Estimation of Acitretine in softule dosage form by HPTLC method

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Received on:11-01-2012; Revised on: 17-02-2012; Accepted on:19-04-2012

ABSTRACT

A simple, accurate, low cost and specific HPTLC method for estimation of Acitretin in capsule dosage form has been developed. It was performed on Silica gel G60 F254 aluminium foil using acetonitrile : chloroform in the ratio of 1:9 as mobile phase. The mobile phase having chamber was saturated for 20 minutes at room temperature. The Rf value of Acitretin was found to be 0.45. The plate was scanned and quantified at 239 nm. The correlation coefficient(r) = 0.998. The percent recovery was found to be 100.0 ± 0.01. The developed method was validated for its accuracy and precision with suitable parameters.

Key words: HPTLC, Acitretin, Rf value, Silica gel G60 F254.

INTRODUCTION

Acitretin is chemically 2E,4E,6E,8E)-9-(4-methoxy-2,3,6-trimethylphenyl)-3,7-dimethylnona-2,4,6,8-tetraenoic acid1-3. It is a second generation retinoid and a metabolite of etretinate. It is used for the treatment of psoriasis4-24.

Structure of Acitretin

From the literature review many analytical methods have been reported for the determination of Acitretin such as spectrofluorimetry25, HPLC26-35 and LC-MS/MS36-45. There is no reported HPTLC method for the determination of Acitretin in softule dosage form. The objective of this work is report a simple, precise, accurate and cost effective HPTLC method for the estimation of Acitretin is quantified at 239 nm.

MATERIALS AND METHODS

A Camag, Linomat 5 sample applicator was used. The scanner used was Camag TLC Scanner3 and CATS4 software for interpretation of data. Acitretin standard pure drug was supplied by Dr.Reddy’s laboratories, Hyderabad. Acetonitrile and Chloroform used were of AR grade purchased from S.D Fine Chemicals Ltd. All other chemicals used in the analysis were AR grade.

Standard preparation

Weigh accurately 10 mg of Acitretin was transferred into10 ml volumetric flask and then add methanol to get (1mg/ml). From that 1ml was withdrawn and it is diluted upto 1000ml with methanol to get (1µg/1µl).

Chromatographic conditions

Stationary phase-silica gel G60F254 TLC precoated plates (10x10), Mobile phase acetonitrile: chloroform in ratio of 1:9, saturation time - 20 Minutes, Migration distance - 56 cm, Band width - 6mm, Source of radiation - Deuterium lamp, Detection wavelength – 239 nm using slit dimension 5 x 6.5 mm.

Calibration curve response

Aliquots of 5, 10, 15, 20 and 25 µl of standard solution of Acitretin were applied on the chromatographic plates. The plate was developed using acetonitrile: chloroform (1:9) dried and scanned at 239 nm between peak areas/concentration was observed for Acitretin.

Sample preparation

Twenty tablets (acetec) were taken and average weight was calculated. The content of Acitretin was weighed equivalent to 100 mg and it was taken in a 50 ml volumetric flask and dissolved with small portion of methanol. The solution was shaken well and filtered through whattman filter paper then the volume was made up to mark using methanol.

Assay

From the sample solution aliquots was spotted (10 µl and 15 µl) on the plate by using Linomat 5 applicator. Developed chromatogram was scanned. A triplicate of those was carried out the peak areas were noted and the amount present formulation was calculated using standard calibration curve. The result of assay is displayed in Table 1.

Recovery study

To study the accuracy and precision of the method recovery experiment to determine if there are positive or negative interferences from excipients present in formulation. The recovery of added standard was studied at 3 different levels, each being analyzed in a manner similar to described for assay. Each set of addition was reported seven times and the recovery of added standard was calculated.

Table 1: Analysis for formulation

<table>
<thead>
<tr>
<th>S.No</th>
<th>Drug</th>
<th>Label claim (mg/capsule)</th>
<th>Assay</th>
<th>% label claim</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Acitretin</td>
<td>100</td>
<td>99.9 ± 0.05</td>
<td>99.5 ± 0.04</td>
</tr>
</tbody>
</table>

Validation

The developed method was validated as per ICH guidelines for specificity and accuracy (Table 2). The method is found to be specific for Acitretin since it resolved the peak (RF = 0.45) in presence of other excipients in the formulation (Fig.1). The correlation co-efficient (r) and other validation parameters
RESULTS AND DISCUSSION
The developed method was precise and drug is resolved in well chromatographic system. From the standard deviation, it was observed that the method was precise. The content of Acitretin was found to be 99.9 ± 0.05 and the percent recovery 100.0 ± 0.01 using precoated silica gel G60F254 on aluminium foil and a mobile phase comprising acetonitrile: chloroform (1:9) which gives good separation of Acitretin (Rf = 0.45). The result of assay is displayed in Table 1. The detector response of Acitretin was found to be linear in the range of 5-25 µg/spot. The correlation co-efficient obtain for the linearity Acitretin was 0.998. The result of assay is displayed in Table 2. Low standard deviation indicated that the present method is more accurate, so the method can be used for routine analysis of Acitretin in dosage forms (Fig.1).

CONCLUSION
There are several methods existing for the estimation of Acitretin viz Spectrofluorimetry, HPLC and LC-MS/MS. These methods are either costlier or cannot detect impurity whereas the HPTLC method developed can simultaneously run standards and formulation. Therefore it is concluded that the HPTLC method is cost effective, less time consuming, precise and accurate.

REFERENCES


Source of support: Nil, Conflict of interest: None Declared