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# Spectrophotometric Methods for the Estimation of Meloxicam in Dosage Forms

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For the estimation of meloxicam from dosage forms two simple, quick, sensitive, visible and UV spectrophotometric methods were developed. The visible method is based on the formation of an intense brownish green coloured complex with ferric ammonium sulfate, which was stable at room temperature. The coloured product is quantitated spectrophotometrically at 396 nm and was found to obey the Beer's law in the concentration range of 5-30 μg $mL^{-1}$. The UV method is based on the ionization of meloxicam and the maximum absorption was found at 354 nm and obeyed the Beer's law in the concentration range of 3-12 μg $mL^{-1}$. The methods were applied for estimation of different meloxicam tablets with mean percentage accuracies of 99.28 ± 0.2802, 99.28 ± 0.1997 and 99.81 ± 0.6040, respectively with visible spectrophotometric method and 99.47 ± 0.5297, 99.73 ± 0.1300 and 98.91 ± 0.6810, respectively with UV spectrometric method.

**Key Words: Meloxicam, Spectrophotometer, Recovery study, Ferric(III) ammonium sulfate.**

## INTRODUCTION

Meloxicam is a newer non-steroidal antiinflamatory drug of the enolic carboxide class, found to preferentially inhibit cyclooxygenase-2 (cox-2). It is used in the treatment of rheumatoid arthritis and osteoarthritis. Several methods were reported for the determination of this drug[1-10]. Meloxicam is official in the BP (2002). The method of analysis for the bulk drug is based upon non-aqueous titration[11]. From the review of the literature, it is evident that there is a need to develop a simple and sensitive method(s) for the estimation of meloxicam from its dosage forms. Hence in this study an attempt has been made to develop two simple, sensitive, quick and economic methods for the determination of meloxicam in pharmaceutical dosage forms using visible and UV spectrophotometric techniques.

## EXPERIMENTAL

Beckman DU 64 single beam spectrophotometer with 10 mm quartz cell attached to a scanning speed of 250 nm/min was employed for all absorbance measurements.

Meloxicam BP was gifted by Sun Pharmaceuticals Ltd. Other reagents included ferric(III) ammonium sulfate (anhydrous) A-grade (Merck), methanol spectroscopic grade (SRL), lactose, (Merck), di-calcium phosphate (Merck), sodium starch glycolate IP (Merck), starch (Merck), ethylcellulose (CDH) and polyvinyl pyrrolidine K30 (SRL). M-cam (Unichem Laboratories Ltd.) and muvera (Sun Pharmaceutical Industries Ltd) were purchased from local pharmacy. 0.1 N sodium hydroxide (Merck) solutions were prepared using double distilled water. 1 or 0.5 % (w/v) solution of ferric(III) ammonium sulfate (anhydrous) was prepared freshly by dissolving the appropriate amount in distilled water.

**Method A (visible method)**

**Preparation of standard curve:** Methanol (spectroscopic-grade) was used to prepare a solution of meloxicam BP (100 µg/mL). From this solution, varying volumes (0.5-3.0 mL) were transferred into 10 mL volumetric flasks so as to obtain final concentrations in the range, 5-30 µg/mL. 1 mL of freshly prepared 0.5 % w/v ferric ammonium sulphate solution was added to each of the above flasks and the contents shaken and made up to volume with methanol in each case. The absorption spectra of the above solutions were recorded between 350-450 nm against a blank using a 10 mm quartz cell in a Beckman DU64 single beam spectrophotometer. The observed values of the ΔA were then plotted against the concentrations to obtain the calibration curve.

**Determination of drug content in tablet dosage form using method A:** 10 Tablets of each of the three-marketed preparations of meloxicam were weighed separately and finely powdered using a mortar and pestle. A quantity equivalent to 6 mg of meloxicam from the powdered tablets was taken in a 50 mL volumetric flask and about 40 mL of methanol was added to it and the contents shaken. Then the volume was made up with methanol and the contents were filtered using a Whatman No. 1 filter paper. Varying volumes (1, 2, 3 and 4 mL) of above solutions were transferred into different 10 mL volumetric flasks. 1 mL of freshly prepared 1 % w/v ferric ammonium sulphate solution was added to each of the flasks, the contents shaken and made up to volume with methanol. From the absorbance values the drug content was determined.

**Method B (UV spectrophotometric method)**

**Preparation of standard curve:** 15 mg of pure drug meloxicam BP was dissolved in 100 mL of 0.1 N NaOH. From the above solution varying volumes 2-6 mL of the above solution were transferred into different 100 mL volumetric flasks and made up to volume with double distilled water, so as to obtain concentration in the range 3-12 µg/mL. The absorption spectra of the above solutions were recorded in the wavelength range of 300-400 nm against the blank. The absorbances obtained were plotted against the corresponding concentrations to obtain the calibration curve.

**Determination of drug content in tablet dosage form using method B:** 10 Tablets of each marketed preparations of meloxicam were weighed and finely powdered using mortar and pestle. A quantity equivalent to 7.5 mg of meloxicam from

the powdered tablets was taken in a 100 mL volumetric flask and the volume was made up to 100 mL of 0.1 N NaOH was added to it and the contents shaken. The contents were filtered using a Whatman No. 1 filter paper. From above solution varying volumes (10, 12 and 15 mL) were transferred into a 100 mL volumetric flask and made up to volume with double distilled water. From the absorbance values the drug content was determined.

**Determination of drugs in the presence of additives:** 20 mg of meloxicam BP and starch (tablet additive) were taken in a 100 mL volumetric flask. 0.1 N NaOH was added, contents mixed and filtered and then estimated for drug content in a manner similar to that of as mentioned in the determination of drug content in tablet dosage forms of method A and B.

The same method as mentioned above was employed with other tablet additives such as lactose, dicalcium phosphate, sodium starch glycoate, ethylcellulose, and polyvinyl pyrrolidineK30.

**Validation of the methods:** To validate the proposed methods, recovery studies were performed. Solutions containing a known amount of meloxicam were added to a specified volume of the solution prepared for the determination of drug content for meloxicam tablets .The amount of meloxicam present in the above mixture was determined and the percentage recovery calculated.

**Statistical analysis:** Experimental results were expressed as mean ± SD analysis of variance was performed by ANOVA procedures (SSPS 9.0 for Windows).

## RESULTS AND DISCUSSION

Compounds with a -C=O and -COOH groups are capable of forming complexes as given in Fig. 1.

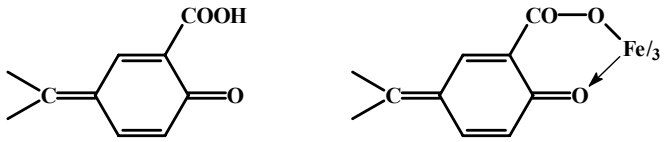


Fig. 1. Compound with a -C=O and -COOH groups are capable of forming complexes

In case of meloxicam, formation of similar complexes with a trivalent $Fe^{3+}$ ion can form the basis for its spectrophotometric determination.

For the visible method, the complex between ferric ions and the meloxicam was formed instantaneously at room temperature upon mixing the solutions and the complex was brownish-green in colour. The complex formed was stable upto 24 h for the quantitative determination. As a result of possible complexation between meloxicam and ferric ion that obeyed Beer's law at the $\lambda_{max}$ of 396 nm within a concentration range of 5-30 µg/mL. Linear regression equation was also obtained for the same. Thus the above-mentioned coloured complex could be used for the determination of meloxicam.

Studies undertaken using tablet additives such as lactose, dicalcium phosphate, sodium starch glycoate, starch, ethylcellulose, polyvinyl pyrrolidine K30, indicate that they did not interfere with the estimation of meloxicam for both the visible and UV method.

The applicability of the proposed methods was tested by the determination of meloxicam in commercially available tablets. The study was carried out on the same batch of samples and the results obtained are presented in Table-1.

TABLE-1
ESTIMATION OF MELOXICAM FROM TABLET DOSAGE FORM

| Tablet | Visible absorption | | | | UV absorption | | |
|---|---|---|---|---|---|---|---|
| | Claim as per label (mg) | Amount estimated (mg) | Mean ± SD | Standard error | Amount estimated (mg) | Mean ± SD | Standard error |
| M-cam | 7.5 | 7.478 | 7.45±0.0231 | 0.0133 | 7.450 | 7.46±0.0400 | 0.0231 |
| | 7.5 | 7.436 | | | 7.427 | | |
| | 7.5 | 7.440 | | | 7.505 | | |
| Muvera | 7.5 | 7.436 | 7.44±0.0115 | 0.0066 | 7.490 | 7.48±0.01 | 0.0057 |
| | 7.5 | 7.459 | | | 7.480 | | |
| Muvera | 15 | 14.89 | 14.97±0.090 | 0.0523 | 14.720 | 14.83±0.102 | 0.0589 |
| | 15 | 15.07 | | | 14.880 | | |
| | 15 | 14.96 | | | 14.830 | | |

The advantage of the visible method is that it is quick and simple method.

For the UV spectrophotometric method compliance with Beer's law at the $\lambda_{max}$ 354 nm and linear regression equation was obtained over a concentration range of 3-12 µg/mL.

The applicability of the proposed methods was tested by the determination of meloxicam in commercially available tablets. The study was carried out on the same batch of samples and the results obtained are presented in Table-1. The recovery studies for both the methods are presented in Table-2.

TABLE-2
REPEATED ESTIMATION OF MELOXICAM FROM THE TABLETS FOR VALIDATION OF ANALYTICAL METHOD A (VISIBLE) AND B (UV) N = 3

| Tablet | Visible | | UV | |
|---|---|---|---|---|
| | Recovery (%) Mean ± SD | Standard error | Recovery (%) Mean ± SD | Standard error |
| M-cam (7.5 mg) | 99.101±0.934 | 0.53967 | 99.49±0.4950 | 0.28618 |
| Muvera (7.5 mg) | 99.800±0.395 | 0.22800 | 99.79±0.8166 | 0.47149 |
| Muvera (15 mg) | 100.753±0.666 | 0.38450 | 99.28±0.0900 | 0.05196 |

Mean recovery percentages of the three meloxicam concentrations in tablets were found to be 99.101, 99.80 and 100.75, respectively [n = 3] in visible method and 99.49, 99.79 and 99.82, respectively [n = 3] in UV method.

The results show that both the methods are accurate, precise, very simple and statistically significant results were obtained for both the visible method and UV spectrophotometric method. Both the methods proposed for the determination of meloxicam in bulk and pharmaceutical formulations have the advantage of being fast, simple, inexpensive, and also show good precision.

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