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Derivative spectrophotometric estimation of azathioprine in bulk drug and pharmaceutical dosage forms

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ABSTRACT

A simple, precise and economical spectrophotometric method have been developed for the estimation of Azathioprine in bulk and pharmaceutical formulations. Azathioprine shows a sharp peak at 301.0 nm in first order derivative spectrum with n=1. The drugs follows Beer-Lambert's law in the concentration range of 2-20μg mL^{-1} in both the methods. Result of the analysis was validated statistically and found to be satisfactory.

KEYWORDS

Azathioprine;
UV spectrophotometry;
Derivative spectroscopy.

1. INTRODUCTION

Azathioprine is a Chemically 6-[(1-methyl-4 nitro-1H-imidazole-5yl)sulphanyl]-7H purine (Figure 1). It is clinically useful in the treatment of cancer. It is official in Indian pharmacopoeia[1]. It is listed in The Merck Index and Martindale[2]. Literature survey reveals that only few RP-HPLC[3] and Visible Spectrophotometric[4] methods are reported for the determination of Azathioprine.The objective of the present work is to develop new spectrophotometric methods for estimation of Azathioprine in bulk and formulation with good accuracy, simplicity, precision and economy[7].

2. EXPERIMENTAL

2.1. Materials

Pure sample of Azathioprine was obtained from Alkem Lab. Ltd., Mumbai as a gift sample and tablet of Azathioprine (50 mg) procured from local market. The 0.1M NaOH prepared in distilled water used as solvent. JASCO V-630 UV/VIS spectrophotometer was used with 1cm matched quartz cells.

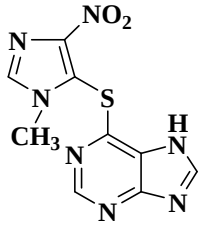

Figure 1: Structure of azathioprine

2.2.Method

Accurately about 100mg of the pure drug was weighed and dissolved in sufficient quantity of 0.1M NaOH and volume made up to100ml with 0.1M NaOH to give standard stock solution (1 mg/ml).Aliquots of standard stock solution were pipette out and suitably diluted with 0.1M NaOH to get final concentration of 2-20μg mL^{-1} of standard solution. The solution were scanned in the spectrum mode from 400 nm to 200 nm wavelength range and the first order derivative spectra were obtained at n =1 a sharp peak was obtained at 301.0 nm (Figure 2). The absorbance difference at n=1 ($dA/d\lambda$) was calculated by the inbuilt software of the instrument which is directly proportional to the concen-

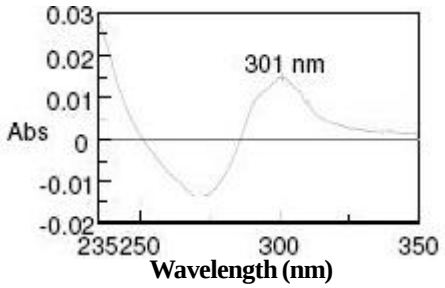

Figure 2 : First derivative spectrum of azathioprine

TABLE 1 : Optical characteristic and other parameters

Parameters	Derivative spectroscopy
λmax (nm)/wavelength range (nm)	301.0
Beer-Lambert's range μg mL^{-1}	2-20
Coefficient of correlation (r)	0.9989
a. Slope (m)	0.0187
b. Intercept (c)	0.5242

TABLE 2 : Estimation of azathioprine in tablet formulation

Method	Tablet	Label claim (mg)	Amount Found (mg)	%	S.D	S.E
Derivative spectroscopy	T1	50	49.96	99.92	0.037	0.0164

TABLE 3: Recovery studies

Excess drug	*Recovery	%RSD
80	99.83	0.2953
100	99.72	0.2026
120	100.58	0.9702

tration of the standard solution .A calibration curve was plotted taking the absorbance difference (dA/dλ) against the concentration of the standard solutions. The method was applied for the sample solution of known concentration and was found be satisfactory for analysis of tablet formulation. Optical characteristics of azathioprine indicated in TABLE 1.

2.3. Analysis of pharmaceutical dosage forms

To determine the content of Azathioprine tablets (label claim: 50 mg of Azathioprine, core tablet) twenty tablets were weighed, their average weight determined and were finely powdered. The weight equivalent to 50 mg of Azathioprine was taken and amount of powder was dissolved 0.1M NaOH by stirring for 30 min. The excipients were separated by filtration. After filtration, an appropriate amount of internal standard was added and diluted up to mark with 0.1M NaOH. Appropriate aliquots were subjected to above methods and the amount of Azathioprine were determined. The results are reported in TABLE 2.

2.4. Recovery studies

To check the accuracy of the developed methods and to study the interference of formulation additives, analytical recovery experiments were carried out by standard addition method, at 80, 100 and 120 % level. From the total amount of drug found, the percentage recovery was calculated. The results are reported in TABLE 3.

3. RESULT AND DISCUSSION

The developed method for estimation of Azathioprine in tablet dosage form were found to be simple, accurate and reproducible. Beer-Lambert's law obeyed in the concentration range of 2-20 μg mL^{-1}. The values of standard deviation were satisfactory and recovery studies were close to 100 %. It also eliminates the interference caused by the excipients and the degradation product present, if any, in the formulation. Hence these methods can be useful in the routine analysis of Azathioprine in bulk drugs and formulations.

4. ACKNOWLEDGEMENTS

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5. REFERENCES

[1] Indian Pharmacopoeia, Govt of India Ministry of Health and Family Welfare, Published By The Indian Pharmacopoeia Commission, Ghaziabad, **1,2**, 144,756,757 **(2007)**.

[2] C.Sean, Sweetman; Martindale, 'The Complete Drug Reference', 34th Edn., The Pharmaceutical Press, London, **875**, 510 **(2002)**.

[3] M.L.Umrethia, P.K.Ghosh, R.J.Majithiya, R.S.R. Murthy; J.Liquid Chrom., and related tech., **29**,**1**, 55-67 **(2006)**.

[4] S.Chilukuri, R.Lakshmi, Manda N.Reddy; Talanta: The International Journal of Pure and Applied Analytical Chemistry, **47(5)**, 1279-1286.

[5] M.S.Palled, M.Chatter, P.M.N.Rajesh, A.R.Bhat; Indian J.Pharma.Sci., **68(5)**, 685-686 **(2006)**.

[6] S.J.Daharwal, G.Garg, R.B.Saudagar, S.Saraf; Indian J.Pharma.Sci., **19(8)**, 102 **(2006)**.