

Separation of Budesonide and its Organic Impurities per USP Monograph

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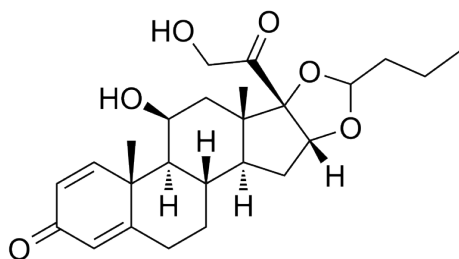
Overview

Budesonide is a corticosteroid that prevents swelling. The development of a quick and efficient analysis of Budesonide and its related organic impurities is of interest for generic drug manufacturers. In this application note, we report the separation of Budesonide and its related organic impurities using a Luna™ 3 µm C18(2) column and a Hypersil® BDS 3 µm C18 column according to the USP monograph for Budesonide Organic Impurities.

System suitability per USP Monograph for the Budesonide Organic Impurities is a resolution no less than (NLT) 1.2 between Budesonide Related Compound E and Budesonide Related Compound L and NLT 3.0 between Budesonide epimer A and the first epimer of Budesonide Related Compound G. A symmetry factor value no more than (NMT) 1.5 for Budesonide epimer B and a percent Relative Standard Deviation (%RSD) NMT 5.0 % for the sum of the peak areas of the two Budesonide epimers are also requirements of system suitability. Finally, a signal-to-noise (S/N) ratio NLT 10 for Budesonide epimers A and B is a system suitability requirement per USP Monograph for the Budesonide Organic Impurities. All requirements for System Suitability for Organic Impurities were met by the Luna 3 µm C18(2) column and the Hypersil BDS 3 µm C18 column (Figure 2, 3, and 4).

All solutions were prepared as indicated in the USP Monograph for Budesonide. USP Budesonide CRS (Catalog No. 1078201), USP Budesonide Related Compound E CRS (Catalog No. 1078245), USP Budesonide Related Compound G CRS (Catalog No. 1078267), and USP Budesonide Related Compound L CRS (Catalog No. 1078289) were purchased from USP.

Figure 1. Budesonide



LC-UV Conditions

Column: Luna 3 µm C18(2) (00G-4251-E0)
Hypersil BDS 3 µm C18

Dimension: 250 x 4.6 mm

Mobile Phase: A: 0.5 mL of Glacial Acetic Acid in 1 L of water. Adjust with Potassium Hydroxide to a pH of 3.9.
B: Acetonitrile

Flow Rate: 1.5 mL/min

Gradient:	Time (min)	%B
	0	25
	5	25
	35	32
	42	41
	59	75
	60	25
	70	25

Injection Volume: 50 µL

Temperature: 50 °C

Detector: UV @ 240 nm

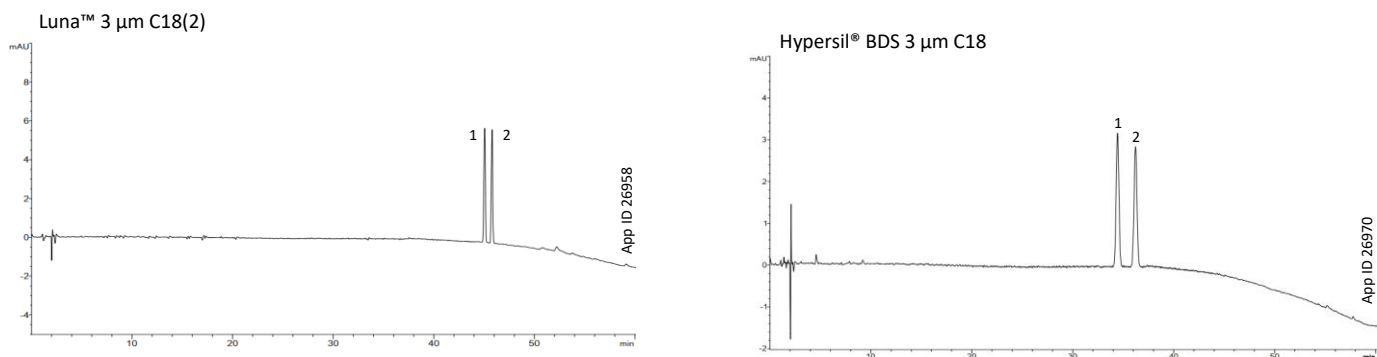
System: Agilent® 1260 Binary UHPLC

Table 1. Preparation of Solutions

Solution	Composition
Diluent	Acetonitrile/water (3:7, v/v)
System Suitability Solution	0.6 mg/mL of USP Budesonide CRS and 3 µg/mL each of: USP Budesonide Related Compound E CRS, USP Budesonide Related Compound G CRS, and USP Budesonide Related Compound L CRS in Diluent
Standard Solution	6 µg/mL of USP Budesonide CRS in Diluent
Sensitivity Solution	0.3 µg/mL of USP Budesonide CRS in Diluent

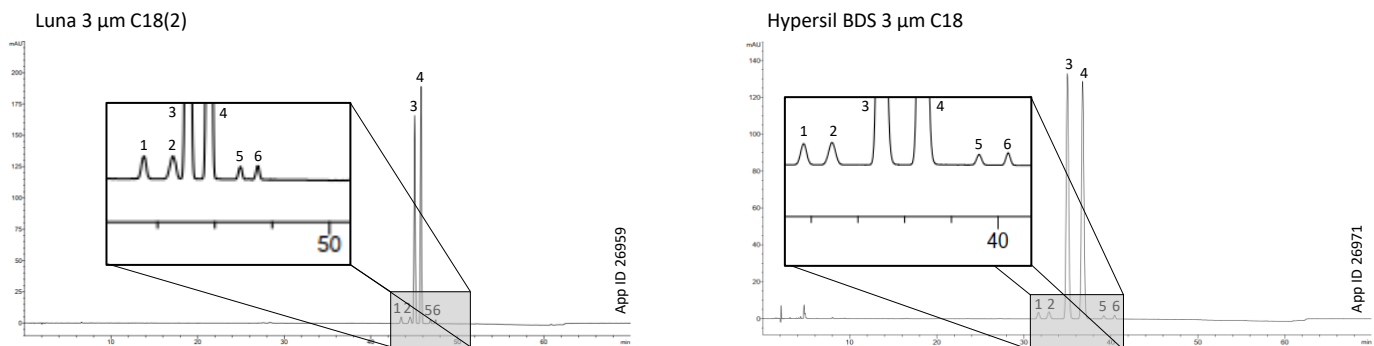


Figure 2. Standard Solution



Peak No.	Analyte	Luna 3 μm C18(2)		Hypersil BDS 3 μm C18	
		Sum Area %RSD	Symmetry Factor	Sum Area %RSD	Symmetry Factor
1	Budesonide Epimer B	0.4	1.089	0.5	0.995
2	Budesonide Epimer A		-		-
N = 6 Injections					

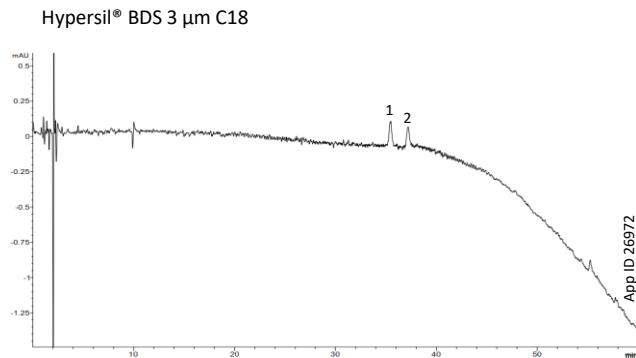
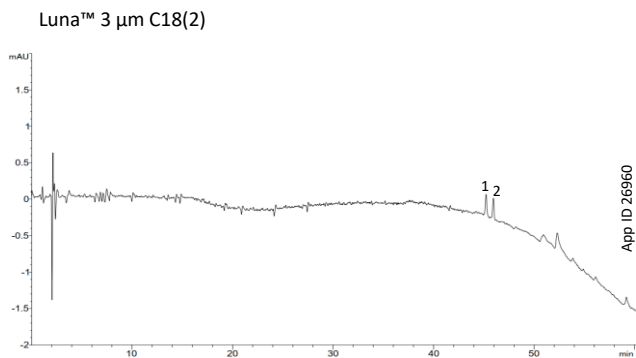
Figure 3. System Suitability Solution



Peak No.	Analyte	Luna 3 μm C18(2)	Hypersil BDS 3 μm C18
		Resolution	Resolution
1	Budesonide Related Compound E	2.82	2.26
2	Budesonide Related Compound L		
3	Budesonide Epimer B	-	-
4	Budesonide Epimer A	4.37	5.15
5	Budesonide Related Compound G Epimer 1		
6	Budesonide Related Compound G Epimer 2	-	-
N = 6 Injections			



Figure 4. Sensitivity Solution



		Luna 3 μm C18(2)	Hypersil BDS 3 μm C18
Peak No.	Analyte	S/N Ratio	S/N Ratio
1	Budesonide Epimer B	19.61	16.42
2	Budesonide Epimer A		
N = 6 Injections			



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