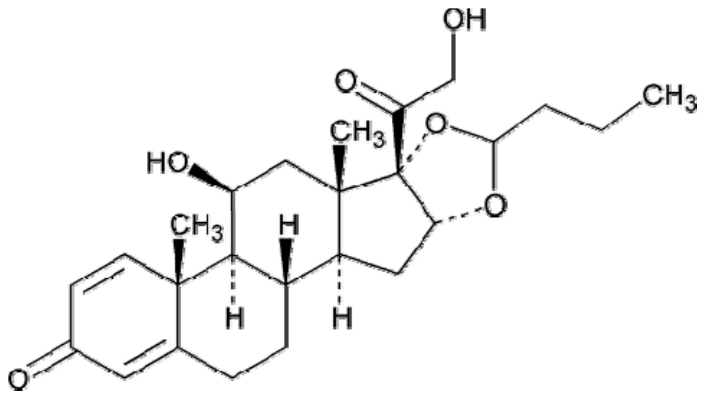
Budesonide

USP Method Budesonide Assay



Original Manufacturer: AstraZeneca (patent expired)

Original Brand Name: Rhinocort (nasal inhalant)
Pulmicort (oral inhalant)
Entocort (enema or oral capsule)

Generic Names: Budanase AQ, Budate, Budecort , Budez, Budvent, Buovent, Derinide

Combination Drugs: Symbicort (Budesonide and Formoterol)

Budesonide is a glucocorticoid steroid for the treatment of asthma and non-infectious rhinitis . In addition, it is used for Crohn's disease (inflammatory bowel disease).

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Budesonide

USP34 – NF29 S1

USP Columns :

Supelcosil LC-18, 4.6 mm x 15 cm, 5 µm.

ZORBAX SB-C18, 4.6 mm x 15 cm, 3.5 µm.

Equivalent Column:

Purospher®STAR RP-18 endcapped (5 µm) 150x4.6 mm (1.51455.0001)

Recommended Solvents and Reagents:

Acetonitrile	isocratic grade for liquid chromatography LiChrosolv®	(1.14291)
Water	Water for chromatography LiChrosolv® or freshly purified water from Milli-Q water purification system	(1.15333)

Phosphoric Acid	Use ACS reagent grade
Sodium di-hydrogen phosphate	Use ACS reagent grade

USP Standards

Budesonide (200 mg)	USP Product Number:	1078201

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Mobile phase
Solution A: 3.17 mg/mL of monobasic sodium phosphate and 0.23 mg/mL of phosphoric acid.
The pH is 3.2 ± 0.1. Mix Acetonitrile and Solution A (32:68)

Standard solution
Dissolve a quantity of USP Budesonide RS in acetonitrile and dilute quantitatively with Solution A to obtain a solution having a concentration of 0.5 mg/mL, keeping the proportion of acetonitrile in this solution to not more then (NMT) 30%.

Sample solution
Dissolve 25 mg of Budesonide in 15 mL of acetonitrile in a 50-mL volumetric flask, and dilute with Solution A to volume.

Chromatographic system (*See Chromatography 621, System Suitability.*)
Detector: UV 254 nm
Flow rate: 1.5 mL/min
Column: 4.6-mm × 15-cm; 5-µm packing L1
Injection size: 20 µL

System suitability (Sample = Standard solution)
Relative retention time for epimer A is 1.1 with respect to epimer B
Resolution: Not less then (NLT) 1.5 between Budesonide epimer A and B
Column efficiency: NLT 5500 theoretical plates, determined from the Budesonide epimer B peak

Analysis (Samples: Standard solution and Sample solution)
Calculate the percentage of epimer A ($C_{25}H_{34}O_6$) in the portion of Budesonide taken:
Result = $[r_{UA}/(r_{UA} + r_{UB})] \times 100$
r_{UA} = = epimer A peak area from Sample solution
r_{UB} = = epimer B peak area from Sample solution

Calculate the percentage of $C_{25}H_{34}O_6$ in the portion of Budesonide taken:
Result = $[(r_{UA} + r_{UB})/(r_{SA} + r_{SB})] \times (C_S/C_U) \times 100$
r_{UA} = = epimer A peak area from Sample solution
r_{UB} = = epimer B peak area from Sample solution
r_{SA} = = epimer A peak area from Standard solution
r_{SB} = = epimer B peak area from Standard solution
C_S = = concentration of USP Budesonide RS in the Standard solution (mg/mL)
C_U = = concentration of Budesonide in the Sample solution (mg/mL)

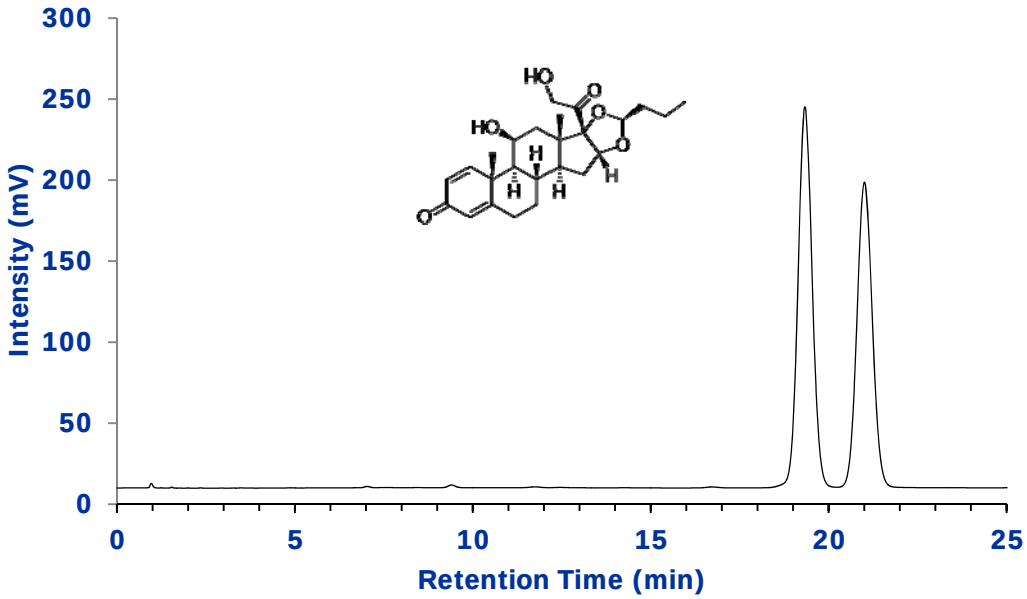
Acceptance criteria
Epimer A: 44.0%–51.0% on the dried basis Both epimers: 98.0%–102.0% on the dried basis

USP Method for Budesonide Assay

Purospher®STAR RP-18 endcapped

Chromatographic Conditions

Column:	Purospher®STAR RP-18 endcapped (5 µm) 150x4.6 mm	1.51455.0001
Injection:	20 µL	
Detection:	Shimadzu Prominence 2010, UV 254 nm	
Cell:	8 µL	
Flow Rate:	1.5 mL/min	
Mobile Phase (v/v):	Buffer: 3.17 gram of sodium di-hydrogen phosphate in water. Add 0.23 gram of Ortho-phosphoric acid and dilute with water to 1000 ml. pH of solution should be 3.2 +/-0.1 Mix buffer and acetonitrile: 68:32	
Temperature:	Ambient	
Diluent	Solution A (phosphate buffer with an adjusted pH to 3.2 ± 0.1).	
Sample:	500 ppm (0.5 mg/mL) Budesonide	
Pressure Drop:	180 Bar (2610 psi)	



Chromatographic Data

No.	Compound	Time (min)	Resolution	Relative Retention Time (RRT)	Plates (N)
1	Budesonide Epimer B	19.3	-	1.1	10695
2	Budesonide Epimer A	21.0	2.2	1.1	10128

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