



PHARMA DEVILS

QUALITY CONTROL DEPARTMENT

ANALYTICAL SPECIFICATION FOR TERPIN HYDRATE

S.No.	Test	Procedure
1.	Isopropyl Alcohol Content (In House)	<p><i>Equipment:</i> Balance GC – 17 A or equivalent.</p> <p>Examine by Gas Chromatography.</p> <p><i>Reagents:</i></p> <ol style="list-style-type: none">1. Biphenyl, AR Grade.2. Chloroform Spectroscopic Grade.3. Ethyl Alcohol, Spectroscopic Grade. <p><i>Reference Standard:</i> Terpin Hydrate Working Standard.</p> <p><i>Chromatographic System :</i> <i>Column:</i> BP-5 capillary column, I.D.=0.53 mm, length=30 meter, Film thickness = 1 µm. or Equivalent.</p> <p><i>Carrier gas</i> : Nitrogen at flow rate of 4.0 ml/min.</p> <p><i>Split ratio</i> : 1:23.</p> <p><i>Column Temperature:</i> Isothermal at 160°C.</p> <p><i>Detector</i> : FID set at 200 °C .</p> <p><i>Injector</i> : Set at 200°C.</p> <p><i>Injection volume</i> : 3 µl.</p> <p><i>Preparation of Diluent Blank, Internal Standard, Standard Solution and Test Solution:</i></p> <p><i>Preparation of Diluent Blank Solution:</i> Use Chloroform as such.</p> <p><i>Preparation of Instrument Blank:</i> Run Temperature Program without any injection.</p> <p><i>Preparation of Internal standard:</i> Weigh accurately about 1.0 g of Biphenyl and transfer to a 50ml volumetric flask, add about 30ml of chloroform and mix the solution vigorously on vortex mixer for 5 minutes. Dilute to volume with chloroform and mix for 1 minute.</p> <p><i>Preparation of Standard solution:</i> Weigh accurately about 175mg Terpin Hydrate Working Standard and transfer to a 100ml volumetric flask, add in this flask 5ml of alcohol, 5ml of internal standard. Add, about 50ml of chloroform and mix the solution vigorously on vortex mixer for 5 minutes. Dilute to volume with chloroform and mix for 1 minute.</p> <p><i>Preparation of Test Solution:</i> Weigh accurately about 180mg substance to be examined and transfer to a 100ml volumetric flask, add in this flask 5ml of alcohol, 5ml of internal standard. Add, about 50ml of chloroform and mix the solution vigorously on vortex mixer for 5 minutes. Dilute to volume with chloroform and mix for 1 minute.</p>



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	Isopropyl Alcohol Content (In House)	<p><i>Procedure :</i> Before analysis, equilibrate chromatography system with Carrier gas for 30 minutes till a stable base line is observed to eliminate/minimize system noise. Inject the solutions in the following sequence, record the chromatograms and interpret the results and mentioned in the following table 1:</p> <p>Table :1</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th style="width: 30%;">Injection Solutions</th> <th style="width: 15%;">No. of Injections</th> <th style="width: 55%;">Interpretation / Calculations</th> </tr> </thead> <tbody> <tr> <td>Instrument Blank Solution</td> <td>1</td> <td>Disregard any peak obtained due to Instrument blank solution.</td> </tr> <tr> <td>Diluent Blank</td> <td>1</td> <td>Disregard any peak obtained due to blank Solution.</td> </tr> <tr> <td>Standard Solution</td> <td>1</td> <td>The retention times of Terpin Hydrate, Biphenyl is about 4.6 and 6.0 minutes respectively.</td> </tr> <tr> <td>Standard Solution</td> <td>*5</td> <td>Calculate the ratio of peak area due to terpin Hydrate to peak area obtained due to Biphenyl for each injection. Calculate the mean ratio and relative standard deviation of ratio, is not more than 5.0%.</td> </tr> <tr> <td>Test Solution(T1)</td> <td>2</td> <td>Calculate the ratio of peak area due to Terpin Hydrate to peak area due to Biphenyl for each injection. Calculate the mean ratio for two injections. Calculate the % Assay of Terpin Hydrate against the standard using the formula as given below.</td> </tr> </tbody> </table> <p>*5 replicate injections are to be considered to meet the requirements. No. Of injections should be increased till a constant peak area is obtained.</p> <p>Calculations:</p> <p>The content of Terpin Hydrate (C)in percent is given by,</p> $\% \text{ Assay (As such)} = \frac{R_u \times W_s \times 100 \times P \times 100}{R_s \times 100 \times W_t \times 100}$ <p>Where,</p> <p>R_u = Mean ratio obtained for Test Solution. R_s = Mean ratio obtained for Standard Solution for each injection. W_s = Weight of Terpin Hydrate Working Standard P = Potency of Terpin Hydrate Working Standard (% As such). W_t. = Weight of Test in mg.</p> <p>The content of Terpin Hydrate in percent on anhydrous basis is given by,</p> $\% \text{ Assay (OAB)} = \frac{\% \text{ Assay (As such)} \times 100}{(100 - \% \text{ M.C.})}$ <p>Where, % M.C. = % moisture content of Test.</p>	Injection Solutions	No. of Injections	Interpretation / Calculations	Instrument Blank Solution	1	Disregard any peak obtained due to Instrument blank solution.	Diluent Blank	1	Disregard any peak obtained due to blank Solution.	Standard Solution	1	The retention times of Terpin Hydrate, Biphenyl is about 4.6 and 6.0 minutes respectively.	Standard Solution	*5	Calculate the ratio of peak area due to terpin Hydrate to peak area obtained due to Biphenyl for each injection. Calculate the mean ratio and relative standard deviation of ratio, is not more than 5.0%.	Test Solution(T1)	2	Calculate the ratio of peak area due to Terpin Hydrate to peak area due to Biphenyl for each injection. Calculate the mean ratio for two injections. Calculate the % Assay of Terpin Hydrate against the standard using the formula as given below.
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