Extractive spectrophotometric determination of Amlodipine using Eriochrome black-T and Indigo Carmine

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Two sensitive spectrophotometric methods for the determination of Amlodipine besylate based on the formation of coloured complexes of the drug with reagents such as Erichrome Black - T at pH 3.0 and Inglgo caramine at pH 2.8 are described for bulk samples and pharmaceutical preparations. The ion-pair complexes formed are qantitatively extracted into chloroform under the experimental conditions.

FEW analytical methods[1-3] appeared in the literature for the determination of amlodipine besylate (ADB) in biological fluids and pharmaceutical preparations. There are no visible spectrophotometric methods for the determination of ADB, the present extraction spectrophotometric methods are based on the formation of ion-pair complexes of ADB with Eriochrome Black - T or Indigo caramine and these complexes are quantitatively extracted into chloroform.

An accurately weighed amount (100 mg) of ADB was dissolved in 0.1 M HCl with slight warming and the volume was made upto 100 ml with the same solvent. The stock solution was further diluted with distilled water to get working standard solutions. Tablet powder equalent to 100 mg of the drug was dissolved with slight warming and diluted with 0.1 M HCl, filtered and the total volume was brought to 100 ml with 0.1 M HCl. Further diluted with distilled water for working sample solutions.

Aqueous solutions of 0.2% Erichrome Black - T (EBT) and 0.2% Indigo caramine (IC) 0.1 M HCl and phthalate buffer[4] of pH 3.0 and 2.8 were prepared. Aliquots of the drug solutions representing 50-500 mcg (method A) or 250-1500 mcg (method B) were transferred into a series of separating funnels, 2ml of pH 3.0 buffer solution and 2 ml of 0.2% EBT solution (method A) or 2ml of 2.8 buffer and 2 ml of 0.2% IC solution (method B) were added to each seperating funnel and the total volume of the aqueous phase was made upto 10 ml with distilled water. Ten ml of chloroform was added to each flask and the contents were shaken for 2 minutes. The two phases were allowed to separate and the absorbance of the chloroform layer was measured at 495 nm (method A) or 590 nm (method B) against reagent blank. The amount of ADB in the sample solution was computed from its calibration curve in each method.

The Beer's law limits (mcg/ml), molar extinction coefficient (1 $mole^{-1}$ cm^{-1}), Sandell's sensitivity (mcg/cm^2/0.001 absorbance unit), percent relative standard deviation and percent range of error (0.05 level) for the proposed methods were found to be 5-50, 6.5424×10^3, 0.06, 0.881 and 0.737 for method A and 25-150, 1.962×10^3, 0.20, 0.868 and 0.726 for method B, respectively. The accuracy of the method was ascertained by comparing the results with proposed and reported methods in case of each dosage form (Table 1). In order to justify the reliability and suitability of the proposed methods, known quantities of pure drug was added to various preanalysed dosage forms and the mixtures were analysed using the proposed methods and results are incorporated in the Table. The stoichiometric relationship of drug:

Table 1: Analysis of Dosage Forms

Drug	Labelled amount (mg)	Reported[2] method (mg)	Proposed method		% Recovery of the proposed method	
			Method A (mg)	Method B (mg)	Method A	Method B
ADB						
Tablet I	5	4.99	5.01	5.02	100.2	100.1
Tablet II	10	9.99	10.02	10.04	100.5	100.4
Tablet III	10	9.97	10.06	9.99	100.25	100.6

dye was obtained by slope analysis[5] and were found to be 1:1 for method A and 2:1 for method B.

Both the proposed methods are reported for the first time as visible spectrophotometric assays of ADB. The usual excipients and adjuncts present in the formulations were found not to interfere in the proposed methods. The results indicated that the proposed methods are simple, sensitive, reproducible and can be used for the routine determination of amoldipine besylate and its dosage forms.

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