Spectrophotometric Determination of Some Phenolic Drugs in Pure Form and in their Pharmaceutical Preparations.

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ABSTRACT

Two methods were described for the determination of etilefrine HCI (I), hexoprenaline HCI (II), levodopa (III) and carbidopa (IV) either in pure form or in pharmaceutical preparations. Method (A) is based on oxidative coupling reaction between (I), (II) and (III) and 4-amino antipyrine (4-AAP) to produce coloured species. The wavelengths for maximum absorption were 509, 494, and 530 nm for (I), (II) and (III), respectively. Beer's law was obeyed in the concentration ranges 1-7, 5-50 and 2.5-20 ug/ml, while those obtained when applying Ringbom were found to be 1.5-4.3, 9.7-31.6 and 3.9-15.8 ug/ml for (I), (II) and (III), respectively. The corresponding molar absorptivities were 1.78 x 10^4, 0.802 x 10^4 and 0.597 x 10^4 L. mol^{-1}. cm^{-1} for (I), (II) and (III), respectively. Method (B) depends on nitration of (I), (II) and (III) and (IV), using sodium cobalt nitrite in acetic acid medium. The reaction products show maximum absorption at 360, 460, 320 and 360 nm for (I), (II), (III) and (IV), respectively. Linear correlations were obtained between absorbance at the specific wavelength and concentrations in the range of 2-16, 8-64, 5-25 and 2.5-20 ug/ml, while Ringbom concentrations were found to be 2.51-12.58, 10-50.11, 5.623-22.38 and 3.16-15.84 ug/ml for (I), (II) and (III) and (IV), respectively. The corresponding molar absorptivities were 1.248 x 10^4, 0.7303 x 10^4, 0.7327 x 10^4 and 1.052 x 10^4 L. mol^{-1}.cm^{-1} for (I), (II), (III) and (IV), respectively. Results of analysis of pure drugs by the proposed methods were in good agreement with the official B.P. and reference methods. The proposed procedures were successfully applied to determine the studied drugs in their pharmaceutical dosage forms.

Keywords: Etilefrine HCI, Hexoprenaline HCI, Levodopa, Carbidopa, 4-amino antipyrine, Sodium cobalt nitrite, Colorimetry.

INTRODUCTION

Etilefrine HCl (RS) 2–ethylamino–1–(3–hydroxyphenyl) ethanol hydrochloride and hexoprenaline HCl N,Ń–bis[2–(3,4–dihydroxyphenyl)–2–hydroxyethyl] hexamethylenediamine hydrochloride[(1)] are direct–acting sympathomimetic drugs. Etilefrine HCl is used for the treatment of hypotensive states. Hexoprenaline HCl is a bronchodilator used in the treatment of reversible airways obstruction which occurs with asthma.[(2)] Levodopa (–)–3–(3,4–dihydroxy phenyl)–L– alanin, a naturally occuring amino acid is the intermediate precursor of the neurotransmitter dopamine and used in the treatment of Parkinson's disease, which is associated with the depletion of dopamine in the brain. Carbidopa (–)–L–α–hydrazino–3,4–dihydroxy–α–metyl hydrocinnamic acid monohydrate is a peripheral decarboxylase inhibitor which inhibits the peripheral decarboxylation of levodopa to dopamine. Carbidopa is used in admixture with levodopa in the treatment of Parkinsonism to decrease the dose of the later, to obtain a more rapid response and to decrease side-

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effects.[2]

Various spectrophotometric methods were applied for the determination of carbidopa alone [3-5] or for levodopa alone.[6-10] Levodopa and carbidopa were also analyzed by column chromatography,[11] capillary electrophoresis[12]and HPLC methods.[13-14]

Spectrophotometric methods[15-17] were applied for the determination of etilefrine HCl. These methods are time consuming (heating for 25 minutes at 100ºC and 20 minutes at 80ºC) and not suitable for routine analyses. Capillary electrophoresis,[18]GC-MS,[19] HPLC,[20] (PMR) spectroscopy,[21] glass capillary column GC[22] and ion exchange[23] methods were also used for etilefrine HCl.

Few methods were found in the literature for the determination of hexoprenaline HCl including reversed phase liquid chromatography with UV detection,[24] HPTLC[25] and spectroscopic methods.[26-27] Accordingly, the development of simple and reliable spectrophotometric methods for the determination of the cited drugs is still important.

This work aimed to find simple, rapid and sensitive methods suitable for routine analysis of the above mentioned drugs.

EXPERIMENTAL

Apparatus

SHIMADZU UV-1201 UV-Vis spectrophotometer with 10 mm matched quartez cells was used for all spectral measurments.

Materials and reagents

Pharmaceutically pure etilefrine HCl (CID Co., for pharmaceuticals, Cairo, Egypt), hexoprenaline HCl (Arabic Co., for pharmaceuticals, Cairo, Egypt), levodopa and carbidopa (Elnasr Co., for chemicals, Cairo, Egypt) were used as working standards.

The following pharmaceutical preparations were analyzed :-

Effortil tablets (CID Co., for pharmaceuticals, Cairo, Egypt) each tablet labelled to contain 5 mg of etilefrine HCl. Effortil drops (CID Co.) each 10 drops labelled to contain 5 mg of etilefrine HCl. Asmadol and gynebral tablets (Arabic Co. for pharmaceuticals, Cairo, Egypt) each tablet labelled to contain 0.5 mg of hexoprenaline sulphate. Levodopa tablets (250 mg/tablet) and carbidopa tablets (25 mg/tablet) were prepared in laboratory and contain the usually included excepients such as starch, talc, avicel (micro crystalline cellulose) and magnesium stearate.

4-Aminoantipyrine(4-AAP) solution, 0.1 % w/v in water was freshly prepared from material obtained from Aldrich (USA). Potassium dihydrogen phosphate solution, 0.1 and 0.05% aqueous solution was prepared. Sodium hydroxide 0.1% solution was prepared in water. Potassium ferricyanide solution, 0.1% w/v was freshly prepared in water and supplied by Aldrich (USA). Sodium cobaltinitrite solution (Sigma USA), 0.3 % and 1% w/v solutions in water were freshly prepared. Acetic acid (Adwic), 6 % v/v solution was prepared in water.

Stock solutions

Stock solutions of 100 µg/ml in water were prepared for etilefrine HCl, 400 and 500 µg/ml for hexoprenaline HCl, while for levodopa and carbidopa, 250 µg/ml in 0.1 N HCl solutions were used.

General procedure

4-Amino antipyrine method

a- Etilefrine HCl and Hexoprenaline HCl:-

Etilefrine HCl, 10-70 µg or hexoprenaline HCl, 50-500 µg, were transferred into 10-ml calibrated flasks, then 2ml of 0.05% potassium dihydrogen phosphate solution, for etilefrine HCl or 1.5 ml of 0.1% potassium dihydrogen phosphate solution for hexoprenaline HCl were added, followed by 4-AAP solution 0.5 and 1 ml for etilefrine HCl and hexoprenaline HCl, respectively) then 2 ml of potassium ferricyanide solution were added. Mixed well, leaved to stand for 5 min at room temperature (hexoprenaline HCl) or for 10 min (etilefrine HCl), then diluted to volume with water. Absorbances were measured at 509 nm (etilefrine HCl) or 494 nm (hexoprenaline HCl) against reagent blank.

b- Levodopa:-

Levodopa 25-200 µg were transferred into 10-ml calibrated flasks, then 1.5 ml of 0.1% 4-AAP solution and 1 ml of 0.1% potassium ferricyanide solution were added

followed by 1 ml of 0.1% sodium hydroxide solution. Mixed well, leaved to stand for 5 min at room temperature, then diluted to 10 ml with water. Absorbances were measured at 530 nm against reagent blank.

Sodium cobaltinitrite method

a-Etilefrine HCl:-

Accurately measured aliquots containing from 20-160 µg etilefrine HCl were transferred into a series of test tubes. Then, 2 ml of 1% sodium cobaltinitrite solution and 0.3 ml of acetic acid solution were added, mixed well, heated in a boiling water bath for 30 min. The mixtures were transferred into 10-ml calibrated flasks, cooled, completed to volume with water. Absorbances were measured at 360 nm against reagent blank.

b- Hexoprenaline HCl, levodopa and carbidopa:-

Accurately measured aliquots containing from 80–640 µg of hexoprenaline HCl, 50-250 µg of levodopa or 25-200 µg of carbidopa were transferred into a series of test tubes. Then, 1.5 ml (hexoprenaline HCl), 0.5 ml (levodopa) and 3.5 ml (carbidopa) of 0.3% sodium cobaltinitrite solution were added followed by the addition of acetic acid solution (0.5 and 0.4 ml) for hexoprenaline HCl, and both levodopa and carbidopa, respectively, mixed well. Heated at 60 ºC for 5 min (hexoprenaline HCl) or for 20 min (levodopa and carbidopa). Mixtures were cooled, transferred into 10-ml calibrated flasks, and completed to volumes with water. Absorbances were measured at 360, 460, 320 and 360 nm for hexoprenaline HCl, levodopa and carbidopa, respectively, against reagent blank.

Procedure for pharmaceutical preparation:-

4-Amino antipyrine method:-

a-Effortil and asmadol tablets :-

30 Tablets were weighed, finely powderd and a portion of the powder equivalent to 10 mg (etilefrine HCl) or 50 mg (hexoprenaline HCl) were accurately weighed, extracted with water, and then filtered. The residue was washed twice with distilled water. The filtrate and washings were collected in 100-ml calibrated flasks, completed to volume with water. Aliquots of the solution equivalent to 10-70 µg (etilefrine HCl) or 50-500 µg (hexoprenaline HCl) were transferred into 10-ml calibrated flask, then proceeded as described under the general procedure.

b-Levodopa tablets:-

20 Tablets were weighed, finely powderd and a portion of the powder equivalent to 25 mg of levodopa was weighed, extracted twice with 0.1 N HCl, then filtered. The residue was washed with 0.1 N HCl, The filtrate and washings were collected in 100 ml calibrated flasks, completed to volume with the mentioned solvent. Aliquots of the solution equivalent to 25-200 µg were transferred into 10-ml calibrated flasks, then proceeded as described under the general procedure.

Sodium cobaltinitrite method

a- Effortil tablets: -

20 Tablets were weighed, finely powdered and a portion of the powder equivalent to 10 mg effortil HCl was weighed, extracted with water and filtered. The residue was washed twice with water. The filtrate and washings were collected in 100-ml calibrated flask and completed to volume with distilled water. Aliquots of the tablets solution equivalent to 20-160 µg of etilefrine HCl were transferred into a series of test tubes, then proceeded as described under the general procedure.

b- Gynebral, levodopa and carbidopa tablets: -

20 Tablets were weighed, finely powdered and portions of the powder equivalent to 40 mg and 25 mg for hexoprenaline HCl and both levodopa and carbidopa, respectively, were weighed. Then, it was extracted twice with water (hexoprenaline HCl) or with 0.1 N HCl (levodopa and carbidopa). And the residue was then filtered and washed with distilled water (hexoprenaline HCl) or with 0.1 N HCl (levodopa and carbidopa). The filtrate and washings were collected100 ml calibrated flasks and completed to volume with the mentioned solvent. Aliquots of the tablets solution equivalent to 80–640 µg of hexoprenaline HCl, 50-250 µg of levodopa or 25-200 µg of carbidopa were transferred into series of test tubes, then proceeded as described under the general procedure.

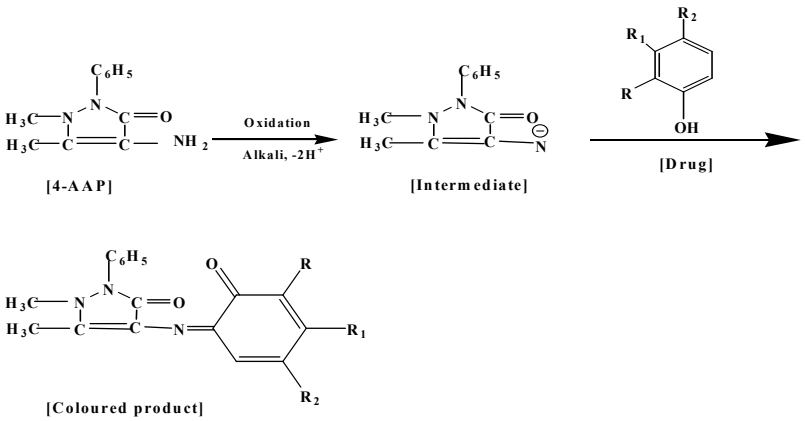
c-Effortil drops: -

Accurately measured volume of the preparation equivalent to 10 mg of the drug, diluted to 100 ml with water, then proceeded as described under the general procedure.

RESULTS AND DISCUSSION

Mechanism of the reaction

Scheme 1:



4-Amino antipyrine method

It has been reported that 4-AAP upon oxidation with alkaline potassium ferricyanide loses 2 protons forming a nucleophilic intermediate[28]. It is suggested that the intermediate undergoes nucleophilic substitution with the phenolic moieties of the drugs to produce the colored product (Scheme 1).

The absorption spectra of the colored reaction product at the optimum conditions recorded in the general procedures show characteristic λ_{max} at 509, 494, and 530 nm for etilefrine HCl, hexoprenaline HCl and levodopa, respectively (Figure 1).

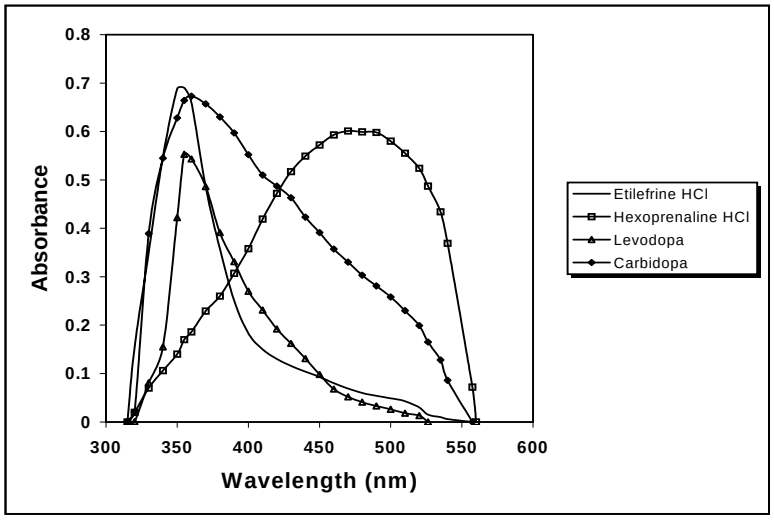
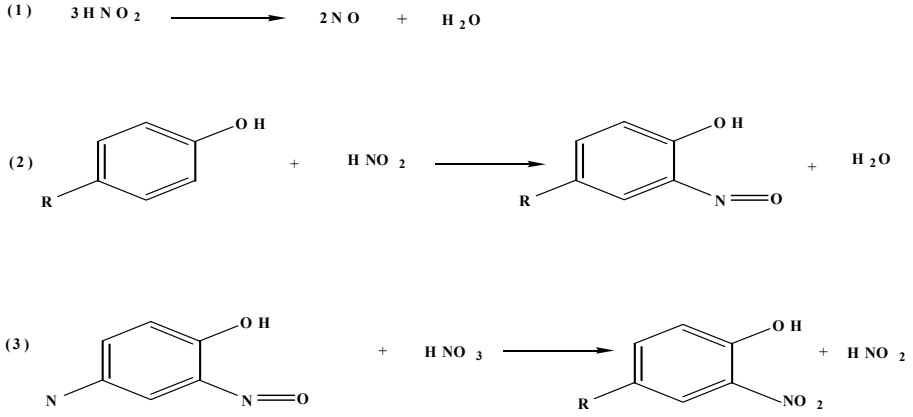


Fig. (1): Absorption spectra of the reaction product of etilefrine HCl (12 µg ml[1]), hexoprenaline HCl (40 µg/ ml), levodopa (15 µg/ ml) and carbidopa (15 µg/ml) with sodium cobaltinitrite (method B).

Sodium cobaltinitrite method:-

Etilefrine HCl, hexoprenaline HCl, levodopa and carbidopa have a phenolic group, which can be converted into o-nitro-derivative[29&30]. (Scheme 2).

Scheme 2:



This can be achieved by the use of sodium cobaltinitrite in acetic acid solution. The reaction products show absorption maxima at 360, 460, 320 and 360 nm for etilefrine HCl, hexoprenaline HCl, levodopa and carbidopa, respectively (Figure 2).

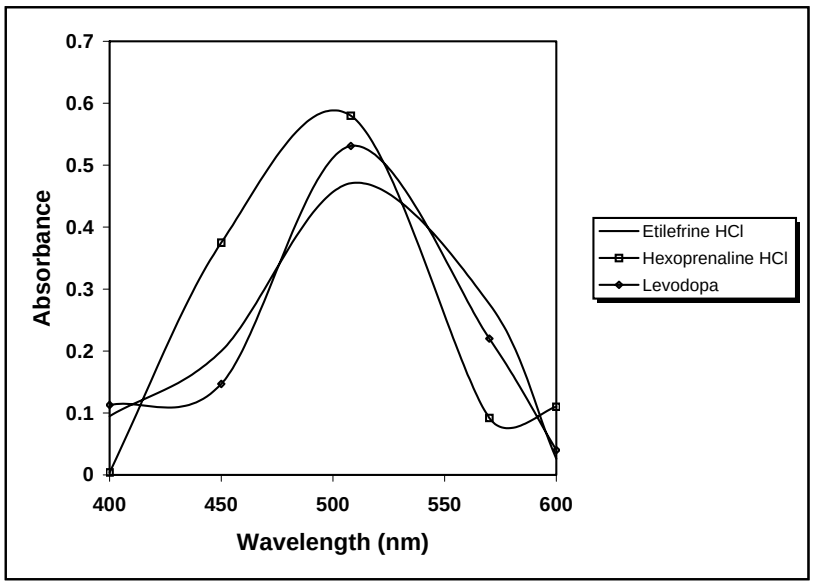


Fig. (2): Absorption spectra of the reaction product of etilefrine HCl (7 µg ml^{-1}), hexoprenaline HCl (40 µg/ ml) and levodopa (17.5 µg/ ml) with 4-AAP (method A).

Effect of reaction variables

4-Amino antipyrine method

The optimum concentration of 4-AAP leading to maximum color intensity was found to be 0.5 ml for etilefrine HCl, 1 ml for hexoprenaline HCl and 1.5 ml for levodopa. Several oxidizing agents were investigated e.g iron ammonium sulphate, hydrogen peroxide, potassium dichromate, potassium chromate, cerric ammonium

sulphate, potassium periodate, potassium ferricyanide and ferric chloride. potassium ferricyanide succeeded to perform the oxidation process. Potassium ferricyanide (0.1% w/v) solution was used and 2 ml for etilefrine HCl and hexoprenaline HCl or 1 ml for Levodopa were found to be optimum for the maximum color production.

Several types of alkalinzing agents were investigated for the four mentioned reaction including NaOH, Na_2CO_3, ammonia buffer pH 9 and potassium dihydrogen phosphate. It was found that the addition of 1.0 ml and 1.5 ml (0.1% w/v) potassium dihydrogen phosphate solution was optimum for a complete color production regarding etilefrine HCl and hexoprenaline HCl, respectively. However, levodopa required the addition of 1 ml (0.1% w/v) NaOH solution for a maximum color production.

The color product developed rapidly and attained maximum intensity after about 10 min (etilefrine HCl) and 5 min (hexoprenaline HCl and levodopa) at room temperature. The color was stable for about 60 min for the studied drugs.

Sodium cobaltinitrite method:-

When different volumes of sodium cobaltinitrite solution were added to a fixed concentration of the drug, it was found that 2 ml (0.1% w/v) sodium cobaltinitrite solution for etilefrine HCl, however, 1.5, 0.5 and 3.5 ml (0.3% w/v) sodium cobaltinitrite solution for hexoprenaline HCl, levodopa and carbidopa were the optimum volumes for maximum color intensity. When different volumes of 6% v/v acetic acid solution were added to a fixed concentration of the drug, 0.3 ml for etilefrine HCl, 0.5 ml for hexoprenaline HCl and 0.4 ml for levodopa and carbidopa were found to be sufficient for maximum color intensity.

It was found that heating the reaction mixture in a boiling water bath for 30 min (etilefrine HCl) and at 60 ºC for 5 min (hexoprenaline HCl) or for 20 min (levodopa and carbidopa) were optimum for a maximum absorption intensity of the colored product.

Job's method of continous variation is applied to study the stoicheiometry of the reaction. Results reveal a 1 : 1 ratio (for etilefrine HC, levodopa and carbidopa) and 1: 2 ratio for (hexoprenaline HCl).

Quantification

4-Amino antipyrine method:-

Under the described experimental conditions, standard calibration curves for etilefrine HCl, hexoprenaline HCl and Levodopa were constructed by plotting the absorbance versus concentration. Conformity to Beer's law was evident over the concentration range of the final dilution 1-7μg/ml, 5-50 μg/ml and 2.5-20 μg/ml μg/ml, respectively. Sandell's sensitivities of the reactions, as calculated from Beer's law data were 0.012, 0.062 and 0.033 μg cm^{-2} and the corresponding molar absorptivities were 1.78×10^4, 0.802×10^4 and 0.597×10^4 $L.mol^{-1}.cm^{-1}$ for etilefrine HCl, hexoprenaline HCl and levodopa, respectively (Table 1).

Ringbom were found to be 1.5-4.3 μg/ml, 9.7-31.6 μg/ml and 3.9-15.8 μg/ml for etilefrine HCl, hexoprenaline HCl and levodopa, respectively. The results obtained showed recoveries of 99.96± 0.551, 100.528 ± 0.538 and 100.39 ± 0.491, with relative standard deviations of 0.551, 535 and 0.489 for etilefrine HCl, hexoprenaline HCl and levodopa, respectively.

Sodium cobaltinitrite method:-

Linear correlations were obtained between absorbance at the specific wavelength and concentrations in the range of 2 - 16 μg/ml, 8 - 64 μg/ml, 5-25 μg/ml and 2.5-20 μg/ml, while Ringbom concentrations were found to be 2.51-12.58 μg/ml, 10-50.11 μg/ml, 5.623-22.38 μg/ml and 3.16-15.84 μg/ml for etilefrine HCl, hexoprenaline HCl, levodopa and carbidopa, respectively. Molar absorpativities and Sandell's sensitivity for the cited drugs were calculated and presented (Table 1).

Table (1): Spectral characteristics of the products from the reaction between 4-AAP and sodium cobaltnitrite and the studied drugs.

	4-AAP method			Sodium cobaltnitrite method			
	Etilefrine HCI	Hexoprenaline HCI	Levodopa	Etilefrine HCI	Hexoprenaline HCI	Levodopa	Carbidopa
λ max (nm)	509	494	530	360	460	320	360
Linear range (ug/ml)	1-7	5-50	2.5-20	2-16	8-64	5-20	2.5-20
Quantitative parameters Intercept Slope Correlation coefficient	0.0014 0.08 0.9999	-0.0003 0.0161 0.9999	0.00075 0.03 0.9999	0.0011 0.057 0.9999	-0.0019 0.0149 0.9999	-0.0035 0.0372 0.9999	-0.00067 0.043 0.999
Sandell's sensitivity (ug/ml -2)	0.012	0.061	0.033	0.017	0.067	0.026	0.023
Molar absorbitivity (mol-1cm-1)	**1.78 × 10**	**0.802 × 10**	**0.5971×10**	**1.248× 10**	**07303 × 10**	**07327 ×10**	**1.0521 × 10**

CONCLUSION

The proposed methods were adventageous over the other reported visible spectrophotometric methods with respect to higher sensitivity which permits the determination of down to 1 µg/ml, simplicity, reproducibility, precision, accuracy and stability of the coloured products more than 2 hours. The interferance of the associated excipients and additives was not observed. The proposed methods can be applied for routine analysis and in quality control laboratories for the quantitative determination of the studied drugs in pure form and in their pharmaceutical formulations depending on the availability of the chemicals.

The obtained results were compared with those of official and reference methods using student (t- test) and variance ratio (F- test). The calculated values didn't exceed the theoretical ones indicating that there is no significant difference (Tables 2).

Table (2): Statistical analysis of results of the determination of the cited drugs using the 4-AAP and sodium cobaltinitrite methods compared with the official methods which include non aquous titration for etilefrine HCI and carbidopa (31) and spectrophotometric methods for hexoprenaline HCI (26) and levodopa (6).

	Proposed methods								Official method	
	4-Amino antipyrine method				Sodium cobaltnitrite method					
	Mean Recovery %	RSD %	t	F	Mean Recovery %	RSD %	t	F	Mean Recovery %	RSD %
Etilefrine HCI	99.96	0.551	0.38 (2.201)	2.36 (4.39)	100.17	0.486	0.466 (2.17)	1.85 (3.97)	100.06	0.357
Hexoprenaline HCI	100.528	0.535	0.226 (2.12)	2.31 (3.29)	100.13	0.508	0.167 (2.08)	2.58 (2.83)	100.6	0.817
Levodopa	100.39	0.489	1.26 (2.131)	1.1 (3.5)	100.09	0.673	0.036 (2.12)	2.07(3.44)	100.1	0.468
Carbidopa					100.28	0.466	1.24 (2.13)	2.8 (3.5)	100.05	0.279

*Average of 6 determinations.

Validity of the methods were tested by the determination of the studied drugs in their pharmaceutical preparations, applying the standard addition technique and the obtained results demonstrate good precision and accuracy as shown in table (3).

Table (3): Statistical analysis of results of the determination of the cited drugs in their pharmaceutical preparation using the 4-AAP and sodium cobaltinitrite methods applying the standard addition technique

	4-Amino antipyrine method					Sodium cobaltnitrite method				
	Mean Recovery %	SD	RSD %	SE	V	Mean Recovery %	SD	RSD %	SE	V
Effortil tab. Effortil drops	100.67 100.38	0.54 0.351	0.537 0.35	0.22 0.143	0.292 0.123	100.73 99.79	0.619 0.611	0.614 0.613	0.252 0.249	0.383. 216
Asmadol or gynebral tab.	100.49	0.386	0.384	0.146	0.149	99.829	0.337	0.338	0.119	0.113
Levodopa tab.	101.14	0.46	0.46	0.189	0.216	99.965	0.559	0.56	0.228	0.313
Carbidopa tab.						100.54	0.454	0.451	0.185	0.206

*Average of 6 determinations.

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التقدير الطيفى لبعض العقاقير المحتوية على مجموعة فينول سواء فى الحالة النقية أو فى مستحضراتها الصيدلية

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ملخـص

تم وصف طريقتين لتقدير كل من هيدروكلوريد الإيتليفرين وهيدروكلوريد هيكسوبرينالين وليفودوبا و كاربيدوبا سواء فى حالتها النقية أو فى مستحضراتها الصيدلية. تعتمد الطريقة الأولى على الأكسدة ثم التفاعل مع كاشف 4-AAP لينتج مركب ملون يتم قياسه عند طول موجى 509 ، 494 ، 530 نانوميتر للعقاقير السابق ذكرها على الترتيب. و قد وجد أن التفاعل يتبع قانون بيير عند تركيزات تتراوح بين 1 – 7 ، 5 – 50 ، 2,5 – 20 ميكروجرام للعقاقير السابق ذكرها على الترتيب و تعتمد الطريقة الثانية على نيترة كل من العقاقير السابق ذكرها باستخدام صوديوم كوبالتى نيتريت فى وسط حمض الخليك لينتج مركبات ملونة يتم قياسها طيفيا. و قد وجد أيضا أن التفاعل يتبع قانون بيير عند تركيزات 2 – 16 ، 8 – 64 ، 5 – 25 ، 2,5 – 20 ميكروجرام لكل مل للعقاقير السابق ذكرها على الترتيب .

تم تقدير الامتصاص الجزيئى الظاهرى ومقارنة نتائج الطرق المقترحة بالطرق الدستورية و المنشورة و كذلك تم تطبيق الطرق المقترحة بنجاح لتقدير العقاقير المذكورة فى مستحضراتها الصيدلية.

الكلمـات الدالـة: هيدروكلوريد الإيتليفرين، هيدروكلوريد هيكسوبرينالين، ليفودوبا، كاربيدوبا، 4-AAP، صوديوم كوبالتى نيتريت.

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