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ISSN: 0973-4945; CODEN ECJHAO
E-Journal of Chemistry
2010, **7(4),** 1463-1467

# Development and Statistical Validation of Spectrophotometric Methods for the Estimation of Nabumetone in Tablet Dosage Form

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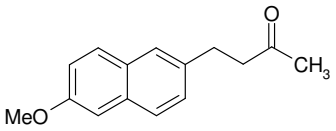
Received 26 January 2010; Accepted 20 March 2010

**Abstract:** Three new simple, economic spectrophotometric methods were developed and validated for the estimation of nabumetone in bulk and tablet dosage form. First method includes determination of nabumetone at absorption maxima 330 nm, second method applied was area under curve for analysis of nabumetone in the wavelength range of 326-334 nm and third method was First order derivative spectra with scaling factor 4. Beer law obeyed in the concentration range of 10-30 µg/mL for all three methods. The correlation coefficients were found to be 0.9997, 0.9998 and 0.9998 by absorption maxima, area under curve and first order derivative spectra. Results of analysis were validated statistically and by performing recovery studies. The mean percent recoveries were found satisfactory for all three methods. The developed methods were also compared statistically using one way ANOVA. The proposed methods have been successfully applied for the estimation of nabumetone in bulk and pharmaceutical tablet dosage form.

**Keywords*:*** Area under curve method, Derivative spectroscopy, Nabumetone, UV spectrophotometric.

## Introduction

Nabumetone (I) is frequently prescribed as non-acidic non-steroidal anti-inflammatory drug (NSAID) for the symptomatic treatment of rheumatic and inflammatory conditions[1,2]. Nabumetone is chemically known as (4-(6-methoxy-2-naphthyl)-butan-2-one. It is official in United States Pharmacopoeia and British Pharmacopoeia. Literature survey revealed that several analytical techniques like Colorimetric[3] liquid chromatographic[4-6], HPLC[7,8,] spectrophotometric[9] methods have been reported for the determination of nabumetone. The proposed research work describes three new UV-spectrophotometric methods for the estimation of nabumetone in bulk and tablet dosage form.




I (Nabumetone)

## Experimental

Shimadzu UV-2450 double beam spectrophotometer with 1 cm path length supported by Shimadzu UV-Probe software, version 2.21 was used for all spectrophotometric estimations. Shimadzu balance (AUW-120D) was used for all weighings.

Nabumetone was obtained from IPCA Lab. Ratlam, Gujarat, India. Formulation of nabumetone in tablet dosage form was purchased from local market. Methanol was purchased from Fischer Scientific (India).

### *Standard stock solution*

Solution containing 200 µg/mL of pure drug was prepared by dissolving 20 mg of nabumetone in sufficient methanol to produce 100 mL solution in volumetric flask. From this aliquot solution was pipetted out and diluted with methanol to obtained working standard stock solution of 50 µg/mL.

### *Analysis of the tablet formulation*

Ten tablets were accurately weighed and powdered. A portion of tablet powder equivalent to 20 mg of nabumetone was accurately weighed and transferred into a 100 mL volumetric flask and then added 25 mL of methanol to dissolve contents of tablet formulation. Then, solution was sonicated for 20 minutes and filtered through Whatman filter paper 41. The final volume was made up to 100 mL with methanol to obtain concentration of 200 µg/mL nabumetone. From this aliquot solution was pipetted out and suitably diluted with methanol to obtained working standard stock solution of 50 µg/mL. Various dilutions of the tablet solution were prepared and analyzed for four times and the concentration was calculated by using the calibration curve for three methods.

### *Recovery*

A recovery study was carried out by addition of known amount of standard drug in the pre-analysed tablet formulation, in 80%, 100% and 120% of label claim. At each level of amount three determinations were performed.

## Results and Discussion

### *Absorption maxima method*

For selection of analytical wavelength 30 µg/mL solution of nabumetone was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400-200 nm. From the spectra $\lambda_{max}$ of nabumetone 330 nm was selected for the analysis (Figure 1). The calibration curve was prepared in the concentration range of 10-30 µg/mL at 330 nm. By using calibration curve, the concentration of the sample solution was determined.

### *Area under curve method*

For selection of analytical wavelength, 30 µg/mL solution of nabumetone was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400-200 nm. From the spectra of drug, area under the curve in the range of 326-334 nm was selected for the analysis (Figure 2). The calibration curve was prepared in the concentration range of 10-30 µg/mL at their respective AUC range. By using calibration curve, the concentration of the sample solution can be determined.

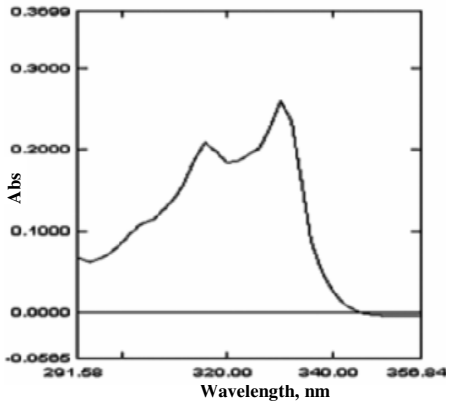



**Figure 1.** Zero order UV-spectrum of nabumetone (30 µg/mL) showing absorptaion maxbma at 330 nm

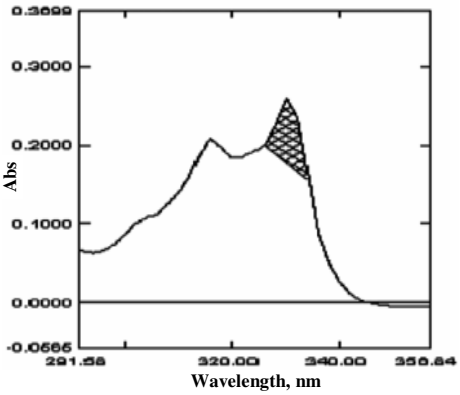



**Figure 2.** Zero order UV-spectrum of area under curve method for nabumetone, 30 µg/mL

*First order derivative spectroscopic method*

In this method, 30 µg/mL solution of nabumetone was prepared by appropriate dilution of standard stock solution and scanned in the spectrum mode from 400-200 nm. The absorption spectra obtained was derivatized to first order derivative spectra. The First order derivative spectra at scaling factor 4 shows maxima and minima at 310 and 338 nm respectively (Figure 3). The absorption difference is calculated which was directly proportional to the concentration of the standard solution. The calibration curve for nabumetone was plotted in the concentration range of 10-30 µg/mL and scanned in the first order derivative spectra. The calibration curve of dA/dt against concentration of drug showed linearity.

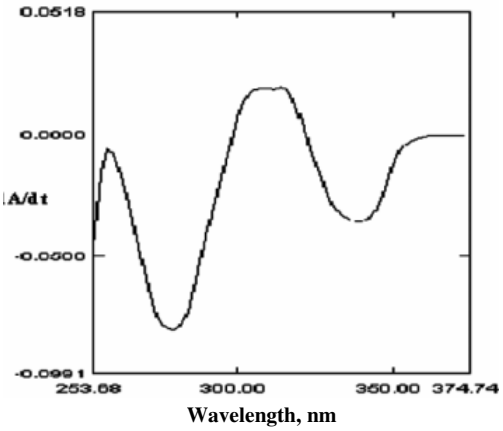



**Figure 2.** First order derivative spectrum of nabumetone (30 µg/mL)

**Method Validation**

*Linearity*

From standard stock solution of 50 µg/mL of nabumetone 1, 2, 3, 4, 5, 6 mL were transferred into series of 10 mL volumetric flasks to obtain concentration range of 5 to 30 µg/mL for nabumetone (Table 1).

**Table 1.** Statistical parameters of linearity of nabumetone

| Parameters | Absorption Maxima | Area Under Curve | First Derivative Spectroscopy |
|---|---|---|---|
| Beer's law range. | 10-30 µg/mL | 10-0 µg/mL | 10-30 µg/mL |
| Coefficient of Correlation ($r^2$) | 0.9997 | 0.9998 | 0.9998 |
| Slope(m). | 0.00888 | 0.01263 | 0.00188 |
| Intercept(c). | -0.00716 | -0.0054 | -0.00088 |
| LOD, µg/mL. | 0.23 | 0.34 | 0.26 |
| LOQ, µg/mL. | 0.71 | 1.03 | 0.79 |

$Y=mX+c$

## *Accuracy and precision*

Precision of the method was evaluated by using tablet powder equivalent to 100% of the label claim of nabumetone. Method repeatability was obtained from R.S.D. value by repeating assay of four replicates of single concentration three times in a same day. Intermediate precision was assessed by assay of four replicates of single concentration of nabumetone on three consecutive days. The accuracy of the methods was assessed by recovery studies at three different levels, 80%, 100% and 120%. The values of standard deviation and recovery studies were found satisfactory (Table 2).

**Table 2.** Results of analysis of nabumetone in tablet formulation and recovery studies

| Method | Label Claim Mg/tab | % estimated* | %Recovery* | S.D.± | %RSD | SEM |
|---|---|---|---|---|---|---|
| Absorption Maxima | 500 . | 99.63 . | 100.6 . | .0.9515 . | 0.9458 . | 0.5494 . |
| Area Under Curve | 500 . | 99.87 . | 100.2 . | 0.9169 . | 0.9150 . | 0.5294 . |
| First Order Derivative | 500 . | 99.78 . | 100.4 . | 0.6803 . | 0.6775 . | 0.3928 . |

**denotes n=4*

## *Limit of detection and limit of quantisation*

The detection limit and quantisation limit was computed for lower limit of detection and minimum quantity of analyte measured and was found to be satisfactory by proposed spectrophotometric methods.

## *Statistical evaluation*

The developed methods statistically compared using one way ANOVA and indicate no significant difference between three methods. Hence these methods can be useful in routine analysis of nabumetone in bulk drug and tablet formulation (Table 3).

**Table 3.** Results of one way ANOVA (Tukey-Kramer multiple comparison test)

| Comparison | Mean Difference | q Value | P Value |
|---|---|---|---|
| Absorption maxima *vs*. AUC | 0.4 | 0.605 | P> 0.05 |
| Absorption maxima *vs*. First order Derivative spectroscopic | 0.2 . | 0.342 . | P> 0.05 . |
| AUC *vs*. First order Derivative Spectroscopic | -0.2 | 0.350 . | P> 0.05 . |

## Conclusion

The developed new three methods proved to be simple in procedure and it produced more accurate results. Hence all three methods effective for the routine analysis of nabumetone in bulk and tablet dosage form.

*Acknowledgments*

The authors gratefully acknowledge the IPCA Laboratories., Ratlam, and Gujrat, India for providing gift sample of nabumetone. The authors are also thankful to Management and Principal of M.G.V's Pharmacy College, Panchavati, Nashik for providing necessary facilities and constant support for research work.

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