ELSEVIER

Journal of Pharmaceutical and Biomedical Analysis
23 (2000) 1045–1055

JOURNAL OF
PHARMACEUTICAL
AND BIOMEDICAL
ANALYSIS

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Determination of some aminobenzoic acid derivatives: glafenine and metoclopramide

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Received 1 June 1999; received in revised form 2 May 2000; accepted 26 May 2000

Abstract

Simple, sensitive and accurate spectrophotometric methods for the determination of glafenine and metoclopramide hydrochloride are described. The first method is based on the oxidation of glafenine with iodic acid in strong acid medium to give a coloured diphenylbenzidine derivative and subsequent measurement of the coloured product at 509 nm. Beer's law is obeyed over the concentration range 2.5–20 μg ml^{-1}. The second method depends on the interaction of metoclopramide hydrochloride with *p*-dimethylaminocinnamaldehyde, to give a red coloured schiff's base with an absorbance maximum at 548 nm. Obedience to Beer's law is achieved over the concentration range 5–30 μg ml^{-1}. First-derivative method is used to overcome the slight interference of *p*-dimethylaminocinnamaldehyde reagent blank at the wavelength of measurement. Linearity between the peak heights at 576 nm versus concentration range 5–25 μg ml^{-1} metoclopromide hydrochloride is obtained. The proposed methods have been successfully applied to the determination of these drugs in commercial products without interference. The validity of the methods is assessed by applying the standard addition technique, the relative standard deviation is less than 1%. The proposed methods are compared with reference methods with good agreement.

Keywords: Glafenine; Iodic acid; Metoclopramide; Hydrochloride; *p*-Dimethylaminocinnamaldehyde; Spectrophotometry; First-derivative

1. Introduction

Glafenine, 2,3-dihydroxypropyl-(*N*-(chloro-4-quinolyl)-anthranilate, is a non-narcotic analgesic Fig. 1. Several methods that have been reported for its determination include spectrophotometric [1], gravimetric [2], polarographic [3] and chromatographic methods [4,5]. In addition to the alkalimetric [6] and potentiometric methods [7], an oxygen flask combustion method for the determination of chlorine content is cited in the literature [8]. Colorimetric methods have also been described using 2,3-dichloro-5,6-dicyano-*p*-benzoquinone and *p*-chloranilic acid reagents [9]. Metoclopramide hydrochloride, 4-amino-5-chloro-*N*-(2-diethyl-aminoethyl)-2-methoxybenzamide

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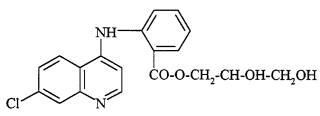
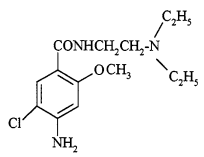
PII: S0731-7085(00)00384-8

hydrochloride, is an antiemetic and gastroprokinetic agent currently used in the treatment of gastrointestinal disorders. Among the several reported methods, colorimetric [10–14], flameless atomic absorption spectrometry [15] and proton magnetic resonance spectroscopy have



Glafenine



Metoclopramide

Fig. 1. Structural formulae of glafenine and metoclopramide.

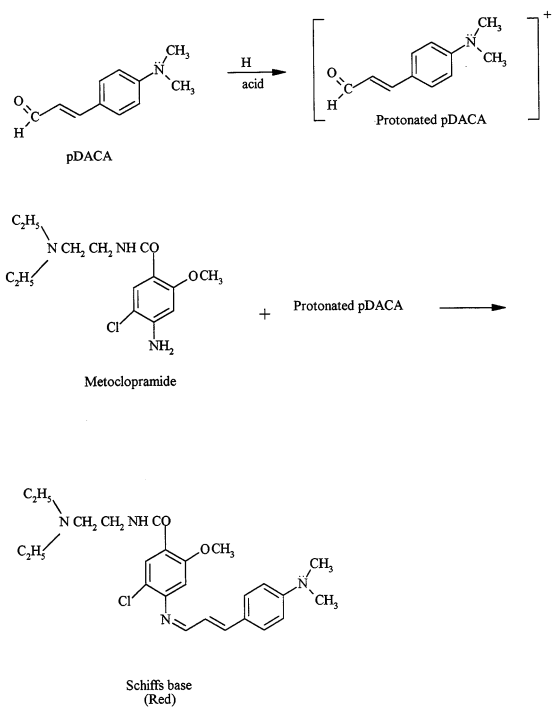


Fig. 2. Reaction of metoclopramide with *p*DACA.

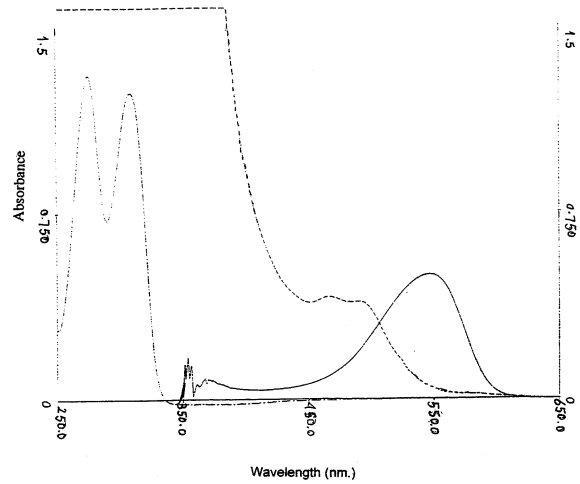


Fig. 3. Absorption spectra of metoclopramide HCl, 26.4 μg ml^{-1} (–·–·–); metoclopramide-*p*DACA., 20 μg ml^{-1} (—) and *p*DACA reagent (---) in methanol.

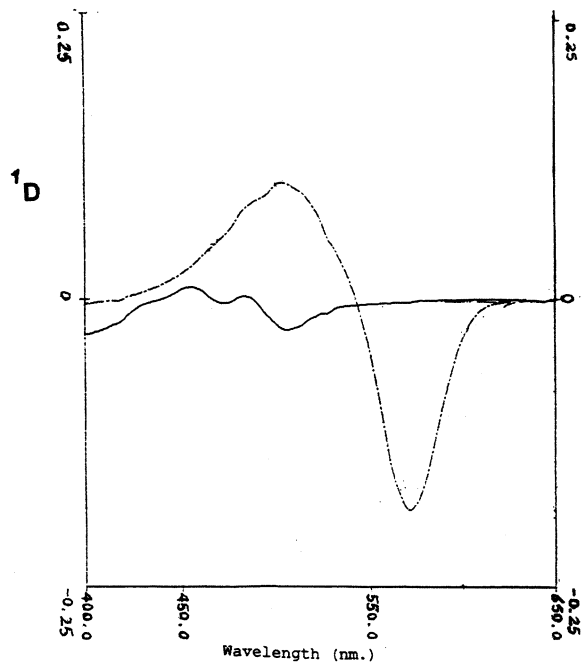


Fig. 4. First-derivative spectra of metoclopramide-*p*DACA, 10 μg ml^{-1} (–·–·–); and *p*DACA reagent, 0.2×10^{-3} M (—).

been described [16]. Liquid chromatography [17], gas chromatography [18] and high performance liquid chromatography [19,20] have been mentioned in the literature. The BP 1998 reported

Table 1
Analytical data and results of linearity study

Drug	Method	Wavelength (nm)	Concentration range (μg ml^{-1})	Regression equation (y)[a]			RSD (%)[b]
				Intercept (a)	Slope (b)	Correlation coefficient (r)	
Glafenine	Iodic acid	509	2.5–20	0.1310	0.0514	0.998	0.63
Metoclopramide HCl	pDACA: zero-order	548	5–30	0.0156	0.0254	0.996	0.51
	First-derivative	576	5–25	−2.0000	5.4400	0.999	0.48

[a] $y = a + bC$, where C is the concentration in μg ml^{-1} and y is the absorbance at 509 or 548 nm for the iodic acid method or pDACA method (zero-order), respectively, or ^{1}D (peak height) at 576 nm for pDACA method (first-derivative).

[b] Relative standard deviations ($n = 5$).

Table 2
Assay results of the accuracy study

Glafenine			Metoclopramide HCl					
Iodic acid			*p*DACA					
			Zero-order at 548 nm			First-derivative at 576 nm		
Taken (μg ml^{-1})	Found (μg ml^{-1})	Recovery (%)	Taken (μg ml^{-1})	Found (μg ml^{-1})	Recovery (%)	Taken (μg ml^{-1})	Found (μg ml^{-1})	Recovery (μg ml^{-1})
3.5	3.431	98.03	6.0	5.93	98.80	6.00	6.042	100.70
7.0	6.940	99.14	12.0	11.97	99.75	12.00	12.050	100.42
10.5	10.470	99.71	18.0	17.95	99.72	18.00	18.000	100.00
17.5	17.290	98.80	22.0	21.93	99.68	22.00	22.022	100.10
20.0	19.800	99.00	25.5	25.41	99.65	25.00	25.073	100.29
Mean		98.94			99.52			100.30
SD		0.61			0.40			0.28
RSD (%)		0.62			0.40			0.28

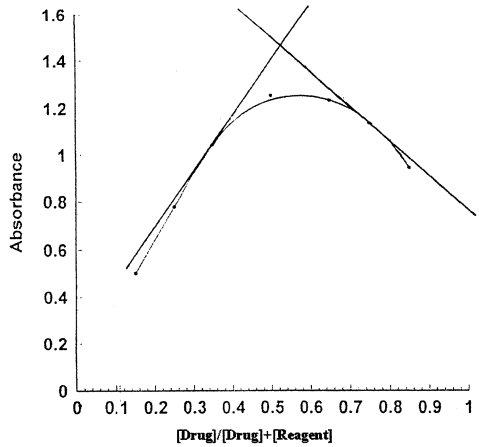


Fig. 5. Continuous variation plot for metoclopramide hydrochloride and *p*DACA in methanol (2×10^{-3} M).

a potentiometric method for the determination of metoclopramide hydrochloride powder and UV method for tablets and apmoules [21]. The potentiometric method requires about 250 mg metoclopramide hydrochloride. The UV method is liable to interferences from tablet excipients and requires pre-extraction of metoclopramide with chloroform. The aim of this investigation was to develop simple, accurate, sensitive and reproducible methods of analysis for the determination of glafenine and metoclopramide hydrochloride in microquantities both in pure and dosage forms. The suggested methods dealt with the interaction of glafenine with iodic acid to produce a violet coloured reaction product with an absorbance maximum at 509 nm. Metoclopramide hydrochloride via its primary aromatic amino group reacted instantaneously with *p*-dimethylaminocinnamaldehyde (*p*DACA) giving a red coloured schiff's base with an absorbance maximum at 548 nm. At this wavelength, *p*DACA showed slight interference and could be overcome by using the first-derivative mode. It was found that the peak height at 576 nm (zero value of *p*DACA) was proportional to metoclopramide hydrochloride concentration. The proposed methods have been applied successfully to the assay of these drugs in dosage forms.

2. Experimental

2.1. Apparatus

A digital pH/MV/,temperature ATC meter with double junction glass electrode (Jenco model 5005) was used for carrying out the potentiometric titration of metoclopramide hydrochloride by the BP 1993 method.

A Shimadzu UV–visible recording spectrophotometer, UV 265, (made in Japan) was used for

Table 3
Assay results of precision study

Replicate	Glafenine		Metoclopramide HCl *p*DACA			
			Zero-order at 548 nm		First-derivative at 576 nm	
	Taken ($\mu g\ ml^{-1}$)	Found ($\mu g\ ml^{-1}$)	Taken ($\mu g\ ml^{-1}$)	Found ($\mu g\ ml^{-1}$)	Taken ($\mu g\ ml^{-1}$)	Found ($\mu g\ ml^{-1}$)
1	10.00	10.12	20.00	20.08	20.00	20.05
2	10.00	10.09	20.00	19.96	20.00	20.10
3	10.00	9.97	20.00	19.90	20.00	19.95
4	10.00	9.90	20.00	19.97	20.00	19.98
5	10.00	10.10	20.00	20.15	20.00	19.92
Mean		10.036		20.01		20.00
SD		0.096		0.102		0.074
RSD (%)		0.957		0.51		0.369

Table 4
Assay results for the determination of glafenine in Glifarelax tablets

Glifarelax tablets[a]			Standard addition		
Claimed (μg tablet^{-1})	Found (μg tablet^{-1})	Recovery (%)[b]	Added (μg ml^{-1})	Found (μg ml^{-1})	Recovery (%)
200	195.92	97.96 ± 0.78	2.5	2.504	100.16
			3.5	3.508	100.23
			4.5	4.503	100.07
			5.00	4.994	99.88
			5.00	4.996	100.92
Mean					100.05
SD					0.15
RSD (%)					0.15

[a] Batch No.3943356.
[b] Average of five measurements ± SD.

absorbance measurements. For the first-derivative method, the following parameters were used: Mode,[1]D (first-derivative); scan speed, fast; scale, 20 nm cm^{-1}; photometric range, −0.25–0.25 absorbance; slit width, 2 nm; wavelength range; 400–650 nm.

2.2. Materials

All chemicals and reagents used were of analytical or pharmaceutical grade and distilled water was used throughout. Solvents were of spectroscopic grade.

Glafenine and Glifarelax tablets (batch number 394356) nominally containing 200 mg glafenine and 1 mg thiochlochicoside, were supplied by Memphis Chemical Company, Cairo, Egypt (under licence of Les Laboratoires Roussel, France).

Metoclopramide hydrochloride was supplied by Memphis Chemical Company, Cairo, Egypt. Plemazol tablets (batch number 898105) and ampoules (batch number 101) nominally containing 10 mg metoclopramide hydrochloride per tablet or ampoule, were supplied by CID Co., Cairo, Egypt.

2.3. Reagents

Iodic acid (BDH) reagent: 0.15% w/v solution in sulfuric acid was used.

p-Dimethylaminocinnamaldehyde (Merck–Schuchardt) reagent: 0.2% w/v solution, was prepared by dissolving 0.2 g of *p*-dimethylaminocinnamaldehyde in about 20 ml methanol and 10 ml of 65% sulfuric acid, into a 100 ml volumetric flask and the volume was completed with methanol.

Methanol (Prolabo), ethanol absolute (prolabo) and sulfuric acid (Prolabo) were used.

2.4. Preparation of standard drugs solutions

Standard solutions of glafenine (100 μg ml^{-1}) in methanol and metoclopramide hydrochloride (1 mg ml^{-1}) in ethanol were prepared.

2.5. Recommended procedures

2.5.1. Determination of glafenine using iodic acid

2.5.1.1. Construction of calibration graph. Into a series of test tubes, transfer aliquot portions of standard glafenine solution (0.25–2 ml) equivalent to (25–200 μg). Evaporate to dryness and cool. To each tube add 1 ml iodic acid solution and transfer the contents of the tubes to a series of 10 ml volumetric flasks. Wash the tubes with three quantities, each of 2 ml sulfuric acid and complete to volume with sulfuric acid. Determine the absorbances at 509 nm against a reagent blank.

Table 5
Assay results for the determination of metoclopramide hydrochloride in commercial products

Commercial product	Claimed taken (μg ml^{-1}) (%)	Zero-order at 548 nm				First-derivative at 576 nm			
		Found (%)[a]	Added (μg ml^{-1})	Recovered (μg ml^{-1})	Recovery (%)	Found (%)[a]	Added (μg ml^{-1})	Recovered (μg ml^{-1})	Recovery (%)
Plemazol tablets batch number 898105	15.00	99.48 ± 0.58	2.5	2.46	98.40	99.66 ± 0.53	2.5	2.482	99.28
			5.0	4.91	98.20		5.0	5.015	100.30
			5.0	4.97	99.40		5.0	5.023	100.46
			8.0	7.92	99.00		8.0	8.042	100.53
			10.0	9.89	98.90		10.0	10.025	100.25
	Mean				98.78				100.16
	SD				0.48				0.51
	RSD (%)				0.49				0.51
Plemazol ampoules batch number 101	15.00	100.35 ± 0.3	2.5	2.533	100.92	100.75 ± 0.33	2.5	2.511	100.44
			2.5	2.520	100.80		2.5	2.504	100.16
			4.0	4.010	100.25		4.0	4.012	100.30
			4.0	4.013	100.33		4.0	4.038	100.95
			5.0	5.006	100.12		5.0	5.035	100.70
	Mean				100.48				100.51
	SD				0.35				0.32
	RSD (%)				0.35				0.32

[a] Average of five measurement ± SD.

Table 6
Results of statistical comparison study of the cited drugs

Statistical term	Glafenine		Metoclopramide HCl		
	Iodic acid	Reference[a]	*p*DACA		
			Zero-order at 548 nm	First-derivative at 576 nm	Reference[b]
Confidence limit	98.94 ± 0.76	99.39 ± 1.2	99.52 ± 0.50	100.30 ± 0.35	99.92 ± 0.48
SD	0.61	0.97	0.40	0.28	0.39
SE	0.27	0.43	0.18	0.13	0.17
n	5	5	5	5	5
Student's *t**	0.88		1.62	1.78	
*F***	2.53		0.95	1.90	

[a] Les Laboratiores Roussel method [22]: dissolve glafenine in dioxane and titrate with 0.1 N HCl in ethanol using thymol blue indicator and carry out a blank experiment.% glafenine = $((N\text{-}n) \times 372.8)/(P(100-\text{n}))$ where N is Volume of 0.1 N HCl for the sample. n is Volume of 0.1 N HCl for the blank. P = mass for the assay sample.

[b] B.P 1998 method [20].

* Tabulated t (n_1,$n_2 = 5$) for 8 df and ($P = 0.05$) is 2.306.

** Tabulated F (n_1,$n_2 = 5$) for(4,4) df and ($P = 0.05$) is 6.39.

2.5.1.2. Determination of glafenine in Glifarelax tablets. Tranfer a quantity of mixed powdered tablets equivalent to 10 mg glafenine into a 100 ml volumetric flask. Add about 50 ml of methanol with shaking thoroughly, complete to volume with methanol and filter. Tranfer aliquot portions equivalent to 50–200 μg glafenine into a series of test tubes and proceed as mentioned under calibration graph (Section 2.5.1.1).

2.5.2. Determination of metoclopramide hydrochloride using p-dimethylaminocinnamaldehyde

2.5.2.1. Construction of calibration graphs. Into a series of 10 ml volumetric flasks, transfer aliquot portions of standard metoclopramide hydrochloride solution (0.05–0.3 ml) equivalent to (50–300 μg). Add 6.5 ml of *p*DACA reagent, complete to

Table 7
Assay results for the determination of the cited drugs in pharmaceutical preparations

Drug	Name of preparation	Mean recovery% ± SD ($n = 5$)	
	Added Authentic	Proposed methods[a]	Reference methods[b]
Glafenine	Glifarelax tablets	97.96 ± 0.78	98.63 ± 1.14
	Added authentic	100.05 ± 0.15	
Metoclopramide hydrochloride	Plemazol tablets	99.48 ± 0.58[c]	99.27 ± 0.73
	Plemazol tablets	99.66 ± 0.53[d]	
	Added authentic	98.78 ± 0.48[c]	
	Added authentic	100.16 ± 0.51[d]	
	Plemazol ampoules	100.35 ± 0.35[c]	100.17 ± 0.13
	Plemazol tablets	100.75 ± 0.33[d]	
	Added authentic	100.48 ± 0.35[c]	
	Added authentic	100.51 ± 0.32[d]	

[a] Iodic acid method for glafenine and its tablets and *p*DACA method for metoclopramide hydrochloride and its preparations.

[b] Les Laboratiores Roussel method for glafenine and its tablets [22] and B.P 1998 method for metoclopramide and its preparations [20].

[c] Zero-order method at 548 nm.

[d] First-derivative method at 576 nm.

volume with methanol and allow to stand for 10 min. Determine the absorbances at 548 nm and record the first-derivative spectrum against a reagent blank. Measure the peak height (^{1}D) at 576 nm.

2.5.2.2. Determination of metoclopramide hydrochloride in plemazol tablets. Transfer an accurately weighed quantity of powdered tablets equivalent to 100 mg metoclopramide hydrochloride to a 100 ml volumetric flask. Add about 75 ml ethanol with shaking thoroughly, complete to volume with the same solvent and filter.

2.5.2.3. Determination of metoclopramide hydrochloride in plemazol ampoules. Dilute the contents of five ampoules equivalent to 50 mg methoclopramide hydrochloride to 50 ml with ethanol.

Transfer the appropriate aliquots of the solutions obtained from (Section 2.5.2.2) or (Section 2.5.2.3) into a series of 10 ml volumetric flasks and proceed as mentioned under calibration graphs (Section 2.5.2.1).

3. Results and discussion

Iodic acid has been used as a reagent to oxidize glafenine in strong acid medium with the formation of a violet chromogen with an absorbance maximum at 509 nm. This reaction depends on the oxidation reaction of diphenylamine which is a redox indicator in acid medium to give a coloured diphenylbenzidine derivative [21].

In designing the reaction conditions for the determination of metoclopramide hydrochloride with *p*DACA, it was postulated that the lone pair on the *N*-dimethylamino group of the reagent will weaken the electrophilicity of the carbonyl carbon. Therefore, it was resorted to the use of acid medium to increase the reactivity of the aldehydo group. This postulate was proven experimentally. In neutral medium nearly no reaction took place, while if the reagent was prepared in the presence of sulfuric acid medium (as described under reagents), the reaction had proceeded quantitatively with the formation of a red coloured schiff's base as shown in Fig. 2.

Fig. 3 shows the absorption spectra of metoclopramide hydrochloride, metoclopramide-*p*DACA and *p*DACA in methanol. Metoclopramide–*p*DACA exhibits maximum absorption at 548 nm; metoclopramide hydrochloride, however, has negligible absorbance at this wavelength. Meanwhile, *p*DACA shows slight interference and could be overcome using first-derivative mode (Fig. 4). Thus the colour product was quantified by measuring the absorbance at 548 nm (zero-order method) and, the^{1}D values (peak height) at 576 nm (first-derivative method) where *p*DACA displayed zero value.

3.1. Optimum conditions for drugs determinations

The optimization of the methods was carefully studied to achieve complete reaction formation, highest sensitivity and maximum absorbances. For the determination of glafenine, different concentrations of iodic acid reagent ranging from 0.05 to 0.2% w/v in sulfuric acid were studied, 1 ml of 0.15% w/v reagent gave maximum absorbance value. The development of glafenine–iodic acid colour product was instant and the absorbance readings were constant and stable for at least $2\frac{1}{2}$ h.

For the determination of metoclopramide hydrochloride, different amounts ranging from 3–8 ml of 0.2% w/v *p*DACA reagent in methanol containing 10 ml of 65% v/v sulfuric acid were tried, 6.5 ml of the reagent was found optimum for complete colour formation. Trichloroacetic acid was tried but erroneous results were obtained. Effect of heat at 60–70°C for 15–20 min was studied but higher absorbances were obtained at room temperature. The formation of metoclopramide–*p*DACA coloured product was rapid, hence maximum absorbance was attained after about 10 min and remained stable for at least 40 min.

3.2. Stoichiometric relationship

The mole ratio of metoclopramide–*p*DACA reaction product was determined by applying Job's method of continuous variations. The concentration of metoclopramide hydrochloride in

ethanol absolute and *p*DACA reagent was (2×10^{-3} M). Fig. 5 indicates the formation of 1:1 mole ratio.

3.3. Linearity

Under the experimental conditions described for drugs determinations, standard calibration curves for glafenine and metoclopramide hydrochloride (zero-order method) were constructed by plotting absorbances versus concentrations. For the first-derivative method of metoclopramide hydrochloride, the standard calibration curve was constructed by plotting the peak heights at 576 nm versus concentrations. Linear relationships were obtained in the concentration ranges 2.5–20 μg ml^{-1} glafenine, 5–30 μg ml^{-1} and 5–25 μg ml^{-1} metoclopramide hydrochloride for the zero-order and the first-deivative methods, respectively. The regression line equations for both drugs are tabulated in Table 1. The correlation coefficients were between 0.999–0.996 indicating good linearity.

3.4. Accuracy and reproducibility

Table 2 shows the accuracy of the proposed methods. Recovery studies were performed with five different concentrations of each of the studied drug. The mean recoveries were 98.94% for glafenine, 99.52 and 100.3% for metoclopromide hydrochloride with the zero-order and the first-derivative methods, respectively. The relative standard deviations (RSD) were found to be less than 1%. The reproducibility of the proposed methods was tested by running five replicate samples each containing 10 μg ml^{-1} glafenine or 20 μg ml^{-1} metoclopramide hydrochloride. At this concentration levels the RSD were 0.957% for glafenine, 0.51 and 0.369% for metoclopramide hydrochloride using the zero-order and the first-derivative methods, respectively. Results in Tables 2 and 3, demonstrate that the proposed methods are accurate in addition of being precise and reproducible.

3.5. Quantification and detection limits

The limits of quantification were taken as the lower limits of the concentration ranges of the methods, i.e. 2.5 μg ml^{-1} for glafenine and 5 μg ml^{-1} for metoclopramide hydrochloride. The detection limits were 1.5 μg ml^{-1} for glafenine and 5 μg ml^{-1} for metoclopramide hydrochloride. Comparing the A (1%, 1 cm) of metoclopramide hydrochloride with the ^1D (1%, 1 cm), the first-derivative procedure would show an exceedingly higher sensitivity.

Average A (1%, 1 cm) = 249

Average D_1 (1%, 1 cm) = 53240

In other words, the first-derivative mode was at least 214 times more sensitive than the zero-order one.

3.6. Application to pharmaceutical preparations

The proposed methods were successfully applied to the analysis of commercial products of glafenine (Glifarelax tablets) and metoclopramide hydrochloride (plemazol tablets and ampoules), results are found in Tables 4 and 5. No interferences were observed in the determination of the studied drugs in the presence of the common excipients of the tablets, (e.g. starch, magnesium stearate, sucrose, lactose and glucose). The concomitantly present thlocolchicoside in Glifarelax tablets with glafenine didn't interfere with the iodic acid method and required no prior separation. Thiocolchicoside was experimentally checked that it produced no reaction with iodic acid under the experimental conditions. Furthermore, the validity of the proposed methods was assessed by applying the standard addition technique. Results found in Tables 4 and 5 showed statisfactory recovery and confirmed the validity and the accuracy of the methods. The results given by the proposed and the reference methods [20,22] were statistically compared and found not to differ significantly Tables 6 and 7.

4. Conclusions

It can be concluded that, the present methods have the advantages of high sensitivity over the official method, since the minimum quantifiable limits were taken as 2.5 μg ml^{-1} for glafenine and 5 μg ml^{-1} for metoclopramide hydrochloride by

the proposed methods. Concerning the published UV methods, necessitate pre-treatment procedures involving extraction of the active ingredient to avoid interference from tablet excipients. However the present methods are simple as there is no need for solvent extraction or separation steps before the analysis, since no interferences were observed either from tablet excipients or the co-existing thiocolchicoside.

Additionally to these advantages, the proposed methods are accurate and precise as indicated by the good recoveries of both drugs and low RSD values.

The above findings substantiate the usefulness of the proposed methods for the assay and quality control of glafenine and metoclopramide hydrochloride both in the pure and dosage forms.

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