RP-HPLC method development and validation of valizodone in pure and tablet dosage form

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Abstract
A simple, precise, rapid, accurate and economic reverse phase high performance liquid chromatographic method has been developed for the estimation of Valizodone in tablet dosage form. The separation was achieved by using octadecylsilane column (C18) and acetonitrile: water in proportion of 60:40 v/v as mobile phase, at a flow rate of 1.0 ml/min. The detection was carried out at 240 nm. The retention time of Valizodone was found to be 4.3 min. The limit of detection and limit of quantitation were found to be 0.11 μg/ml and 0.33 μg/ml respectively. The accuracy and reliability of the proposed method was carried out by evaluating various validation parameters like linearity, precision, accuracy and specificity according to ICH guidelines.

Key words: Valizodone, HPLC, Validation

INTRODUCTION
Valizodone is chemically 5-{4-[4-(5-cyano-1H-indol-3-yl) butyl] piperazin –1-yl} benzofuran -2-carboxamide. It is a strong dopamine antagonist. It has high affinity for D2 dopaminergic receptors. The literature survey reveals that the drug can be estimated by RP-HPLC [1], LCMS [2,3]. The aim of the study was to develop a simple, precise and accurate RP-HPLC method for the estimation of Valizodone in pure drug and in pharmaceutical dosage form.

EXPERIMENTAL METHODS
All the reagents used were HPLC grade. HPLC experiments were performed on a Waters HPLC system equipped with Phenomenex Luna C18, 5μm (4.6×250 mm) column, waters pumps, dual wavelength UV detector and Empower 2 software was used. The mobile phase consisted of acetonitrile: water (60:40, v/v) that was set at a flow rate of 1 mL/min.

Procedure
Stock solution of Valizodone was prepared by dissolving accurately weighed 100 mg of the drug in 100 mL of mobile phase (final concentration, 1 mg/mL). Calibration plot were constructed by analysis of appropriate working solutions (concentration 10, 50, 100, 150, 200 and 250 μg/mL) of Valizodone in the mobile phase and plotting concentration against peak area response for each injection. Unknown samples were quantified by reference to this Beers plot.

Sample analysis
Twenty tablets were weighed and powdered. An amount of powder equivalent to 100 mg Valizodone was accurately weighed and transferred to a 100 mL volumetric flask. Mobile phase (50 mL) was added and the mixture was sonicated for 10 min, for complete extraction of the drug, and the solution was diluted to volume with mobile phase. The solution was centrifuged at 4000 rpm for 10 min, and the clear supernatant was collected and filtered through a 0.2 μm membrane filter. This solution was taken and diluted to 10 mL with mobile phase, to furnish a 100 μg/mL solution, of which 20 μL was injected for HPLC analysis.

RESULTS AND DISCUSSION
The retention time of the drug was 4.3 min (figure 1). There was no interference from diluents used for preparation of tablets (figure 2). Chromatographic parameters such as peak asymmetry and capacity factor were found to be...
1.03 and 0.921 respectively. The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.11 and 0.33 µg/mL respectively. Analytical recovery studies were carried out from a series of spiked concentrations added to the pre-analysed dosage form (Table – 1). The accuracy of the method was carried out by adding standard to pre-analysed pharmaceutical formulation at 50, 100 and 150% levels and report is in Table 2.

Table 1. Determination of Vilazodone in tablets

<table>
<thead>
<tr>
<th>Sample</th>
<th>Label claim/tablet (mg)</th>
<th>Amount found* (mg/tab)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vilazodone Tablets</td>
<td>50</td>
<td>49.98</td>
</tr>
</tbody>
</table>

* Mean of 5 determinations

Table 2. Recovery study data of Vilazodone.

<table>
<thead>
<tr>
<th>Drug</th>
<th>Amount added (µg)</th>
<th>Amount recovered (µg)</th>
<th>Percentage recovery (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vilazodone</td>
<td>50</td>
<td>49.97</td>
<td>99.94</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>100.06</td>
<td>100.07</td>
</tr>
<tr>
<td></td>
<td>150</td>
<td>150.12</td>
<td>100.01</td>
</tr>
</tbody>
</table>

CONCLUSION
The developed RP-HPLC method was simple, sensitive, precise and accurate and hence can be used in routine for the determination of Vilazodone in pure as well as pharmaceutical preparations.

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REFERENCES