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Spectrophotometric Analysis of some Pharmaceuticals by the Absorbancy Ratios Method

Absorbans Oranı Metodu Yardımı ile Bazı İlâçların Spektrofotometrik Miktar Tayini

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The analysis of multicomponent systems requires multiple steps and often include special methods of separation. One of the first steps in the analysis of a pharmaceutical is the separation of one or more ingredients from the bulk form of the multicomponent system. This is carried out by solvent-solvent extraction process or by column chromatography. Sometimes the simultaneous analysis does not require the total speration, but a partial one is sufficient. Many methods are available for the analysis of mixtures. Spectrophotometric methods for the quantitation of compounds often employ a measurement of the absorbancy of solutions in the ultraviolet region.

A series of papers were published on the use of the absorbancy ratios technique and their application to the analysis of binary or ternary mixtures (1-14).

Pernarowski and his co-workers (1-7) have described the application of spectrophotometric assay for the multicomponent pharmaceuticals based on the absorbancy ratio, using the Q analysis method.

The present paper describes a spectrophotometric method, for the binary or ternary mixtures containing pheniramine, chlorpheniramine, brompheniramine and pyrilamine maleates with salicylamide, phenylpropanolamine, caffeine and phenacetine, based on the measurement of UV absorbancy of the mixtures on two wavelengths, using the absorbance maximum and isoabsorptive points.

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EXPERİMENTAL

Apparatus. VSU I Model Zeiss spectrophotometer was used.

Solutions. I) Standard solutions of pheniramine (PH), chlorpheniramine (CPH), brompheniramine (BPH), pyrilamine (P) maleates; salicylamide (SAL), phenylpropanolamine (PP), caffeine (C), phenacetine (PC) were prepared by dissolving 1,2,3 and 5 mg of each of the samples in water and diluting to 100 ml.

II) Binary and ternary mixtures (solutions in water): 1. pheniramine maleate-salicylamide, 2. chlorpheniramine maleate-salicylamide, 3. brompheniramine maleta-salicylamide, 4. pyrilamine maleate-salicylamide, 5. caffeine-salicylamide, 6. phenacetine-salicylamide, 7. a) pheniramine maleate-pyrilamine maleate, b) chlorpheniramine maleate-pyrilamine maleate, c) brompheniramine maleate-pyrilamine maleate, 8. salicylamide-phenacetine-chlorpheniramine maleate, 9. solutions prepared by adding phenylpropanolamine to the mixtures mentioned above.

Spectral characteristics of the substances. Solution of PH, CPH, BPH showed absorption maximum at 262 mμ, the absorption maxima for (P) occur at 250 mμ and 310 mμ, for SAL at 300 mμ, for PP 242 mμ, for C 274 mμ and for PC 246 mμ. For the mixtures containing SAL the chosen absorption maximum is 300.015 mμ and for the mixtures containing P is 309.85 mμ. This choice of these wavelengths results in optimum conditions and does not interfere with the other substances which absorbe at shorter wavelengths.

Locations of the isoabsorptive points. The isoabsorptive points were located first approximately by superimposing the spectra of each pair of the substances being analzyed, and then fixing the exact wavelength by comparing the solutions of the compounds with the interval of 0.002 mμ.

The isoabsorptive points determined for the mixtures are given below:

1 — 270.665 mμ

2 — 270.80 mμ

3 — 270.50 mμ

4 — 270.76 mμ

5 — 288.62 mμ

6 — 279.625 mμ

7 — a) 270.74 mμ

b) 270.58 mμ

c) 270.54 mμ

8 — 279.625 mμ

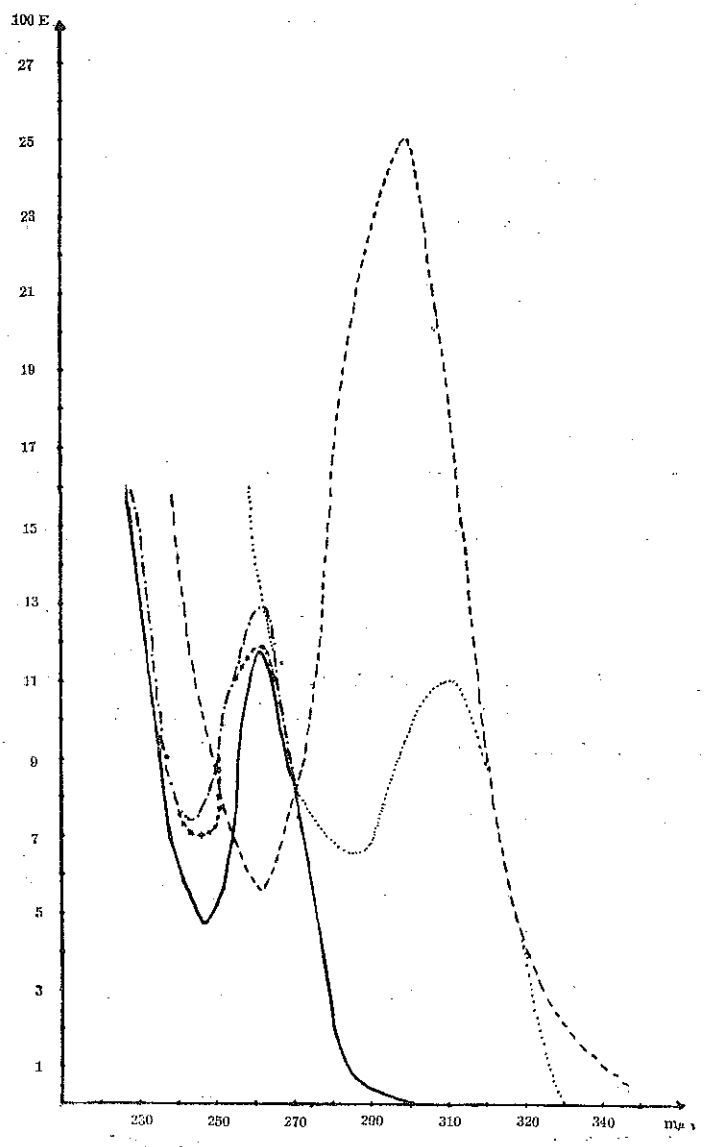

Fig. 1. Absorption curves. - - - - - - salicylamide, ... pheniramine maleate, —·—·— brompheniramine maletae, ++++ chlorpheniramine maleate, ——— pynilamine maleate.

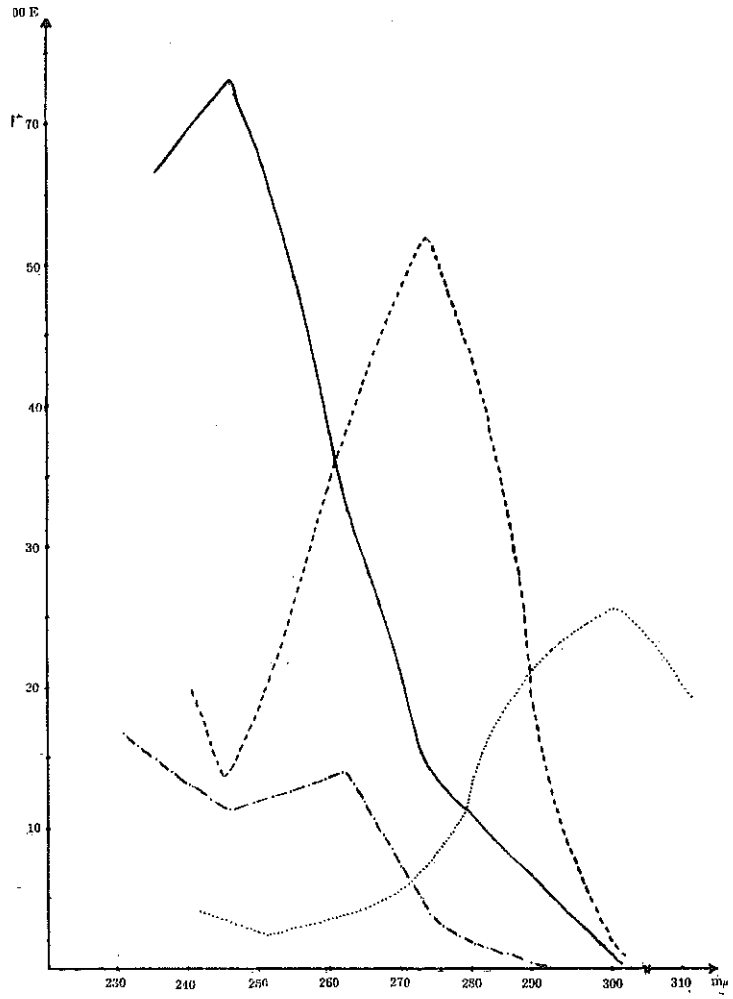

Fig. 2. Absorption curves, ——— phenacetine, ... Caffeine, - - - - - - Salicylamide, —·—·—·— chlorpheniramine maleate.

In the mixtures containing phenylpropanolamine, the isoabsorptive points are as same as in the binary mixtures (Fig. 1 and 2).

Procedure. The mixtures were prepared from 0.001, 0.002, 0.003 and 0.005 % solutions of each compound in water. The absorbances were measured at the absorption maxima and at the absorptive points using water as a blank. The relative concentrations of the components in the sample were calculated by substituting the observed values into the following equations:

$$C_x = \frac{Q_0 - Q_y}{Q_x - Q_y} \cdot \frac{A_3}{a} \quad \text{or} \quad C_y = \frac{Q_0 - Q_y}{Q_y - Q_x} \cdot \frac{A_3}{a}$$

$$C_y = \frac{A_3}{a} - C_x \qquad C_x = \frac{A_3}{a} - C_y$$

C_x and C_y are the concentrations of the substances x and y in the mixture.

Q_x and Q_y are the ratio of the extinction coefficients at λ max. to the isoabsorptive point for x and y respectively.

Q_0 is the absorbancy ratio of the mixtures at λ max. and at isoabsorptive point.

A_3 is the absorbancy of the mixture at the isoabsorptive point.

a is the extinction coefficient of the mixture at the isoabsorptive point.

$\frac{A_3}{a}$ gives the total concentration of x and y in the sample.

The results are given in Table I-VIII (concentrations are given in mg/100 ml).

Table I. Analysis of the mixture 1.

solution	*pheniramine maleate*		*salicylamide*	
	present	*found*	*present*	*found*
I	1.07	0.998	2.50	2.59
II	1.25	1.16	1.87	1.96
III	1.47	1.37	1.47	1.56
IV	1.50	1.40	3.50	3.59
V	1.84	1.86	0.79	0.82
VI	2.00	1.98	0,50	0.56
VII	2.00	1.92	3.00	3.07
VIII	2.50	2.37	2.50	2.62
IX	3.00	2.86	2.00	2.11
X	3.50	3.39	1.50	1.60
XI	4.00	3.90	1.00	1.07

Table II. Analysis of the mixture 2.

solution	*chlorpheniramine maleate*		*salicylamide*	
	present	*found*	*present*	*found*
I	0.05	0.049	0.95	0.95
II	0.10	0.10	0.90	0.90
III	0.30	0.32	3.50	3.64
IV	0.50	0.49	2.50	2.68
V	0.57	0.48	0.60	0.61
VI	0.80	0.80	1.00	1.09
VII	1.00	0.95	4.00	4.04
VIII	1.20	1.20	0.40	0.42
IX	2.00	2.02	3.00	2.97
X	3.00	2.96	2.00	2.03
XI	4.00	3.88	1.00	1.11
XII	4.50	4.43	0.50	0.57
XIII	4.75	4.81	0.25	0.28

Table III. Analysis of the mixture 3.

solution	*brompheniramine maleate* *present*	*found*	*salicylamide* *present*	*found*
I	1.00	1.01	4.00	3.997
II	1.07	1.03	2.50	2.53
III	1.25	1.23	1.87	1.93
IV	1.47	1.44	1.47	1.53
V	1.66	1.69	1.11	1.16
VI	1.50	1.49	3.50	3.52
VII	2.00	2.02	3.00	2.99
VIII	2.50	2.51	2.50	2.50
IX	3.00	2.99	2.00	2.01
X	3.50	3.51	1.50	1.51
XI	4.00	3.99	1.00	1.02

Table IV. Analysis of the mixture 4.

solution	*pyrilamine maleate* *present*	*found*	*salicylamide* *present*	*found*
I	0.84	0.86	3.30	3.28
II	1.00	1.15	4.00	3.84
III	1.25	1.24	1.87	1.89
IV	1.47	1.38	1.47	1.49
V	1.50	1.57	3.50	3.43
VI	1.66	1.74	1.11	1.09
VII	2.00	1.93	0.50	0.51
VIII	2.00	2.02	3.00	2.98
IX	2.50	2.54	2.50	2.46
X	3.00	2.93	2.00	1.97
XI	3.50	3.45	1.50	1.45
XII	4.00	3.95	1.00	0.95

Table V. Analysis of the mixture 5.

solution	*caffeine*		*salicylamide*	
	present	*found*	*present*	*found*
I	0.50	0.44	2.50	2.50
II	0.60	0.57	2.00	2.00
III	1.00	1.06	4.00	3.89
IV	2.00	1.89	3.00	3.06
V	3.00	2.90	2.00	2.05
VI	3.50	3.39	1.50	1.56

Table VI. Analysis of the mixture 6.

solution	*phenacetine*		*salicylamide*	
	present	*found*	*present*	*found*
I	0.60	0.61	2.40	2.37
II	0.90	0.905	2.10	2.07
III	1.25	1.24	2.25	2.21
IV	1.50	1.46	1.50	1.52
V	1.80	1.76	1.20	1.21
VI	2.10	2.06	0.90	0.91
VII	2.40	2.37	0.60	0.61
VIII	2.20	2.14	1.66	1.66
IX	2.70	2.68	0.30	0.30
X	3.75	3.62	0.75	0.74

Table VII a. Analysis of the mixture 7a.

solution	*pheniramine maleate* *present*	*found*	*pyrilamine maleate* *present*	*found*
I	1.0	1.09	2.33	2.32
II	1.31	1.36	0.65	0.67
III	1.33	1.38	1.33	1.31
IV	1.40	1.49	0.38	0.40
V	1.50	1.60	1.00	1.01
VI	1.50	1.61	3.50	3.42
VII	2.00	2.07	3.00	2.94
VIII	2.50	2.53	2.50	2.48
IX	3.00	3.03	2.00	1.97
X	2.30	2.34	1.15	1.05
XI	2.58	2.60	0.69	0.72

Table VII b. Analysis of the mixture 7b.

solution	*chlorpheniramine maleate* *present*	*found*	*pyrilamine maleate* *present*	*found*
I	1.16	1.22	2.13	1.86
II	1.25	1.37	3.74	3.61
III	1.76	1.84	3.23	3.14
IV	2.25	2.45	1.75	1.78
V	2.50	2.54	2.50	2.46
VI	2.80	2.78	2.18	2.20
VII	3.12	3.11	1.87	1.87

Table VII c. Analysis of the mixture 7c.

solution	*brompheniramine maleate*		*pyrilamine maleate*	
	present	*found*	*present*	*found*
I	1.00	0.98	4.00	3.98
II	1.07	1.03	2.50	2.52
III	1.25	1.29	1.99	1.85
IV	1.47	1.38	1.47	1.54
V	1.66	1.62	1.11	1.14
VI	1.84	1.84	0.79	0.83
VII	2.00	1.97	0.50	0.58
VIII	2.00	1.98	3.00	2.99
IX	2.50	2.47	2.50	2.50
X	3.00	2.95	2.00	2.02
XI	3.50	3.45	1.50	1.50
XII	4.00	3.94	1.00	1.00

Table VIII. Analysis of salicylamide and phenacetine in the mixture 8.

solution	*salicylamide*		*phenacetine*	
	present	*found*	*present*	*found*
I	0.78	0.76	1.80	1.84
II	1.30	1.34	1.25	1.28
III	1.53	1.52	0.91	0.93
IV	2.00	1.90	0.30	0.34

The results obtained for the mixtures 9 are same as given in table I-VIII.

DİSCUSSİON

Some antihistaminics and some antipyretics were assayed in their mixtures by applying the absorbancy ratio method of analysis to the solutions, without a special extraction.

The spectral characteristics of the compounds are illustrated in Figure 1 and 2. These characteristics indicated that binary mixtures containing antihistaminics such as pheniramine, chlorpheniramine, brompheniramine maleates or pyrilamine maleate could be analysed in the presence of salicylamide, phenacetine or phenylpropanolamine. In addition, binary mixtures containing salicylamide with caffeine or pyrilamine maleate with pheniramine, chlorpheniramine or brompheniramine maleates may be analysed by applying this method. Addition of phenylpropanolamine does not interfere with the determinations since it showed no absorbance at the region of 260 $m\mu$ to 310 $m\mu$.

The results obtained for each sample which were analysed are given in Tables I-VIII. Duplicate absorbance readings were made on each sample. Results were calculated as described above. The examination of the results in Tables I-VIII indicate that the components present in the mixtures can be determined with accuracy and precision within the limits of concentration of 1-4 mg/100 ml for pheniramine maleate, brompheniramine maleate and pyrilamine maleate, 0.05-4.75 mg/100 ml for chlorpheniramine maleate, 0.25-4 mg/100 ml for salicylamide, 0.5-3.5 mg/100 ml for caffeine and 0.60-3.75 mg/100 ml for phenacetine.

The proposed method of analysis, appear to be preferable to the other methods which involve solvent-solvent extraction.

SUMMARY

A spectrophotometric method of analysis was described for determination of some antihistaminics and antipyretics. The procedure was used successfully on the binary and ternary mixtures. The results were accurate and reproducible.

ÖZET

Bu çalışmada feniramin, klorfeniramin, bromfeniramin, pirilamin maleatlar, salisilamid, fenasetin, kafein ve fenilpropanolamin ihtiva eden biner ve terner karışımlara, absorbans oranı esasına dayanarak

spektrofotometrik Q analiz metodu tatbik edilmiştir. Karışımların absorbansları λ maksimum ve isoabsorpsiyon noktalarında ölçülmüş ve bulunan değerlerin Q formülüne tatbiki ile konsantrasyonlar hesaplanmıştır. Tekrarlanabilen neticeler elde edilmiştir.

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