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Full Length Original Research Paper

Analytical method development and validation of Drotaverine Hydrochloride and Aceclofenac in bulk and pharmaceutical dosage forms by UV-Spectrophotometer

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Abstract:
New simple, precise, rapid and reproducible UV-spectrophotometric method has been developed for the estimation of Drotaverine Hydrochloride and Aceclofenac in both bulk and tablet formulation. Drotaverine and Aceclofenac in combined tablet formulation were estimated by using the multicomponent mode at 307 nm for Drotaverine and 276 nm for Aceclofenac in their solution in ethanol: distilled water in the ratio of 50:50 (v/v %), With correlation coefficient of 0.999 for the both the drugs. The Beer's law obeyed the concentration range of 4-40 µg/mL for Drotaverine and 5-40 µg/mL for Aceclofenac. Mean recovery of 99.54% for Drotaverine and 98.23% for Aceclofenac signifies the accuracy of the method. This method was validated with respect to linearity, accuracy (recovery), precision, Limit of detection (LOD) and limit of quantification (LOQ) as per *ICH* guidelines and successfully applied for the estimation of Drotaverine Hydrochloride and Aceclofenac in commercially available tablet dosage form.

Keywords: Drotaverine Hydrochloride(DRT), Aceclofenac(ACE), spectrophotometric method.

Introduction

Drotaverine hydrochloride (DRT), 1-[(3, 4-diethoxy phenyl) methylene]-6, 7-diethoxy-1, 2, 3, 4-tetra hydro isoquinolene is an analogue of papaverine [The Merck Index 13th, 2001]. It acts as an antispasmodic agent by inhibiting phosphodiesterase IV enzyme, which is specific for smooth muscle spasm and pain, used to reduce excessive labor pain. A few UV-spectrophotometric[1][2] and HPLC[3][4][5] methods have been reported individually or in combination with other drugs for estimation of Drotaverine Hydrochloride.

Aceclofenac (ACE) is chemically designated as 2-(2, 6- Dichloroanalino) phenyl acetoxy acetic acid. It is a NSAID, used in the management of osteoarthritis, rheumatoid arthritis, and ankylosing spondylitis [The Merck Index 13th, 2001]. Aceclofenac is official in Indian pharmacopoeia [Indian pharmacopoeia 2007]. Various UV-spectrophotometric[6][7] and HPLC[8][9] methods for aceclo-fenac have been reported for its estimation individually or in combination with other drugs.

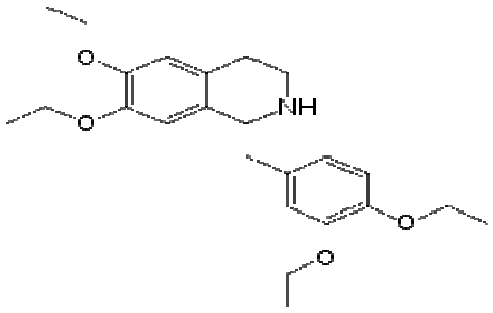


Fig. 1: Structure of Drotaverine

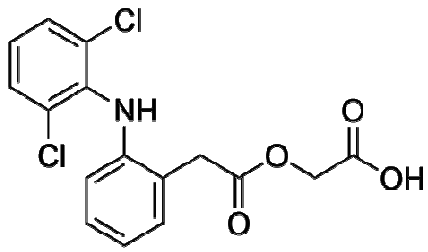


Fig. 2: Structure of Aceclofenac

MATERIALS AND METHODS

Chemicals & Reagents:

Analytically pure DRT and ACE were obtained as gift samples Commercial tablet formulation (DROLGAN) was purchased from the local market. All chemicals and reagents used were of Analytical Grade obtained from Merck.

Instrument:

SHIMADZU double beam UV/Visible recording spectrophotometer (Model: UV-1800) with 2 nm spectral bandwidth was employed for all spectrophotometric measurement. A 10mm matchedquartz cell and Borosil glass wares were used for the study. Calibrated electronic single pan balances Sartorius CP 225 D, pH Meter (LABINDIA), Enertech Fast Clean Ultrasonicator were also used during the analysis.

Standard Stock Solution

The standard stock solutions of DRT and ACE were prepared by dissolving accurately weighed 100 mg of drug in 100 ml of a mixture of ethanol and distilled water (50:50) in two separate 100 ml volumetric flasks to get a concentration of 1000 µg/mL. Both were appropriately diluted with mixture of ethanol and distilled water (50:50) to get a concentration of 100µg/ml and were kept as the stock solutions.

Determination of λmax

The standard solution of both DRT and ACE (10 µg/mL) were scanned in the wavelength region of 200-400 nm and the λmax was found to be 307nm and 276nm respectively.

Preparation of calibration curve

For each drug, appropriate aliquots were pipettedout from each standard stock solution into a series of 10 ml volumetric flasks. The volume was made up to mark with mixture of ethanol and distilled water (50:50) to get set of solutions having concentration range 4-40 µg/mL for DRT and 5-40 µg/mL for ACE. The prepared solutions of DRT and ACE were scanned at 307 nm and 276 nm respectively. The respective absorbances were recorded and were plotted against the concentrations to obtain their respective calibration curves.

Preparation of sample solution

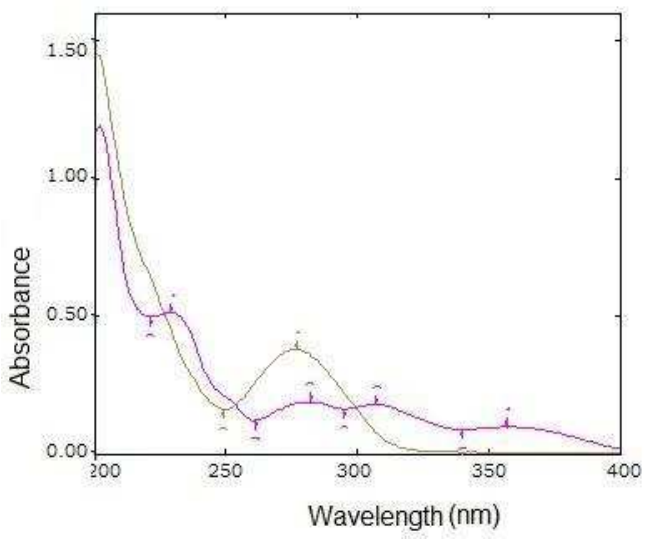
The proposed method was successfully applied for the determination of DRT and ACE in tablet dosage form. 20 tablets were weighed and powdered. The amount of tablet powder equivalent to 100 mg of DRT and 80 mg of ACE was weighed accurately and transfered to 50mL mixture of methanol and water and kept for 2 min in sonicator and volume was made up to 100 mL with the same solvent. The solution was then filtered through whatmann f ilter paper. This filtrate was diluted suitably with mixture of methanol and water to get the solution of 8 µg/mL of DRT and 10 µg/mL of ACE concentration. The absorbance was measured against blank. The drug content of the preparation was calculated using standard calibration curve.

RESULTS AND DISCUSION

The analytical method was developed in multicomponent mode of analysis and validated according to *ICH* guidelines for determination of

DRT and ACE in their pharmaceutical dosage form at selected wavelength of 307 nm and 276 nm respectively where there is no interference among the drugs as shown in the overline spectra (Fig.3).

Figure 3: Overline spectra Drotaverine Hydrochloride and Aceclofenac



Linearty

Developed analytical method showslinearity response over the range of 4-40 µg/mL for DRT and 5-40 µg/mL for ACE, With correlation coefficient of 0.999 for the both the drugs.

Table1: Linearity table for Drotaverine Hydrochloride at 307 nm

S. No	Concentration(µg/mL)	Absorbance at 307nm
1	04	0.097
2	08	0.176
3	12	0.259
4	16	0.329
5	20	0.407
6	24	0.500
7	28	0.575
8	32	0.653
9	36	0.740
10	40	0.819

Figure 4: Calibration curve of Drotaverine Hydrochloride at 307 nm

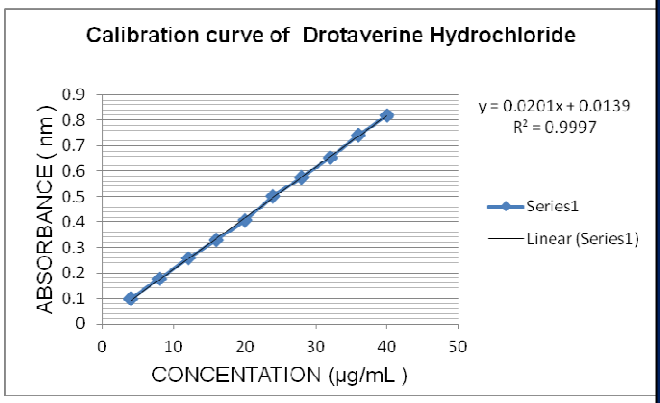
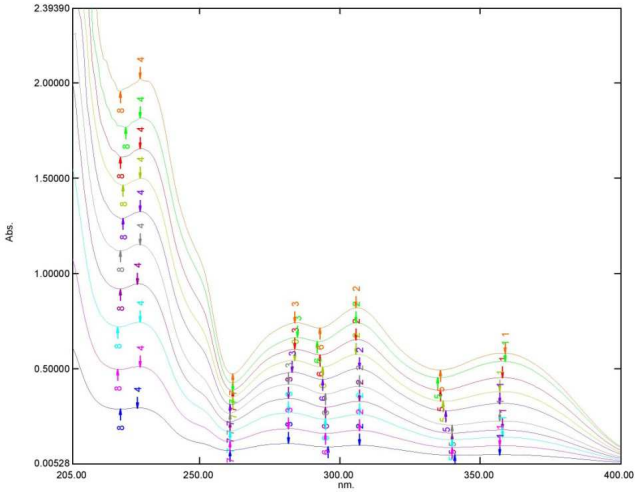


Figure 5: Over line spectra of Drotaverine Hcl



Hydrochloride

Table 2: Linearity table for Aceclofenac at 276 nm

S. No	Concentration(µg/mL)	Absorbance at 276nm
1	05	0.223
2	10	0.375
3	15	0.557
4	20	0.716
5	25	0.903
6	30	1.067
7	35	1.268
8	40	1.413

Figure 6: Calibration curveof Aceclofenac at 276nm

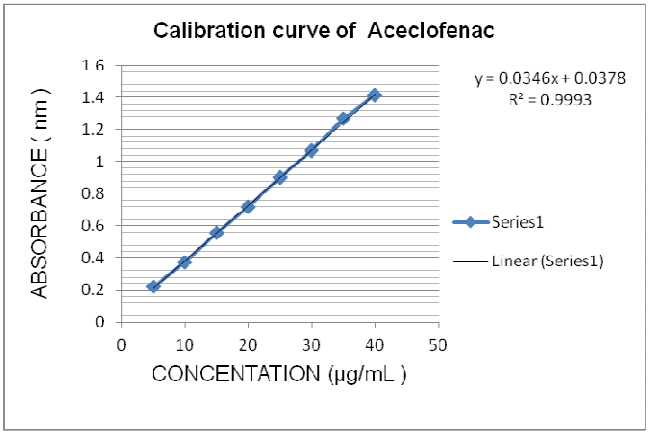
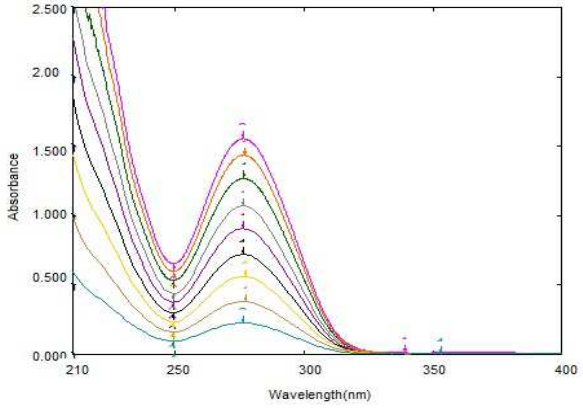


Figure 7: Over line spectra of Aceclofenac



Precision

Precision was determined and the results are represented in the form of % RSD which was found to be below 2% and shows that the test was highly précised and results given in table 3.

Table 3: Repeatability data

Drug	Concentration (µg/ml)	%RSD (n=6)
DrotaverineHydrochloride	20 µg/ml	0.1159
Aceclofenac	15 µg/ml	0.4714

Recovery studies

The accuracy of the proposed method was evaluated by adding known amounts of pure drug to its tablet formulation. Three samples were prepared for each recovery level of 80%, 100% and 120% by adding standard solutions of DRT and ACE. The average recovery of marketed formulation was found to be 99.54% and 98.23%for DRT and ACE respectively shown in table 4.

Table 4: Recovery studies for Drotaverine Hydrochloride (DRT) and Aceclofenac (ACE) in the pharmaceutical dosage form

Formulation	% Level	% recovery	%RSD
Drotaverine Hydrochloride	80%	100.9	0.87
	100%	99.46	0.18
	120%	98.27	0.92
Aceclofenac	80%	99.07	0.22
	100%	98.34	0.91
	120%	97.29	0.97

LOD & LOQ

The absorbance of ten solutions containing 10 µg/mL were measured at 307 nm for DRT and 276 nm for ACE calculated according to equation of LOD [3.3 x MSD/ average response] and LOQ [10 x MSD/ average response].

Table 5: LOD and LOQ

Drug	LOD	LOQ
Drotaverine Hydrochloride	0.0381 µg/mL	0.115 µg/mL
Aceclofenac	0.0146 µg/mL	0.472 µg/mL

Table 6: Summary of Validation Parameters

S. No	Validation Parameter	Drotaverine Hydrochloride	Aceclofenac
1	Absorption maxima (nm)	307	276
2	Linearity range (µg/ml)	4-40	5-40
3	Regression equation	y = 0.020x + 0.013	y = 0.034x + 0.037
4	Correlation coefficient (R^2)	0.999	0.999
5	LOD (µg/mL)	0.0381	0.0146
6	LOQ (µg/mL)	0.115	0.472
7	% Recovery	99.54%	98.23%
8	Precision(%RSD)	0.1159	0.4714

Conclusion

The optimized method is simple, precise, accurate for simultaneous determination of DRT

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and ACE in combined tablet dosage form. The method was validated as per *ICH* guidelines in terms of linearity, accuracy, precision, LOD and LOQ. This method can be used for routine analysis of DRT and ACE both in bulk and tablet dosage forms.

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