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UV-spectrophotometric estimation of thiocolchicoside by derivative method in pharmaceutical dosage form

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ABSTRACT

The objective of the study was to develop a simple, accurate, precise and rapid a UV spectrophotometric i.e. first and second order derivative methods for the determination of thiocolchicoside in pharmaceutical dosage form i.e. capsules by using methanol as a solvent. The method was further validated by ICH guidelines. The proposed derivative methods involve the measurement of absorbance at 251.8 nm for first order and 224 nm for second derivative for the estimation of thiocolchicoside respectively. The linearity of the proposed method was found in the concentration range of 1 to 14 µg /ml (r^2= 0.9993) for first order derivative and 1 to 14 µg /ml (r^2= 0.9987) for second order derivative methods respectively. The percentage mean recovery was found to be 100.365 % for first order derivative and 100.31% for second order derivatives methods respectively. The methods were also statistically validated for its linearity, accuracy and precision. Both intra and inter day variations showed less percentage (%) RSD values indicating high grade of precision of this methods.

Keywords: UV spectrophotometric estimation, derivative method, thiocolchicoside, methanol

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INTRODUCTION

Thiocolchicoside, a semi synthetic derivative of naturally occurring compound of colchicoside from the seeds of various species of colchicum antumnale (autumn crocus, meadow saffron, Gloriosa upuba), chemically,*N*-[(7*S*)-3-(β-D-Glucopyranosyl oxy)-1,2-dimethoxy-10-(methyl sulfanyl)-9-oxo-5,6,7,9-tetrahydro benzo[*a*]heptalen-7-yl] acetamide It is centrally acting muscles relaxant and it also show analgesic activity. It is used in treatment of muscular pain and gout.

In literature survey reveals, spectrofluometric [1], spectrophotometric [2], HPLC [3,4] and HPTLC methods [5] for determination of thiocolchicoside in pharmaceutical dosage form.

MATERIALS AND METHODS

Instrument and reagents

Spectral scan was made on a Shimadzu UV-spectrophotometer, model 1800 (Shimadzu, Japan) with spectral band width of 0.5 nm with automatic wavelength corrections by using a pair of 10 mm quartz cells. All spectral measurements were done by using UV-Probe 2.42 software.

Reference standard of thiocolchicoside was obtained from reputed firm with certificate of analysis.

Preparation of standard drug solution
A10 mg standard thiocolchicoside was weighed accurately and transferred to a 10 ml volumetric flask and sonicated with 5 ml methanol for 15 minutes. The volume was made up to the mark with methanol to give a stock solution of thiocolchicoside of concentration 1000 µg /ml. From this solution, 1 ml of solution was pipetted out and transferred into 10 ml volumetric flask. The volume was made up to mark with methanol to give a working standard solution of concentration 100 µg/ml.

Estimation from capsules
Twenty capsules were weighed accurately and average weight of powder containing in each capsule was determined. Powder equivalent 4 mg of thiocolchicoside was weighed and transferred in 100 ml of volumetric flask. A 30 ml of methanol added and sonicated for 15 minutes and filtered. The filtrate and washing were diluted up to the mark with methanol to give concentration as 40 µg /ml of thiocolchicoside respectively. For working sample solution 1 ml of such solution was diluted to 100 ml and such solution was used for analysis.

Experimental
Method:
(a)For first order derivative method
For the selection of analytical wavelength, 100 µg/ml solution of thiocolchicoside was scanned in the spectrum mode from 350 nm to 200 nm by using methanol as blank. The first order derivative spectrum was obtained by using derivative mode by UV probe 2.42 software. From the spectrum, the amplitude of the first derivative spectrum was measured at 251.8 nm.

(b) For second order derivative method
For the selection of analytical wavelength, 100 µg/ml solution of thiocolchicoside was scanned in the spectrum mode from 350 nm to 200 nm by using methanol as blank. The first order derivative spectrum was obtained by using derivative mode by UV probe 2.42 software. From the spectrum, the amplitude of the second derivative spectrum was measured at 224 nm.

Preparation of calibration curves
Series of solutions containing 1 – 14 µg/ ml of thiocolchicoside were used to determine linearity of the proposed method respectively. Solutions were scanned in the spectrum mode and absorbance spectra were converted to first order derivative spectra. The overlain spectra of first order and second order derivatives of thiocolchicoside were given in Fig. 1(a), 1(b) respectively.

Fig. 1(a): Overlay spectra of first order derivative of thiocolchicoside in the concentration range of 2 and 14 µg/ ml at 251.8 nm

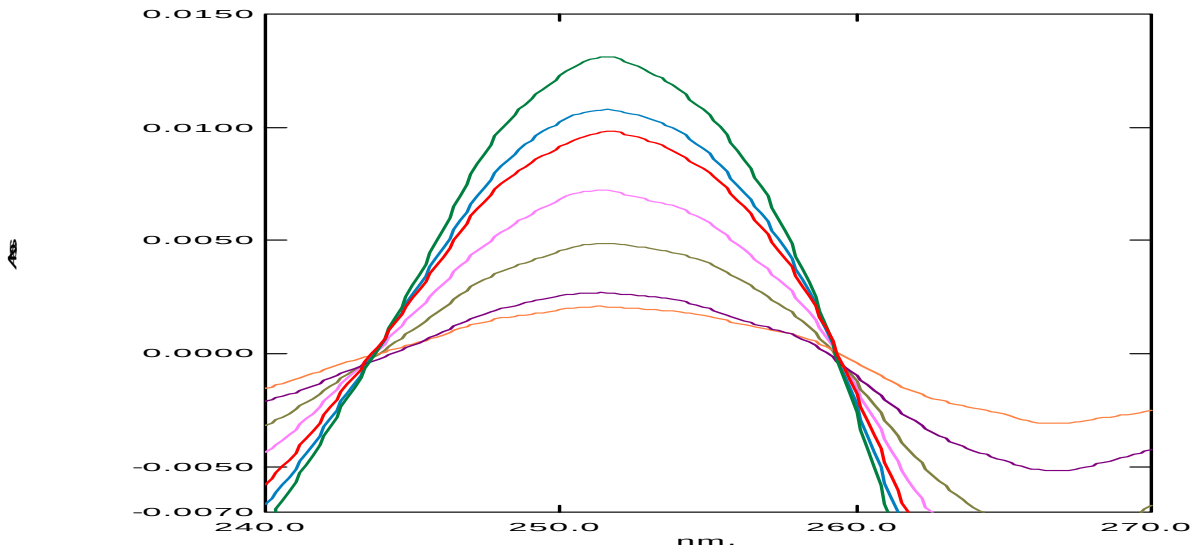
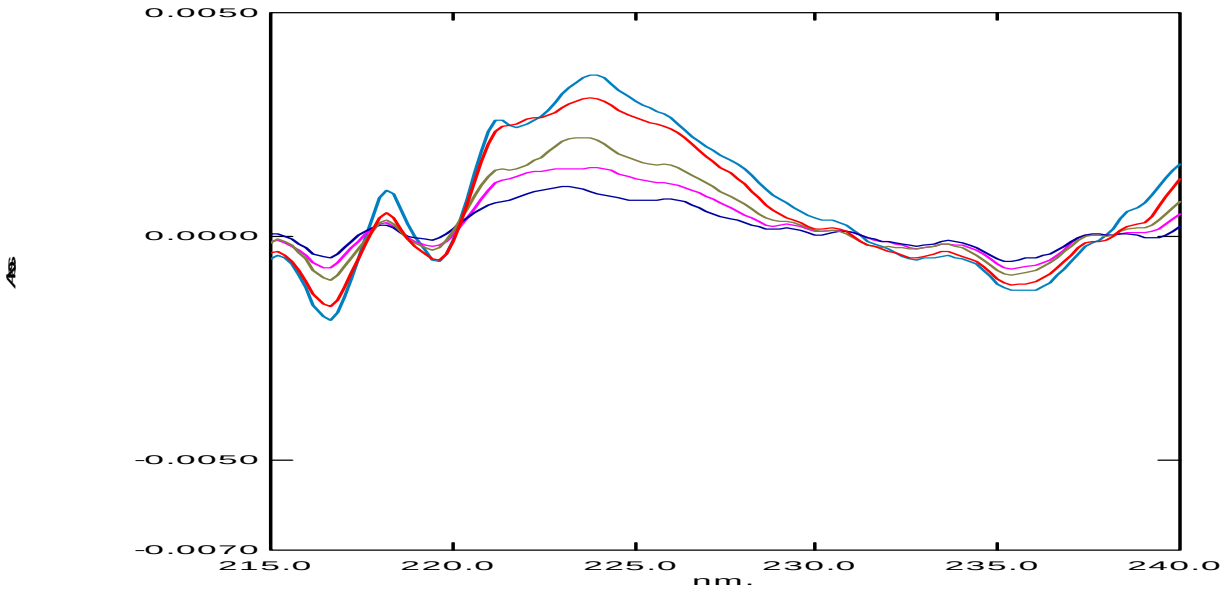


Fig. 1(b): Overlay spectra of second order derivative of thiocolchicoside in the concentration range of 2 and 14 µg/ ml at 224 nm



After observing the overlain first order and second order derivative spectra of thiocolchicoside, the wave length selected was 251.8 nm, for first order derivative method and 224 nm for second order derivative method. The calibration curves were plotted of amplitude against concentrations [Fig. 2 (a), 2(b)].

Fig.2 (a): Calibration curve of first order derivative method in the concentration range of 2-14 µg/ml.

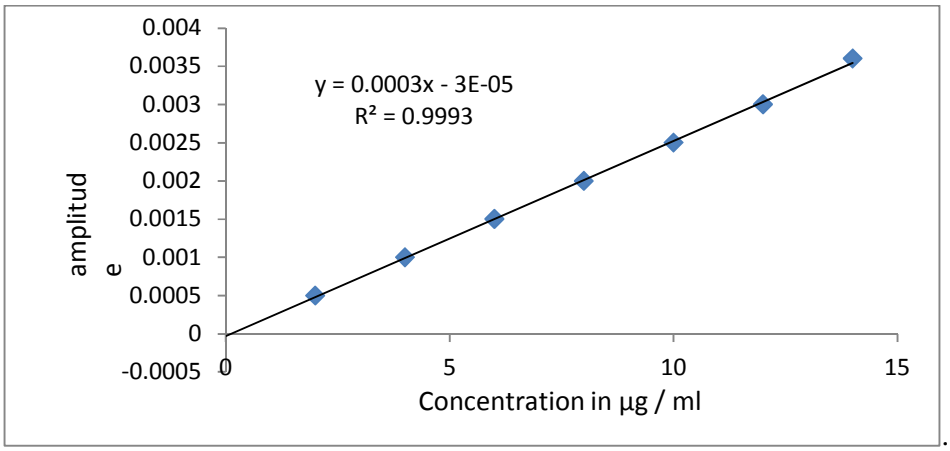
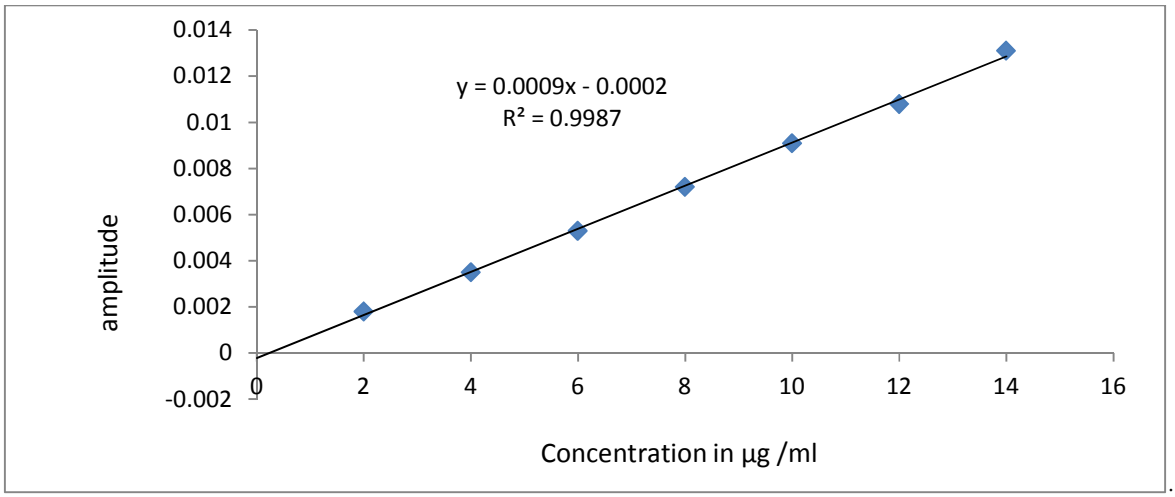


Table 1: Values of results of optical and regression of drugs

Parameter	First order derivative method	Second order derivative method
Detection Wavelength (nm)	251.8	224
Beer Law Limits (µg/ml)	1-14	1-14
Correlation coefficient(r^2)	0.9993	0.9987
Regression equation (y=b+ac)		
Slope (a)	0.0003	0.0009
Intercept (b)	-0.00003	-0.0002

Fig.2 (b): Calibration curve of second order derivative method in the concentration range of 2-14 µg/ml



Results of the analysis are given in table 1.

Estimation from capsules

Powdered from twenty capsules were collected and weighed accurately and average weight of powder from each capsule was determined. Powder equivalent to 4 mg of thiocolchicoside was weighed and transferred in 100 ml of volumetric flask. A 30 ml of methanol was added and sonicated for 15 minutes and filtered. The filtrate and washing were diluted up to the mark with methanol to give concentration as 40 µg /ml of thiocolchicoside respectively. A 10 ml of such solutions was diluted to 100 ml. It was scanned in the range of 200-400 nm against methanol. The absorbance spectra were converted to first and second order derivative spectra. Calculations were done as per the equations. The concentrations of thiocolchicoside present in capsules were calculated by substituting the values of absorbance in linearity equations.

(a) For aceclofenac Y = 0.0003x - 0.00003
(b) For thiocolchiciside Y = 0.0009x - 0.0002

Method Validation

These methods were validated according to ICH guidelines.

Accuracy

To ascertain the accuracy of proposed methods, recovery studies were carried out by standard addition method at three different levels (80%, 100% and 120%). Percentage recoveries for thiocolchicoside were 100.11 % to 100.34% and 100.11 % to 100.37 % for first order and second order derivatives respectively. (Table2).

Table 2(a): Statistical evaluation of the data subjected to accuracy for first order derivative method

Level of % recovery	Amount present in µg/ml	Amount added in µg/ml	Amount found in µg/ml	% Recovery	Mean % recovery
80%	4	3.2	7.215	100.21	100.20
	4	3.2	7.218	100.26	
	4	3.2	7.210	100.14	
100%	4	4	8.015	100.19	100.21
	4	4	8.027	100.34	
	4	4	8.008	100.11	
120%	4	4.8	8.814	100.17	100.25
	4	4.8	8.822	100.25	
	5	4.8	8.829	100.34	

Table 2(b): Statistical evaluation of the data subjected to accuracy for second order derivative method

Level of % recovery	Amount present in µg/ml	Amount added in µg/ml	Amount found in µg/ml	% Recovery	Mean % recovery
80%	4	3.2	7.222	100.31	100.24
	4	3.2	7.218	100.26	
	4	3.2	7.210	100.15	
100%	4	4	8.015	100.19	100.22
	4	4	8.029	100.37	
	4	4	8.008	100.11	
120%	4	4.8	8.814	100.17	100.19
	4	4.8	8.813	100.15	
	5	4.8	8.822	100.26	

Linearity

The linearity of measurement was evaluated by analyzing different concentration of the standard solutions of thiocolchicoside. For both the methods concentration range was found to be 1-14 µg/ml for thiocolchicoside respectively.

Precision

The method precision was established by carrying out the analysis of powder blend from capsules containing 4 mg of thiocolchicoside. The assay was carried out for the drugs by using proposed analytical method in six replicates. The values of relative standard deviation were 0.4574 % for first order derivative method and 0.2455 % for second order derivative method in respectively indicating the sample repeatability of the methods. The results obtained are tabulated in table 3

Table 3: Statistical evaluation of the data subjected to method of precision

Sr..No.	Sample No.	% Assay	
		First order derivative metho	Second order derivative method
1	1	100.25	100.09
2	2	100.62	100.25
3	3	100.67	100.55
4	4	99.65	100.00
5	5	100.85	100.35
6	6	100.15	100.62
Mean % assay		100.365	100.31
%R.S.D.		0.4574	0.2455

Intra-day precision was estimated by assaying capsules powder blend containing 4 mg of thiocolchicoside. The assay was carried out for the drugs by using proposed analytical method in six replicates. The results were average for statistical evaluation.

Inter-day precision was estimated by assaying capsules powder blend containing 4 mg of thiocolchicoside for three consecutive days (i.e. 1st, 3rd and 5th days). The statistical validation data for intra and inter day precision is summarized in table 4.

Table 4: Summary of validation parameter for intra-day and inter-day

Sr. No.	Parameters	First order derivative method	Second order derivative method
1	Intra-day precision (N=3)amount found ± % R.S.D.	100.27% 0.4865	100.47% 0.2789
2	Inter-day precision (N=3)amount found ± % R.S.D.	98.81 0.5675	99.77% 0.4765

Both intra- day and inter-day precision variation found to be less in % RSD values. It indicates high degree of precision of the methods.

RESULTS AND DISCUSSION

The developed derivative spectrophotometric methods for determination of thiocolchicoside in capsules formulation were found to be simple and convenient for the routine analysis of drug. The proposed methods are accurate, precise and reproducible. It is confirmed from validation data as given in tables 1 to 4. The % RSD was found to be less than 1, which indicates validity of method. Linearity was observed by linear regression equation methods for thiocolchicoside in different concentration range. The correlation coefficient of these drugs was found to be close to 1.00, indicating good linearity figure 2 (a) and 2 (b).

The assay results obtained by proposed method is shown in table 2 are in good agreement. Hence proposed methods can be used for routine analysis of the drug in pharmaceutical dosage form. Methods are simple, accurate, precise, reliable, rapid, sensitive, reproducible and economical. They are validated as per ICH guidelines.

CONCLUSION

The proposed methods are simple, precise, accurate and rapid for the determination of thiocolchicoside in pharmaceutical dosage form. These methods can be adopted as an alternative to the existing methods. It can be easily and conveniently adopted for routine quality control analysis.

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