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NOTE

Extractive Spectrophotometric Methods for the Determination of Amlodipine Besylate

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Two simple and sensitive extractive spectrophotometric methods (Methods A and B) have been developed for the determination of amlodipine besylate (ADB) in bulk samples and pharmaceutical preparations. It forms green coloured complex with fast green FCF exhibiting maximum absorption at 625 nm (Method A). In Method B, ADB forms yellowish orange coloured complex with orange II showing maximum absorption at 485 nm. Both the methods obeyed Beer's law limits in the concentration range of 2.5 to 20 μg/mL of ADB.

Amlodipine besylate (ADB) is 2-[(2-amino-ethoxy)-methyl)-4-(2-chlorophenyl), 1,4-dihydroxy-6-methyl-3,5-pyridine dicarboxylic acid, 3-ethyl-5-methyl ester[1]. Besylate is an anti-hypertensive drug belonging to the class of calcium channel blocker. Literature survey revealed a few analytical methods for the estimation of ADB which include HPLC[2-5], GC[6], LC[7], colorimetric[8] and difference spectrophotometric[9] methods. In the present investigation, ADB forms green coloured molecular ion complex with fast green FCF extractable into chloroform showing maximum absorption at 625 nm (Method A). In the second method (Method B) ADB forms yellowish orange coloured ion pair complex with orange II (extractable into chloroform) exhibiting absorption maximum at 485 m.

All the reagents used were of analytical grade. Solutions of fast green FCF (0.5%), orange II (0.5%), HCl (0.1 N and 0.01 N), were prepared in distilled water.

Spectral and absorbance measurements were made on systronics UV-visible spectrophotometer model 117 with spectral band width of 1 nm and using a pair of 10 mm matched quartz cells.

Standard and sample solutions

100 mg of ADB (pure or formulation) was accurately weighed and dissolved in 100 mL of distilled water. Further dilution was made with distilled water to get working standard solution of 100 μg/mL of ADB.

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Assay Procedures

Method A: Volumes of standard ADB solution ranging from 0.25 to 2.0 mL (1 mL = 100 μg) were transferred into a series of 150 mL separating funnels. Two mL of HCl (0.01 N) and 0.5 mL of fast green FCF (0.5%) were added to each separating funnel and the total volume of the aqueous phase was made up to 10 mL with distilled water. 10 mL of chloroform was added to each funnel and the contents were shaken for 2 min. The two phases were allowed to separate and the absorbance of chloroform layer was measured at 625 nm against reagent blank. The amount of ADB present in the sample solution was computed from its calibration curve.

Method B: To a series of 150 mL separating funnels, aliquot samples of ADB ranging from 0.25 to 2.0 mL (1 mL = 100 μg), 2 mL of HCl (0.1 N), 0.5 mL of orange II (0.5 %) were added successively and the aqueous phase was made up to 10 mL with distilled water. 10 mL of chloroform was added and the contents were shaken for 2 min. The two phases were allowed to separate and the absorbance of yellowish orange coloured chloroform layer was measured at 485 nm against reagent blank. The amount of ADB present in the sample solution was calculated from its calibration curve.

The optical characteristics such as Beer's law limits, Sandell's sensitivity, molar extinction coefficient, stability of the colored species, percent relative standard deviation (calculated from eight separate samples containing $3/4^{th}$ amount of the upper Beer's law limits of ADB in each method), per cent range of error (0.05 and 0.01 confidence limits), correlation coefficient, slope and intercept of regression analysis using least square method were calculated and are summarized in Table-1.

TABLE-1
OPTICAL CHARACTERISTICS AND PRECISION

Parameters	Method A	Method B
Beer's law limit (μg/mL) (C)	2.5 to 20	2.5 to 20
Sandell's sensitivity (μg/ cm^2/0.001 absorbance unit)	0.02380	0.02739
Molar extinction coefficient (1 $mole^{-1}$ cm^{-1})	2.257×10^4	1.916×10^4
Correlation coefficient	0.9999	0.9999
Regression equation (b + aC):		
Slope (a)	4.23×10^{-3}	3.66×10^{-3}
Intercept (b)	–0.000518	–0.001268
Percent relative standard deviation	0.72528	0.98640
Per cent range of error:		
Confidence limit with 0.05 level	±0.6064	±0.8248
Confidence limit with 0.01 level	±0.8972	±1.2200

The values obtained for the determination of ADB in several pharmaceutical formulations (tablets) by the proposed and reported methods[6] are compared in Table-2. To evaluate the validity and reproducibility of the methods, known amount of pure drug was added to the previously analyzed pharmaceutical

formulations and the mixtures were analyzed by the proposed methods and the recoveries (average of six determinations) are given in Table-2. Interference studies revealed that the common excipients and other additives usually present in the dosage forms did not interfere in the proposed methods.

TABLE-2
ESTIMATION OF AMLODIPINE IN PHARMACEUTICAL FORMULATIONS

Sample	Labelled amount (mg)	Amount obtained (mg)			% recovery of the proposed method	
		Reported method[6]	Proposed methods			
			A	B	A	B
1.	5	4.98	5.02	4.99	100.4	99.8
2.	5	5.01	5.03	4.97	100.6	99.4
3.	5	5.03	5.01	5.02	100.2	100.4
4.	5	4.96	5.04	5.01	100.8	100.2

The stoichiometric relationships of the drug : dye have been found to be 1 : 3 and 1 : 2 for method A and method B respectively through the slope analysis method[10]. The results indicate that the proposed methods are simple, sensitive, reproducible and accurate and can be used for the routine determination of ADB in bulk and dosage forms.

REFERENCES

1. **Martindale, The Extra Pharmacopoeia, 26th Edn., The Royal Pharmaceutical Society, London, p. 1492 (1989).**
2. **Barbalo, Francesco, Coppelo, Bronella, Grumetto, Lucia, Morrica and Patrizia, *Farmaco*, 48, 417 (1993).**
3. **A.B. Avadhanulu, J.S. Srinivas and Y. Anjaneyulu, *Indian Drugs*, 33, 36 (1996).**
4. **Patel, P. Yagnesh, Patil, Suvarna, Bhoir, C. Indravedan and M. Sundreshan, *J. Chromatogr. Anal.*, 828, 283 (1998).**
5. **(Ms.) V.J. Dhora and N.B. Shatkar, *Indian Drugs*, 36, 638 (1999).**
6. **A.P. Beresford, P.V. Macrae, D.A. Stopher and B.A. Wood, *J. Chromotogr.*, 420, 178 (1987).**
7. **Yeung, K.F. Pollen Mosher, J. Sugan, P. Pollak and Timothy, *J. Pharm. Biomed. Anal.*, 9, 565 (1991).**
8. **I. Singhvi and S.C. Chaturvedi, *Indian J. Pharm. Sciences*, 60, 309 (1998).**
9. **A. Surekha, Khopade and N.K. Jain, *Indian Drugs*, 37, 351 (2000).**
10. **H. Irving, E.J.C. Rossotti and R.J.P. Williams, *J. Chem. Soc.*, 157, 1906 (1955).**

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