SPECTROPHOTOMETRIC ESTIMATION OF MELOXICAM IN BULK AND ITS PHARMACEUTICAL FORMULATIONS

B.DHANDAPANI, S.ESWARA MURALI, N. SUSRUTHA, RAMA SWETHA, S K. SONIA RANI, T. SARATH BABU, G.V. SEETHARAMANJANEYULU, AND R.V.CELESTIN BABOO

Department of Pharmaceutical Analysis, A.M. Reddy Memorial College of Pharmacy, Petlurivaripalem, Narasaraopet (Mdl), Guntur District – 522601. Andhrapradesh, India.

ABSTRACT

Three simple, accurate, rapid and sensitive methods developed for the determination of meloxicam in bulk drug and in its tablets by UV Spectroscopy (Method - A), Visible (Method -B) and Hydrotropic method (Method - C). In Method A meloxicam estimated at 269 nm using 0.1M NaOH as a solvent. The linearity was observed in the concentration range of 5 – 30 µg/ml with correlation co efficient of 0.9995. In Method B 0.1M NaOH solution using as a solvent and 5% ferric chloride used as a chromogenic agent and the resulting green colour chromogen was measured at 476 nm in the linearity range and correlation co -efficient of 50 – 250 µg/ml and 0.9986. In Method C 10% trisodium citrate solution used as a hydrotropic agent to dissolve the meloxicam which is water insoluble, and it is estimated at 269 nm with the linearity range and correlation co -efficient of 5 – 30 µg/ml and 0.9987. The result of analysis for all the methods was validated statistically and by recovery studies.

Key Words: Meloxicam, Uv-Spectrophotometry, Ferric chloride and Hydrotropic agent

INTRODUCTION

Meloxicam is chemically 4-hydroxy-2-methyl-1-N (5-methyl-2-thiazolyl)-2H-1, 2- benzothiazine-3-carboxamide-1, 1-dioxide, **[Fig-1]** used as a non steroidal anti-inflammatory drug (NSAID) with analgesic and antipyretic properties [1]. Prostaglandins are substances that contribute to inflammation of joints. Meloxicam inhibits prostaglandin synthetase (cylooxygenase 1 and 2) and leads to a decrease of the synthesis of prostaglandins, therefore, inflammation is reduced. Survey of literature reveals that the drug is determined by using HPLC [2-3], HPTLC [4] and few spectrophotometric method [5-6]. The present study describes simple, sensitive, accurate, rapid and economical spectrophotometric methods A, B and C for the estimation of meloxicam in bulk drug and its pharmaceutical formulations.

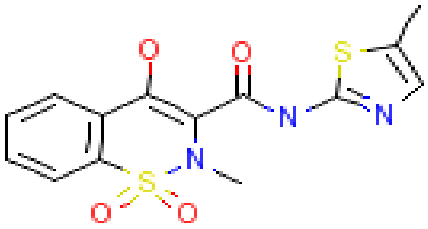

Fig 1: Structure of Meloxicam

EXPERIMENTAL

ELICO UV Spectrophotometer SL 150 with 1cm matched quartz cells was used for the spectra measurements.

All the chemicals used were of analytical reagent grade: (i) Ferric chloride (ii) sodium hydroxide (iii) Tri sodium citrate (iv) All Reagents were prepared by using distilled water

ASSAY PROCEDURE

METHOD A: Meloxicam stock solution was prepared by weighing 100mg of Meloxicam, transferred in to 100ml volumetric flask (previously calibrated) and dissolve it in 40ml of solvent by shaking for 10 min and volume was made up to 100ml with 0.1N NaOH to get a concentration of 1mg/ml (solution A). From this solution an aliquot of 10ml was withdrawn and it was diluted to 100ml with distilled water to get a concentration of 100µg/ml (solution-B). From this aliquots of 0.5ml, 1ml, 1.5ml, 2ml, 2.5ml, 3.0ml were pipetted out in to a 10 ml volumetric flask (Previously Calibrated) and diluted to 10ml using distilled water to get concentrations of 5 µg/ml, 10 µg/ml, 15 µg/ml, 20 µg/ml, 25 µg/ml, 30 µg/ml respectively. Absorbance of this solution was measured at 269nm using UV Spectrophotometer against blank (0.1N NaOH).

Similarly the absorbance of sample solution was measured and amount of meloxicam was determined by using calibration curve.

METHOD B: Meloxicam stock solution was prepared by weighing 100mg of meloxicam, transferred in to a 100ml volumetric flask and volume was made up to 100ml with 0.1N NaOH ,to get a concentration of 1000 µg/ml was obtained (**solution-A).** From this aliquots of 0.5ml, 1ml, 1.5ml, 2ml and 2.5ml were pipetted out in to a 10 ml volumetric flask (Previously Calibrated) and add 1ml of 5% ferric chloride solution (acidic medium) finally diluted to 10ml using distilled water to get concentrations of 50µg/ml, 100 µg/ml, 150 µg/ml, 200 µg/ml and 250 µg/ml. the absorbance of the green colored chromogen was measured at 476nm against blank solution.

Similarly the absorbance of sample solution was measured and amount of meloxicam was determined by using calibration curve.

METHOD C: By using hydrotropic solubilising agent (10% Tri sodium citrate in distilled water), the drug-Meloxicam (insoluble in water) is made water soluble due to a complex formation which is completely water soluble. The absorbance of resulting solution was measured at 269 nm.

Meloxicam stock solution was prepared by weighing 100mg of meloxicam, transferred in to a 100ml volumetric flask, then volume was made up to 100ml with 10% Tri sodium citrate solution and heated in a water bath at 70^0C for 10 min, to get a concentration of 1000 µg/ml (**solution-A).** From this solution an aliquot of 10ml was withdrawn and it was diluted to 100ml with distilled water to get a concentration of 100µg/ml (solution-B). from this aliquots of 0.5ml, 1ml, 1.5ml, 2ml, 2.5ml, 3.0ml were pipetted out and diluted to 10ml using distilled water to get concentrations of 5 µg/ml, 10 µg/ml, 15 µg/ml, 20 µg/ml,25 µg/ml, 30 µg/ml respectively and the absorbance the resulting solution (green color) was measured at 269nm against blank.

Similarly the absorbance of sample solution was measured and amount of meloxicam was determined by using calibration curve.

ANALYSIS OF FORMULATION

The methods were extended for the determination of meloxicam in tablets. 20 tablets of brand M-com, label claim 7.5mg of meloxicam was weighed, powdered, average weight was determined and equivalent weight was calculated based on label claim equal to 100mg, transferred into a 100ml volumetric flask and make up to the mark with suitable solvent. The contents of the flask thoroughly mixed and filtered by using what Mann's filter

paper. The sample solution was analyzed as described in the above mentioned methods. The analysis procedure was repeated six times and the results of analysis were shown in **Table 1, 2&3.**

Table 1: (Method – A)

Analysis of Formulation:

S.No	Sample	Labelled Amount (mg)	Concentration Taken For Analysis (µg/ml)	Amount Of Substance (µg/ml)	% Recovery •
1.	Tablet	7.5	10	97.82	97.82
2.	Tablet	7.5	15	14.905	99.36
3.	Tablet	7.5	20	19.813	99.06
				Mean Recovery	**98.746**

- Each value is mean of six observations

Table 2: (Method – B)

Analysis of Formulation:

S.No	Sample	Labelled Amount (mg)	Concentration Taken For Analysis (µg/ml)	Amount of Substance (µg/ml)	% Recovery •
1.	Tablet	7.5	100	101.87	101.87
2.	Tablet	7.5	150	146.97	97.98
3.	Tablet	7.5	200	196.91	98.45
				Mean %Recovery	**99.43**

- Each value is mean of six observations

Table 3: (Method – C)

Analysis of Formulation:

S.No	Sample	Labelled Amount (mg)	Concentration Taken For Analysis (µg/ml)	Amount Of Substance (µg/ml)	% Recovery
1.	**T**ablet	7.5	10	10.572	105.72
2.	Tablet	7.5	15	14.54	96.93
3.	Tablet	7.5	20	20.04	100.2
				Mean% Recovery	**100.74**

- Each value is mean of six observations

RECOVERY STUDIES

To ensure the accuracy and reproducibility of the results obtained, adding known amounts of pure drug to the previously analyzed formulated samples and reanalyzed by the proposed methods and also performed by recovery experiments. The results of percentage recovery shown in **Table 4, 5&6.**

Table 4: (Method – A)

Recovery Studies:

Level	Concentration of Standard Solution Used	Concentration of Sample Used	Amount found (µg/ml)	% Recovery •
A	10(µg/ml)	10(µg/ml)	10.220	102.20
B	15 (µg/ml)	15(µg/ml)	14.359	98.726
C	20 (µg/ml)	20(µg/ml)	19.453	97.265
			Mean % Recovery	99.397

- Each value is mean of six observations

Table 5: (Method – B)

Recovery Studies:

Level	Concentration of Standard Solution Used	Concentration of Sample Used	Amount found (µg/ml)	% Recovery
A	100 µg/ml	100 µg/ml	104.20	104.20
B	150 µg/ml	150 µg/ml	147.44	98.29
C	200 µg/ml	200 µg/ml	197.44	98.72
			Mean %Recovery	100.40

- Each value is mean of six observations

Table 6: (Method – C)

Recovery Studies:

Level	Concentration of Standard Solution Used	Concentration of Sample Used	Amount found (µg/ml)	% Recovery •
A	10(µg/ml)	10(µg/ml)	9.880	98.80
B	15 (µg/ml)	15(µg/ml)	14.524	96.82
C	20 (µg/ml)	20(µg/ml)	20.274	101.37
			Mean % Recovery	97.33

- Each value is mean of six observations

RESULTS AND DISCUSSION

Estimation of Meloxicam in dosage forms by UV, Visible Spectrophotometry and hydrotropic solubilisation method was carried out using optimized conditions, the percentage recovery of drug found in formulations and the results of analysis shows that the amount of drug was in good agreement with the label claim of the formulation. The proposed method for quantification of Meloxicam in tablets was simple, precise, accurate, rapid and sensitive. The methods are linear in the concentration range reported. The developed methods are free from interference due to the excipients present in the tablets and can be used for quantitative estimation of meloxicam in tablets. Statistical analysis was carried out and the result of which satisfactory. The optical characteristics such as absorption maxima, Beer's law limits, molar absoptivity and sand ell's sensitivity were reported in **Table - 7.**

Table 7: Validation and Statistical Parameters

S.NO	PARAMETER		RESULTS		
			UV (Method A)	COLORIMETRY (Method B)	HYDROTROPIC (Method C)
1	λ max		269 nm	476 nm	269 nm
2	Linearity range (µg/ml)		5-30	50-250	5-30
3	Correlation Coefficient (r)		0.9995	0.9992	0.9987
4	r^2		0.9992	0.9986	0.9976
5	Intercept (c)		0.1588	-5.297	-0.7343
6	Slope (m)		0.02204	0.003470	0.02043
7	Standard Deviation (SD)		0.2382	0.3248	0.2209
8	Standard Error (SE)		0.09003	0.1326	0.08350
9	Limit Of Detection (LOD) (ng/ml)		37.8	327.60	37.84
10	Limit Of Quantification (LOQ) (ng/ml)		108.07	936.02	108.1
11	Intra Day (%RSD)	10µg/ml	0.0051	0.0046	0.0051
		15µg/ml	0.0060	0.0042	0.0060
		20µg/ml	0.0095	0.0049	0.0095
12	Inter Day (%RSD)	10µg/ml	0.0140	0.0194	0.0140
		15µg/ml	0.0029	0.0701	0.0029
		20µg/ml	0.0068	0.0060	0.0068
13	Repeatability (% RSD)		0.0130	0.0068	0.0294
14	Accuracy		99.39	100.40	97.33
15	Sand ell's Sensitivity (µg/ml)		0.4756	2.752	0.5745
16	Molar Absoptivity (g/lit/mole)		2.1066×10^4	3.633×10^3	2.5648×10^4

The regression analysis using the method of least squares was made for slope (m), intercept (c) and correlation obtained from the different concentration and the results were shown in **Table – 7.**

The reproducibility and precision of the methods are very good shown by the low values of % RSD. Recovery studies were close to 100% that indicates the accuracy and precision of the proposed methods and also indicates non interference from the formulation excipients. The results of analysis were shown **Table – 7.**

In conclusion the developed methods are simple, accurate, sensitive and economical for the routine estimation of meloxicam in bulk drug and its pharmaceutical formulations.

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