

King Saud University

Arabian Journal of Chemistry

www.ksu.edu.sa



ORIGINAL ARTICLE

Spectrophotometric determination of carbamazepine and mosapride citrate in pure and pharmaceutical preparations

Eman Y.Z. Frag, M.A. Zayed *, M.M. Omar, Sally E.A. Elashery, Gehad G. Mohamed

Chemistry Department, Faculty of Science, Cairo University, Gamaa Str., 12613 Giza, Egypt

Received 2 January 2011; accepted 20 February 2011 Available online 24 February 2011

KEYWORDS

Carbamazepine and mosapride citrate; Ion-pair formation; Charge transfer; Spectrophotometry Abstract Simple, rapid and sensitive spectrophotometric methods were developed for the determination of carbamazepine and mosapride citrate drugs in pure and pharmaceutical dosage forms. These methods are based on ion pair and charge transfer complexation reactions. The first method is based on the reaction of the carbamazepine drug with Mo(V)-thiocyanate in hydrochloric acid medium followed by an extraction of the coloured ion-pair with 1.2-dichloroethane and the absorbance of the ion pair was measured at 470 nm. The second method is based on the formation of ionpairs between mosapride citrate and two dyestuff reagents namely bromothymol blue (BTB) and bromocresol green (BCG) in a universal buffer of pH 4 and 3, respectively. The formed ion-pairs are extracted with chloroform and methylene chloride and measured at 412 and 416 nm for BTB and BCG reagents, respectively. The third method is based on charge transfer complex formation between mosapride citrate (electron donor) and DDQ (π-acceptor reagent) and the absorbance of the CT complexes was measured at 450 nm. All the optimum conditions are established. The calibration graphs are rectilinear in the concentration ranges 10-350 for carbamazepine using Mo(V)thiocyanate and 4-100, 4-60 and 10-150 µg mL⁻¹ for mosapride citrate using BTB, BCG and DDQ reagents, respectively. The Sandell sensitivity (S), molar absorptivity, correlation coefficient, regression equations and limits of detection (LOD) and quantification (LOQ) are calculated. The law values of standard deviation (0.04-0.09 for carbamazepine using Mo(V)-thiocyanate and 0.022-0.024,

E-mail address: mazayed429@yahoo.com (M.A. Zayed).

1878-5352 © 2011 King Saud University. Production and hosting by Elsevier B.V. All rights reserved.

Peer review under responsibility of King Saud University. doi:10.1016/j.arabjc.2011.02.023



Production and hosting by Elsevier

^{*} Corresponding author. Tel.: +20 2 22728437; fax: +20 2 35728843.

0.013–0.018 and 0.013–0.020 for mosapride citrate using BTB, BCG and DDQ, respectively) and relative standard deviation (0.630–2.170 for carbamazepine using Mo(V)–thiocyanate and 0.123–1.43, 0.102–0.530 and 0.226–1.280 for mosapride citrate using BTB, BCG and DDQ, respectively) reflect the accuracy and precision of the proposed methods. The methods are applied for the assay of the two investigated drugs in pharmaceutical dosage forms. The results are in good agreement with those obtained by the official method.

© 2011 King Saud University. Production and hosting by Elsevier B.V. All rights reserved.

1. Introduction

Carbamazepine (CBZ) has the IUPAC name 5-H-dibenz[b,f]azepine-5-carboxamide (Scheme 1) of molecular weight 236.269 g mol $^{-1}$ and molecular formula $C_{15}H_{12}N_2O$. Carbamazepine (CBZ) is an anticonvulsant and mood stabilizing drug used primarily in the treatment of epilepsy and bipolar disorder, as well as trigeminal neuralgia (http://en.wikipedia.org/wiki/main_page). It is also used off-label for a variety of indications, including attention-deficit hyperactivity disorder (ADHD), schizophrenia, phantom limb syndrome, paroxysmal extreme pain disorder, and post-traumatic stress disorder.

The methods available for the determination of carbamazepine included liquid chromatography-electrospray ionization mass spectrometry (Breton et al., 2005), high performance liquid chromatography (Oh et al., 2006; Yoshida et al., 2006; Ha et al., 2006; Patil and Bodhankar, 2005), flow injection analysis (Zhang et al., 2006), micellar electrokinetic capillary chromatography (Liu et al., 2006), solid phase extraction (Xiu et al., 2003), chemiluminescence (Lee et al., 2003) and gas chromatographic methods (Minkova and Getova, 2001).

Mosapride citrate (MOC) has the IUPAC name 4-amino-5-chloro-2-ethoxy-*N*-[[4-[(4-fluorophenyl)methyl]-2-morpholinyl]methyl]-benzamide-2-hydroxy-1,2,3-propanetricarboxylate (Scheme 2) of molecular weight 650.05 g mol⁻¹ and molecular

Scheme 1 Structure of carbamazepine.

Scheme 2 Structure of mosapride citrate.

formula $C_{27}H_{37}ClFN_3O_{12}$ (http://www.chemblink.com/products/112885-42-4.htm). Mosapride is a selective serotonin 5-HT4 receptor agonist drug used for a short-term treatment of erosion and ulceration of the esophagus caused by the gastroesophageal reflux disease.

A number of studies were described for the determination of mosapride citrate in both pure and pharmaceutical samples including high-performance liquid chromatography (Rao et al., 2006), liquid chromatography—tandem mass spectrometry (Aoki et al., 2007) and spectrophotometric methods (Rajput et al., 2005; Bhatt et al., 2009; Shamkant et al., 2009; Revanasiddappa and Veena, 2007).

This work is undertaken in order to test the sensitivity, accuracy and selectivity of new spectrophotometric methods and to use these methods for the determination of carbamazepine and mosapride citrate and also to study the analytical aspects of the reaction between these drugs and different reagents. The proposed methods are applied successfully to the determination of carbamazepine and mosapride citrate either in pure or in dosage forms, with good accuracy and precision. The results were compared with those given by the official methods.

2. Experimental

2.1. Materials and solutions

All chemicals and reagents used were of analytical reagent grade. They included carbamazepine (CBZ) provided by Unipharma, Egypt, and mosapride citrate (MOC) provided by Western Pharmaceutical Industries, Egypt. Reagents used included 2,3-dichloro-5,6-dicyano-p-benzoquinone (DDQ) supplied from Arcos-USA. Dyestuffs included bromocresol green (BCG) and bromothymol blue (BTB) (they were purchased from Win lab, UK).

Nitric, sulfuric and hydrochloric acids were supplied from Merck. Absolute ethanol and sodium hydroxide were supplied from Adwic, while propan-2-ol and acetonitrile (AR) were supplied from Aldrich. Chloroform, methanol, acetone, ethylene chloride, methylene chloride, dimethyl formamide, tetrahydrofuran, ascorbic acid, ammonium thiocyanate and ammonium molybdate were supplied from El-Nasr Company, Egypt.

Stock (1 mg mL⁻¹) solutions of CBZ and MOC drugs were prepared by dissolving the accurate weighed amount in a definite volume of acetonitrile and methanol, respectively, to get the required concentration. Dilute solutions were prepared by accurate dilution from the stock solution to get the desired concentrations.

10% (w/v) Solutions of each of ascorbic acid and ammonium thiocyanate were prepared by dissolving the accurate

weight (10 g) of each substance in 100 mL bidistilled water. 0.02% (w/v) ammonium molybdate solution was prepared by dissolving the accurately weighed (0.02 g) of ammonium molybdate in bidistilled water.

Universal buffer solutions of different pH values ranging from 2 to 6 were prepared by adjusting 100 mL solution of the acid mixture to the desired pH value using 0.1 N NaOH solutions. Borax and acetate buffer solutions were prepared using the recommended method by Britton and Robinson (Collector, 1960).

0.1% (w/v) of 2,3-dichloro-5,6-dicyano-*p*-benzoquinone (DDQ) reagent was prepared by dissolving 10 mg of DDQ in 100 ml acetonitrile. The dyestuffs were used as 0.1% solution of BTB and 0.02% solution of BCG, all in 10% (v/v) methanol. 4 mol L^{-1} acid solutions (HCl, H_2SO_4 and HNO_3) were prepared by accurate dilution with bidistilled water from concentrated solutions.

The water was always twice distilled from all glass equipments. Redistillation was carried out from alkaline permanganate solution.

Tegretol was supplied from Novartis Pharma, Cairo, Egypt. Each tablet contains 200 mg carbamazepine. Mosapride was produced by Western Pharmaceutical industries, Cairo, Egypt. Each tablet contains 5 mg mosapride citrate.

2.2. Apparatus

The spectrophotometric measurements were carried out using a manual Unico 1200 spectrometer (United Products and Instruments, Inc.) in the wavelength range from 325 to 1000 nm.

2.3. Procedure

2.3.1. Determination of carbamazepine (CBZ) using Mo(V)-thiocyanate

1 mL of 0.02% (w/v) of Mo(VI) solution, 1 mL of 4 M HCl, 1 mL of 10% (w/v) ascorbic acid and 1 mL of 10% (w/v) ammonium thiocyanate solution were placed in a 100 mL capacity separating funnel. The mixture was left for 15 min at 40 °C. Different volumes (0.1–3.5 mL) of carbamazepine (1 mg mL $^{-1}$) were added and diluted with bidistilled water up to 10 mL. After another 10 min, 10 mL of dichloroethane was added twice with 5 mL portions and the solution mixture was shaken vigorously for 1 min. The solution was allowed to be separated into two phases. The organic layer was collected in a 10 mL measuring flask. The absorbance of the formed ion-pair was measured at 470 nm, against a blank solution.

2.3.1.1. Procedure for the tablets. The content of 10 tablets of carbamazepine was weighed and ground into a fine powder. An accurately weighed portion of the powder equivalent to 0.5 tablet (100 mg) was transferred into a 100 mL calibrated measuring flask and the volume was made up to the mark with bidistilled water. The solution was filtered in a 100 mL calibrated measuring flask and then the solution was completed to 100 mL with bidistilled water. The procedure mentioned above was followed where different concentrations of CBZ in the range of $10-350~\mu g~mL^{-1}$ were added. The drug concentrations were calculated from the standard calibration graph prepared under identical conditions.

2.4. Determination of mosapride citrate (MOC) using dyestuff reagents (BTB and BCG)

Different volumes of 1 mg mL⁻¹ of mosapride citrate (0.02–0.5 and 0.02–0.2 mL in case of BTB and BCG, respectively) were transferred into a 50 mL separating funnel then 0.7 mL or 1 mL of universal buffer of pH 4 or pH 3 in case of using BTB or BCG reagents, respectively, were added. Then 1 mL of 1 mg mL⁻¹ BTB or 1.5 mL of 0.02% BCG was added to the mixture. The solution was completed to 10 mL with bidistilled water and shaken well. After the reaction mixture was left for a selected period of time for each complex formation, the ion-pairs were collected in a 10 ml measuring flask using 10 mL methylene chloride in the case of BCG and 10 mL (5 mL × two times) chloroform in the case of BTB, after shaking for one minute. The calibration curves were constructed at 412 and 416 nm using BTB and BCG reagents, respectively, against a blank solution.

2.4.1. Procedure for the tablets

The content of 10 tablets of mosapride citrate was weighed and ground into a fine powder. A mass of powder containing approximately 50 mg of the drug was weighed accurately, dissolved in 50 mL of methanol. The solution was filtered in a 50 mL calibrated measuring flask and then the solution was completed to 50 mL with methanol. The procedure mentioned above was followed where different concentrations of mosapride citrate in the range of 4–100 and 4–60 µg mL⁻¹ were added for BTB and BCG reagents, respectively. The drug concentrations were calculated from the standard calibration graph prepared under identical conditions.

2.5. Determination of mosapride citrate using DDQ

 $1.5\,ml$ of $1\,mg\,mL^{-1}$ DDQ was added to different concentrations of MOC (10–150 $\mu g\,ml^{-1}$). The mixtures were completed up to 10 ml with acetonitrile. The absorbance of the coloured CT complexes was measured at 450 nm, against a blank solution.

2.5.1. Procedure for the tablets

The content of 10 tablets of mosapride citrate was weighed and ground into a fine powder. A mass of powder containing approximately 50 mg of the drug was weighed accurately, dissolved in 50 mL of methanol. The solution was filtered in a 50 mL calibrated measuring flask and then the solution was completed to 50 mL with methanol. The procedure mentioned above was followed where different concentrations of mosapride citrate in the range of 10–150 $\mu g\,m L^{-1}$ were added. The drug concentrations were calculated from the standard calibration graph prepared under identical conditions.

3. Results and discussion

3.1. Determination of carbamazepine using Mo(V)-thiocyanate reagent

This work is undertaken from the view that an ion-pair is formed between the amine group of carbamazepine and Mo(V)—thiocyanate binary complex (Abdel-Gawad and El-Guindi, 1995). The ion-pair formed is soluble in 1,2-dichlo-

roethane, while Mo(V)-thiocyanate binary complex is insoluble. The absorption spectrum of the ion-pair shows a maximum at 470 nm against a blank reagent as shown in Fig. 1. It is found that, the reduction probability of Mo(VI) to Mo(V) may occur by ascorbic acid or by SCN in acidic medium. The sensitivity and stability of Mo(V)-thiocyanate binary complex are enhanced considerably by using ascorbic acid. Ascorbic acid gives reproducible value and masks many interfering ions (Mohamed et al., 2006). It is found that 5 mg mL $^{-1}$ of 10% ascorbic acid is sufficient for a complete conversion of 0.01 mg mL^{-1} of 0.02% (w/v) Mo(VI) to Mo(V). Also, it is found that 5 mg mL⁻¹ of 10% ammonium thiocyanate is required for maximum absorbance in a final volume of 10 mL aqueous solution. The maximum absorbance of the formed ion-pair is obtained using 1 mL of 4 M hydrochloric acid (Thimmaiah et al., 1986). Eq. (1) represents the reaction of Mo(VI) with ammonium thiocyanate in 1 mL 4 M HCl and in the presence of ascorbic acid. In this method, the complete formation of the ion-pair needs 10 min before extraction with 1,2-dichloroethane at 40 °C as shown in Fig. 2. The absorbance of Mo(V)-thiocyanate binary complex is stable after 15 min while the ion-pair needs another 10.0 min for its complete formation.

$$Mo(VI) \xrightarrow{Ascorbic acid} Mo(V) \xrightarrow{6SCN^{-}} Mo(SCN)_{6}^{-}$$
 (1)

3.1.1. Stoichiometry of the ion-pair

The stoichiometry of Mo(V) thiocyanate-carbamazepine ion pair in the presence of excess amount of ammonium thiocyanate is determined by the continuous variation and molar ratio methods (Vosburgh and Cooper, 1941; Job, 1928) in order to check the ratio between Mo(V) and carbamazepine. The data obtained are shown in Fig. 3. The results indicate that a 1:1 [Mo(V)-thiocyanate]: [drug] ion-pair is formed through the electrostatic attraction between positive protonated drug CBZ^+ and thiocyanate negative complex as shown in Scheme 3.

3.1.2. Validity of Beer's law

Under the optimum conditions described above, the calibration graph can be constructed for the investigated drug. Table 1 shows the analytical parameters obtained for the determina-

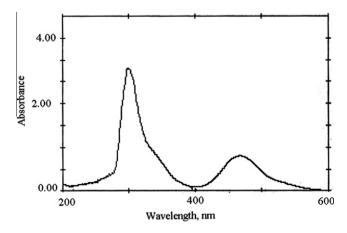
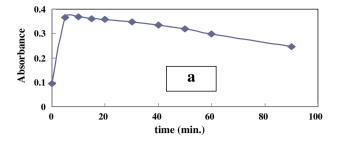


Figure 1 Absorption spectrum of CBZ-Mo(V)–thiocyanate ion pair in 1,2-dichloroethane.



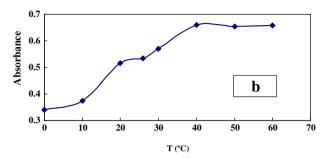


Figure 2 Effect of time (a) and temperature (b) on the determination of CBZ using Mo(V)-thiocyanate-drug ion pair at $\lambda_{max} = 470$ nm.

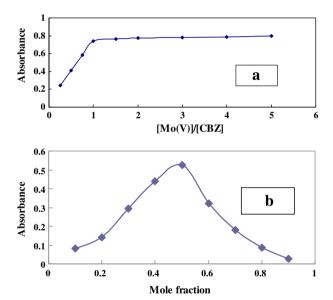
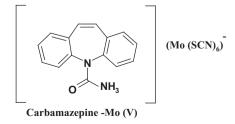


Figure 3 Stoichiometric ratio of the reaction of Mo(V)–thiocyanate with the CBZ at $\lambda_{\text{max}} = 470 \text{ nm}$ using (a) molar ratio and (b) continuous variation methods.



Scheme 3 Proposed structure of Mo(V)-carbmazepine ion pair.

Parameters		Drug					
		Mosapride citrate	Carbamazepine				
		ВТВ	BCG	DDQ	MO(V)		
λ_{\max} (nm)		412	416	450	470		
Time (min)		5	10	5	10		
T (°C)		20	28	20	40		
[Drug] ($\mu g m L^{-1}$)		4–100	4–60	10-150	10-350		
$\varepsilon (L \text{ mol}^{-1} \text{ cm}^{-1})$		3.57×10^{3}	1.87×10^{3}	3.45×10^{3}	1.90×10^{3}		
$S (\mu g cm^{-2})$		0.430	0.823	0.446	2.22		
Percent recovery (%)		99.87-100.0	100-101.3	100-100.2	100-100.3		
A = mC + z	M	0.0186	0.0275	0.0047	0.0039		
	z	0.0943	0.1552	0.0581	0.0663		
r		0.9997	0.9991	0.9999	0.9998		
SD		0.022-0.024	0.013-0.0182	0.013-0.02	0.04-0.09		
RSD (%)		0.123-1.43%	0.102-0.53%	0.226-1.28%	0.63-2.17%		
LOD $(\mu g mL^{-1})$		0.371	0.169	0.956	3.36		
$LOQ (\mu g m L^{-1})$		1.236	0.566	3.189	11.21		

tion of carbamazepine using Mo(V)-thiocyanate reagent. The absorbance-concentration curve is found to be rectilinear and Beer's law is obeyed in the concentration range 10-350 µg ml ⁻¹. The mean recovery values obtained amount in the range 100-100.3%. The correlation coefficient of the data obtained is 0.9998. The Sandell sensitivity (S) is found to be $2.22 \,\mathrm{\mu g} \,\mathrm{cm}^{-2}$. The limit of detection (LOD) and quantification (LOQ) are found to be 3.36 and 11.21 μ g mL⁻¹, respectively. The SD is found to be 0.04-0.09 and the RSD is 0.63-2.17%. The low values of the relative standard deviation indicate the high accuracy and precision of the method. This is supported also by the calculated values of Sandell sensitivity which indicates the high sensitivity of the method.

3.2. Determination of mosapride citrate (MOC) using BTB and BCG reagents

Mosapride citrate drug is present in a positively charged protonated form and dyestuff reagents present mainly in an anionic form at pH \geqslant 3. So by adding 0.7 and 1 mL of pH 4.0 and 3.0 of universal buffer using BTB and BCG reagents, respectively, the ion-pairs which are extracted with 10 mL of chloroform (5 mL \times 2) and 10 mL of methylene chloride (10 mL \times 1) using BTB and BCG, respectively, are formed. The spectra of the ion-pair reaction products show characteristic λ_{max} at 412 and 416 nm for mosapride citrate using BTB and BCG reagents, respectively (Fig. 4). The optimum reaction conditions for the determination of the ion-pair complexes were established. The effect of varying the concentration of 1 mg mL⁻¹ BTB or 0.02% w/v BCG on the intensity of the coloured product was studied. It is found that maximum absorption was obtained by adding 140 and 300 µg mL⁻¹ of BTB and BCG, respectively. The optimum reaction time is determined at an ambient temperature. It is found that the optimum time for the completion of the reaction of mosapride citrate is 5 and 10 min using BTB and BCG reagents, respectively.

3.2.1. Stoichiometry of the formed ion-pairs

Job's method of continuous variation (Job, 1928) of equimolar solution was employed. Series solutions were prepared in which the total volume of drug and reagent is kept constant.

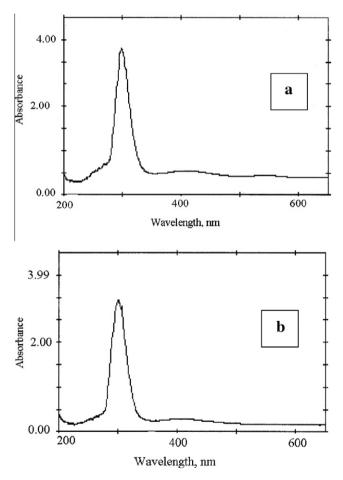


Figure 4 Absorption spectra of MOC ion pairs using (a) BTB and (b) BCG reagents in chloroform and methylene chloride, respectively.

The molar ratio of the reagents (drug:dye) in the ion pair complexes was determined (Vosburgh and Cooper, 1941). The results indicate that 1:1 (drug:dye) ion-pairs are formed through the electrostatic attraction between positive

protonated MOC⁺ and negative BTB⁻ and BCG⁻ as shown in Scheme 4. The data obtained are illustrated in Fig. 5. The extraction equilibrium can be represented as follows:

$$MOC_{(aq)}^+ + D_{(aq)}^- \leftrightarrow MOC^+D_{(aq)}^- \leftrightarrow MOC^+D_{(org)}^-$$
 (2)

where MOC⁺ and D⁻ represent the protonated mosapride citrate and the anion of the dye, respectively, and the subscript (aq) and (org) refer to the aqueous and organic phases, respectively.

3.2.2. Validity of Beer's law

After optimizing experimental conditions for mosapride citrate determination, the calibration graph can be constructed by plotting absorbances versus concentrations. Table 1 shows the analytical parameter data obtained for the determination of mosapride citrate drug using BTB and BCG reagents. The absorbance-concentration curves are found to be rectilinear and Beer's law is obeyed in the concentration ranges 4-100 and 4-60 µg mL⁻¹ for BTB and BCG reagents, respectively. The mean recovery values, correlation coefficients, Sandell sensitivity (S), limit of detection (LOD) and quantification (LOO), standard deviation (SD) and relative standard deviation (RSD) are calculated and listed in Table 1. The low values of the relative standard deviation indicate the high accuracy and precision of the method. This is supported also by the calculated values of Sandell sensitivity; it indicates the high sensitivity of the method.

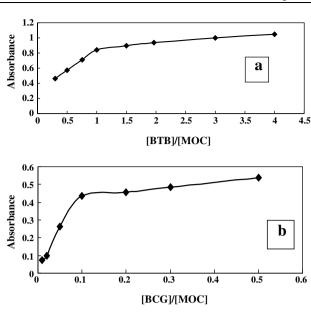
3.3. Determination of mosapride citrate using DDQ

This method is based on determination of mosapride citrate via charge transfer complex formation reaction between the

Mosapride citrate-BTB ion

Mosapride citrate -BCG ion pair

Scheme 4 Structures of MOC-BTB and -BCG ion pairs.



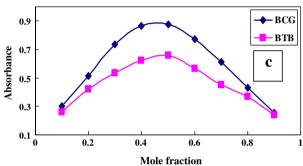


Figure 5 Stoichiometric ratio of the reaction of MOC with (a) BTB and (b) BCG reagents using molar ratio and (c) continuous variation methods.

drug (electron donor) and DDQ (π -acceptor). The spectrum of the reaction product shows characteristic λ_{max} at 450 nm. The experimental conditions were established by varying one variable and observing its effect on the absorbance of the coloured species. The effect of the concentration of DDQ on the intensity of the coloured product was studied. It is found that maximum absorption is obtained by adding 1.5 mg mL⁻¹. Several organic solvents were tried, such as acetonitrile, methanol, ethanol, acetone, tetrahydrofuran, dimethyl formamide, chloroform and propan-2-ol. The results obtained are shown in Table 2. Although ethanol and dimethyl formamide have high molar absorptivity than acetonitrile but the stability

Table 2 The molar absorptivity values of MOC-DDQ CT complex in different solvents.

Organic solvents	A	$\varepsilon (\mathrm{L} \mathrm{mol}^{-1} \mathrm{cm}^{-1})$
Acetonitrile	0.667	4.34×10^{3}
Acetone	0.515	3.35×10^{3}
Methanol	0.248	1.61×10^{3}
Dimethyl formamide	0.719	4.67×10^{3}
Tetrahydrofuran	0.084	0.55×10^{3}
Ethanol	1.094	7.11×10^{3}
2-Propanol	0.358	2.33×10^{3}

Scheme 5 Structure of MOC-DDQ CT complex.

and reproducibility of the absorbance values of the CT complex are stable and reproducible in acetonitrile solvent. It is found that, the optimum time for the completion of the reaction of mosapride citrate is 5 min at 20 °C.

3.3.1. Stoichiometry of the formed ion-pairs

Continuous variation and molar ratio methods are applied in order to determine the suitable ratio between MOC drug and DDQ reagent. It is found that the interaction between this drug and reagent occurs in equimolar basis, i.e., 1:1 CT complex is formed between the drug and DDQ reagent. A π - π * CT complex is formed through the benzene ring (electron rich group) of the MOC as electron donor and the electron-acceptor reagent (DDQ) (Frag and Mohamed, 2010). The structure of the CT complex formed between the drug under study and DDQ reagent is shown in Scheme 5.

3.3.2. Validity of Beer's law

After the selection of suitable solvents, reagent concentrations, reaction time, temperature, and ratio, the calibration curve can be constructed by plotting absorbances versus concentrations. Beer's law was valid over the concentration range $10-150 \, \mu \mathrm{g} \, \mathrm{mL}^{-1}$ for the DDQ reagent. Table 1 shows the different analytical parameters obtained such as slope, intercept, correlation coefficient, Sandell sensitivity, molar absorptivity (ε),

Table 3 Between-day precision of the determination of CBZ using Mo(V)-thiocyanate reagent and MOC using BTB, BCG and DDQ reagents.

Compound [Drug], taken (µg mL		[Drug], found ($\mu g \ mL^{-1}$)	Percentage recovery (%)	SD^*	RSD* (%)	
CBZ	50.00	50.28	100.6	0.021	1.96	
	100.00	100.4	100.4	0.017	1.08	
	150.00	150.4	100.3	0.022	1.12	
MOC	20.00	19.83	99.15	0.05	0.695	
BTB	50.00	49.81	99.61	0.01	0.684	
MOC	20.00	20.08	100.4	0.03	0.479	
BCG	40.00	40.01	100.0	0.04	2.980	
MOC	10.00	10.19	102.0	0.017	0.914	
DDQ	100.0	101.0	101.0	0.077	1.288	

Mean values for five experiments carried out on four days.

Table 4 Spectrophotometric determination of CBZ and MOC in different pharmaceutical preparations by proposed and official methods.

Sample	Proposed		Official		% Recovery		SD		
	[Drug] μg mL ⁻¹		[Drug] μg	[Drug] μg mL ⁻¹					
	Taken	Found	Taken	Found	Proposed	Official	Proposed	Official	
Tegretol	50.00	49.71	20.00	19.87	99.43	99.35	0.011	0.298	
	100.0	99.46			99.46		0.011		
	150.0	149.8			99.87		0.025		
MosaprideBTB	20.00	20.4	15.00	14.89	102.12	99.27	0.008	0.300	
_	50.00	51.16			102.32		0.027		
	80.00	80.85			101.06		0.018		
Mosapride BCG	10.00	9.79	15.00	14.89	97.92	99.27	0.003	0.300	
•	20.00	19.86			99.29		0.005		
	40.00	39.94			99.84		0.003		
Mosapride DDQ	10.00	9.850	5.00	4.92	98.50	98.40	0.017	0.030	
	50.00	50.34			100.7		0.050		
	100.0	100.6			100.6		0.026		

standard deviation, and relative standard deviation, limit of quantification and limit of detection. The small value of Sandell sensitivity indicates the high sensitivity of the proposed method in the determination of the drug under investigation.

4. Quantification, accuracy and precision of the proposed methods

A linear correlation is found between absorbance and concentration in the ranges given in Table 1. The correlation coefficients, intercepts and slopes for the calibration data for the two cited drugs were calculated. The accuracy and precision of the proposed methods were established by measuring the content of carbamazepine and mosapride citrate in pure form at different concentration levels. The between day precision of the proposed methods is performed by carrying out four replicate experiments at each concentration level within four days (Table 3). The results of standard deviation (SD), relative standard deviation (RSD) and recoveries by the proposed methods in Table 1 can be considered to be very satisfactory. Thus the proposed methods are very effective for the assay of carbamazepine and mosapride citrate in drug formulations. The applicability of the proposed methods for the determination of carbamazepine and mosapride citrate has been tested on commercially available pharmaceutical formulations and the data obtained are listed in Table 4. The results of the proposed methods were compared with those obtained by the official method (Krishnaia et al., 2002; Comoglu et al., 2006). Statistical analysis of the results did not detect any significant difference in the performance of the proposed method to the reference method with respect to accuracy and precision as revealed by the values of percentage recovery.

5. Conclusion

The data given above reveal that the proposed methods are simple, accurate and sensitive with good precision and accuracy. Also, the reagents utilized in the proposed methods are cheaper, readily available and the procedures do not involve any critical reaction conditions or tedious sample preparation. Thus, this proposed spectrophotometric method can be successfully applied for the determination of carbamazepine and mosapride citrate in pure form and in pharmaceutical preparations.

References

Abdel-Gawad, F.M., El-Guindi, N.M., 1995. Anal. Lett. 28, 1437. Aoki, Y., Hakamata, H., Igarashi, Y., Uchida, K., Kobayashi, H., Hirayama, N., Kotani, A., Kusub, F., 2007. J. Chromatogr. B 858, 135

Bhatt, H.S., Mehta, R.S., Christian, M., Maradiya, R., 2009. Int. J. Pharm. Res. 1, 29.

Breton, H., Cociglio, M., Bressolle, F., Peyriere, H., Blayac, JP., Buys, H.D., 2005. J. Chromatogr. B 828, 80.

Collector, J.E., 1960. Ann. Chim. 5, 415.

Comoglu, T., Gonul, N., Sener, E., Dal, A.G., Tuncel, M.J., 2006. Liq. Chromatogr. Related Technol. 29, 2677.

Frag, E.Y., Mohamed, G.G., 2010. J. Mol. Struct. 979, 46.

Ha, D.K., Wang, L., Wang, Y.C., 2006. Yaowu. Fenxi. Zazhi 26, 212. http://en.wikipedia.org/wiki/main_page > .

< http://www.chemblink.com/products/112885-42-4.htm > .

Job, P., 1928. Ann. Chim. 9, 113.

Krishnaia, Y.S.R., Murthy, T.K., Sankar, D.G., Satvanrayana, V., 2002. Anal. Sci. 18, 1269.

Liu, Y.H., Yang, X.H., Niu, Z.J., Wang, M.Z., Tian, B.Y., 2006. J. Fenxi. Kexue. Xuebao 22, 464.

Lee, S.H., Li, M., Suh, J.K., 2003. J. Anal. Sci. 19, 903.

Minkova, G., Getova, D., 2001. Methods Find. Exp. Clin. Pharmacol. 23, 481.

Mohamed, G.G., Nour El-Dien, F.A., Khalil, S.M., Mohamed, N.A., 2006. Spectrochim. Acta, Part A 65, 1221.

Oh, E.K., Ban, E., Woo, J.S., Kim, C.K., 2006. Anal. Bioanal. Chem. 386, 1931.

Patil, K.M., Bodhankar, S.L., 2005. J. Pharm. Biomed. Anal. 39, 181.
Rajput, S., Sankalia, M.G., Patel, F.T., 2005. Indian J. Pharm. Sci. 67, 582

Rao, R.N., Nagaraju, D., Narasaraju, A., 2006. J. Pharm. Biomed. Anal. 40, 338.

Revanasiddappa, H.D., Veena, M.A., 2007. J. Ecl. Quím. São Paulo 32. 71.

Shamkant, P.S., Pandurang, D.N., Bhanudas, K.S., 2009. Int. J. Pharm. Technol. Res. 1, 1458.

Thimmaiah, K.N., Chandrappa, G.T., Sekhar, V.C., 1986. Mikrochim. Acta 111, 227.

Vosburgh, W.C., Cooper, G.R., 1941. J. Am. Chem. Soc. 63, 437.

Xiu, S.M., Metcalfe, Chris D., 2003. Anal. Chem. 75, 3731.

Yoshida, T., Imai, K., Motohashi, S., Hamano, S., Sato, M., 2006. J. Pharm. Biomed. Anal. 41, 1386.

Zhang, Z.Q., Liang, G.X., Ma, J., Lei, Y., Lu, Y.M., 2006. Anal. Lett. 39, 2417.