1-

Spectrophotometric determination of tizanidine and orphenadrine via ion pair complex formation using eosin Y

Abstract
A simple, sensitive and rapid spectrophotometric method was developed and validated for the determination of two skeletal muscle relaxants namely, tizanidine hydrochloride (I) and orphenadrine citrate (II) in pharmaceutical formulations. The proposed method is based on the formation of a binary complex between the studied drugs and eosin Y in aqueous buffered medium (pH 3.5). Under the optimum conditions, the binary complex showed absorption maxima at 545 nm for tizanidine and 542 nm for orphenadrine. The calibration plots were rectilinear over concentration range of 0.5-8 \( \mu g/mL \) and 1-12 \( \mu g/mL \) with limits of detection of 0.1 \( \mu g/mL \) and 0.3 \( \mu g/mL \) for tizanidine and orphenadrine respectively. The different experimental parameters affecting the development and stability of the complex were studied and optimized. The method was successfully applied for determination of the studied drugs in their dosage forms; and to the content uniformity test of tizanidine in tablets.

2-


Abstract
Two rapid, simple, sensitive, selective and economic derivative spectrophotometric (first \([D1]\) and second \([D2]\)) and synchronous spectrofluorimetric (FDSFS and SDSFS) methods have been developed for the analysis of fexofenadine hydrochloride (FXD) in the presence of its different degradation products. Derivative spectrophotometry \((D1)\) was used to measure FXD at 223 nm in the presence of its alkaline or acidic degradation products, and at 211 nm in the presence of its oxidative degradation product. Derivative spectrophotometry \((D2)\) was used to determine FXD at 217 nm in the presence of its alkaline or acidic degradation products, and at 215 nm in the presence of its oxidative degradation product; the UV degradation product was measured at 211 nm. Synchronous spectrofluorimetry \((FDSFS)\) was used to measure FXD in the presence of its alkaline or acidic degradation products at 406 nm, and at 367 nm in the presence of its oxidative or UV degradation products. Synchronous spectrofluorimetry \((SDSFS)\) was applied to determine the drug at
225 nm in the presence of its alkaline, acidic, oxidative or UV degradation products. The proposed methods were successfully applied for the determination of the studied compound in its commercial tablets. The results obtained were in good agreement with those obtained by the comparison method.

3-

â€¢ Validated stability-indicating spectrofluorimetric methods for the determination of ebastine in pharmaceutical preparations.

Abstract
Two sensitive, selective, economic, and validated spectrofluorimetric methods were developed for the determination of ebastine (EBS) in pharmaceutical preparations depending on reaction with its tertiary amino group. Method I involves condensation of the drug with mixed anhydrides (citric and acetic anhydrides) producing a product with intense fluorescence, which was measured at 496 nm after excitation at 388 nm. Method (IIA) describes quantitative fluorescence quenching of eosin upon addition of the studied drug where the decrease in the fluorescence intensity was directly proportional to the concentration of ebastine; the fluorescence quenching was measured at 553 nm after excitation at 457 nm. This method was extended to (Method IIB) to apply first and second derivative synchronous spectrofluorimetric method (FDSFS & SDSFS) for the simultaneous analysis of EBS in presence of its alkaline, acidic, and UV degradation products. The proposed methods were successfully applied for the determination of the studied compound in its dosage forms. The results obtained were in good agreement with those obtained by a comparison method. Both methods were utilized to investigate the kinetics of the degradation of the drug.

4-

â€¢ Validated stability indicating liquid chromatographic determination of ebastine in pharmaceuticals after pre column derivatization: Application to tablets and content uniformity testing.

Abstract
An accurate, simple, sensitive and selective reversed phase liquid chromatographic method has been developed for the determination of ebastine in its pharmaceutical preparations. The proposed method depends on the complexation ability of the studied drug with Zn2+ ions. Reversed phase chromatography was conducted using an ODS C18 (150 1—4.6 mm id) stainless steel column at ambient temperature with UV-detection at 260 nm. A mobile phase containing 0.025%w/v Zn2+ in a mixture of (acetonitril/methanol; 1/4) and Britton Robinson buffer
adjusted to pH 4.2, has been used for the determination of ebastine at a flow rate of 1 ml/min. The calibration curve was rectilinear over the concentration range of 0.3 - 6.0 μg/ml with a detection limit (LOD) of 0.13 μg/ml, and quantification limit (LOQ) of 0.26 μg/ml. The proposed method was successfully applied for the analysis of ebastine in its dosage forms, the obtained results were favorably compared with those obtained by a comparison method. Furthermore, content uniformity testing of the studied pharmaceutical formulations was also conducted. The composition of the complex as well as its stability constant was also investigated. Moreover, the proposed method was found to be a stability indicating one and was utilized to investigate the kinetics of alkaline and ultraviolet induced degradation of the drug. The first-order rate constant and half life of the degradation products were calculated.

5-

**Validated spectrofluorimetric determination of some H1 receptor antagonist drugs in pharmaceutical preparations through charge transfer complexation.**

**Abstract** A validated simple, rapid, and selective spectrofluorimetric method was developed for the determination of some antihistaminic H1 receptor antagonist drugs namely ebastine (EBS), cetirizine dihydrochloride (CTZ), and fexofenadine hydrochloride (FXD). The method is based on the reaction of the cited drugs with some acceptors namely p-chloranilic acid (CLA), tetracyanoethylene (TCNE), and 2,3-dichloro-5,6-dicyano-pbenzoquinone (DDQ) to give highly fluorescent derivatives. The fluorescence intensity—concentration plots were rectilinear over the concentration ranges of 0.2 μg/ml to 3.0, 0.2 μg/ml to 2.5, and 0.15 μg/ml to 2.0 μg/ml for EBS with CLA, DDQ, and TCNE respectively; 0.5 μg/ml to 7.0, 0.5 μg/ml to 6.0, and 0.2 μg/ml to 4.0 μg/ml for CTZ with the previously mentioned reagents, and 0.2 μg/ml to 3.5, 0.5 μg/ml to 6.0, and 0.2 μg/ml to 3.5 μg/ml for FXD. The factors affecting the formation of the reaction products were carefully studied and optimized. The method was applied for the determination of the studied drugs in their dosage forms. The results obtained were in good agreement with those obtained by the comparison methods. Reactions Stoichiometries of the complexes formed between the studied drugs and acceptors were defined by the Job’s method of the continuous variation and found in 1:1 in all cases.

6-

**Validated stability indicating liquid chromatographic method for the...**
determination of fexofenadine hydrochloride in presence of its degradation products. Application to tablets and content uniformity testing.

A simple, stability-indicating, reversed phase liquid chromatographic method has been developed for the determination of fexofenadine hydrochloride in the presence of its forced alkaline, acidic and oxidative degradation products. Reversed phase chromatography was conducted using an ODS C18 (150 x 4.6 mm id) column at ambient temperature with UV-detection at 225 nm. A mobile phase consisting of potassium dihyrogen phosphate buffer: acetonitrile (35:65, v/v) adjusted to pH 5.5 with phosphoric acid, has been used for the separation of the studied drug and its degradation products at a flow rate of 1 ml/min. The calibration curve was rectilinear over the concentration range of 2-20 $\mu$g/ml with a detection limit (LOD) of 0.92 $\mu$g/ml, and quantification limit (LOQ) of 1.5 $\mu$g/ml. The proposed method was successfully applied for the analysis of fexofenadine hydrochloride in its dosage forms, the obtained results were favorably compared with those obtained by a comparison method. Furthermore, content uniformity testing of the studied pharmaceutical formulations was also conducted. The drug was exposed to forced alkaline, acidic and oxidative degradation according to the ICH Guidelines. Moreover, the method was utilized to investigate the kinetics of the different degradation products of the drug. The first-order rate constant, half-life time, and activation energy of the degradation reactions were calculated.

7-

Spectrofluorimetric determination of some H1 receptor antagonist drugs in pharmaceutical formulations and biological fluids.

A validated simple, economic, selective, and stability indicating spectrofluorimetric method was developed for the determination of some antihistaminic H1 receptor antagonist drugs namely ebastine (EBS), cetirizine dihydrochloride (CTZ), and fexofenadine hydrochloride (FXD). The method is based on the reaction of the cited drugs with 2-cyanoacetamide in alkaline medium to give highly fluorescent derivatives measured at 365 nm after excitation at 312 nm. The method was applied for the determination of the studied drugs in their dosage forms. Furthermore, the method was applied for the determination of the drugs in spiked human plasma, and used to reveal the pharmacokinetic characters in a healthy volunteer treated with oral administration of the different dosages of the drugs. The method was utilized to investigate the kinetics of the alkaline, acidic, oxidative, and ultraviolet degradation of the drugs. The apparent first order rate constants and half life times of the degradation products were calculated.

8-

Simultaneous determination of methocarbamol and aspirin by RP-HPLC using fluorescence detection with time programming: its application to pharmaceutical dosage form.

A new simple, rapid and sensitive reversed-phase liquid chromatographic method was developed and validated for the simultaneous determination of methocarbamol (MET) and aspirin (ASP) in their combined dosage form. The separation of these compounds was achieved within 6.0 minutes on a CLC Shim-pack C(8) column (250â€‰mm).—
Simultaneous Determination of Methocarbamol and Ibuprofen in their Binary Mixtures using HPLC Method with Fluorescence Detection. Application to Combined Tablets.

A new simple, rapid and sensitive reversed-phase liquid chromatographic method was developed and validated for the simultaneous determination of methocarbamol (MET) and ibuprofen (IBU) in their combined dosage form. The separation of these compounds was achieved within 7.0 min on a CLC Shim-pack C8 column (250 x 4.6 mm, 5 µm particle size) using isocratic mobile phase consisting of acetonitrile and 0.02 M dihydrogenphosphate buffer (30:70, v/v) at pH 5.0. The analysis was performed at a flow rate of 1.0 mL/min with fluorescence detection at 277/313 nm for MET and 298/410 nm for ASP using real-time programming. The selectivity, linearity of calibration, accuracy, inter- and intra-day precision and recovery were examined as parts of the method validation. The concentration-response relationship was linear over concentration ranges of 0.02-0.20 and 0.02-0.40 µg/mL for MET and ASP, respectively, with a limit of detection of 6 and 32 ng/mL for MET and ASP, respectively. The proposed method was successfully applied for the analysis of both MET and ASP in prepared tablets with average recoveries of 99.88 ± 0.65% for MET and 100.44 ± 0.78% for ASP. The results were favourably compared to those obtained by a reference method.

Development and Validation of RP- HPLC Method for Simultaneous Determination of Ascorbic Acid and Salicylamide in their Binary Mixtures: Application to Combined Tablets

A new simple, rapid and sensitive reversed-phase liquid chromatographic method was developed and validated for the simultaneous determination of ascorbic acid (ASC) and salicylamide (SAL) in their combined dosage form. The analysis was carried out on CLC Shim-pack C8 column (250 x 4.6 mm, 5 µm particle size) using a mobile phase consisting of methanol: 0.03 M phosphate buffer mixture (55: 45, v/v) of pH 4.0. The mobile phase was pumped at a flow rate of 1 mL/min with ultraviolet detection at 255 nm. The selectivity, linearity of calibration, accuracy,
intra and inter day precision and recovery were examined as parts of the method validation. The concentration–response relationship was linear over a concentration range of 0.50-10.00 and 5.00-50.00 µg/mL for ASC and SAL, respectively with limits of detection of 0.048 and 0.676 µg/mL. The proposed method was applied for the simultaneous determination of the two studied drugs in their combined tablets with average recoveries of 100.04 ± 0.75% and 100.11 ± 1.04% for ASC and SAL, respectively. The results were favorably compared to those obtained by the comparison methods.