Simple and rapid HPLC method for simultaneous determination of atenolol and chlorthalidone in spiked human plasma

Mohamed S. Elgawish, Samia M. Mostafa, Abdalla A. Elshanawane

Abstract

A simple, sensitive and rapid chromatographic method was developed and validated for the simultaneous quantification of atenolol and chlorthalidone in human plasma using hydrochlorothiazide as internal standard (IS). The method utilized proteins precipitation with acetonitril as the only sample preparation involved prior to reverse phase-HPLC. The analytes were chromatographed on Shim-pack cyanopropyl column with isocratic elution with 10 mM KH2PO4 (pH 6.0) – methanol (70:30, v/v) at ambient temperature with flow rate of 1 mL min⁻¹ and UV detection at 225 nm. The chromatographic run time was less than 10 min for the mixture. The calibration curves were linear over the range of 0.1–10 lg mL⁻¹. The method was validated in terms of accuracy, precision, absolute recovery, freeze–thaw stability, bench-top stability and re-injection reproducibility.

The within- and between-day accuracy and precision were found to be within acceptable limits <15%. The analytes were stable after three freeze–thaw cycles (deviation <15%). The proposed method was specific for the simultaneous determination of atenolol and chlorthalidone in human plasma where there was no interference from endogenous biological substances.
Application of a Validated, Stability-Indicating LC Method to Stress Degradation Studies of Ramipril and Moexipril.HCl

Abdalla A. Elshanaawane¹, Samia M. Mostafa²,², Mohamed S. Elgowish²
¹ Medicinal Chemistry Department, Faculty of Pharmacy, Zagazig University, Zagazig, Egypt
² Pharmaceutical Chemistry Department, Faculty of Pharmacy, Suez Canal University, Ismailia, Egypt;
E-Mail: medicinalchemistry@yahoo.com

Abstract

A stability-indicating reversed-phase liquid chromatographic (RPLC) method has been established for analysis of ramipril (RAM) and moexipril hydrochloride (MOEX.HCl) in the presence of the degradation products generated in studies of forced decomposition. The drug substances were subjected to stress by hydrolysis (0.1 m NaOH and 0.1 m HCl), oxidation (30% H₂O₂), photolysis (254 nm), and thermal treatment (80 °C). The drugs were degraded under basic and acidic conditions and by thermal treatment but were stable under other stress conditions investigated. Successful separation of the drugs from the degradation products was achieved on a cyanopropyl column with 40:60 (v/v) aqueous 0.01 m ammonium acetate buffer (pH 6)-methanol as mobile phase at a flow rate of 1 mL min⁻¹. Detection was by UV absorption at 210 nm. Response was a linear function of concentration over the range 5–50 μg mL⁻¹ (r > 0.9995), with limits of detection and quantitation (LOD and LOQ) of 0.04 and 0.09 μg mL⁻¹, respectively, for RAM and 0.014 and 0.32 μg mL⁻¹, respectively, for moexipril. The method was validated for specificity, selectivity, solution stability, accuracy, and precision. Statistical analysis proved the method enabled reproducible and selective quantification of RAM and MOEX as the bulk drug and in pharmaceutical preparations. Because the method effectively separates the drugs from their degradation products, it can be used as stability-indicating.
Development and Validation of LC Method for Simultaneous Determination of Two Binary Mixtures Containing Indapamide

Abdalla A. Elshanawone, Samia M. Mostafa, Mohamed S. Elgowish

1 Medicinal Chemistry Department, Faculty of Pharmacy, Zagazig University, Zagazig, Egypt
2 Pharmaceutical Chemistry Department, Faculty of Pharmacy, Suez Canal University, Ismailia 41522, Egypt;
E-Mail: medicinalchemistry@yahoo.com

Abstract

A new sensitive, simple, rapid, and precise RP LC method with hydrochlorothiazide as internal standard has been developed for resolving two binary mixtures, perindopril with indapamide and captopril with indapamide, in pharmaceutical formulations. The drugs were separated at room temperature on a 250 mm x 4.6 mm, 5-μm particle, cyanopropyl column with 10 mM KH₂PO₄, pH 6.0-methanol 55:45 (v/v) as mobile phase at a flow rate of 1 mL min⁻¹. Detection was at 210 nm. Factors affecting the separation process were studied and optimized. The linearity, accuracy, and precision of the method were good, and the method was successfully applied to the determination of the two binary combinations in synthetic mixtures and commercial pharmaceutical products.