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ORIGINAL ARTICLE

Sensitive spectrophotometric determination of metoclopramide hydrochloride in dosage forms and spiked human urine using vanillin

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Abstract A new spectrophotometric method which is simple, sensitive, selective and rapid is described for the determination of metoclopramide hydrochloride (MCP) in bulk drug and in dosage forms using vanillin as the chromogenic agent. The method is based on the condensation reaction between primary aromatic amine group present in MCP with aromatic aldehyde, vanillin to produce an intense yellow colored product. The resulting Schiff's base shows an absorption maximum at 410 nm and the reaction product is stable for more than one day. The reaction was carried out in acetic acid and perchloric acid medium. Beer's law was obeyed in the concentration range 1.5–15.0 μg ml^{-1} MCP with a molar absorptivity of 1.89×10^4 l mol^{-1} cm^{-1}. The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.51 and 1.55 μg ml^{-1}, respectively. The method was statistically evaluated by calculating percent relative error (% RE) for accuracy and percent relative standard deviation (% RSD) for precision, and was applied successfully to the determination of MCP in tablets, in injection and also in spiked human urine. No interference was observed from common additives found in pharmaceutical preparations. The results obtained by the proposed method were validated statistically by comparing the results with those of the reference method by applying the Student's *t*-test and *F*-test. The accuracy and reliability of the method were further ascertained by performing recovery tests *via* standard-addition technique.

1. Introduction

Metoclopramide hydrochloride, 4-amino-5-chloro-2-methoxy-*N*-(2-diethylamino-ethyl) hydrochloride, is a dopamine-receptor antagonist. It is a substituted benzamide used for its prokinetic and antiemetic properties in disorders of decreased gastrointestinal motility such as gastroparesis and ileus, as well as in gastroesophageal reflux disease, dyspepsis, nausea and vomiting, and for the prevention of cancer therapy-induced emesis (American Hospital Formulary Service, 1989). It is

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official in British Pharmacopoeia (2009) and United States Pharmacopoeia (2007) which recommend acid-base titrations with potentiometric end point detection.

The wide use of MCP has prompted the development of several analytical methods for its determination in pharmaceuticals when present alone or in combination with other drugs and include HPLC (Aqeel and Gregory, 1989; Besner et al., 1989; Chang et al., 2000; Nagwa, 1994; Aboul-Enein and Rafiqul, 1990; Suleiman et al., 1989; Tian et al., 2009), LC–MS (Yan et al., 2010), NMR spectrometry (Hanna and Lau-Cam, 1991) differential scanning calorimetry and X-ray powder diffractometry (Pabon et al., 1996), voltammetry (Farghaly et al., 2005; Wang et al., 2001), potentiometry (Badwan et al., 1986; Cannelo et al., 1989; Farnoush et al., 2009; Metrohm Application Bulletin, 2001; Mostafa, 2003), flow-injection chemiluminescence spectrometry (Bao-xiu et al., 2010; Hun and Zhang, 2008; Nawal, 2004; Shunli et al., 2002), fluorimetry (Attia and Aboaly, 2010; Buna et al., 1996), UV-spectrophotometry (Dudhane et al., 2010; Groszkowski et al., 1984a,b; Wadher et al., 2008) and flow-injection spectrophotometry (Guo et al., 2009; Jing et al., 2005; Leda de Souza et al., 2007; Royo Herrero et al., 1998). Some analytical techniques have also been reported for the determination of MCP in biological matrices and include HPLC (Takahashi et al., 1987; Mahasen, 1998; Fiorenzo et al., 1987; Brenda and Janis, 1990), LC–MS (Jaswanth et al., 2010), voltammetry (Farghaly et al., 2005), potentiometry (Farnoush et al., 2009), flow-injection chemiluminescence spectrometry (Nawal, 2004) and fluorimetry (Attia and Aboaly, 2010; Buna et al., 1996). Although the above methods are sensitive, they are time-consuming and complicated and require expensive instrumental set up.

Despite the availability of sophisticated and sensitive instruments for the assay of MCP, visible spectrophotometry continues to be the technique of choice in many laboratories, particularly in developing and underdeveloped nations, because of its simplicity, cost effectiveness, sensitivity and selectivity, and fair accuracy and precision. Many visible spectrophotometric methods based on different reaction schemes are found in the literature for the assay of MCP, especially for the analysis of pharmaceutical preparations. Methods based on redox reactions and using reagents such as sodium vanadate (Ramappa and Shivakumara, 1990), ammonium metavanadate (Singh et al., 1990) and Folin-Ciocalteu (Rao et al., 1990) have been reported. Ahmad El-Gendy (1992) has reported charge-transfer complex formation reaction using chloranil or bromanil as π acceptors. There are two reports based on ion-pair complex formation reaction of MCP with bromothymol blue (Shingbal and Velingkar, 1988) and Mo (V) and Co (II) thiocyanates (Fatma Abdel-Gawad and Nabawia El-Guindi, 1995). Shingbal and Kudchadkar (1987) have reported a method based on condensation reaction with sodium 1,2-naphthoquinone-4-sulfonate to form a orange-red colored product in the presence of borax. Methods based on coupling reaction with reagents like 8-anilino-1-naphthalene sulfonic acid, resorcinol and β-naphthol (Zarapkar and Mehra, 1989); and 1–2 napthoquinone, hydroquinone and resorcinol (Zarapkar and Deshmukh, 1990) have also been reported. The most widely used reaction for the assay of MCP in pharmaceuticals has been the diazo-coupling reaction and based on this reaction several workers have reported methods using different reagents like imipramine hydrochloride (Revanasiddappa and Veena, 2006), benzoyl acetone (Omran, 2005), sodium nitrite and aniline (Shah et al., 2005), dibenzoyl methane (Revanasiddappa and Manju, 2001), acetyl acetone (Revanasiddappa and Manju, 2002), chromotropic acid (Ramappa et al., 1999), 3-α, γ-dicarboxypropylrhodanine (Kalaschnikov et al., 1997), resorcinol (Shingbal and Joshi, 1984), 4-aminosalicylic acid (Emmanuel and Naik, 1983) and sodium nitrite and β-napthol (Patel et al., 2006) . Other color forming reactions such as oxidative coupling of MCP with MBTH and cerium (IV) (Ramappa et al., 1999), Schiff base formation with 4-dimethyl amino-benzaldehyde (Patel et al., 2006), *p*-dimethylaminocinnamaldehyde (Moussa, 2000) have also been reported.

However, most of the reported visible spectrophotometric methods suffer from one or the other disadvantage like poor sensitivity (Ramappa and Shivakumara, 1995; Singh et al., 1990; Shingbal and Kudchadkar, 1987; Ahmad El-Gendy, 1992; Fatma Abdel-Gawad and Nabawia El-Guindi, 1995; Zarapkar and Mehra, 1989; Revanasiddappa and Manju, 2002; Ramappa et al., 1999; Kalaschnikov et al., 1997; Moussa, 2000), drastic experimental conditions like heating (Ramappa and Shivakumara, 1995; Singh et al., 1990; Shingbal and Kudchadkar, 1987; Ahmad El-Gendy, 1992; Shah et al., 2005), strict pH control (Shingbal and Velingkar, 1988; Omran, 2005), liquid-liquid extraction step (Shingbal and Velingkar, 1988; Fatma Abdel-Gawad and Nabawia El-Guindi, 1995), use of organic solvents (Shingbal and Velingkar, 1988; Fatma Abdel-Gawad and Nabawia El-Guindi, 1995; Omran, 2005; Revanasiddappa and Manju, 2001), narrow linear range (Zarapkar and Mehra, 1989; Zarapkar and Deshmukh, 1990; Shingbal and Joshi, 1984; Moussa, 2000) etc.

The objective of this investigation was to devise simple, rapid, sensitive and economically viable spectrophotometric method that could be used to determine MCP in tablets, injection and spiked human urine. The determination involves the Schiff's base formation between MCP and vanillin in acid medium, the product of the chemical association of an aldehyde with aromatic primary amine group was present in MCP. The proposed method has been demonstrated to be superior to the reported methods with respect to speed, simplicity, sensitivity, selectivity and cost-effectiveness.

2. Experimental

2.1. Apparatus

A Systronics model 106 digital spectrophotometer (Ahmedabad, India) equipped with 1-cm matched quartz cells was used to measure the absorbance readings.

2.2. Materials and reagents

All chemicals used were of analytical-reagent grade. MCP certified to be 99.74% was obtained as gift from IPCA Laboratories Ltd., Mumbai, India. Vanillin was purchased from Loba Chemie, Mumbai, India. Acetic acid and perchloric acid were of analytical-reagent grade and they were purchased from Merck, Mumbai, India.

Two brands of tablets containing 10 mg MCP per tablet, Perinorm-10 (IPCA Laboratories Ltd., Mumbai, India) and Reglan-10 (Cosme farma laboratories Ltd., Karnataka, India); and one brand of injection containing 10 mg MCP per 2 ml,

perinorm (IPCA Laboratories Ltd., Mumbai, India) used in the investigation were purchased from local commercial sources.

A stock standard solution of pure drug equivalent 300 μg ml^{-1} MCP was prepared in glacial acetic acid and diluted to get 30 μg ml^{-1} MCP solution with the same acid. A 10% solution of vanillin was made by dissolving 10 g of vanillin in 100 ml glacial acetic acid.

2.3. Procedures

2.3.1. Procedure for calibration curve

Different aliquots (0.25, 0.5, 1.0, 1.5, 2.0 and 2.5 ml) of 30 μg ml^{-1} MCP solution were accurately transferred into a series of 5 ml calibrated flasks using a micro burette and the total volume was adjusted to 2.5 ml by adding glacial acetic acid. One milliliter of 0.1 M perchloric acid and 1 ml of 10% vanillin were added, the content was mixed and made up to the mark with glacial acetic acid. The absorbance of each solution was measured at 410 nm against a reagent blank.

2.3.2. Procedure for spiked human urine

A 50 ml volume of MCP – free human urine taken in a 125 ml separating funnel was spiked with 2.5 mg MCP. Ten milliliters of 1 M NaOH was added, mixed and kept aside for 3 min. Then, 25 ml of ethyl acetate was added, shaken well for about 15 min and the upper organic layer was collected in a beaker containing anhydrous sodium sulfate. The water-free organic layer was transferred into a dry beaker and solvent removed by evaporation on a hot water bath. The dry residue was dissolved in glacial acetic acid and transferred into a 25 ml calibrated flask, and diluted to the mark with the same acid. The resulting solution equivalent to 100 μg ml^{-1} MCP was diluted with the same acid to get 30 μg ml^{-1} MCP and analyzed by following the procedure described above.

2.3.3. Procedure for tablets

Twenty tablets were powdered and mixed thoroughly. An amount of powder equivalent to 15 mg of MCP was weighed into a 50 ml calibrated flask, 30 ml of glacial acetic acid was added and the mixture was shaken for 20 min; then the volume was made up to the mark with the same acid, mixed well and filtered using Whatman No. 42 filter paper. The resulting tablet extract (300 μg ml^{-1} in MCP) was diluted appropriately with the same acid to get a working concentration of 30 μg ml^{-1} MCP and subjected to analysis following the procedure described above.

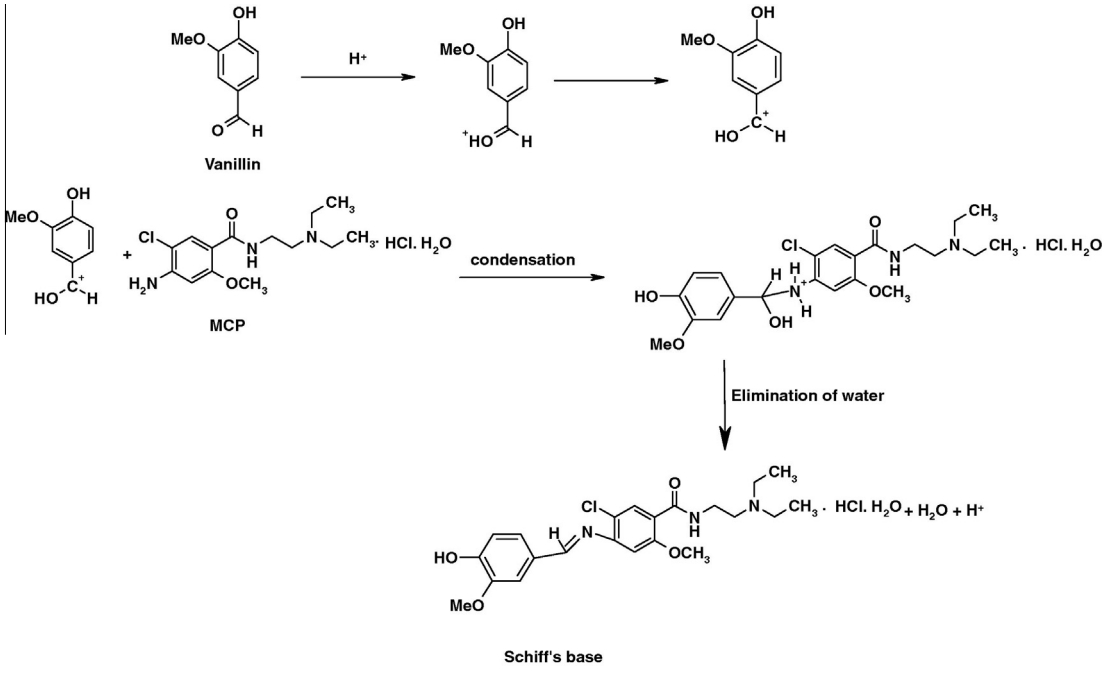
2.3.4. Procedure for injection

The contents of ten injection tubes were pooled in a dry beaker and mixed. An aliquot containing 15 mg of MCP was measured accurately and transferred to a 50 ml calibrated flask and diluted with glacial acetic acid. The resulting solution (300 μg ml^{-1} in MCP) was further diluted to get 30 μg ml^{-1} MCP and then subjected to analysis following the procedure described above.

2.3.5. Placebo blank and synthetic mixture analyses

A placebo blank of the composition: talc (20 mg), starch (15 mg), acacia (15 mg), methyl cellulose (20 mg), sodium citrate (15 mg), magnesium stearate (15 mg) and sodium alginate (25 mg), was transferred to a 50 ml calibration flask and its solution was prepared as described under ''procedure for tablets'' and then subjected to analysis using the procedure described above.

For synthetic mixture analysis, to the placebo blank of the composition described above, 1.5 mg of MCP was added and homogenized, transferred to a 50 ml calibrated flask and the solution was prepared as described under ''procedure for tablets'', and then subjected to analysis by the procedure described above. The analysis was used to study the interferences



Scheme 1 Probable reaction scheme.

of excipients such as talc, starch, acacia, methyl cellulose, sodium citrate, magnesium stearate and sodium alginate.

3. Results and discussion

3.1. Chemistry

The Schiff's base linkages are known to form easily by a condensation reaction of aromatic amine with an aldehydic group at the terminus of an organic compound (Ki and Dong, 1999). Based on this reaction many pharmaceutical compounds containing aromatic amine group have been determined (Annapurna et al., 2009; Adegoke and Nwoke, 2008; Siddappa et al., 2008; Claire et al., 1947; Tehseen et al., 2005) and the same reaction was adopted for the assay of MCP, since it contains primary aromatic amine group. The proposed method utilized vanillin which contains an aldehyde group.

The condensation reaction requires the presence of an acid for the protonation of the carbonyl oxygen and thereby leaving the carbonyl carbon fully positive charge. The NH_2 group in MCP donates a lone pair of electrons to the carbon present in the carbonyl group of vanillin. Internal rearrangement thereafter results in the formation of imine (Schiff's base) and then giving water and proton as by-products (Annapurna et al., 2009). The tentative reaction scheme is shown in Scheme 1.

3.2. Spectral characteristics

As shown in Fig. 1a, the condensation product as well as the blank absorb maximally at 340 nm. However, the maximum difference in absorbance between the sample and blank was observed at 410 nm as shown clearly in Fig. 1b. Hence, all measurements were made at this wavelength against reagent blank.

3.3. Optimization of experimental variables

Various experimental variables were optimized to achieve maximum sensitivity.

3.3.1. Effect of vanillin

Vanillin is insoluble in water and H_2SO_4. Even though it is soluble in methanol, there was no development of color in this medium. In acetic acid, both vanillin and MCP were found to dissolve and the yellow colored reaction product was also obtained in this medium. Hence, acetic acid was used to prepare vanillin and MCP solutions. The effect of vanillin on the sensitivity of the reaction was studied by using 10% vanillin and it was observed that when 1–2 ml was used, the absorbance readings were nearly constant; below and above this range there was a decrease in absorbance (Fig. 2). Blank absorption was found to increase with increasing concentration of vanillin. Hence, considering minimum blank absorption and maximum chromogen absorption, 1 ml of 10% vanillin was used as optimum in a total volume of 10 ml.

3.3.2. Effect of acid, reaction time and color stability

In aqueous medium, MCP failed to give condensation product with vanillin even in the presence of H_2SO_4. Yellow colored product was developed only in glacial acetic acid medium, hence, acetic acid was used as the solvating solvent. The intensity of the yellow color was found to increase on addition of concentrate H_2SO_4, however even the blank itself showed

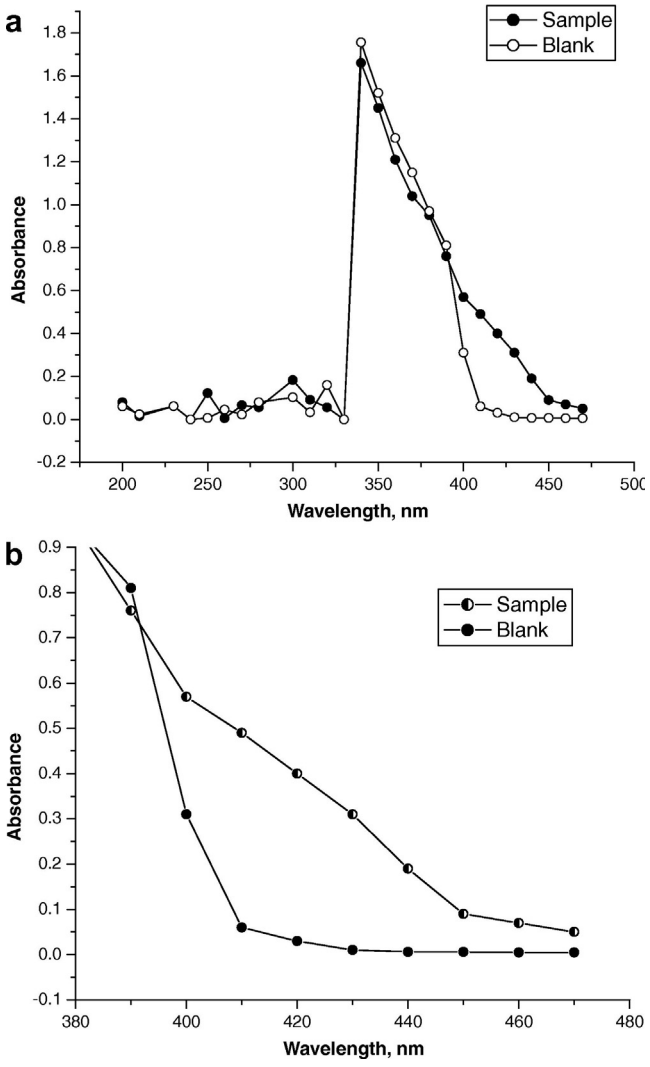


Figure 1 Absorption spectra (9 μg ml^{-1} MCP).

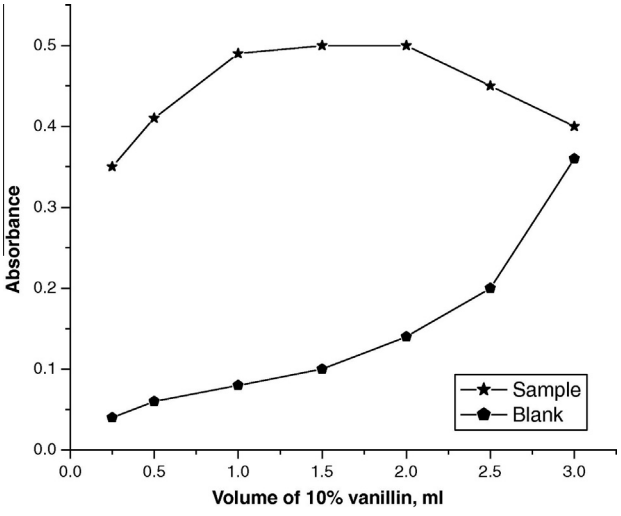


Figure 2 Effect of vanillin (9 μg ml^{-1} MCP).

intense yellow color. Hence, the use of H_2SO_4 was avoided. Fortunately, the sensitivity and the stability of the reaction product were enhanced in perchloric acid medium. The effect of perchloric acid was studied by using 0.1 M perchloric acid. The sensitivity of the color increased with increase in perchloric acid concentration and then decreased and blank absorption was found to increase with increasing concentration of perchloric acid (Fig. 3). Hence, 1 ml of 0.1 M acid in a total volume of 10 ml was fixed as the optimum.

Reaction was found to be instantaneous and the colored product remained stable for more than one day.

3.3.3. Order of addition

After optimizing all other experimental variables, further experiments were performed to ascertain the influence of order of addition of reactants on the color development. Maximum sensitivity was achieved when perchloric acid was added before adding vanillin as shown in Fig. 4. Hence, the method was performed in the order: MCP + perchloric acid + vanillin.

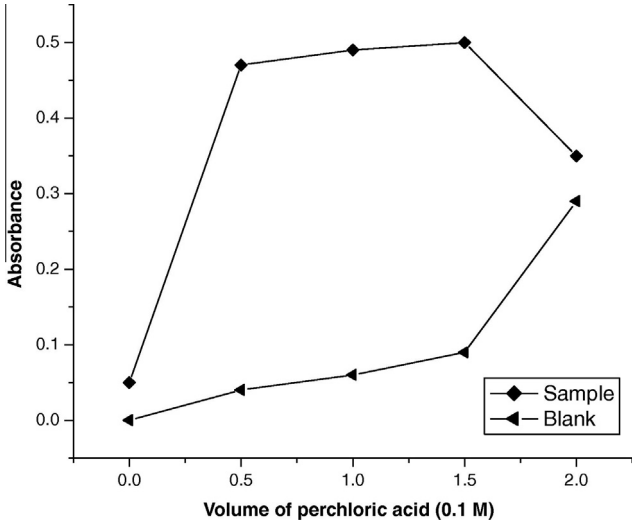


Figure 3 Effect of perchloric acid (9 μg ml^{-1} MCP).

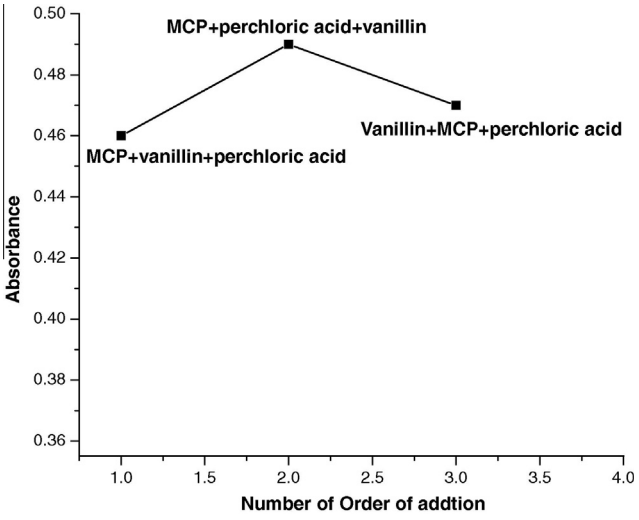


Figure 4 Effect of order of reactants (9 μg ml^{-1} MCP).

3.4. Method validation

3.4.1. Analytical parameters

A linear relation was found to exist between absorbance and the concentration of MCP in the range (1.5–15.0) μg ml^{-1} (Fig. 5). The calibration graph is described by the equation:

$$Y = a + b\,X$$

(where Y is the absorbance, a is the intercept, b is the slope and X is the concentration in μg ml^{-1}) obtained by the method of least squares. Correlation coefficient, intercept and slope for the calibration data are summarized in Table 1. Sensitivity parameters such as apparent molar absorptivity and Sandell's sensitivity values, the limits of detection and quantification calculated as per the current ICH guidelines (International Conference on Harmonization, 1996) are compiled in Table 1 and are indicative of the excellent sensitivity of the method. The limits of detection (LOD) and quantification (LOQ) were calculated according to the same guidelines using the formulae:

$$\mathrm{LOD} = 3.3\sigma/b \text{ and } \mathrm{LOQ} = 10\sigma/b$$

where σ is the standard deviation of five reagent blank determinations and b is the slope of the calibration curve.

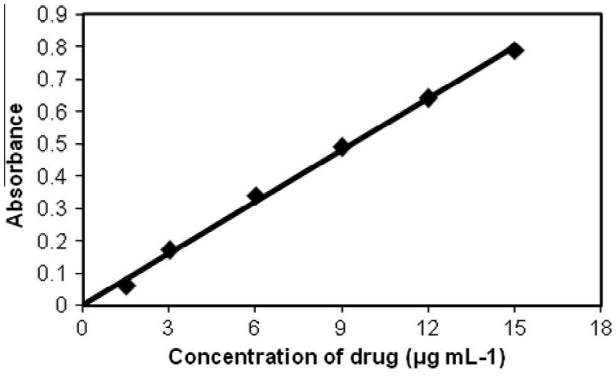


Figure 5 Calibration curve.

Table 1 Sensitivity and regression parameters.

Parameter	Proposed method
λ_{max} (nm)	410
Linear range (μg ml^{-1})	1.5–15.0
Molar absorptivity (ε) l mol^{-1} cm^{-1}	1.89×10^4
Sandell sensitivity[a] (μg cm^{-2})	0.019
Limit of detection (LOD) (μg ml^{-1})	0.51
Limit of quantification (LOQ) (μg ml^{-1})	1.55
Regression equation Y[b]	
Intercept (a)	0.008
Slope (b)	0.053
Standard deviation of a (S_a)	0.035
Standard deviation of b (S_b)	0.003
Regression coefficient (r)	0.999

[a] Limit of determination as the weight in μg ml^{-1} of solution, which corresponds to an absorbance of $A = 0.001$ measured in a cuvette of cross-sectional area 1 cm^2 and l = 1 cm.

[b] $Y = a + b\,X$, where Y is the absorbance, X is concentration in μg ml^{-1}, a is intercept, b is slope.

Table 2 Evaluation of intra-day and inter-day accuracy and precision.

MCP taken (μg ml^{-1})	Intra-day accuracy and precision			Inter-day accuracy and precision		
	MCP found	% RE	% RSD	MCP found	% RE	% RSD
6.0	6.06	1.00	1.02	6.09	1.50	1.12
9.0	9.08	0.89	0.99	9.13	1.44	1.24
12.0	12.05	0.417	1.08	12.15	1.25	1.16

RE: relative error and RSD: relative standard deviation.

Table 3 Robustness and ruggedness expressed as intermediate precision (% RSD).

MCP taken (μg ml^{-1})	Method robustness		Method ruggedness	
	Parameters altered			
	Perchloric acid[a] (0.1 M) ml RSD, % (n = 3)	Vanillin[b] (10%) ml, RSD, % (n = 3)	Inter-analysts' RSD, % (n = 4)	Inter-instruments' RSD, % (n = 3)
6.0	1.01	1.07	0.99	1.29
9.0	1.13	1.17	1.04	1.32
12.0	1.05	0.98	1.08	1.37

[a] Perchloric acid.
[B] Vanillin volumes used were 0.8, 1.0 and 1.2 ml.

Table 4 Recovery of the drug from synthetic mixture.

MCP in synthetic mixture taken (μg ml^{-1})	MCP recovered[a] (percent ± SD)
6.0	95.38 ± 0.66
9.0	96.18 ± 0.71
12.0	96.75 ± 0.69

[a] Mean value of five determinations.

3.4.2. Accuracy and precision

The accuracy and precision of the method were evaluated by performing seven replicate analyses on pure drug solution at three different concentration levels (within the working range). The relative error (%), an indicator of accuracy was $\leqslant$1.50 and within day precision, also called the repeatability, expressed as relative standard deviation (RSD %) was $\leqslant$1.24 indicating high accuracy and repeatability of the method. The results of the study are given in Table 2. The reproducibility of the method also known as the intra-day precision was evaluated by performing replicate analyses on pure drug solution at three levels over a period of five days, preparing all solutions afresh. The intra-day RSD values were less than 2% reflecting the usefulness of the method in routine analysis.

3.4.3. Robustness and ruggedness

Method robustness was tested by making small incremental changes in perchloric acid and vanillin concentrations. To check the ruggedness, analysis was performed by four different analysts and on three different spectrophotometers by the same analyst. The robustness and the ruggedness were checked at three different drug levels. The intermediate precision,

Table 5 Results of analysis of tablets and injection by the proposed method.

Tablet/injection brand name	Label claim, (mg/tablet)	MCP in tablet/injection taken (μg ml^{-1})	MCP found[a] (μg ml^{-1}) ± SD		Found[a] (percent of label claim ± SD)	
			Reference method	Proposed method	Reference method	Proposed method
Perinorm-10[b]	10	6.00	5.95 ± 0.04	5.90 ± 0.04	99.18 ± 0.68	98.27 ± 0.73 t = 2.04 F = 1.15
Raglan-10[c]	10	6.00	6.01 ± 0.04	6.08 ± 0.05	100.17 ± 0.63	101.3 ± 0.76 t = 1.63 F = 1.46
Injection perinorm-10[b]	10	6.00	6.07 ± 0.04	6.12 ± 0.04	101.12 ± 0.59	102.1 ± 0.65 t = 2.49 F = 1.21

The value of t (tabulated) at 95% confidence level and for four degrees of freedom is 2.77.
The value of F (tabulated) at 95% confidence level and for four degrees of freedom is 6.39.
[a] Mean value of five determinations.
[b] IPCA Laboratories Ltd., Mumbai, India.
[c] Cosme farma laboratories Ltd., Karnataka, India.

Table 6 Accuracy assessment by recovery experiments.

Tablet studied	MCP in tablet (μg ml^{-1})	Pure MCP added (μg ml^{-1})	Total found (μg ml^{-1})	Pure MCP recovered[a] Percent ± SD
Raglan-10	6.08	3.0	9.13	101.67 ± 0.73
	6.08	6.0	12.05	99.50 ± 0.85
	6.08	9.0	14.92	98.22 ± 0.77

[a] Mean value of three measurements.

expressed as percent RSD, which is a measure of robustness and ruggedness was within the acceptable limits as shown in the Table 3.

3.4.4. Effect of interferences

The effect of interferences was tested by placebo blank and synthetic mixture analyses. A convenient aliquot of the placebo blank solution was subjected to analysis according to the recommended procedure. There was no interference by the inactive ingredients as indicated by the near blank absorbance.

The analysis of synthetic mixture solution yielded percent recoveries which ranged between 95.38 and 96.75 with standard deviation of 0.66–0.71. The results of this study are presented in Table 4 indicating that the inactive ingredients did not interfere in the assay.

3.4.5. Application to the analysis of spiked urine sample, tablets and injection

The proposed method was successfully applied to the determination of MCP in spiked urine sample. The percent recovery of pure MCP added was 86.37 with standard deviation of 0.87 ($n = 5$). In order to evaluate the analytical applicability of the proposed method to the quantification of MCP in commercial tablets and injection, the results obtained by the proposed method were compared to those of the reference method (British Pharmacopoeia, 2009) by applying Student's *t*-test for accuracy and *F*-test for precision. In reference method, 0.2500 g of MCP was dissolved in 5.0 ml of 0.01 M HCl and 50 ml of alcohol; and the resulting solution was titrated with 0.1 M NaOH to potentiometric end point detection. The results (Table 5) show that the Student's *t*- and *F*-values at 95% confidence level are less than the theoretical values, indicating that there is a good agreement between the results obtained by the proposed method and the reference method with respect to accuracy and precision.

3.4.6. Recovery studies

The accuracy and validity of the proposed method were further ascertained by performing recovery studies. Pre-analyzed tablet powder was spiked with pure MCP at three levels (50%, 100% and 150% of that found in tablet powder) and the total was determined by the proposed method. The percent recovery of pure MCP added was in the range of 98.22–101.67% with standard deviation of 0.73–0.85 (Table 6) indicating that the recovery was good, and that the co formulated substance did not interfere in the determination.

4. Conclusions

A simple, sensitive and rapid method for the determination of MCP in its dosage forms and in spiked human urine is described, involving the use of vanillin as a chromogenic agent. The method is rapid and less tedious than many reported spectrophotometric methods. The present method can be applied at ambient temperature, color development is instantaneous and does not require strict pH control or tedious liquid-liquid extraction step. The method employs inexpensive and easily available chemicals and instrument. As most relevant features of the method, high sensitivity and a wide linear dynamic range compared to many existing methods can be emphasized. The color formed is highly stable leading to very precise results. An addition feature of the method is its applicability to determine the drug in spiked human urine after due sample treatment. These advantages make the method a valuable alternative to many existing methods for the determination of MCP in pharmaceuticals and body fluid.

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