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## Determination of Tamsulosinehydrochloride 0.2% and Tolterodinetartrate 0.2% Combination Pellets by RP HPLC method

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### ABSTRACT

A Simple and precise reverse phase high performance liquid chromatographic method has been developed for the determination of Tamsulosine hydrochloride 0.2% & Tolterodine tartrate 0.2% combination pellets. An Inertsil ODS 3V (4.6 mm X 150 mm) that contain 5 $\mu$ m packing column, gradient mode, with 30.5ml perchloric acid with 95 ml of water and add 10.5gm sodium hydroxide, made up to 1000ml with water and homogenize as mobile phase. The flow rate is 2ml/minute and effluent is monitored at 220nm.

Keywords: Tamsulosine hydrochloride/determination. Tolterodine tartrate/determination. Pharmaceutical forms/analysis. HPLC method/drug's analysis. Liquid chromatographic method. Inertsil ODS 3V.

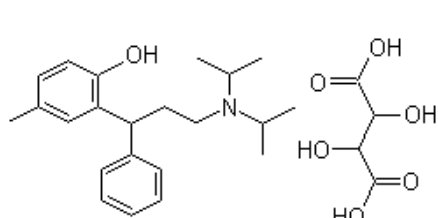
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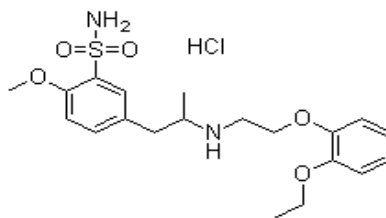


## INTRODUCTION

Tamsulosin hydrochloride & Tolterodine tartarate combination is used for the treatment of a chronic urological disorder. Chemically Tamsulosin hydrochloride is known as 5-[(2R)-2-[[2-(2-Ethoxyphenoxy)ethyl]amino]propyl]-2-methoxybenzenesulfonamide hydrochloride. It is not reported any of the pharmacopoeias. A survey of literature reveals that HPLC methods [1,2] are reported for the determination [3,4] of Tamsulosin hydrochloride. Tolterodine tartarate chemically Known as (+)-R)-2-{a-[2-(Diisopropylamino)ethyl]benzyl}-p-cresol tartrate. A survey of literature reveals that HPLC methods [5,6] were reported for the determination of Tolterodine tartarate. However Determination of Tamsulosine Hydrochloride & Tolterodine tartrate combination commercial dosage forms, by HPLC method is not reported. Hence HPLC method for the determination of Tamsulosine Hydrochloride & Tolterodine tartrate combination solid dosage forms is described.



Molecular Structure of Tolterodinetartarate  
FIGURE 1



Molecular Structure of  
TamsulosineHydrochloride  
FIGURE 2

## EXPERIMENT

### Instrument:

High performance liquid chromatograph, Shimadzu 2010 Rheodyne injector with 100 $\mu$ l loop LC solution computer based data station is used.

### Chemicals and Reagents:

Reference standard Tamsulosine hydrochloride is procured from M/S Suven life sciences, Reference standard Tolterodinetartrate is procured from M/S Fleming laboratories, perchloric acid, Sodium Hydroxide AR grade (E-Merck), HPLC grade D.M water were used.

### Stationery phase:

Inertsil ODS 3V (4.6 mm X 150 mm ) contains 5- $\mu$ m packing.

### Mobile phase preparation:

Mix 100 ml perchloric acid solution with about 565ml water and homogenize. Adjust pH=2.0 with 1N sodium hydroxide solution or with perchloric acid solution make up with 700ml of water. Filtered and degassed.

### Standard preparation:

Transfer about 25mg of Tamsulosin Hydrochloride and 25mg Tolterodine tartrate WS, accurately weighed, to a 50 ml volumetric flask and dissolve and dilute with methanol to volume. Mix and filter. Transfer 2.0ml of this solution to a 100 ml volumetric flask, dilute with mobile phase to volume and mix.



### Assay Preparation:

Weigh accurately 2000mg of Tamsulosin Hydrochloride, Tolterodine tartrate pellets and were taken into a mortar, grind and mix. Transfer accurately weighed quantity of about 2.0g of powder into a 200ml volumetric flask; add 60ml of methanol, sonicate till the powder dissolves. Cool the solution to room temperature. Make up to volume with mobile phase. Sonicate for 5minutes. Homogenize the sample solution and centrifuge it at 3000rpm for 15min. Filter through a 0.45µm nylon filter.

### METHOD

#### Gradient

Pump A : Perchloric acid solution

Pump B : Acetonitrile

TABLE I – TamsulosineHydrochloride,and Tolterodine tartarate of HPLC gradient method given below table.

Time in minutes	Buffer: Pump A	Acetonitrile: Pump B
0	70	30
7.0	50	50
15	50	50
20	70	30

#### Chromatographic System

The liquid chromatograph is equipped with a 220-nm detector and a 4.6 mm X 150 mm inertsil ODS 3V that contains 5-µm packing. The flow rate is about 2.0 ml per minute. Chromatograph the standard preparation and the record the peak responses as directed in the procedure. The relative standard deviation for replicate injections is not more than 2.0% Separately inject equal volumes (about 100µl) of the Standard solution and the test solution into the chromatograph, record the chromatograms, and measure the responses for the major peak .Calculate the quantity in mg , of Tamsulosin hydrochloride  $C_{20}H_{28}N_2O_5S.HCl$  & Tolterodine tartrate  $C_{22}H_{31}NO_6$ ;  $C_{26}H_{37}NO_7$ . Results are tabulated as follows:

TABLE II – TamsulosineHydrochloride,and Tolterodine tartarate of Release rates values given below table.

Semi formulation	S.No	Label claim%		Amount Estimated%		% of label Claim		% deviation		S.D		RSD	
		Tam	Tolt	Tam	Tolt	Tam	Tol	Tam	Tolt	Tam	Tolt	Tam	Tolt
P E L L E T	1	0.2	0.2	0.202	0.201	101	100.5	+1	+0.5	.00089 4	.00089 4	0.4427	0.4427
	2			0.201	0.203	100.5	101.5	+0.5	+1.5				
	3			0.203	0.202	101.5	101	+1.5	+1				
	4			0.201	0.201	100.5	100.5	+0.5	+0.5				
	5			0.202	0.203	101	101.5	+1	+1.5				
	6			0.203	0.202	101.5	101	+1.5	+1				

#### Calibration

100µl of the above working standard solution are injected over a time interval of 20 minutes. Evaluation is performed with U.V detector at 220nm. The retention time for Tamsulosine Hydrochloride is



found to be around 3.82 minutes & Tolterodine tartarate is around 13.24 minutes. Peak areas are recorded and calibration graph is obtained by plotting peak areas versus concentration.

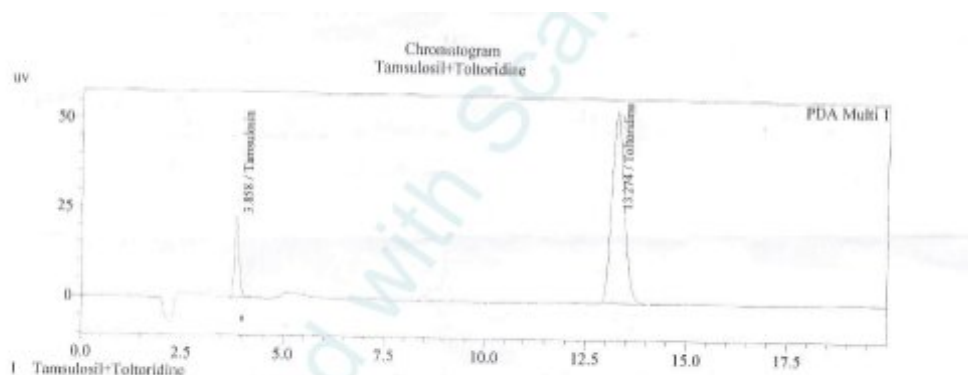
### Assay

100µl of Standard and sample solutions are injected into an injector of liquid chromatograph. Amount of Tamsulosine Hydrochloride, and Tolterodine tartarate calculated by comparing the peak ratio, with that of the standard.

### Recovery studies

To study the linearity, accuracy and Precision of Proposed method, recovery experiments were carried out. Known quantities of standard at two different levels were added to the pre-analyzed sample , the recovery was estimated to be more than 99%.

System suitability test is applied to a representative chromatogram to check various parameters such as efficiency , resolution and peak tailing. The results obtained are shown in table-2 that is in concurrence with the USP requirements.



Chromatogram of sample semi formulation containing Tamsulosine Hydrochloride, and Tolterodine tartarate

## RESULTS AND DISCUSSION

### Linearity

The linearity of Tamsulosine hydrochloride, and Tolterodine tartarate is established by plotting graph of Peak area of standard solutions versus concentration .The linearity is found to be between 100-500µg/ml.

### Chromatography

The mobile Phase mixture is found to be ideal for analysis of Tamsulosine hydrochloride, and Tolterodine tartarate combination. The concentration of Tamsulosine Hydrochloride, and Tolterodine tartarate found to be with in limits and RSD values are reasonable low.

TABLE III – TamsulosineHydrochloride,and Tolterodine tartarate of relative standard deviation,theoretical plates and tailing factor values given below table.

S.No	Parameter	Tamsulosinehydrochloride	Tolterodine tartarate
1	Theoretical Plates	4965.07	8139.52
2	Tailing Factor	1.33	1.19
3	RSD of 6 Injections	0.4427	0.4427



The precision of the method is studied by making 5 injections of standard and very low RSD values indicating good Precision. The Reproducibility and reliability of the method has been tested by Performing recovery studies which showed good results.

The proposed method is very simple, rapid and no where involves use of complicated sample preparation. High percentage of recovery shows that the method is free from interference of the excipients used in the semi formulations. Therefore the method can be useful in routine quality control analysis.

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