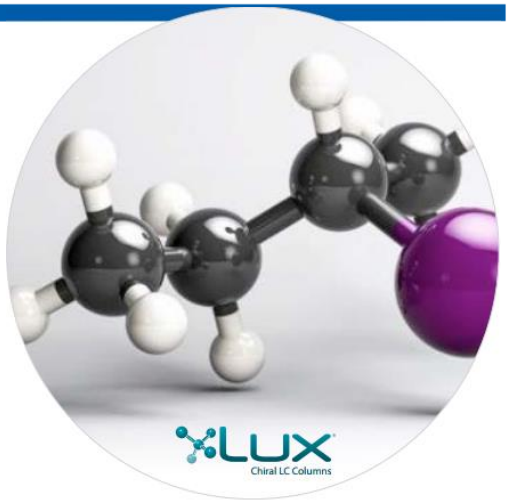


Enantiomeric Purity of Sitagliptin Phosphate per USP Monograph using Lux® 5 µm Amylose-1 Column

Lauren Nakasone, Zeshan Aqeel, and Dr. Bryan Tackett
Phenomenex Inc., 411 Madrid Ave., Torrance, CA 90501, USA

Overview

Sitagliptin Phosphate is a chiral drug which is commercialized as pure enantiomer. It is an anticholinergic drug used for the treatment of patients with overactive bladder. The current trend is to commercialize drugs formulated in their enantiopure form, which means containing only the single enantiomer responsible for pharmaceutical activity. The S-enantiomer of Sitagliptin Phosphate is pharmaceutically inactive and therefore undesirable to have in the presence of active Sitagliptin Phosphate.

In this application note, we report the enantiomeric separation between Sitagliptin Phosphate and its S-enantiomer using the Lux Amylose-1 chiral column according to the USP monograph for Sitagliptin Phosphate.

System suitability per USP Monograph for Sitagliptin Phosphate for resolution of not less than (NLT) 1.5 between the S-enantiomer and Sitagliptin Phosphate. The separation of the S-enantiomer was achieved and peaks corresponding to S-enantiomer and Sitagliptin Phosphate had a resolution of 1.69 (**Figure 1**). This surpassed the required resolution to meet system suitability.

The Lux Amylose-1 column provided a signal to noise ratio (S/N) of 17.5 for the Sitagliptin Phosphate peak, exceeding the required S/N of NLT 10 (**Figure 2**).

All reference solutions were prepared as indicated in the USP Monograph for Sitagliptin Phosphate. Sitagliptin Phosphate (200 mg) (Catalog No. 1612903), and Sitagliptin System Suitability Mixture RS (20 mg) (Catalog No. 1612914) were purchased from USP.

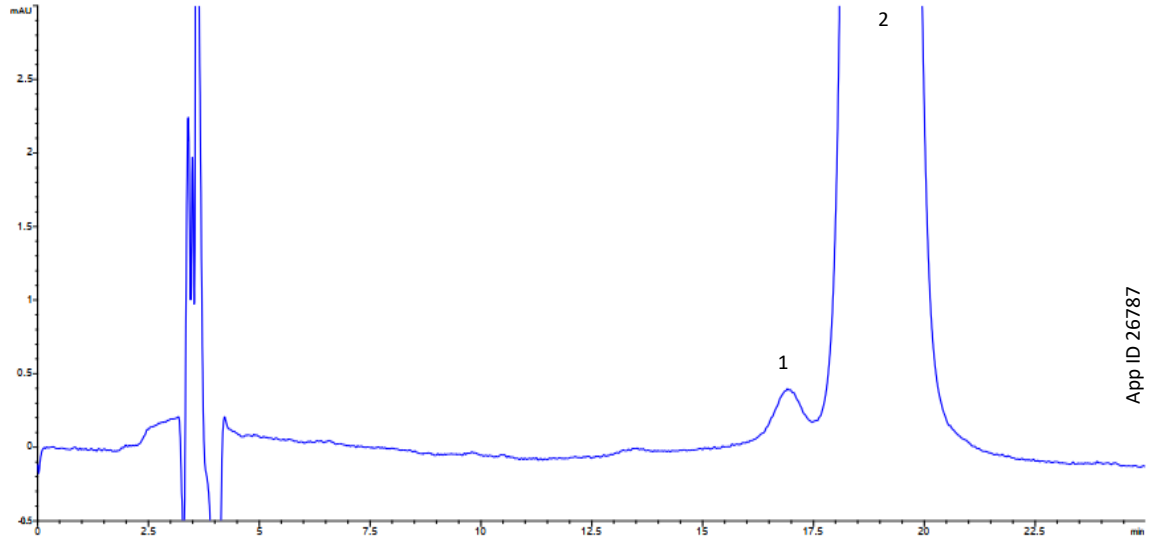
LC-UV Conditions

Column: Lux 5 µm Amylose-1 (250 x 4.6 mm)
Part No.: 00G-4732-E0
Mobile Phase: Dehydrated Alcohol:Chromatographic n-Heptane: Diethylamine:Water (600:400:1:1, v/v/v/v)
Pressure (bar): 109
Flow Rate: 0.8 mL/min
Injection: 10 µL
Temperature: 35 °C
Detector: UV @ 268 nm
System: Agilent® 1260 Binary UHPLC

Table 1. Preparation of Solutions

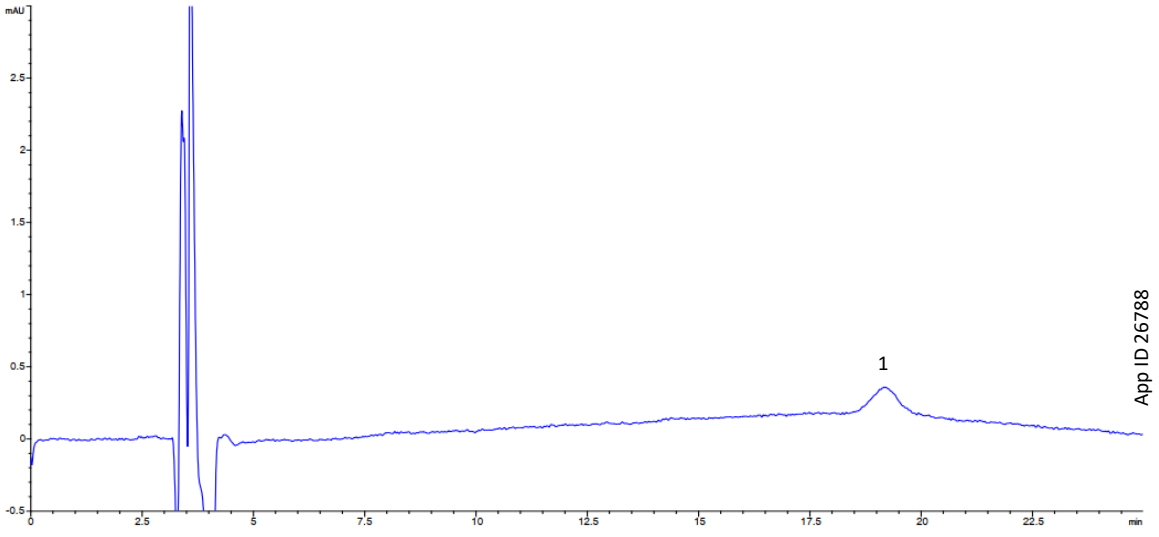
Solution	Composition
System Suitability Solution	Dissolve 8 mg of USP Sitagliptin System Suitability Mixture RS in 1 mL of Diluent (9:1 Methanol:Water)
Sample Solution	Dissolve 8 mg of Sitagliptin Phosphate in 1 mL Diluent
Sensitivity Solution	Dilute 1:1000 of Sample Solution with Diluent to a concentration of 8 µg/mL

Figure 1. System Suitability Solution



Peak	Analyte	Resolution
1	S-Enantiomer	**1.69**
2	Sitagliptin Phosphate	
Number of injections = 6		

Figure 2. Sensitivity Solution



Peak	Analyte	S/N
1	Sitagliptin Phosphate	**17.5**
Number of injections = 6		

Need a different column size or sample preparation format?

No problem! We have a majority of our available dimensions up on www.phenomenex.com, but if you can't find what you need right away, our super helpful Technical Specialists can guide you to the solution via our online chat portal www.phenomenex.com/LiveChat.

Australia
t: +61 (0)2-9428-6444
auinfo@phenomenex.com

Austria
t: +43 (0)1-319-1301
anfrage@phenomenex.com

Belgium
t: +32 (0)2 503 4015 (French)
t: +32 (0)2 511 8666 (Dutch)
beinfo@phenomenex.com

Canada
t: +1 (800) 543-3681
info@phenomenex.com

China
t: +86 400-606-8099
cninfo@phenomenex.com

Czech Republic
t: +420 272 017 077
cz-info@phenomenex.com

Denmark
t: +45 4824 8048
nordicinfo@phenomenex.com

Finland
t: +358 (0)9 4789 0063
nordicinfo@phenomenex.com

France
t: +33 (0)1 30 09 21 10
franceinfo@phenomenex.com

Germany
t: +49 (0)6021-58830-0
anfrage@phenomenex.com

Hong Kong
t: +852 6012 8162
hkinfo@phenomenex.com

India
t: +91 (0)40-3012 2400
indiainfo@phenomenex.com

Indonesia
t: +62 21 5010 9707
indoinfo@phenomenex.com

Ireland
t: +353 (0)1 247 5405
eireinfo@phenomenex.com

Italy
t: +39 051 6327511
italiainfo@phenomenex.com

Japan
t: +81 (0) 120-149-262
jpinfo@phenomenex.com

Luxembourg
t: +31 (0)30-2418700
nlinfo@phenomenex.com

Mexico
t: 01-800-844-5226
tecnicomx@phenomenex.com

The Netherlands
t: +31 (0)30-2418700
nlinfo@phenomenex.com

New Zealand
t: +64 (0)9-4780951
nzinfo@phenomenex.com

Norway
t: +47 810 02 005
nordicinfo@phenomenex.com

Poland
t: +48 22 104 21 72
pl-info@phenomenex.com

Portugal
t: +351 221 450 488
ptinfo@phenomenex.com

Singapore
t: +65 800-852-3944
sginfo@phenomenex.com

Slovakia
t: +420 272 017 077
sk-info@phenomenex.com

Spain
t: +34 91-413-8613
espinfo@phenomenex.com

Sweden
t: +46 (0)8 611 6950
nordicinfo@phenomenex.com

Switzerland
t: +41 (0)61 692 20 20
swissinfo@phenomenex.com

Taiwan
t: +886 (0) 0801-49-1246
twinfo@phenomenex.com

Thailand
t: +66 (0) 2 566 0287
thaiinfo@phenomenex.com

United Kingdom
t: +44 (0)1625-501367
ukinfo@phenomenex.com

USA
t: +1 (310) 212-0555
info@phenomenex.com

All other countries/regions
Corporate Office USA
t: +1 (310) 212-0555
info@phenomenex.com

www.phenomenex.com
Phenomenex products are available worldwide. For the distributor in your country/region, contact Phenomenex USA, International Department at international@phenomenex.com