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Novel First Order Derivative UV- Spectrophotometric Peak Detect Method for the Determination of Nitrofurantoin

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Abstract : A simple, precise and economical procedure has been developed for estimation of Nitrofurantoin in bulk drug and pharmaceutical dosage form, Using UV- spectrophotometer. As there are no reported the UV methods for Quantitative evaluation of first order peak detect method for Nitrofurantoin in bulk as well as tablet dosage form, there was a need to expand novel methods to analyze the drugs using Acetone as a solvent. For the evaluation of Nitrofurantoinin bulk as well as tablet dosage form, first order peak detect method was developed, which is based on absorption at maximum wavelength 333nm using acetone as a solvent. This drug follows the Beer's law in the concentration range of 5-25µg/ml. The recovery studies ascertained the accuracy of the proposed method in addition validated as per ICH guidelines. This process was useful for the assessment of Nitrofurantoin in pure drug form as well as in tablet dosage form.

Keywords : Nitrofurantoin, First order derivative method, peak determination.

Introduction:

Nitrofurantoinis an antibiotic applicable for the care for bladder infections. It is not efficient for kidney infections. It is taken by oral cavity[1]. Nitrofurantoin is an antibiotic that fights bacteria in the body. Nitrofurantoin is used to treat urinary tract infections.You should not take Nitrofurantoin if you have severe kidney disease, urination problems, or a history of jaundice or liver problems caused by nitrofurantoin.[2]

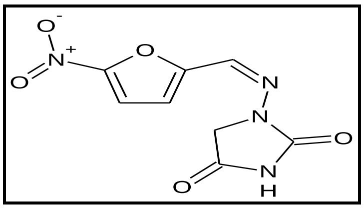

Fig.1: Chemical Structure of Nitrofurantoin

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It is used in the management of type 2 diabetes mellitus. It improves sensitivity to insulin in muscle and adipose tissue and inhibits hepatic gluconeogenesis also improves glycemic control while reducing circulating insulin levels.The most important use is to treat UTI. [3]

On literature survey, it was found that rapid HPLC method for determination of Nitrofurantoin, analysis of Nitrofurantoin using HPLC in tablet dosage form[4], UV spectrophotometric method [5] have been reported for determination of Nitrofurantoin. But first order derivative UV Spectrophotometric peak detect method not yet reported for the determination of Nitrofurantoin. Hence, investigation of new analytical methods was in need for the quantitative estimation of Nitrofurantoin.

Materials and Methods:

Materials:

Shimadzu 1800 spectronic UV Spectrophotometer with 1cm matched quartz cells was used for data collection andanalysis. Methanol was used as a solvent for drug substance.

Methodology:

Preparation of Standard Stock Solution:

The standard stock solution was prepared by transferring 25 mg Nitrofurantoin in to a 100 ml volumetric flask. 100 ml Acetone $(CH_3)_2CO$ was shifted in to this volumetric flask and then shakes well and dissolved. Then the quantity was prepared up to the mark with$(CH_3)_2CO$ to give a solution containing 1000 µg/ml Nitrofurantoin. From this volume 0.5 ml was move to 25 ml volumetric flask and the volume was adjusted to the mark with the help of Acetone to give a solution containing 100 µg/ml of Nitrofurantoin.

Determination of λ max:

Accurate volume 1.0 ml of standard stock solution of Nitrofurantoin was shifted to 25 ml volumetric flask & the volumes were adjusted to the mark with similar solvent to get the solution of concentration 10 µg/ml. After that the prepared solution was scanned in theUV range 330-400 nm. The λ max was found to be 333 nm. The spectrum of Nitrofurantoinwas recorded.

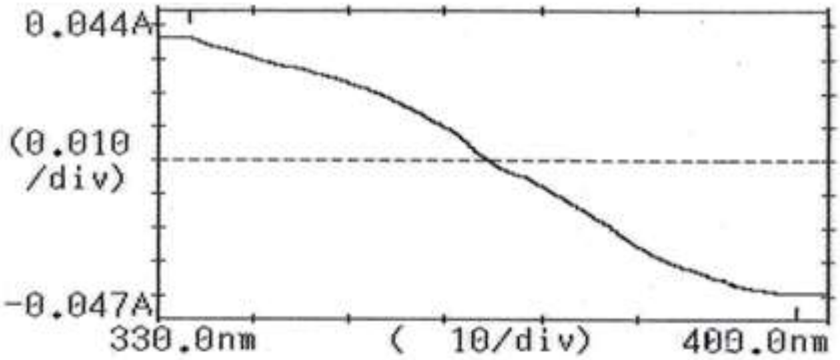


Fig2: Spectrum of λ max of Nitrofurantoin

Selection of Analytical Wavelength Range:

By the use of Shimadzu 1800 spectronic UV-Visible Spectrophotometer the derivative spectra of Nitrofurantoin were taken at N=8 & the standard solutions of Nitrofurantoin in Acetone (5µg/ml each) subjected to a scan 330 nm to 400nm. The λ max was found to be at 330nm. For the calibration curve of Nitrofurantoin was found to be linear at 333 nm. Beer’s law obeyed in the concentration range of 5-25µg/ml.

Stability of Drug in Selected Solvent:

The stability of the drugs in the selected solvent was determined by measuring the absorbance of the drug solutions (20µg/ml) at different time interval. The absorbance was measured after every 5 and 15min. The stability data is given in Table 1.

Table 1: Stability Data of Nitrofurantoin

Sr. No.	**Time (min)**	**Absorbance**
1	0	0.032
2	5	0.040
3	10	0.040

Table 2: Standard Calibration Table for Nitrofurantoin330nm.

Sr. No.	**Concentration of Nitrofurantoin (µg/ml)**	**Absorbance at 330nm**
1	5	0.007
2	10	0.013
3	15	0.022
4	20	0.032
5	25	0.038

Linearity

By using of the standard stock solution of Nitrofurantoin diverse volumes 0.5, 1, 1.5, 2, 2.5, ml were transferred to five separate 25 ml volumetric flask and volume were made up to the mark along with $(CH_3)_2CO$ i.e. Acetone solvent to obtain the concentrations5, 10,15, 20, 25 µg/ml after that calibration curve was assembled. (Table 2, Figures 3 to 7).

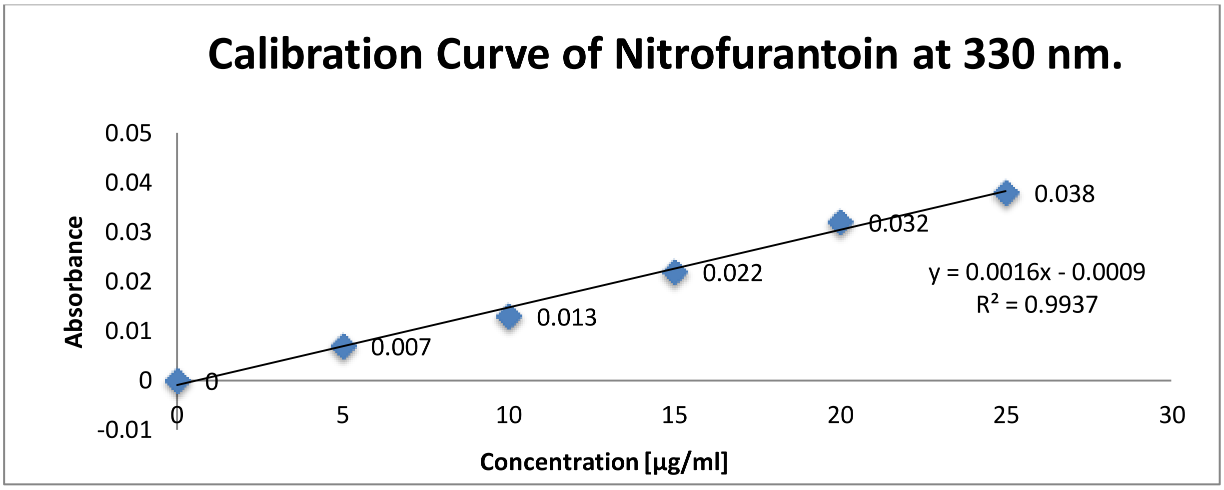
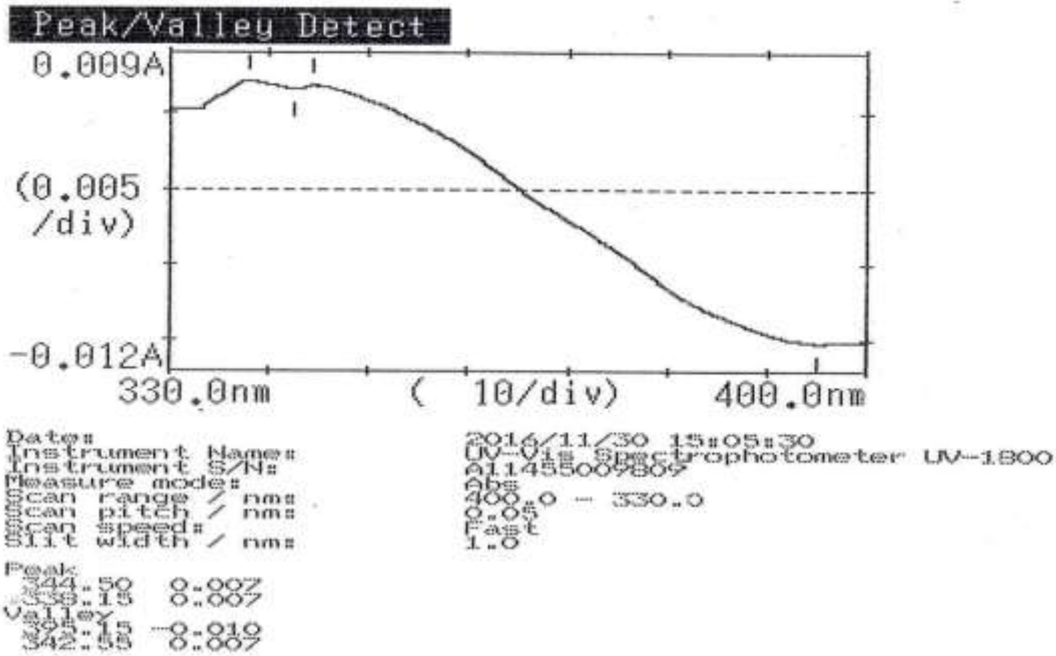


Fig. 3:Standard Calibration Curve of Nitrofurantoin at 330 nm.

The following Figures represent the linearity of Nitrofurantoinat 330 nm.



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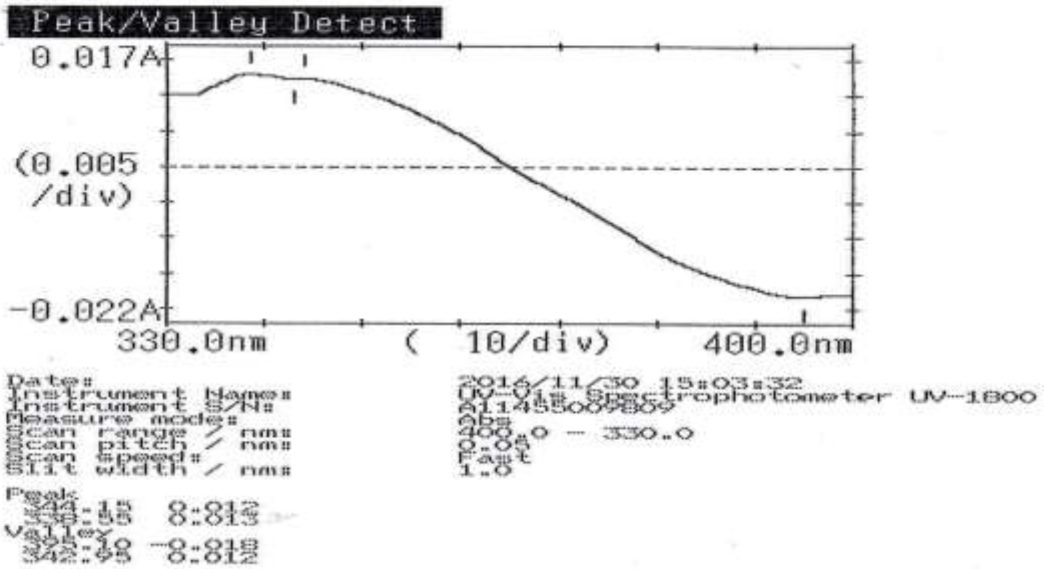
Date:                          2016/11/30 15:05:30
Instrument Name:               UV-Vis Spectrophotometer UV-1800
Instrument S/N:                A11455009809
Measure mode:                  Abs
Scan range / nm:               400.0 - 330.0
Scan pitch / nm:               0.05
Scan speed:                    Fast
Slit width / nm:               1.0
Peak
 344.50   0.007
 338.15   0.007
Valley
 395.15  -0.010
 342.55   0.007
```

Fig. 4: First derivative spectrum of Nitrofurantoin conc. 5μg/ml



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Instrument S/N:                A11455009809
Measure mode:                  Abs
Scan range / nm:               400.0 - 330.0
Scan pitch / nm:               0.05
Scan speed:                    Fast
Slit width / nm:               1.0
Peak
 344.15   0.012
 338.55   0.013
Valley
 395.10  -0.018
 342.95   0.012
```
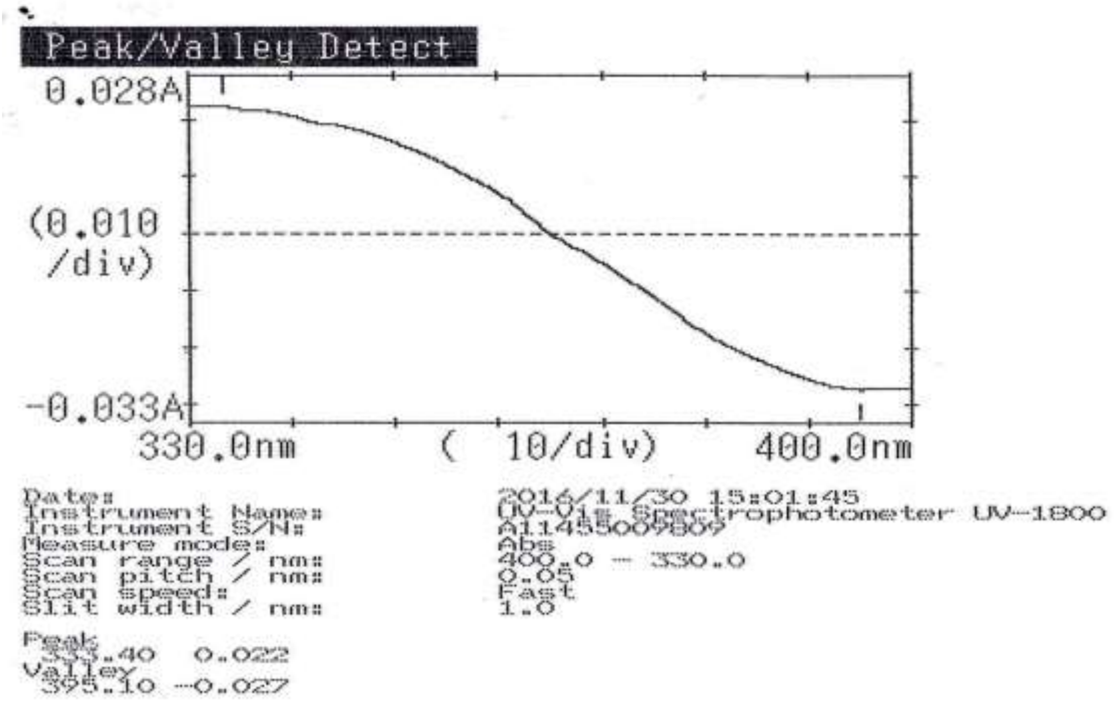

Fig. 5: First derivative spectrum of Nitrofurantoin conc. 10 μg/ml



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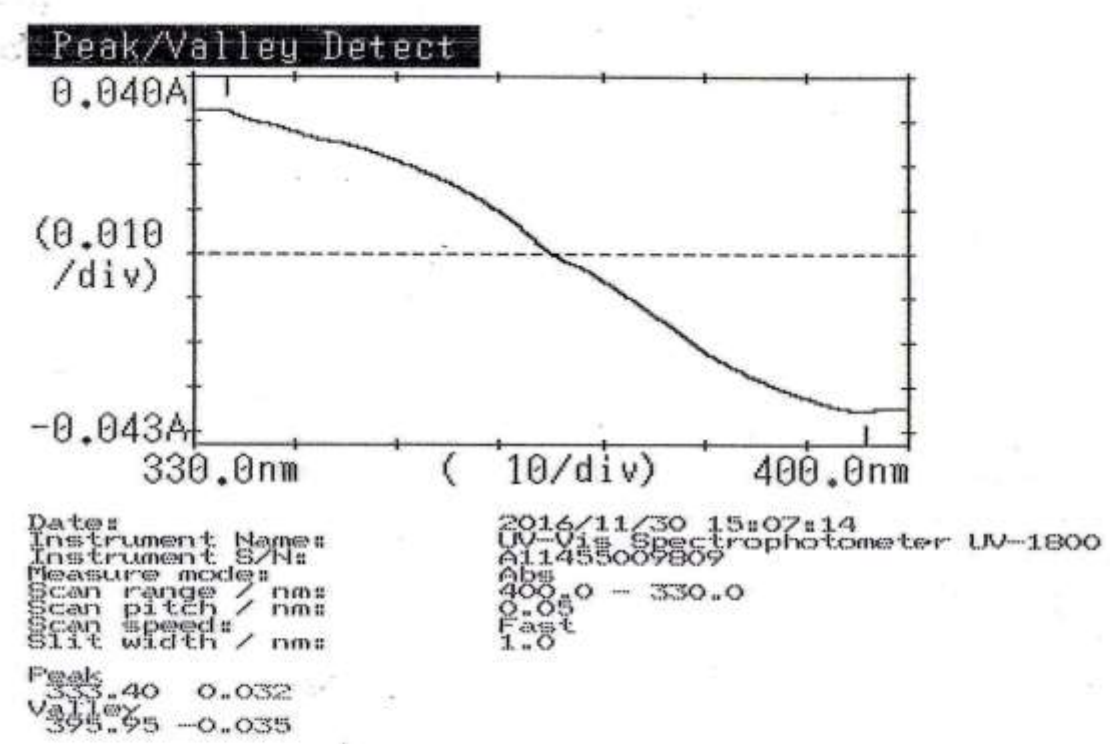
Date:                          2016/11/30 15:01:45
Instrument Name:               UV-Vis Spectrophotometer UV-1800
Instrument S/N:                A11455009809
Measure mode:                  Abs
Scan range / nm:               400.0 - 330.0
Scan pitch / nm:               0.05
Scan speed:                    Fast
Slit width / nm:               1.0

Peak
  333.40   0.022
Valley
  395.10  -0.027
```

Fig. 6: First derivative spectrum of Nitrofurantoin conc. 15 µg/ml



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Instrument Name:               UV-Vis Spectrophotometer UV-1800
Instrument S/N:                A11455009809
Measure mode:                  Abs
Scan range / nm:               400.0 - 330.0
Scan pitch / nm:               0.05
Scan speed:                    Fast
Slit width / nm:               1.0

Peak
  333.40   0.032
Valley
  395.95  -0.035
```
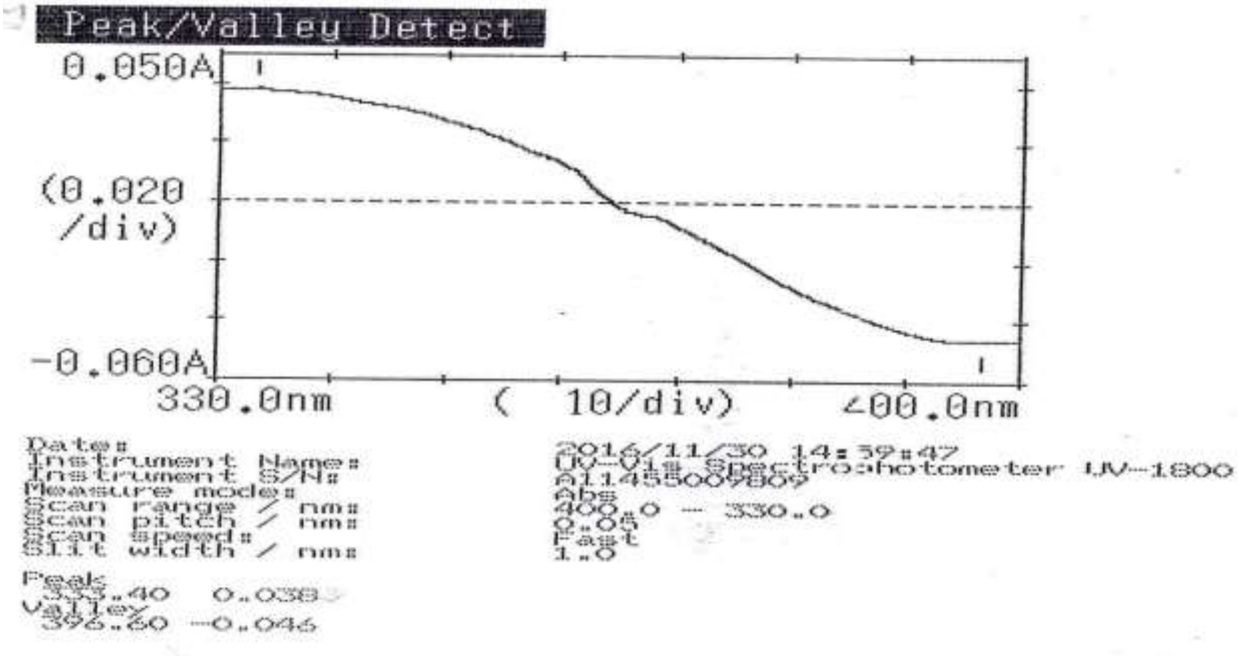

Fig. 7: First derivative spectrum of Nitrofurantoin conc. 20 µg/ml



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Date:                      2016/11/30 14:59:47
Instrument Name:           UV-VIS Spectrophotometer UV-1800
Instrument S/N:            A11455009809
Measure mode:              Abs
Scan range / nm:           400.0 - 330.0
Scan pitch / nm:           0.05
Scan speed:                Fast
Slit width / nm:           1.0
Peak
 333.40   0.038
Valley
 396.60  -0.046
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Fig. 8: First derivative spectrum of Nitrofurantoin conc. 25 µg/ml

Validation of Proposed Method:

Estimation of Drug from Dosage Form: (Tablet AssayStudy)

Brand name- **NIFTAS 50**

Standard:

For the possibility of projected method for evaluation of Nitrofurantoinin marketed pharmaceutical formulations, the process is preliminary tried for evaluation of drugs in standard bulk sample.

Exactly weighed 25 mg of Nitrofurantoin was conveying to 100 ml volumetric flask, after that dissolved in acetone by shaking and quantity was adjusted to mark by the same solvent. Then 5 ml was shifted to 50 ml volumetric flask & then volume was adjusted up to the mark with the same solvent to achieve the concentration 100 µg/ml. From that accurate stock solution 0.5 ml was transferred to 25 ml volumetric flask & volume adjustedup to the mark with the same solvent to obtain the concentration 5µg/ml. At 330 nm the absorbance of the solution was recorded against blank and results are reported.

Sample:

For analysis of marketable tablet formulation; brought the marketed brands tablet strips of Nitrofurantoin. Total weight of the all tablet was recorded. Afterward separately take 1 tablet weights, for the projected work 25 mg drug was required. Crush the tablet by using mortar. Then calculated weight to be taking followed by prepare the 100 µg/ml stock solution. Prepare the 10µg/ml solution and take the absorbance at 330 nm. Results are shown in Table 3.

Table 3: Assay of Nitrofurantoinin Tablet formulation (Brand A)

Amount Taken (mg/tab)	Amount found (mg/tab)	Amount found (%)
50	49.87	98.67
50	49.85	99.65
50	50	100.65
50	49.98	99.78
50	50	100.65
	Mean	**99.88**
	SD	**0.8235**
	CV	**0.0082**

Accuracy (Recovery Test):

For the recovery experiment was studied by Accuracy. By adding known amounts of tablet the recovery experiments were performed. The recovery was performed at three levels, 80, 100, and 120 % of Nitrofurantoinstandard concentration. Recovery samples were prepared in before mentioned procedure. For every recovery level three samples were prepared. The percentage recoveries were calculated by using formula and the solutions were analyzed.

$$\% \text{ Recovery} = \frac{\text{Observed amount of compound in sample}}{\text{Amount of all compound present in sample}} \times 100$$

The Results of accuracy parameter of Nitrofurantoinare shown in Table 4.

Table 4: Results of accuracy parameter of Nitrofurantoin

Level of % Recovery	Label claim (50mg)	Amount of standard added (mg)	Total amount recovered (mg)	% Recovery	% mean Recovery	SD	CV
80	50	40	40.05	100.12	99.9633	0.2136	0.0021
80	50	40	39.89	99.72			
80	50	40	40.02	100.05			
100	50	50	49.90	99.8	100	0.2227	0.0022
100	50	50	50.12	100.24			
100	50	50	49.98	99.96			
120	50	60	59.89	99.81	99.98	0.17	0.0017
120	50	60	60.09	100.15			
120	50	60	59.99	99.98			

Table 5: Determination of Precision of Nitrofurantoinfor the first derivative method

Sample Number	Assay of Nitrofurantoin as % of labeled amount			
	Analyst- 1	**Analyst-2**	**Analyst-3**	**Analyst-4**
1	99.58	99.84	100.04	99.74
2	99.89	99.86	99.86	100.20
3	100.98	99.40	100.16	99.75
4	99.90	99.67	99.53	100.78
5	99.75	100.95	99.74	99.79
Mean	100.02	99.944	99.866	100.052
S.D.	0.552132	0.591802	0.24775	0.449744
CV	0.005520	0.005921	0.002480	0.004495

Precision:

Assay of method precision was evaluated by carrying out three independent assays of test sample of Nitrofurantoin. By the use of four different analysts the intermediate precision of the method also evaluated.The Assay values obtained by four analysts were summarized in the following Table 5.

Results And Discussion:

The standard solutions of Nitrofurantoin in Acetone subjected to a scan at the series of wavelengthsof 330 nm to 400nm. At First order derivative peak detect method and thederivative spectra were taken at N=8 using Shimadzu1800 spectronic UV-Visible spectrophotometer. Andabsorbance maxima found at 330nm. The calibration plot ofNitrofurantoin was found to be linear at conc. Range 5-25µg/ml. at 234nm. There for, it was clear that Nitrofurantoin can be determined in presence of Acetone with no intervention of any irrelevant substance in pharmaceutical products. With the intention of determining the practicability of the developed technique for the assessment of commercially available brands (NIFTAS 50) of medicinal formulations, the technique was initially attempted on bulk drugs in their synthetic mixture sample as well as concentrations were estimated. Then the technique was subjected to the assay of in marketed dosage forms and satisfactory results were attained within the appropriate limits as per the content of the label claim for Nitrofurantoin.

The newly developed method was validated as per the ICH Guidelines and parameters. The novel method for the quantitative investigation of Nitrofurantoin was subjected to different validation parameters like specificity and selectivity in presence of formulation additives and excipients, studied for Linearity and range at different levels of concentrations and calibration standards where the determination range was optimized, accuracy was proved by recovery studies at different concentration levels, precision was established through inter day precision studies, where the samples were subjected to changed conditions other than optimized parameters.

Conclusion:

From the above experimental studies it can be concluded that First Order Derivativepeak detect method by UV spectrophotometry instrument developed for estimation of Nitrofurantoin. The proposed methods for the selected drugs were found to be precise and accurate. The most important features of spectrophotometric methods are their rapidity and simplicity. Results of validation parameters demonstrate that these performed analytical procedures are suitable for its intended purpose and meet the criteria defined in ICHQ2A/B guidelines. The method is an excellent alternative to HPLC methods for routine analysis and accurate and rapid than the zero order UV spectrophotometric method.

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