ABSTRACT

The verapamil was determined by the technique of sequential injection analysis (SIA) with chemiluminescence (CL) detection. The CL was emitted during the oxidation of the analyte by permanganate in aqueous sulphuric acid in the presence of CL enhancer polyphosphate (PFS). Concentration, order, volume of reagents and flow rate were optimised. Also influence of solvents 60% methanol and 60% ethanol was proved. The optimum order, volumes and concentrations of reagents for aqueous solution of verapamil were: 30 μl of 10 mM $KMnO_4$, 50 μl of 2% PFS, 50 μl of 1 mM verapamil, 30 μl of 1 M H_2SO_4 and for 60% ethanolic solution of verapamil were: 20 μl of 10 mM $KMnO_4$, 30 μl of 2% PFS, 50 μl of 1 mM verapamil, 30 μl of 2 M H_2SO_4, 30 μl of 2% PFS and 30 μl/s flow rate. The transient CL signal was recorded at the wavelength ≥ 390 nm. Calibration curves relating the intensity of CL (peak heights) to the concentration of the analyte were curvilinear with rectilinear sections in the range $5 \cdot 10^{-5}$ - $5 \cdot 10^{-3}$ M for aqueous solution and $1 \cdot 10^{-5}$ - $1 \cdot 10^{-3}$ M for 60% ethanolic solution of verapamil. The limits of detection were $2 \cdot 10^{-5}$ M for aqueous solution and $1 \cdot 10^{-5}$ M for 60% ethanolic solution. Repeatability of peak heights (RSD, n = 10) ranged between 4.43 % ($1 \cdot 10^{-4}$ M) and 1.83 % ($1 \cdot 10^{-3}$ M) for aqueous solution, and between 2.11 % ($1 \cdot 10^{-4}$ M) and 4.90 % ($1 \cdot 10^{-3}$ M) for 60% ethanolic solution. The verapamil was assayed in pharmaceutical dosage forms (tablets with nominal content 40, 80 or 120 mg of verapamil) by the proposed method and the SIA results did not show any statistically significant difference from those obtained by HPLC method, except the preparation with nominal content 120 mg of verapamil, where the interference of adjuvants occurs at the SIA method.