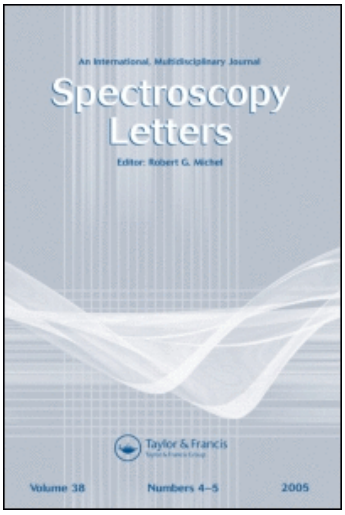

Spectroscopy Letters
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597299

U. V. Derivative Spectrophotometric Determination of Chlorphenoxamine Hydrochloride in Combination with Unhydrous Caffeine

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To cite this Article El-Henawee, Magda M. , Mohamed, H Saleh , El-bolkiny, N. and Ayad, Magda M.(1990) 'U. V. Derivative Spectrophotometric Determination of Chlorphenoxamine Hydrochloride in Combination with Unhydrous Caffeine', Spectroscopy Letters, 23: 3, 273 — 282
To link to this Article: DOI: 10.1080/00387019008054413
URL: http://dx.doi.org/10.1080/00387019008054413

SPECTROSCOPY LETTERS, 23(3), 273-282 (1990)

U.V. DERIVATIVE SPECTROPHOTOMETRIC DETERMINATION OF CHLORPHENOXAMINE HYDROCHLORIDE IN COMBINATION WITH UNHYDROUS CAFFEINE

Key words:
Chlorphenoxamine hydrochloride, unhydrous caffeine, pharmaceutical preparations, first and second derivative spectrophotometry.

BY

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ABSTRACT :

A rapid spectrophotometric method for the analysis of chlorphenoxamine hydrochloride in combination with unhydrous caffeine was described. The method is based on the determination of chlorphenoxamine hydrochloride by $\frac{d^2A}{d\lambda^2}$ *at 225 and 232 nm while unhydrous caffeine recorde zero at the same wave lengths. Unhydrous caffeine determined by* $\frac{dA}{d\lambda}$ *at 256 and 283 nm which corresponding to zero reading for chlorphenoxamine hydrochloride.*

INTRODUCTION :

The selectivity of derivative spectrometry has been used advantageously in pharmaceutical analysis to assay drugs with poorly developed maxima[1] *or when excipients, other active ingredients and degradation products in dosage forms interfered with the conventional spectrophotometric determination or when there existed spectral overlapping*[2-5].

Recently, determination of two component mixtures using derivative spectrometry have been reported using the solution of two simaltaneous equations[6] or using zero crossing technique[7].

This paper describes an application of derivative spectroscopy to the determinantion of both chlorphenoxamine hydrochloride and unhydrous caffeine in combination without previous separation.

The mixture of chlorphenoxamine hydrochloride and unhydrous caffeine was frequently used to treat hay fever, urticaria, serum sickness, angioneurotic oedema, pruritus of drug rashes and jaundice, contact dermatitis and insect bites.

MATERIALS AND METHODS :

Instrument :

Shiamadzu recording spectrophotometry uv. 260 with 1 cm quartiz cuvettes, suitable settings are scan speed 40 nm/min., shart speed 60 mm./min. and slite width 2 nm.

Materials :

Analytical standard grade of chlorphenoxamine hydrochloride, unhydrous caffeine (Epico Co. Egypt) were used. Alergex caffeine tablets (Epico Co. Egypt) labelled to containe 20 mg of chlorphenoxamine hydrochloride and 50 mg of unhydrous caffeine per tablet. Other reagents used were of analytical grade.

Calibration graphs :

a. Standard solution were prepared containing 20 mg, chlorphenoxamine hydrochloride per 100 ml distilled water, pipette 0.5 - 6.5 ml of the drug solution into 50 ml separate volumetric flasks, complete to the mark with distilled water $d^2A/d\lambda^2$, was recorded at 225, and 232 nm (Fig. II-A).

b. Standard solution of unhydrous caffeine were prepared containing 100 mg/100 ml distilled water, pipette 0.2-4.8 ml in 100 ml separate

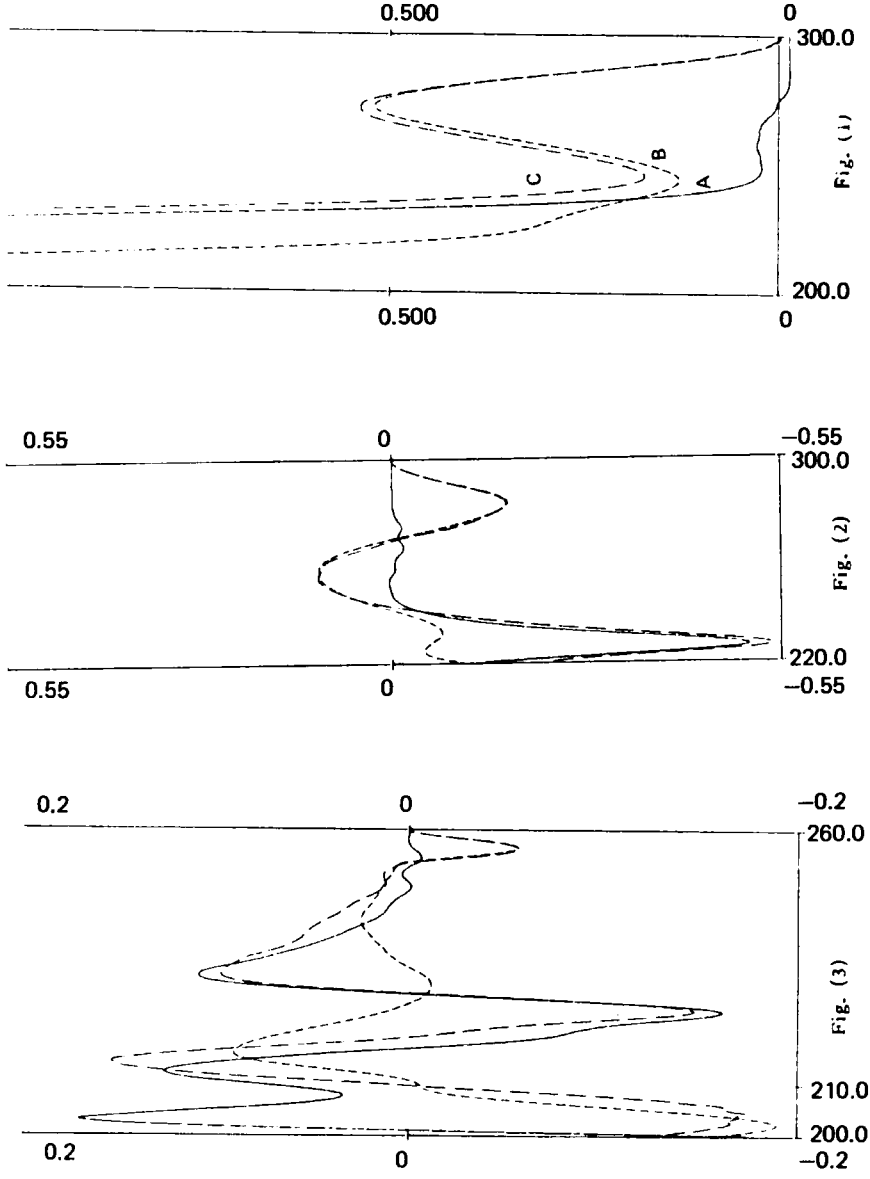

Fig. (1) : Zero absorption curve of (A) 3.2 mg% chlorphenoxamine hydrochloride ——— (B) 1 mg % unhydrous coffeine ---- and (C) Mixture from two concentrations——.——. **Fig. (2)** : 1st derivative spectroscopy of (A, B & C).
Fig. (3) : 2nd derivative spectroscopy of (A, B & C).

Table (I)

Determination of chlorphenoxamine hydrochloride by the proposed D_2 method at 225 & 232 nm. Compared with the official method:[8]

official method (8)		proposed D_2 method *		
mg % taken	% recovery	mg% taken	% recovery	
			D_2 at 225 nm	D_2 at 232 nm
1.6	131.8 *	0.2	77.44 *	106.5
4	94.25	0.4	95.14	97
8	98.63	0.6	104.45	97.83
12	101.83	0.8	103.98	98.4
16	99	1	101.7	101.19
20	97.25	1.2	100.08	98.92
40	95.6	1.4	100.43	100.79
60	99.85	1.6	100.72	100.7
-	-	1.8	99.78	100.61
-	-	2	100.06	100.55
-	-	2.2	100.27	100.50
-	-	2.4	99.63	100.42
-	-	2.6	99.08	98.47
mean	98.06		100.44	100.14
variance	6.642		5.6	5.45
SD	2.577		2.37	2.33
SE	0.974		0.684	0.645
F			1.186(4.74)	1.22(4.74)
T			2 (2.11)	1.78(2.101)

* average of three experiments.

Table (II)

Determination of unhydrous caffeine by the proposed D_1 method at 256 and 283 nm. Compared with the official method.[8]

mg % taken	% recovery *		
	offical method (8)	proposed D_1 method	
		256 nm.	283 nm.
0.2	-	102.83	98
0.4	99.5	97.96	99
0.6	-	97.46	99.5
0.8	97.88	100.75	101.38
1	-	99.2	97.7
1.2	99.08	101.58	99.83
1.4	-	98.93	98.93
1.6	100.94	99.5	100.31
1.8	-	99.94	100.33
2	100.05	99.75	99.97
2.2	-	100.55	100.27
2.4	100.29	-	-
2.6	-	-	-
2.8	101.18	100.43	100.47
3.2	100.22	100.82	100.06
3.6	98.44	99.96	100.07
4	99.75	-	-
4.8	-	99.54	99.38
		99.95	99.68
mean	99.73	99.95	99.68
N	10	15	15
variance	1.083	1.794	0.927
SD.	1.043	1.339	0.963
SE.	0.33	0.346	0.249
T	-	0.4603(2.069)	0.1174(2.069)
F	-	1.657	1.168

* average of three experiments

Table (III)

Determination of chlorphenoxamine hydrochloride by D_2 method at 225 and 232 nm. and unhydrous caffeine by D_1 at 256 and 283 nm. in a synthetic prepared mixture in the ratio of 2:5.

mg % taken	Chlorphenoxamine % recovary **		mg % taken	Unhydrous caffeine % recovary **	
	D_2 at 225 nm	D_2 at 232 nm		D_1 at 256 nm	D_1 at 283 nm.
	71.16 *	101.83	0.40	98	99
0.32	99.69	97.81	0.80	99.38	100.50
0.48	96.46	101.67	1.20	99.92	100.42
0.64	97.97	99.53	1.60	100.75	102
0.80	98.88	95.25	2.00	100.30	100.30
0.96	95.2	100.21	2.40	101.25	100.83
1.12	96.25	99.11	2.80	100.43	102.90
1.28	95.47	98.59	3.20	100.50	100.81
1.44	96.25	101.46	3.60	99.94	100.06
1.60	96.88	100.69	4.00	99.53	100.25
1.76	97.39	100.06	4.40	100.59	100.70
1.92	97.92	99.53	4.80	99.54	99.58
mean	97.12	99.645		100.01	100.61
N	11	12		12	12
variance	1.956	3.442		0.711	1.046
SD	1.399	1.855		0.843	1.023
SE	0.422	0.536		0.243	0.295
T					
F					

* rejected from calculation.

** average of three experiments.

Table (IV)

application of the proposed D_1 & D_2 methods to the determination of unhydrous caffeine and chlorphenoxamine hydrochloride in the ratio of 5:2 in allergex caffeine tablets.

D_2 of chlorphenoxamine hydrochloride			D_1 of unhydrous caffeine		
mg % taken	% recovery **		mg% taken	% recovery **	
	225 nm	232 nm.		256 nm	283 nm.
0.2	87.7*	94*	0.5	92.8*	94*
0.4	100.25	97	1	98.1	95.7
0.6	97.67	97.83	1.5	97.13	98.13
0.8	96.25	95.25	2	98.2	96.95
1	97.5	98.7	2.5	95.52	96.28
1.2	96.67	96.83	3	96.83	98.03
1.4	96.07	97.29	3.5	97.8	96.43
1.6	96.88	96.25	4	97.2	97.58
1.8	97.5	96.67	4.5	97.89	98.67
	97.35	96.98		97.33	97.22
N	8	8		8	8
variance	1.727	1.06		0.781	1.087
DS	1.314	1.03		0.884	1.043
SE	0.465	0.364		0.313	0.369

* rejected from calculation.

** average of three experiments.

Table (V)

Results of linear equations of the proposed methods.

		K	B	R	Range of mg %
D_1 of unhydrous caffeine	256 nm	10.344	- 0.0426	0.9998	0.2 -4.8
	283 nm	6.6794	- 0.0311	0.9998	0.2 -4.8
D_2 of chlorphen-oxamine hydrochloride	225 nm	20.515	-0.0913	0.9992	0.2-2.6
	232 nm	24.971	0.0131	0.9995	0.2-2.6

measuring flasks, complete to the mark with distilled water. Measur D_1 spectra at 256 and 283 nm against distilled water (Fig. II-B).

Preparation of pharmaceutical preparations for allergex caffeine tablets :

Powder and mix twenty tablets. An accurate weight equivalent to one tablet was transfered into 100 volumetric flask, about 70 ml distilled water were added, the contents of the flask were shaken, filter into 100 ml measuring flask and the volume was completed with distilled water to the mark, the procedures was then continued as described under calibration graph.

RESULTS AND DISCUSSION :

It is evident from Fig. I that the zero order absorption curves of chlorphenoxamine hydrochloride and unhydrous caffeine in distilled water were overlapped, it is also evident that chlorphenoxamine hydrochloride was weakly absorbed so we use high concentrations in the

official method, derivative spectroscopic technique dissolves such problems. Fig (II-A) show that chlorphenoxamine hydrochloride possece maximum D_2 value at 225 and 232 nm. while unhydrous caffeine at the same wave lengths show zero. On the other hand unhydrous caffeine had a maximum D_1 value at 256 and 283 nm while chlorphenoxamine hydrochloride record zero at the same wave lengths Fig. (II-B).

The plots of D_2 values of chlorphenoxamine hydrochloride at 225 and 232 nm and D_1 values for unhydrous caffeine at 256 and 283 nm against concentrations showed linear-relationship within the range of 0.2 - 2.6 mg% for chlorphenoxamine hydrochloride and 0.2 - 4.8 mg% for unhydrous caffeine, the four linear equations were tabulated in table (5).

In order to prove the validity and applicability of the proposed method, twelve synthetic mixtures of chlorphenoxamine hydrochloride and unhydrous caffeine were prepared and analysed using the proposed method. The concentration of the intact drugs in this mixtures were in the range 0.32 - 1.92 mg% of chlorphenoxamine hydrochloride and 0.4 - 4.8 mg% of unhydrous caffeine, table (III).

The mean percentage recovery was found 97.12 $\pm$ 0.442 and 99.65 $\pm$ 0.536 for chlorphenoxamine hydrochloride at 225 and 232 when determined by D_2 method table (II), and for caffeine was found to be that 100.01 $\pm$ 0.243 at 256 nm and 100.61 $\pm$ 0.295 at 283 nm table (III).

It is found that the proposed method was agreed well with the official method[8] for chlorphenxamine hydrochloride and unhydrous caffeine [8], table (I, II).

The proposede methods were applied to the determination of chlorphenoxamine hydrochloride and unhydrous caffeine in combina-

tion in the allergex caffeine tablet's, it gave good result' as shown in table IV.

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Date Received: 11/08/89
Date Accepted: 12/06/89