Optimized and validated flow-injection spectrophotometric analysis of topiramate, piracetam and levetiracetam in pharmaceutical formulations

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Abstract

Application of a sensitive and rapid flow injection analysis (FIA) method for determination of topiramate (TP), piracetam (PC), and levetiracetam (LV) in pharmaceutical formulations has been investigated. The method is based on the reaction with ortho-pthalaldehyde (OPA) and 2-mercaptoethanol (ME) in a basic buffer and measurement of the absorbance at 295 nm under flow conditions. Variables affecting the determination such as sample injection volume, pH, ionic strength, reagent concentrations, flow rate of reagent and other FIA parameters were optimized to produce the most sensitive and reproducible results using a quarter-fraction factorial design, for five factors at two levels. The optimization of the method was executed with the concentration of OPA of 50 mg/100 mL borate buffer, the volume of ME of 100 µL, pH of 9.4, and 0.1 ionic strength borate buffer and the length of the tubing was of 0.75 m. Also, the method has been fully validated in terms of linearity and range, limit of detection and quantitation, precision, selectivity and accuracy. The method was successfully applied to the analysis of pharmaceutical preparations.
Validated stability-indicating HPLC method for the determination of dimethyl-4,4'-dimethoxy-5,6,5',6'-dimethylene dioxybiphenyl-2,2'-dicarboxylate (DDB) and its degradation products

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ABSTRACT

High-performance liquid chromatographic method was developed for the quantitative determination of dimethyl-4,4'-dimethoxy-5,6,5',6'-dimethylene dioxybiphenyl-2,2'-dicarboxylate (DDB) and its degradation products. Forced degradation studies were performed on bulk sample of DDB using acid (1N hydrochloric acid), alkaline (0.1N sodium hydroxide), oxidation (33% hydrogen peroxide), heat (70 °C) and photolytic degradation. The chromatographic method was fine tuned using the samples generated from forced degradation studies. Good resolution between the peaks corresponds to degradation products and the analyte was achieved on 5 μm ODS column (Luna, Phenomenex, USA). The mobile phase consists of a mixture of acetonitrile and water (60:40, v/v). Quantitation was achieved with UV detection at 235 nm based on peak area. The proposed HPLC method was utilized to investigate the kinetics of acidic, alkaline and oxidative degradation processes of DDB at different temperatures and the apparent pseudo first-order rate constant, half-life and activation energy were calculated. The pH-rate profiles of degradation of DDB in Britton-Robinson buffer solutions within the pH range 2–11 were studied. The developed method was validated with respect to linearity, accuracy, precision, robustness and forced degradation studies, prove the stability-indicating power of the method.
Validated, Stability-Indicating LC Method for Analysis of Pipenzolate Bromide and Its Hydrolysis Products

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Abstract

A liquid chromatographic method has been developed and validated for quantitative analysis of pipenzolate bromide (PP), its hydrolysis products, and phenobarbitone, sodium benzoate, and sodium saccharine. A 5-µm particle ODS column was used with acetonitrile–KH₂PO₄ (10 mM, pH 3.5) 40:60 (v/v), containing 5 mM heptanesulfonic acid sodium salt, as mobile phase. Quantitation was achieved by UV detection at 210 nm, on the basis of peak area. Forced degradation studies were performed on a bulk sample of PP using 0.1 M hydrochloric acid, 0.01 M sodium hydroxide, 0.33% hydrogen peroxide, heat (70 °C), and photolytic degradation. The proposed LC method was used to study the kinetics of acidic hydrolysis and pH-rate profiles of hydrolysis of PP in Britton–Robinson buffer solutions.

Keywords

Column liquid chromatography
Pipenzolate
Kinetics of hydrolysis
pH-rate profile
Arrhenius plot
New Validated Liquid Chromatographic and Chemometrics-Assisted UV Spectroscopic Methods for the Determination of Two Multicomponent Cough Mixtures in Syrup

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Multivariate spectrophotometric calibration and liquid chromatographic (LC) methods were applied to the determination of 2 multicomponent mixtures containing diprophylline, guaiphenesin, methylparaben, and propylparaben (Mixture 1), or clobutinol, orciprenaline, saccharin sodium, and sodium benzoate (Mixture 2). For the multivariate spectrophotometric calibration methods, principal component regression (PCR) and partial least-squares regression (PLS-1), a calibration set of the mixtures consisting of the components of each mixture was prepared in 0.1 M HCl. Analytical figures of merit such as sensitivity, selectivity, limit of quantitation, and limit of detection were determined for both PLS-1 and PCR. The LC separation was achieved on a reversed-phase C\textsubscript{18} analytical column by using isocratic elution with 20 mM potassium dihydrogen phosphate, pH 3.3–acetonitrile (55 + 45, v/v) as the mobile phase and UV detection at 260 and 220 nm for Mixture 1 and Mixture 2, respectively. The proposed methods were validated and successfully applied to the analysis of pharmaceutical formulations and laboratory-prepared mixtures containing the 2 multicomponent combinations.
HPLC and chemometrics-assisted UV-spectroscopy methods for the simultaneous determination of ambroxol and doxycycline in capsule

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Abstract

High-performance liquid chromatography (HPLC) and multivariate spectrophotometric methods are described for the simultaneous determination of ambroxol hydrochloride (AM) and doxycycline (DX) in combined pharmaceutical capsules. The chromatographic separation was achieved on reversed-phase C18 analytical column with a mobile phase consisting of a mixture of 20 mM potassium dihydrogen phosphate, pH 6-acetonitrile in ratio of (1:1, v/v) and UV detection at 245 nm. Also, the resolution has been accomplished by using numerical spectrophotometric methods as classical least squares (CLS), principal component regression (PCR) and partial least squares (PLS-1) applied to the UV spectra of the mixture and graphical spectrophotometric method as first derivative of the ratio spectra (1DD) method. Analytical figures of merit (FOM), such as sensitivity, selectivity, analytical sensitivity, limit of quantitation and limit of detection were determined for CLS, PLS-1 and PCR methods. The proposed methods were validated and successfully applied for the analysis of pharmaceutical formulation and laboratory-prepared mixtures containing the two component combination.

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Keywords: Ambroxol; Doxycycline; HPLC; Multivariate spectrophotometric methods