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

M.Sc.(Chem.) Semester - 3 (*CBCS*) Examination
Oct/Nov-2022 (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**	**04**
(1)	Define documentation and name the various important documents concern to pharmaceutical manufacturing.	
(2)	What is SOP of SOP?	
Q.1 (b)	**Answer any two questions out of three**	**10**
(1)	Prepare a SOP on calibration of TDS meter in standard SOP format.	
(2)	What is SOP? state its introduction and importance.	
(3)	Prepare Specification of Acebutolol Hydrochloride from given official JP monograph (JP monograph attached here as annexure-1)	
Q.2 (a)	**Answer the following question**	**04**
(1)	Write process for calibration of hot air oven with calibration format.	
Q.2 (b)	**Answer any two questions out of three.**	**10**
(1)	Define precision, discuss its types with acceptance criteria.	
(2)	Write method validation parameters and distinguish between Robustness and Ruggedness.	
(3)	Answer the following a) Define Calibration b) What are Calibrators? give two examples. c) When should be measuring instruments be calibrated as per GLP?	
Q.3 (a)	**Answer the following question**	**04**
(1)	Give brief details of qualification of analyst.	
(2)	When does the qualification of analysts shall be done?	
Q.3 (b)	**Answer any two questions out of three**	**10**
(1)	Explain installation qualification (IQ) in detail.	
(2)	What are the objectives of qualification? distinguish between operational qualification and performance qualification.	
(3)	Enlist phases of qualification and draw qualification chart	
Q.4 (a)	**Answer the following question**	**04**
(1)	Note the requirements of container closer for stability studies of API.	
(2)	what is Shelf-life in stability studies?	
Q.4 (b)	**Answer any two questions out of three**	**10**
(1)	Explain the following in brief (a) Various climatic zones, countries, temperature and %RH values (b) Stability testing frequency	
(2)	Prepare storage conditions table for stability studies of APIs	
(3)	Enlist types of analysis carried out under stress testing? explain any one in detail.	
Q.5 (a)	**Answer the following questions**	**04**
(1)	What is GLP?	
(2)	Draw reagent bottle tag and instrument calibration tag.	
Q.5 (b)	**Answer any two questions out of three**	**10**
(1)	Write dos and don'ts to maintain the GLP practice effective. (8-10 points)	
(2)	Discuss the general requirements for personal qualification and personal hygiene to follow GLP.	
(3)	What is the CPCSEA? Give an account on basic needs of animal house for GLP.	

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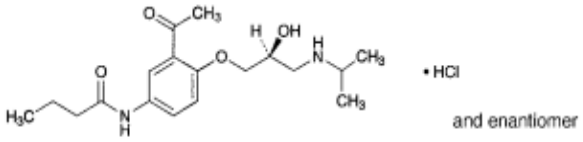
Acebutolol Hydrochloride

アセブトロール塩酸塩



$C_{18}H_{28}N_2O_4.HCl$: 372.89
N-{3-Acetyl-4-[(2*RS*)-2-hydroxy-3-(1-methylethyl)aminopropyloxy]phenyl}butanamide monohydrochloride
[*34381-68-5*]

Acebutolol Hydrochloride, when dried, contains not less than 98.0% and not more than 102.0% of acebutolol hydrochloride ($C_{18}H_{28}N_2O_4.HCl$).

Description Acebutolol Hydrochloride occurs as white to pale yellowish white, crystals or crystalline powder.

It is freely soluble in water, in methanol, in ethanol (95) and in acetic acid (100), and practically insoluble in diethyl ether.

A solution of Acebutolol Hydrochloride (1 in 20) shows no optical rotation.

Identification **(1)** Determine the absorption spectrum of a solution of Acebutolol Hydrochloride in 0.01 mol/L hydrochloric acid TS (1 in 100,000) as directed under Ultraviolet-visible Spectrophotometry <*2.24*>, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(2) Determine the infrared absorption spectrum of Acebutolol Hydrochloride, previously dried, as directed in the potassium bromide disk method under Infrared Spectrophotometry <*2.25*>, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(3) A solution of Acebutolol Hydrochloride (1 in 100) responds to the Qualitative Tests <*1.09*> for chloride.

Melting point <*2.60*> 141 – 145°C

Purity **(1)** Heavy metals <*1.07*>—Proceed with 1.0 g of Acebutolol Hydrochloride according to Method 2, and perform the test. Prepare the control solution with 1.0 mL of Standard Lead Solution (not more than 10 ppm).

(2) Arsenic <*1.11*>—Prepare the test solution with 1.0 g of Acebutolol Hydrochloride according to Method 3, and perform the test (not more than 2 ppm).

(3) Related substances—Dissolve 40 mg of Acebutolol Hydrochloride in 2 mL of methanol, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add methanol to make exactly 25 mL, and pipet 1 mL of this solution, add methanol to make exactly 20 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography <*2.03*>. Spot 5 μL each of the sample solution and standard solution on a plate of silica gel for thin-layer chromatography. Develop the plate with the upper layer of a mixture of water, 1-butanol and acetic acid (100) (5:4:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 365 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Loss on drying <*2.41*> Not more than 1.0% (0.5 g, 105°C, 3 hours).

Residue on ignition <*2.44*> Not more than 0.2% (1 g).

Assay Weigh accurately about 0.25 g of Acebutolol Hydrochloride, previously dried, dissolve in 20 mL of acetic acid (100), add 80 mL of acetic anhydride, and titrate <*2.50*> with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 37.29 mg of $C_{18}H_{28}N_2O_4.HCl$

Containers and storage Containers—Well-closed containers.

Aceglutamide Aluminum

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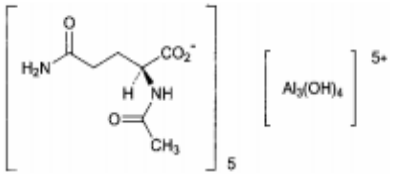

$C_{35}H_{59}Al_3N_{10}O_{24}$: 1084.84
Pentakis[(2*S*)-2-acetylamino-4-carbamoylbutanoato]tetrahydroxotrialuminium
[*12607-92-0*]

Aceglutamide Aluminum contains not less than 85.4% and not more than 87.6% of aceglutamide ($C_7H_{12}N_2O_4$: 188.18), and not less than 7.0% and not more than 8.0% of aluminum (Al: 26.98), calculated on the dried basis.

Description Aceglutamide Aluminum occurs as a white powder, having astringent bitter taste.

It is freely soluble in water, and practically insoluble in ethanol (99.5).

It dissolves in dilute hydrochloric acid.

It is hygroscopic.

Identification **(1)** Dissolve 0.03 g each of Aceglutamide Aluminum and Aceglutamide RS in 5 mL of water, and use these solutions as the sample solution and standard solution. Perform the test with these solutions as directed under Thin-layer Chromatography <*2.03*>. Spot 5 μL each of the sample solution and standard solution on a plate of cellulose for thin-layer chromatography. Develop the plate with a mixture of 1-propanol, water and acetic acid (100) (16:8:1) to a distance of about 10 cm, and air-dry the plate. Spray evenly a solution of bromocresol green in ethanol (95) (1 in 1000), then spray evenly diluted ammonia solution (28) (1 in 100):