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DEVELOPMENT AND VALIDATION OF ANALYTICAL METHOD FOR ESTIMATION OF DICLOFENAC SODIUM IN SWAB SAMPLES

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ABSTRACT

The objective of the current study was to develop and validate simple and precise UV Spectrophotometric method for estimation of Diclofenac Sodium in the swab samples to validate cleaning procedure. The swabbing procedure was optimized in order to obtain a suitable recovery from stainless steel surface using Tex wipe polyurethane swab stick. Detection wavelength selected was 267 nm. The proposed method was validated in terms of Linearity, precision, accuracy, limit of detection and limit of quantitation. Linearity was studied over concentration range of 0.5 -3 µg / ml and correlation coefficient was found to be 0.999 for regression line. A recovery obtained was 94.58 %. LOD and LOQ for developed method were found to be 0.014715 µg/ml and 0.044592 µg/ml respectively.

INTRODUCTION: Cleaning Validation is documented evidence that an approved cleaning procedure will provide equipment that is suitable for processing of pharmaceutical products or active pharmaceutical ingredients (APIs). For validation of cleaning procedure three methods of sampling that are considered to be acceptable, namely direct surface sampling (swab method), indirect sampling (use of rinse solutions) and placebo sampling.

A combination of the first two methods is generally the most desirable, particularly in circumstances where accessibility of equipment parts can mitigate against direct surface sampling. In Swab method, the suitability of the material to be used for sampling and of the sampling medium should be determined. The ability to recover samples accurately may be affected by the choice of sampling material.

It is important to ensure that the sampling medium and solvent are satisfactory and can be readily used. Rinse samples allow sampling of a large surface area. In addition, inaccessible areas of equipment that cannot be routinely disassembled can be evaluated. However, consideration should be given to the solubility of the contaminant. A direct measurement of the product residue or contaminant in the relevant solvent should be made when rinse samples are used to validate the cleaning process. Placebo sampling method provides simulation of actual production of subsequent batches [1-10].

Therefore, the cleaning method was developed by using methanol as cleaning solvent. Diclofenac Sodium is official in IP 2010, USP 2007 and B.P. 2009. Literature survey revealed that only a few HPLC, HPTLC, spectroscopic methods were reported for the estimation of Diclofenac Sodium in the formulation and in bulk. No method is reported for estimation of Diclofenac Sodium in swab samples.

Therefore UV method is developed for the same. The proposed analytical method has been validated with respect to linearity, precision, accuracy, LOD and LOQ.

The present work focuses on development and validation of spectroscopic method for analysis of swab samples of Diclofenac sodium [11].

EXPERIMENTAL:

Reagents and Chemicals: Diclofenac Sodium was obtained as the gift sample from Centurion Laboratories, Vadodara. All other reagents used were of analytical grade. Methanol (AR grade) was used as solvent for swab testing. The sample solution was passed through Whatmann filter paper. Swab sampling was done by using Tip T × Tm. 714 swabs from Tex Wipe Corporation.

Instrumentation: A UV visible spectrophotometer (Shimadzu 1800) with spectral bandwidth 1nm was employed for all spectroscopic measurements, using a pair of 10 mm matched quartz cells. Shimadzu AUX 220 weighing balance, Lab Press tablet compression machine.

Recovery Studies of Diclofenac Sodium from Clean Tip Swabs and Stainless Steel Plate: Stainless steel plate (30cm × 15cm) was used for the surface testing. The spiking solution was prepared by dissolving 25mg Diclofenac Sodium in 25ml methanol to get the concentration of 1000 µg / ml. This was further diluted to get 10 µg / ml. Heads of the TXTM 714 swab sticks were rinsed with methanol (AR grade). Using calibrated graduated pipette, 1.6 ml, 2ml and 2.2ml solution having concentration 10 µg / ml were transferred on the three specified areas of recovery plate. These solutions were spread on the recovery plate in the area of 5 cm ×5 cm and were allowed to dry. Swabs sticks previously placed into glass test tube containing 5 ml of methanol (AR grade) were used for the swabbing the stainless steel plate. Swabbing was done first in horizontal and then in vertical direction.

Finally, swabs sticks were put again into glass test tube containing methanol and sonicated for 10 min at an ambient temperature and volume was made with the methanol (AR grade). Finally, absorbance of these sample solutions was measured at the detection wavelength of 267 nm.

Method for Cleaning the Instrument: Tablet compression machine was cleaned with dry cloth. To remove the traces of residue of drug, machine was then cleaned with 2% SLS solution twice and then wiped with methanol using cotton plug.

Method for Swab Testing: Critical sites were selected and marked with area as shown in Table I. Each swab was dipped in 5 ml methanol. Swabs were taken in selected area using separate swab for different area carefully. Swabbing is done first in horizontal and then in vertical direction. Then swabs were again dipped in 5 ml methanol contained in 10ml test tube. These Test tubes were then sonicated for 10 min and then volume was made. Resultant solutions were filtered using Whatmann filter paper and analyzed at 267 nm.

Preparation of Standard Solution: Stock solution of Diclofenac Sodium was prepared by dissolving 25mg of Diclofenac Sodium in 25 ml methanol. This solution was further diluted suitably to get solution of concentration 10 µg/ml.

Determination of Absorption Maxima: Standard solution 10 µg / ml were scanned between 200-400 nm. Spectrum was recorded and the suitable absorption maxima selected was 267 nm.

Development and Validation of Analytical Method [11]: Spectrophotometric method for the determination of Diclofenac Sodium in swab samples was developed and validated by determining the linearity, precision, accuracy, LOD and LOQ. Detection wavelength selected for analysis was 267 nm.

Linearity: Linearity was studied over a small drug concentration range from 0.5 – 3 µg/ml. The correlation coefficient (R^2=0.999) obtained for regression line showed excellent linearity relationship between absorbance and concentration of Diclofenac Sodium (**fig. 1**).

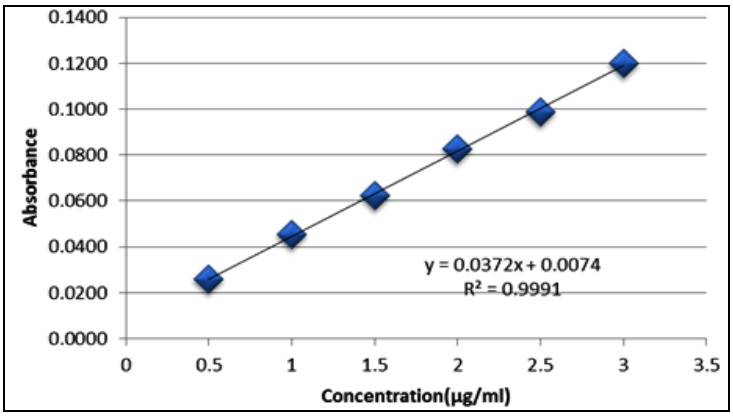

FIG. 1: CALIBRATION CURVE FOR DICLOFENAC SODIUM

Precision: Precision of the method reported as % RSD, was estimated by repeatability, reproducibility and intermediate precision by measuring absorbance of six replicates of 2 µg/ml of Diclofenac Sodium. %RSD value is less than 2% that illustrate the good precision of the analytical method.

Accuracy: Accuracy of the procedure was determined by comparing the analytical amount determined Vs known amount spiked at 80%, 100% and 120% level of LOQ concentration with measurements for each concentration level achieved.

Limit of Detection and Quantitation: The LOD and LOQ of Diclofenac Sodium were estimated from the standard deviation of the response and the slope of the calibration curve by using following formula.

$LOD = 3.3 \times \sigma / S$

$LOQ = 10 \times \sigma / S$

Where σ = the standard deviation of the response, S = the slope of the calibration curve

RESULTS AND DISCUSSION: Developed cleaning method removes even traces of residue of drug present on the instrument. Analytical method developed was found to be linear, precise, accurate and sensitive to detect even small quantity of drug residue.

All the results for validation parameter are tabulated in **Table 1-3**.

TABLE 1: CRITICAL SITES AND AREA SELECTED FOR UV READINGS

Critical sits selected	Areas for swab testing	Absorbance
Turret	2cm×2cm	Not detected
Upper punch (12.5mm)	1cm×1cm	0.005
Lower punch (12.5mm)	1cm×1cm	0.002
Die	1cm×1cm	Not detected
Upper camp tract	2cm×2cm	0.006
Plat form	2cm×2cm	0.010

TABLE 2: LINEARITY FOR DICLOFENAC SODIUM

Sr. No.	Concentration (µg/ml)	Absorbance
1	0.5	0.0261
2	1.0	0.0452
3	1.5	0.0623
4	2.0	0.0825
5	2.5	0.0987
6	3.0	0.1200

TABLE 3: RESULTS FOR VALIDATION PARAMETERS

Sr. No.	Validation Parameter	Results
1	Linearity	0.999
2	Precision(%RSD)	
	Intraday	1.11%
	Intermediate	1.46%
	Interday	1.72%
3	Accuracy (%recovery)	
	80%	92.37
	100%	94.58
	120%	93.87
4	LOD	0.014715 µg/ml
5	LOQ	0.044592 µg/ml

CONCLUSION: The proposed method is simple, rapid, sensitive and economic and hence can be used for the routine analysis of swabs.

REFERENCES:

1. Cleaning Validation. Health Sciences Authority– Health Products Regulation Group 2008; 2-11.
2. FDA: Guide to Inspections of Validation of Cleaning Processes. 1993.
3. Sharma PP: How to Practice GMPs. A Plan for Total Quality Control. Vandana Publication: 1995; 302-314.
4. Dhoka MV, Dumbre SC and Sandage SJ: Spectrophotometric Method for the Determination of Cefpodoxime Proxitil Residue in Swab Samples. Indian Drugs. 2009; 6(9): 702-707.
5. Berry IR and Nash RA: Pharmaceutical Process Validation. Marcel Dekker, New York; 1993; 319-349.
6. Shifflet M and Shapiro M: Development of Analytical Methods to Accurately and Precisely Determine Residual Active Pharmaceutical Ingredients and Cleaning Agents on Pharmaceutical Surfaces. Am. Pharm. Rev. 2002; 4: 35–39.
7. Chudzik GM: General Guide to Recovery Studies Using Swab Sampling Methods for Cleaning Validation. J. Validation Technol. 1998; 5 (1): 77–81.

8. Code of Federal Regulations, Food and Drugs General Services Administration. Washington DC, 1973, Title 21: Part 211.67.
9. Pharmaceutical Inspection Convention, Recommendations on Validation Master Plan, Installation and Operational Qualification, Non-Sterile Process Validation and Cleaning Validation 2004; 2:17-22.
10. United State Pharmacopoeia. 25th Edition. 30-National Formulary 2007; 701- 711.
11. ICH Harmonised – Triplicate Guideline Validation of analytical Procedures text and Methodology. 2005; Q2 [R1].

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