647510 **MSC3AnaC302x** **Seat No :_____**

M.Sc.(Chem.) Semester - 3 (*CBCS*) Examination
Oct/Nov-2021 (NEW COURSE)
Pharma Regulatory Affairs (Core(New))

Time: 2:30 Hours **Marks: 70**

Instructions:

1. All questions are compulsory.
2. Figures to the right indicate marks.

Q.1(A)	**Answer the following question**	**(4)**
(1)	What is SOP of SOP? which department has right to kept the master copies of all SOPs	2
(2)	Enlist elements of SOP with brief details	2
Q.1(B)	**Answer any two questions out of three**	**(10)**
(1)	Prepare SOP for responsibilities of quality control department.	5
(2)	What is SOP? Give any 5 examples of SOP violations.	5
(3)	Prepare Specification of Rupatadine fumarate from given official EP monograph (EP monograph attached here as annexure-1)	5
Q.2(A)	**Answer the following question**	**(5)**
(1)	Define the term calibration, give brief Outline on Calibration activity and draw calibration master plan taking suitable example.	4
Q.2(B)	**Answer any two questions out of three.**	**(10)**
(1)	Explain Linearity study with suitable example.	5
(2)	Give an account of accuracy studies in method validation.	5
(3)	Discuss calibration of hot air oven with calibration format.	5
Q.3(A)	**Answer the following question**	**(4)**
(1)	Briefly explain static documents and dynamic documents	2
(2)	When does the qualification of analysts shall be done?	2
Q.3(B)	**Answer any two questions out of three**	**(10)**
(1)	Give an account of Installation qualification (IQ).	5
(2)	State the objectives of instrument qualification and discuss the difference between operational qualification and performance qualification.	5
(3)	Give an account of analyst qualification in detail.	5
Q.4(A)	**Answer the following question**	**(4)**
(1)	State the objectives of stability studies.	2
(2)	Define dosage form and excipient.	2
Q.4(B)	**Answer any two questions out of three**	**(10)**
(1)	Explain the following (a) Selection of batches for stability studies (API) (b) Stability testing frequency	5
(2)	Prepare Storage Conditions table for General case and storage in refrigeration.	5
(3)	Briefly explain thermal degradation and oxidative degradation.	5
Q.5(A)	**Answer the following questions**	**(4)**
(1)	Give brief introduction and historical account of GLP and list out the elements of GLP.	4
Q.5(B)	**Answer any two questions out of three**	**(10)**
(1)	Discuss requirements for facility and infrastructure under GLP	5
(2)	Give two examples for GLP violations in following criteria: (a) qualification (b) validation (c) Calibration (d) personal (e) reagents & chemicals	5
(3)	Discuss the role of Personal department under GLP	5

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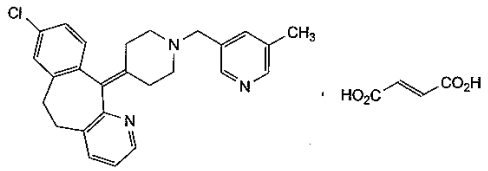
RUPATADINE FUMARATE

Rupatadini fumaras



$C_{30}H_{30}ClN_3O_4$ M_r 532.0
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DEFINITION

8-Chloro-11-[1-[(5-methylpyridin-3-yl)methyl]piperidin-4-ylidene]-6,11-dihydro-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine (2*E*)-but-2-enedioate.

Content: 98.0 per cent to 100.5 per cent (dried substance).

CHARACTERS

Appearance: white or slightly pinkish powder.

Solubility: very slightly soluble in water, slightly soluble in anhydrous ethanol, very slightly soluble in heptane.

IDENTIFICATION

Infrared absorption spectrophotometry (*2.2.24*).

Comparison: *rupatadine fumarate CRS*.

TESTS

Related substances. Liquid chromatography (*2.2.29*). *Prepare the solutions immediately before use.*

Solvent mixture: *acetonitrile R1*, mobile phase A (20:80 *V/V*).

Test solution. Dissolve 32.0 mg of the substance to be examined in the solvent mixture and dilute to 50.0 mL with the solvent mixture.

Reference solution (a). Dilute 1.0 mL of the test solution to 100.0 mL with the solvent mixture. Dilute 1.0 mL of this solution to 10.0 mL with the solvent mixture.

Reference solution (b). Dissolve 3 mg of *rupatadine for system suitability CRS* (containing impurities A and B) in the solvent mixture and dilute to 5 mL with the solvent mixture.

Column:

- *size*: *l* = 0.15 m, Ø = 4.6 mm;
- *stationary phase*: *end-capped octadecylsilyl silica gel for chromatography R* (5 µm);
- *temperature*: 30 °C.

Mobile phase:

- *mobile phase A*: 7.0 g/L solution of *sodium dihydrogen phosphate monohydrate R* in *water for chromatography R*;
- *mobile phase B*: *acetonitrile R1*;

Time (min)	Mobile phase A (per cent *V/V*)	Mobile phase B (per cent *V/V*)
0 - 2	80	20
2 - 27	80 → 50	20 → 50

Flow rate: 1.2 mL/min.

Detection: spectrophotometer at 210 nm.

Injection: 50 µL.

Identification of impurities: use the chromatogram supplied with *rupatadine for system suitability CRS* and the chromatogram obtained with reference solution (b) to identify the peaks due to impurities A and B.

Relative retention with reference to rupatadine (retention time = about 17 min): fumaric acid = about 0.1; impurity A = about 0.6; impurity B = about 0.7.

System suitability: reference solution (b):

- *resolution*: minimum 5.0 between the peaks due to impurities A and B.

Calculation of percentage contents:

- *correction factor*: multiply the peak area of impurity A by 1.3;
- for each impurity, use the concentration of rupatadine in reference solution (a).

Limits:

- *impurity B*: maximum 0.5 per cent;
- *impurity A*: maximum 0.15 per cent;
- *unspecified impurities*: for each impurity, maximum 0.10 per cent;
- *total*: maximum 0.7 per cent;
- *reporting threshold*: 0.05 per cent; disregard the peak due to fumaric acid.

Loss on drying (*2.2.32*): maximum 0.5 per cent, determined on 1.000 g by drying *in vacuo* in an oven at 80 °C for 3 h.

Sulfated ash (*2.4.14*): maximum 0.1 per cent, determined on 1.0 g.

ASSAY

Dissolve 0.150 g in 50 mL of *glacial acetic acid R*. Titrate with *0.1 M perchloric acid*, determining the end-point potentiometrically (*2.2.20*).

1 mL of *0.1 M perchloric acid* is equivalent to 17.73 mg of $C_{30}H_{30}ClN_3O_4$.

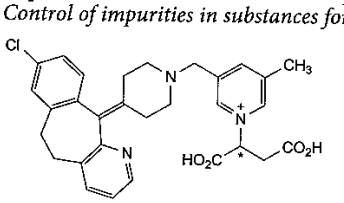
STORAGE

Protected from light.

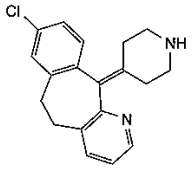
IMPURITIES

Specified impurities: A, B.

Other detectable impurities (the following substances would, if present at a sufficient level, be detected by one or other of the tests in the monograph. They are limited by the general acceptance criterion for other/unspecified impurities and/or by the general monograph *Substances for pharmaceutical use (2034)*. It is therefore not necessary to identify these impurities for demonstration of compliance. See also *5.10. Control of impurities in substances for pharmaceutical use*): C.



A. 3-[[4-(8-chloro-5,6-dihydro-11*H*-benzo[5,6]-cyclohepta[1,2-*b*]pyridin-11-ylidene)piperidin-1-yl]methyl]-1-(1,2-dicarboxyethyl)-5-methylpyridin-1-ium,



B. 8-chloro-11-(piperidin-4-ylidene)-6,11-dihydro-5*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridine,