ANALYTICAL METHOD VALIDATION REPORT FOR ASSAY OF ETORICOXIB TABLETS

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OBJECTIVE: The efficacy & safety of a medicinal product can only be assured by analytical monitoring of its quality.

SCOPE: The scope of analytical validation is to ensure that the procedure under consideration is capable of giving reproducible and reliable results.

Product Name	Etoricoxib Tablets
Ingredient	Etoricoxib.
Label Claim	Each film coated tablet contains Etoricoxib ----------120 mg
Test Method	Liquid Chromatography Etoricoxib

Specificity (Diluents Interference)

Placebo Preparation:

A placebo solution was prepared same as the formulation except for the addition of the active ingredients. Here used as the placebo solution. Area at 220nm, Observation Result: Nil

Conclusion for Specificity:

We observed that at wavelength 220nm there is no significant area for placebo (Diluents) for Etoricoxib tablets assay method. Therefore specificity of the method considered acceptable.

System Accuracy:

The system precision of the above method was carried out by taking area for six times of the sample preparation of exact weight.

Test data collection sheet:

Serial No.	Area of Etoricoxib
1.	
2.	
3.	
4.	
5.	
6.	
Mean	
% RSD	

Acceptance Criteria: RSD is not more than 2.0%.

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Linearity/ Accuracy:

Definition:

The Linearity of an analytical method is its ability to elicit test results that are directly, or by a well defined mathematical transformation, proportional to the concentration of the analyte in samples within a given range. Linearity is usually expressed in terms of the variance around the slope of the regression line calculated according to an established mathematical relationship from test results obtained by the analysis of sample with varying concentration of analyte.

Range:

Definition:

The Range of an analytical method is the interval between the upper & lower level of analyte that have been demonstrated with precision, accuracy & linearity using the method as written. The Range is normally expressed in same units as test results e.g. Percent or Parts per million, obtained by the analytical method.

Assay:

Etoricoxib Tablets (Limit: 90.0 % to 110.0 % of the labeled amount).

Chromatographic condition:-

Column: A stainless steel column 25 cm 4.6 mm packed with octadecylesilane bonded to porous silica (5µm).

Column temperature: ambient

Detector: UV 220 nm

Flow rate: 1.0 ml/min.

Injection volume: 20 µl

Mobile phase:—

A mixture of 55 volume of buffer solution prepared by dissolving 6.8gm of potassium dihydrogen orthophosphate in 1000 ml water, adjusted pH 3.5 with Orthophosphoric acid and 45 volume of Acetonitrile,

Standard Preparation:—

Dissolve an accurately weighed ----------mg (50 mg) of Etoricoxib WS into 100 ml volumetric flask, add 20 ml methanol, sonicate completely to dissolve, shake and dilute with methanol. Take 5.0 ml of this solution and dilute to 50 ml with methanol and shake.

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Sample Preparation:—

Weigh and powdered of 20 tablets. Dissolve a required quantity of tablets powder with 50 ml of methanol with the aid of ultrasound and dilute to 100 ml with methanol. Take 5.0 ml of this solution and dilute to 50 ml with methanol.

Chromatographic system:

Inject the reference solution The test is not valid unless the Coloumn efficiency is not more than 2000 theoretical plates and the tailing factor not more than 2.0 and standard deviation not more than 2.0% for replicate injections.

Procedure

Separately injects equal volumes (about 20 µL) of the Standard preparation and the Assay preparation into the chromatograph, records the chromatograms, and measures the area responses for the major peaks. Calculate the quantity, in %, of Etoricoxib in the portion of tablets taken by the formula:

$$\frac{\text{Sample area} \times \text{WS Weight} \times \text{Potency of WS} \times \text{Average Weight} \times 100}{\text{Standard area} \times \text{Sample weight} \times 100 \times \text{Claim}} = \quad \%$$

Text data collection sheet:

S.No.	Standards	Area of Etoricoxib
1.	Standard-1	
2.	Standard-2	
3.	Standard-3	
4.	Standard-4	
5.	Standard-5	
6.	Standard-6	
7.	Mean	
8.	% RSD	

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Samples	Sample Area Etoricoxib	Mean
Sample-A-01 80%		
Sample-A-02 80%		
Sample-A-03 80%		

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Samples	Sample Area Etoricoxib	Mean
Sample-B-01 90%		
Sample-B-02 90%		
Sample-B-03 90%		
Sample-C-01 100%		
Sample-C-02 100%		
Sample-C-03 100%		
Sample-D-01 110%		
Sample-D-02 110 %		
Sample-D-03 110%		
Sample-E-01 120 %		
Sample-E-02 120 %		
Sample-E-03 120 %		

Calculation:

Data Collection:

Concentration (µg/ml)	Concentration in %	Corr. Coefficient	Sample Mean Area	% Recovery	Corr. Coefficient
	80	1.0			
	90				
	100				
	110				
	120				

Precision:

The precision of an analytical method is the degree of agreement among individual test results when the method is applied repeatedly to multiple sampling of a homogeneous sample. The precision of the analytical method is usually expressed as Standard deviation or relative standard deviation (coefficient of variation) of a series measurement. The precision may be measured of either the degree of reproducibility or of repeatability of the analytical method on the normal operating condition.

Precision: – Method precision

Etoricoxib Tablets (Limit: 90.0 % to 110.0 % of the labeled amount).

Analyst (I): "…….."

Chromatographic condition:-

Column: A stainless steel column 25 cm 4.6 mm packed with octadecylesilane bonded to porous silica (5µm).

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Column temperature: ambient

Detector: UV 220nm

Flow rate: 1.0ml/min.

Injection volume: 20µl

Mobile phase:—

A mixture of 55 volume of buffer solution prepared by dissolving 6.8gm of potassium dihydrogen orthophosphate in 1000ml water, adjusted pH 3.5 with Orthophosphoric acid and 45 volume of Acetonitrile,

Standard Preparation:—

Dissolve an accurately weighed ----------mg (50mg) of Etoricoxib WS into 100ml volumetric flask, add 20ml methanol, sonicate completely to dissolve, shake and dilute with methanol. Take 5.0ml of this solution and dilute to 50ml with methanol and shake.

Sample Preparation:—

Weigh and powdered of 20 tablets. Dissolve a required quantity of tablets powder containing 50mg of Etoricoxib with 50ml of methanol with the aid of ultrasound and dilute to 100ml with methanol. Take 5.0ml of this solution and dilute to 50ml with methanol.

Chromatographic system: —

Inject the reference solution The test is not valid unless the Coloumn efficiency is not more than 2000 theoretical plates and the tailing factor not more than 2.0 and standard deviation not more than 2.0% for replicate injections.

Procedure—

Separately injects equal volumes (about 20 µL) of the Standard preparation and the Assay preparation into the chromatograph, records the chromatograms, and measures the area responses for the major peaks. Calculate the quantity, in %, of Etoricoxib in the portion of tablets taken by the formula:

$$\frac{\text{Sample area X WS Weight X Potency of WS X Average Weight X 100}}{\text{Standard area X Sample weight X 100 X Claim}} = \%$$

Sample Dilutions:

(A) Take ----------mg of the sample and proceed as above.

(B) Take ----------mg of the sample and proceed as above.

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(C) Take ----------mg of the sample and proceed as above.

(D) Take ----------mg of the sample and proceed as above.

(E) Take ----------mg of the sample and proceed as above.

(F) Take ----------mg of the sample and proceed as above.

Text data collection sheet:

S.No.	Standards	Area of Etoricoxib
1.	Standard-1	
2.	Standard-2	
3.	Standard-3	
4.	Standard-4	
5.	Standard-5	
6.	Standard-6	
7.	Mean	
8.	% RSD	

Acceptance Criteria: RSD is not more than 2.0%.

Samples		Area of Etoricoxib	Mean
Sample A	T1		
	T2		
Sample B	T1		
	T2		
Sample C	T1		
	T2		
Sample D	T1		
	T2		
Sample E	T1		
	T2		
Sample F	T1		
	T2		

Calculation:

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Table for Six Replicate Assays:

Sample Number	Estimated % Amount	Mean	Relative Standard Deviation (% RSD)
Sample A			
Sample B			
Sample C			
Sample D			
Sample E			
Sample F			

Acceptance Criteria: NMT 2% (% of Relative Standard Deviation).

Intermediate Precision: – (Within laboratory variations such as different days, analyst & equipments): **Etoricoxib Tablets** (Limit: 90.0 % to 110.0 % of the labeled amount).

Analyst (II): "……..."

Chromatographic condition:-

Column: A stainless steel column 25 cm 4.6 mm packed with octadecylesilane bonded to porous silica (5µm).

Column temperature: Ambient

Detector: UV 220 nm

Flow rate: 1.0 ml/min.

Injection volume: 20 µl

Mobile phase:—

A mixture of 55 volume of buffer solution prepared by dissolving 6.8gm of potassium dihydrogen orthophosphate in 1000 ml water, adjusted pH 3.5 with Orthophosphoric acid and 45 volume of Acetonitrile,

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Chromatographic system: —
Inject the reference solution The test is not valid unless the Coloumn efficiency is not more than 2000 theoretical plates and the tailing factor not more than 2.0 and standard deviation not more than 2.0% for replicate injections.

Procedure—
Separately injects equal volumes (about 20 µL) of the Standard preparation and the Assay preparation into the chromatograph, records the chromatograms, and measures the area responses for the major peaks. Calculate the quantity, in %, of Etoricoxib in the portion of tablets taken by the formula:

$$\frac{\text{Sample area X WS Weight X Potency of WS X Average Weight X 100}}{\text{Standard area X Sample weight X 100 X Claim}} = \quad \%$$

Sample Dilutions:

(A) Take ----------mg of the sample and proceed as above.

(B) Take ----------mg of the sample and proceed as above.

(C) Take ----------mg of the sample and proceed as above.

(D) Take ----------mg of the sample and proceed as above.

(E) Take ----------mg of the sample and proceed as above.

(F) Take ----------mg of the sample and proceed as above.

Text data collection sheet:

S.No.	Standards	Area of Etoricoxib
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5.	Standard-5	
6.	Standard-6	
7.	Mean	
8.	% RSD	

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Samples		Area of Etoricoxib	Mean
Sample A	T1		
	T2		
Sample B	T1		
	T2		
Sample C	T1		
	T2		
Sample D	T1		
	T2		
Sample E	T1		
	T2		
Sample F	T1		
	T2		

Calculation:

Table for Six Replicate Assays:

Sample Number	Estimated % Amount	Mean	Relative Standard Deviation (% RSD)
Sample A			
Sample B			
Sample C			
Sample D			
Sample E			
Sample F			

Acceptance Criteria: NMT 2% (% of Relative Standard Deviation).

Table for Six Replicate Assays analyst by two different Analysts:

Test Data analyst by **…….**-
Table for Six Replicate Assays:

Sample Number	Estimated % Amount	Mean	Relative Standard Deviation (% RSD)
Sample A			
Sample B			
Sample C			
Sample D			
Sample E			
Sample F			

Test Data analyst by **…….**:-

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Table for Six Replicate Assays:

Sample Number	Estimated % Amount	Mean	Relative Standard Deviation (% RSD)
Sample A			
Sample B			
Sample C			
Sample D			
Sample E			
Sample F			

Acceptance Criteria: NMT 2% (% of Relative Standard Deviation).

Robustness:

To demonstrate the analytical method is capable to yield reproducibility results under; small but deliberate variations in method parameters during normal usage such as composition & Flow rate of mobile phase.

Procedure:

Perform the robustness study by injecting single of resolution solution & standard solution for six times for the following parameters.

- Change in ratio of the mobile phase. Record the observation in below observation table.
- Change in Flow rate of mobile phase. Record the observation in below observation table.

OBSERVATION TABLE:-

Change in flow rate at 220 nm					
Mobile phase		**Flow rate ml/min**	**System suitability**		
Buffer	**Acetonitrile**		**Retention time**	**Theoretical plate**	**Tailing Factor**
540ml	460ml	1.0 ml/min.			
550ml	450ml	1.0 ml/min.			
560ml	440ml	1.0 ml/min.			

Change in flow rate at 220 nm					
Mobile phase		**Flow rate ml/min**	**System suitability**		
Buffer	**Acetonitrile**		**Retention time**	**Theoretical plate**	**Tailing Factor**
550ml	450ml	0.8 ml/min.			
550ml	450ml	1.0 ml/min.			
550ml	450ml	1.2 ml/min.			

Acceptance criteria:

Analytical method validation shall be robust (i.e. Theoretical Plates is not less than 2000 & tailing factor is not more than 2.0).

Analysed By/On: **Checked By/On:**