DEVELOPMENT AND VALIDATION OF ANALYTICAL METHOD FOR SIMULTANEOUS ESTIMATION OF DOXOFYLLINE AND TERBUTALINE SULPHATE IN BULK AND ITS FORMULATION

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ABSTRACT

A simple, specific, accurate and precise 1st order derivative UV- spectrophotometry method has been developed for the simultaneous estimation of Doxofylline and Terbutaline sulphate in Bulk and its dosage form. The samples were analyzed in methanol. The validation was carried out according to ICH guidelines. In linearity curve correlation coefficients for Doxofylline and Terbutaline sulphate were above 0.997. The percentage recovery was 99.88 %w/w for Doxofylline and 100.08 % w/w for Terbutaline sulphate. The limit of detection was 5.57 µg/ml and 0.070 µg/ml for Doxofylline and Terbutaline sulphate respectively and the limit of quantification was 17.50 µg/ml and 0.214 µg/ml for Doxofylline and Terbutaline sulphate respectively. Proposed methods were validated as per ICH guidelines for linearity, accuracy, precision and robustness for estimation of Doxofylline and Terbutaline sulphate in commercially available tablet dosage form and results were found to be satisfactory.

So the method can be used for estimation of combination of these drugs in tablet dosage form.

A simple, specific, accurate and precise reverse phase high pressure liquid chromatographic (RP-HPLC) method has been developed for the simultaneous estimation of Doxofylline and Terbutaline sulphate in Bulk and its dosage form by Enable C_{18} Column. The samples were analyzed by using Ammonium dihydrogen orthophosphate buffer (0.05M) : Methanol in the ratio of 35:65 v/v as a mobile phase at the flow rate of 1.2 ml/min and detection wavelength was 290 nm. Both the drugs were eluted within 10 minutes and gave sharp peak with high theoretical plate count and low tailing factor. The retention time for Doxofylline and Terbutaline sulphate was found to be 3.44 and 4.61 min. respectively. The validation was carried out according to ICH guidelines. In linearity curve correlation coefficients for Doxofylline and Terbutaline sulphate were above 0.998. The percentage recovery was 99.60 %w/w for Doxofylline and 99.38 % w/w for Terbutaline sulphate. The limit of detection was 27.19 µg/ml and 0.336 µg/ml for Doxofylline and Terbutaline sulphate respectively and the limit of quantification was 81.57 µg/ml and 1.009 µg/ml for Doxofylline and Terbutaline sulphate respectively. Proposed methods were validated as per ICH guidelines for linearity, accuracy, precision and robustness for estimation of Doxofylline and Terbutaline sulphate in commercially available tablet dosage form and results were found to be satisfactory. So the method can be used for estimation of combination of these drugs in tablet dosage form. Degradation study by the developed RP-HPLC method was successfully carried out. Degraded products were separated sufficiently. Purposeful degradation was studied so as to measure reasonable degradation of 10-30% by acid, alkali, oxidative and thermal stress.

Key words: Doxofylline, Terbutaline sulphate, UV-spectrophotometry, Reverse phase high pressure liquid chromatography, Degradation.