TABLE 1: ANALYSIS RESULTS

Drug	Amount (mg/cap)		% label claim*	% recovery*
	Labeled	Found*		
Cefdinir	300	304.5±0.21	101.5±0.12	98.7±0.15

*Mean of 6 observations

TABLE 2: SYSTEM SUITABILITY PARAMETERS

Parameters	Cefdinir
Resolution	-
Capacity factor	1.2
Asymmetry factor	1
Number of theoretical plates	13,001
LOD (ng/ml)	0.01
LOQ (ng/ml)	0.1

Figures indicate ideal chromatographic separation of cefdinir.

powder equivalent to 1 mg of cefdinir was then extracted with 10 ml buffer. From this 0.6 and 0.9 ml samples were taken and their volume was made up to 10 ml each. A chromatogram of these solutions was obtained by injecting 20 µl of each sample into the chromatographic system. The amount of cefdinir present per capsule and percentage labeled claim was shown in Table 1. There was no interference from diluents and lubricants. Analytical recovery studies were carried out from a series of spiked concentrations added to the pre analyzed dosage form. (Table 1). The drug solution stored under refrigeration was stable up to 12 h, while the solution stored under room temperature was stable up to half an hour only.

The retention time of the drug was 2 min. The system suitability parameters were calculated to confirm the specificity of the developed method and shown in Table 2. The high percentage recovery and low percentage deviation (Table 1) was satisfactory and confirms the accuracy, precision and reliability of the method. The present method can be used for the routine analysis of cefdinir in formulation.

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Visible Spectrophotometric Methods for the Determination of Azithromycin in Tablets

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Two visible spectrophotometric methods have been developed for the estimation of azithromycin in pure and in pharmaceutical formulations. The first method (A), a visible spectrophotometric

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method was based on the formation of a red coloured chromogen with ferric chloride and 1,10-phenanthroline, which showed absorbance maximum at 490 nm and Beer's law was obeyed in the concentration range of 2.5-15 μg/ml. The second method (B) was based on the formation of a blue coloured chromogen with Folin-Ciocalteau reagent, which showed maximum absorbance at 720 nm and Beer's law was obeyed in the concentration range of 25-150 μg/ml. Results of analysis for both the methods were validated statistically and by recovery studies.

Azithromycin, (AZM) (2R,3S,4R,5R,8R,10R,11R,12S, 13S,14R)-13-(2,6-Dideoxy-3-C-3-O-dimethyl-α-L-ribo-hexopyranosyloxy)-2-ethyl-3,4,10-trihydroxy-3,5,6,8,10,12,14-heptamethyl-11-(3,4,6-trideoxy-3-dimethylamino-β-D-xylo-hexopyranosyloxy)-1-oxa-6-azacyclopentadecan-15-one dihydrate, is indicated for the treatment of *Mycobacterium avium* complex infections[1]. It is a new drug and is official in Martindale[2] and British National Formulary[3]. Literature survey revealed a few reverse phase HPLC[4-9] methods available for its determination. No spectrophotometric method has so far been reported. As a part of our continuing efforts to develop simple, sensitive and selective visible spectrophotometric analytical procedure for bulk drugs and their formulations, attention was focused on AZM molecule, keeping in view the relative lack of such methods for its estimation. This paper describes two simple spectrophotometric methods for AZM using ferric chloride and 1,10-phenanthroline for the first method and Folin-Ciocalteau (FC) for the second method.

In the first method, AZM reduces ferric chloride to ferrous, which forms complex with 1,10-phenanthroline to yield a colored chromogen. In the second method, AZM reduces FC reagent in alkaline medium to molybdenum blue, a colored chromogen. FC reagent is the mixture of phosphoric acid, sodium molybdate and sodium tungstate, which is also known as phosphomolybdotungstic acid. The colour formation by FC reagent with azithromycin may be explained in the following manner based on the analogy with the reports of earlier workers[10]. The mixed acids in the FC reagent preparation are the final chromogen and involve the following chemical species; $3H_2O.\ P_2O_5.\ 13WO_3.\ 5MoO_3.\ 10H_2O$ and $3H_2O.\ P_2O_5.\ 14WO_3.\ 4MoO_3.10H_2O$.

All the chemicals used were of analytical grade. Aqueous solution of ferric chloride (0.0033 M) and 1,10-phenanthroline (0.1 M) (S. D. Fine Chem. Ltd., Mumbai) were freshly prepared in distilled water. FC reagent (2 N, CDH Pvt. Ltd, Mumbai) was diluted in the ratio 1:2 and sodium carbonate (10%) was prepared in distilled water. Spectral and absorbance measurements were made on a Shimadzu 1601 UV/Vis Spectrophotometer with 1 cm matched cuvettes. Standard stock solution (Madras Pharmaceuticals) was prepared by dissolving 100 mg of AZM in 2-3 ml of 1.0 M hydrochloric acid and then diluting to the mark in a 100 ml standard flask. Working standard solutions were prepared by diluting the stock solution with water to get 125 μg/ml and 250 μg/ml.

To a series of 25 ml volumetric flasks, aliquots of standard drug solutions ranging from 0.5 to 3 ml (125 μg/ml) were added. This was followed by the addition of 3.75 ml ferric chloride and 3.75 ml of 1,10-phenanthroline and the volume was made up to 25 ml with distilled water. The solutions were warmed on a water bath for 5 min and shaken for 15 min. A red coloured chromogen was obtained, which was measured at 490 nm against a reagent blank.

Aliquots of standard drug solutions ranging from 1 to 6 ml of 250 μg/ml were taken in a series of 10 ml volumetric flasks and to each volumetric flask, 2 ml of FC reagent and 2 ml of sodium carbonate solution were added and heated up to 15 min. Then the volume was made up to 10 ml with distilled water. The absorbance of blue coloured chromogen was measured at 720 nm against a reagent blank after 15 min and a calibration curve was constructed.

Twenty tablets (Azithral-500 mg, Alembic) were weighed and finely powdered. The powder amount equivalent to 100 mg was dissolved in 3 ml of 1 M hydrochloric acid and filtered. The filtrate was made up to 100 ml and appropriate aliquots of solutions were taken and analyzed for AZM using the procedures described earlier.

The optical characteristics such as Beer's law limits, Sandell's Sensitivity, percent relative standard deviations and % range of error were calculated for both the methods and results were summarized in the Table 1. The proposed methods were applied for the analysis of drug in tablets. To evaluate the validity and reproducibility of the method, known amount of pure drug was added to previously analyzed samples and these samples were reanalyzed by the proposed method, the percentage recovery was found to be close to 100% for both the methods. In conclusion, the proposed methods are economical, simple, sensitive and accurate enough for the routine estimation of AZM in bulk

TABLE 1: OPTICAL CHARACTERISTICS AND PRECISION DATA

Parameters	Method A	Method B
λmax	490 nm	720 nm
Beer's law limit (μg/ml)	2.5 to 15.0	25 to 150
Molar absorptivity (l/mol.cm)	$0.96x10^4$	$0.36x10^4$
Correlation coefficient	0.999	0.996
Sandell's sensitivity	0.0811	0.218
(μg/cm² absorbance unit/0.01)		
Regression equation (Y=mx+c)[a]		
Slope (b)	0.0077±0.036	0.0034±0.164
Intercept (a)	0.0472	0.0357
Relative standard deviation	0.97%	0.732%
% Range of error[b]		
Confidence limit with 0.01 level	1.024	0.771
Confidence limit with 0.05 level	0.778	0.581

a -With respect to Y=mx + c, where 'c' is the intercept and 'x' is the concentration in μg/ml, b- Six replicate samples.

as well as in tablet form.

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