two methods for analysis are adopted, the first method based on measuring the absorbance at 285 nm and the amount of piperonyl butoxide can be calculated (Fig. 2), and measuring the absorbance at 225 nm, the amount of the total tetramethrin

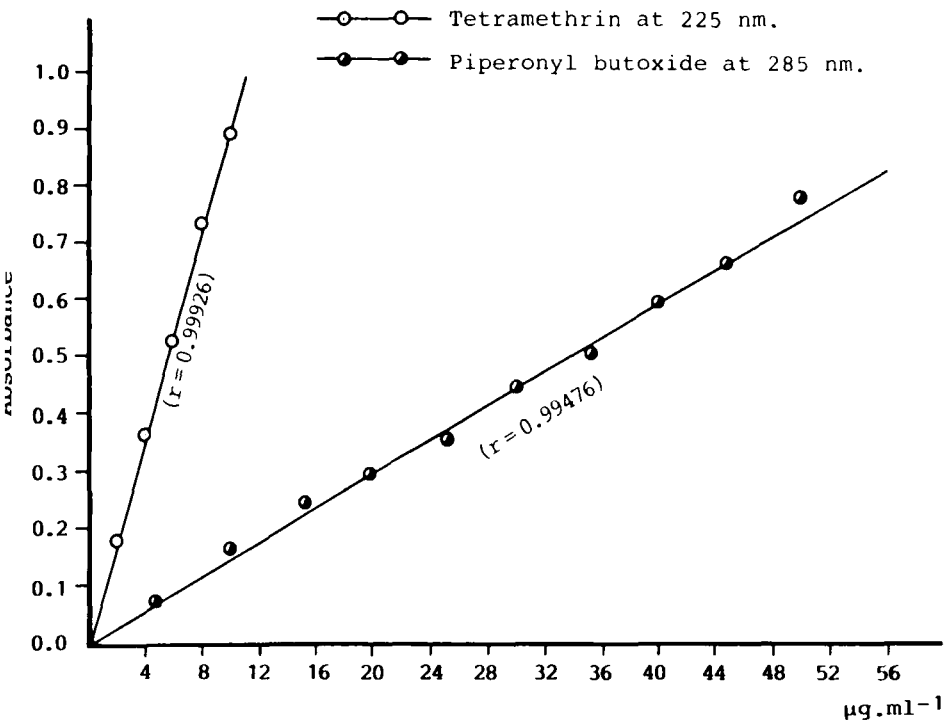


FIGURE (2): Calibration curves of tetramethrin and piperonyl butoxide

and piperonyl butoxide can also be calculated (Fig. 3, curve 4). Tetramethrin content can be calculated by difference. The second method based on determination of piperonyl butoxide by measuring the absorbance at 285 nm then measuring the absorbance at 225 nm against the same amount of piperonyl butoxide present in the mixture this measures the absorbance for tetramethrin only. The amount of each constituent can be calcualted from previously caliberated curve (Fig. 2).

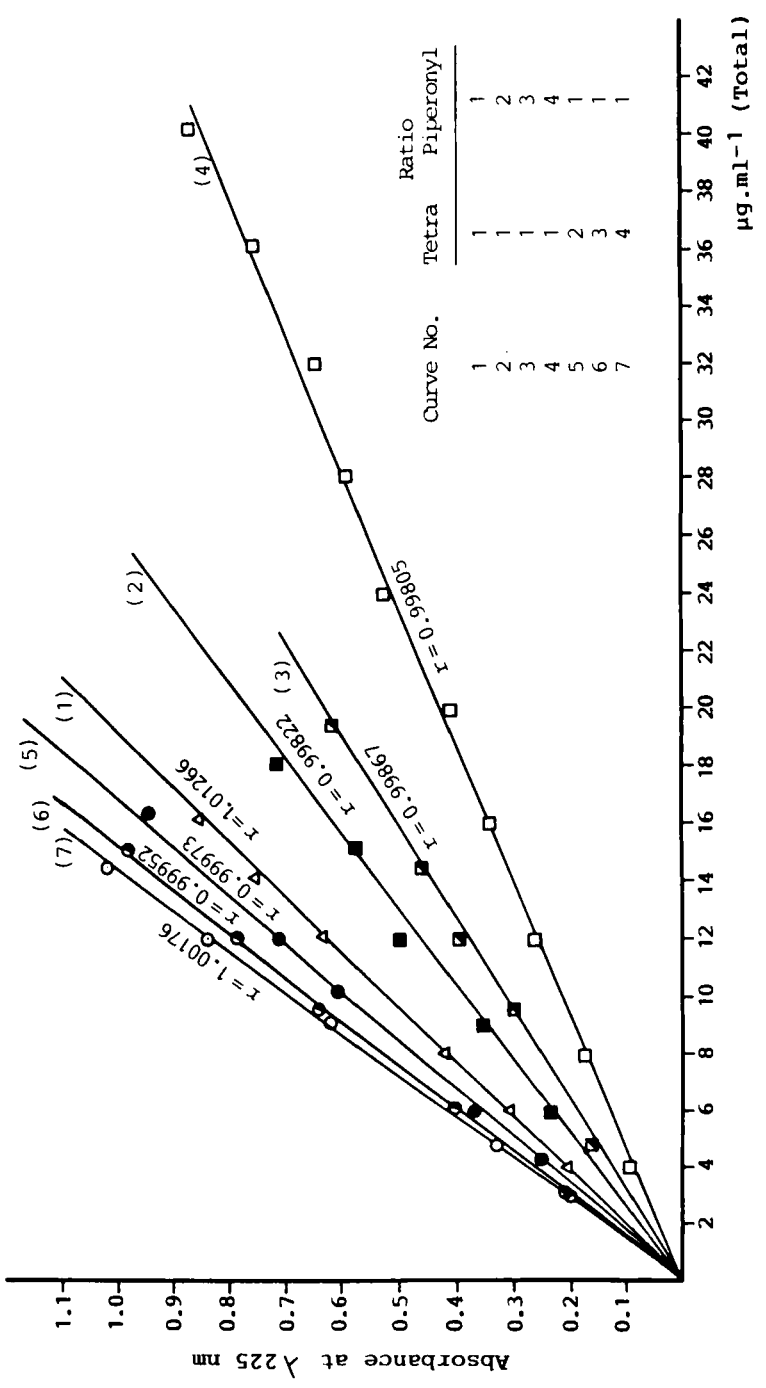


FIGURE (3): Calibration curves of the mixture in different ratios.

Table (2): Determination of Pharmaceutical Preparations.

Method I						Method II	
Amt. added (μg.)		Amt. found (μg.)		Rec., %*		Amt. found (μg.)	Rec., %*
Tetra	Piperonyl	Tetra	Piperonyl	Tetra	Piperonyl	Tetra	
1. Spray:							
1.35	5.4	1.39	5.50	102.96	101.85	1.32	97.72
2.70	10.8	2.73	11.24	101.11	104.04	2.65	98.05
4.05	16.2	4.25	17.02	104.94	105.08	3.99	98.44
5.40	21.6	5.72	22.78	105.93	104.44	5.32	98.42
6.75	27.0	7.05	27.94	104.44	103.49	6.62	98.08
Mean Rec., ± S.D**				103.88 ± 1.88	103.78 ± 1.23		98.14 ±0.30
2. Lotion:							
2.40	9.6	2.43	9.82	101.25	102.29	2.41	100.42
3.60	14.4	3.61	14.83	100.28	102.99	3.63	100.82
4.80	19.2	4.77	19.85	99.38	103.39	4.87	101.42
6.00	24.0	6.12	24.60	102.00	102.50	5.96	99.18
7.20	28.8	7.33	29.01	101.81	100.73	7.29	101.25
Mean Rec., ± S.D**				100.94 ± 1.10	102.38 ± 1.02		100.62 ± 0.89

Method I: Measurements at λ285 nm for piperonyl and 225 nm for total amounts, against n-hexane.
Method II: Measurements at λ225 nm against the same amount of piperonyl (for tetramethrin only).
* Mean of 3 determinations.
** Mean recovery ± Standard deviation.

The two components present in the mixture can be formulated in different ratios giving good linearity (Fig. 3) but in Licid^R the mixture presents in the ratio of 1:4, tetramethrin : piperonyl butoxide.

Table (1) shows the collective data for analysis of the mixture in different ratios.

Table (2) shows the results obtained for the analysis of pharmaceutical preparation Licid^R (spray and lotion) by the proposed method with accurate and convenient recoveries.

The proposed methods are time saving and require neither sophisticated instrument nor special skill, and provide a simple spectrophotometer which is an advantage in developing countries where preparations of such mixtures are most needed.

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