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Application News

No. L580A

High Performance Liquid Chromatography Nexera™ lite / RF-20AXS

Analysis of Voglibose by Post-Column Derivatization Method

Y. Zhou

User Benefits

- The results of system suitability test meet all criteria of system performance and reproducibility described in the Japanese Pharmacopoeia 17th Edition Voglibose Tablets Assay.
- A cooling chamber is not required because the cell temperature of the fluorescence detector can be controlled to 15 ˚C.
- Whole system including the chemical reaction box meets data integrity requirements.

■ Introduction

Voglibose is known as one of the antidiabetic drugs. It can delay the digestion and absorption of sugars and improve post-prandial blood glucose levels by inhibiting the activity of α-glucosidase, which is responsible for the decomposition of disaccharides to monosaccharides, in the intestinal tract.

Since voglibose does not have UV absorption, the Japanese Pharmacopoeia (JP) 17th Edition describes the monographs (purity test for voglibose, assay for voglibose tablets) using the HPLC post-column fluorescence derivatization method with a taurine/sodium periodate reagent.

This article introduces an analysis complying with the assay method for voglibose tablets described in the JP 17th Edition, using the Nexera series HPLC.

■ Analysis of Voglibose Standard Solution

The post-column derivatization reaction was carried out using a Shimadzu chemical reaction box (CRB-40). It can be controlled by LabSolutions™ workstation which complies with data integrity. Fig. 1 shows the LabSolutions screen capture of the control panel window.

The analytical conditions are shown in Table 1, and the flow path diagram is shown in Fig. 2. In this system, the reaction reagent was once cooled to 25 ˚C in the column oven, and then introduced into the fluorescence detector in which the cell was preliminarily kept at specified 15 ˚C. This eliminates the need for a cooling chamber.

Fig. 3 shows the chromatogram when 50 µL of a 1 mg/L voglibose standard solution was injected.

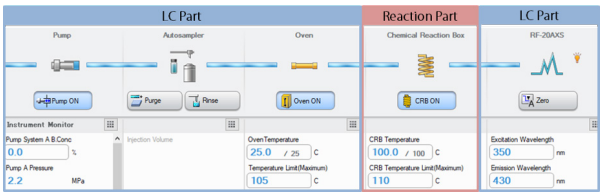

Fig. 1 Screen Capture of Control Panel Window

Table 1 Analytical Conditions

<Separation>	
System	: Nexera lite
Column	: Shim-pack™ GIST NH2 (150 mm × 4.0 mm I.D., 5 µm) *1
Vial	: SHIMADZU LabTotal™ Vial for LC 1.5 mL, Glass *2
Mobile phase	: Sodium phosphate buffer (pH 6.5)/ Acetonitrile = 300 : 600
Flow rate	: 0.37 mL/min
Column temp.	: 25 ˚C
Injection volume	: 50 µL
<Detection>	
Reaction reagent	: Taurine/Sodium periodate aqueous solution
Flow rate	: 0.37 mL/min
Reaction temp.	: 100 ˚C
Cooling temp.	: 25 ˚C (CTO-40C) → 15 ˚C (RF-20AXS)
Detection	: Ex. 350 nm, Em. 450 nm (RF-20AXS)
Cell temp.	: 15 ˚C *3

*1 P/N: 227-30301-06, *2 P/N: 227-34001-01
*3 Room temperature should be controlled below 20 ˚C.

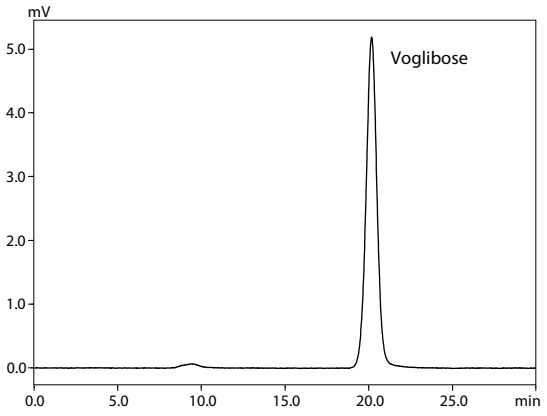

Fig. 3 Chromatogram of Voglibose Standard Solution (1 mg/L)

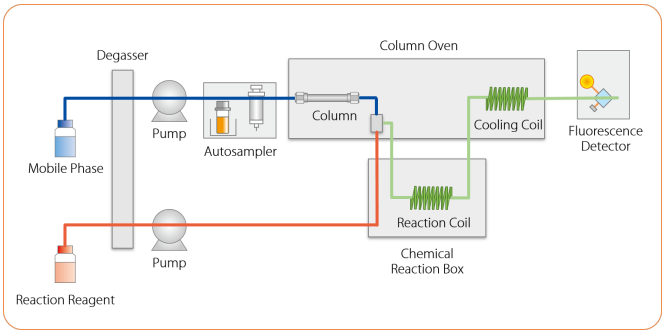

Fig. 2 Flow Path Diagram

■ System Suitability Test

System suitability test was performed based on the assay for voglibose tablets described in the JP 17th Edition.

Fig. 4 shows the chromatogram of standard solution containing 4 g/L of lactose and 40 mg/L of voglibose for evaluation of system performance, and Fig. 5 shows the chromatogram of 40 mg/L of voglibose standard solution for evaluation of system repeatability.

Table 2 shows the results of the system suitability test. Resolution between lactose and voglibose was 5.2, confirming that the system meets the criterion of not less than 4 in the JP. The relative standard deviation (%RSD) of the peak area of voglibose was 0.13% (n = 6), which also meets the criterion of not more than 2% in the JP.

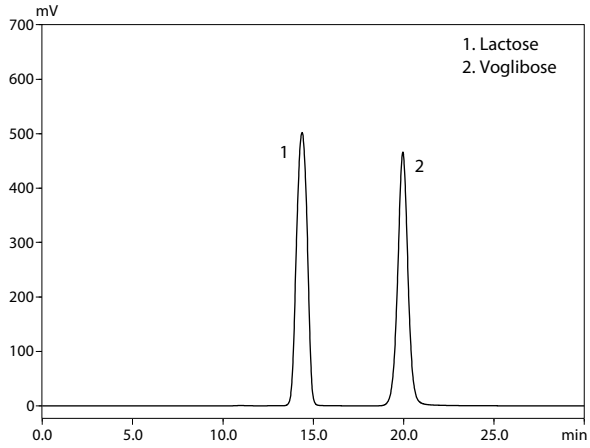

Fig. 4 Chromatogram of Standard Solution of Lactose (4 g/L) and Voglibose (40 mg/L)

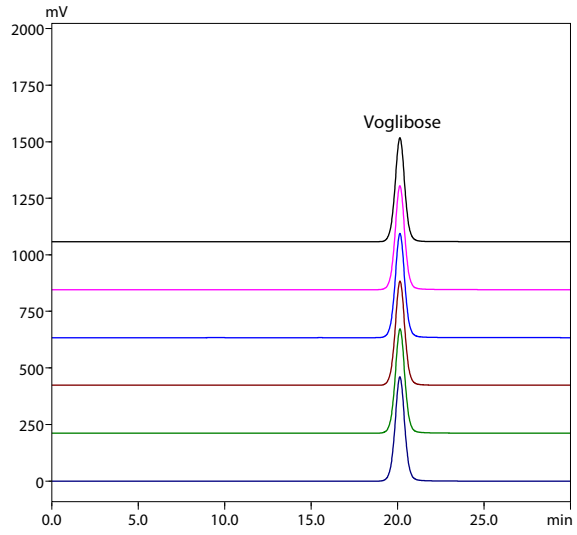

Fig. 5 Chromatograms of Standard Solution of Voglibose (40 mg/L) (n = 6)

Table 2 Results of System Suitability Test

Test item	Criterion	Result	Judgement
Resolution (Between lactose and voglibose)	≥4	5.2	Passed
Relative standard deviation of area (%) (n=6)	≤2	0.13	Passed

■ Linearity

Fig. 6 shows the calibration curve of the voglibose standard solution with concentrations of 25, 40, 100, 250, and 1000 (µg/L). Excellent linearity was obtained, with a r^2 value (coefficient of determination) greater than 0.9999.

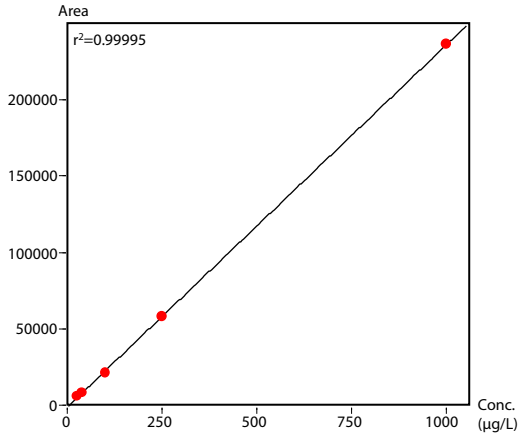

Fig. 6 Calibration Curve of Voglibose Standard Solution (25 µg/L - 1000 µg/L)

■ High Sensitivity Analysis of Voglibose

Fig. 7 shows the chromatogram when 50 µL of 25 µg/L voglibose standard solution was injected. The peak area %RSD (n = 6) was 1.1%, showing excellent repeatability.

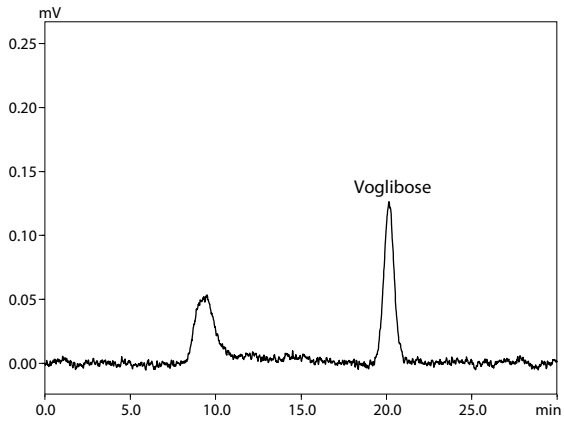

Fig. 7 Chromatogram of Voglibose Standard Solution (25 µg/L)

■ Conclusion

In this article, an analysis was conducted in compliance with the assay method for voglibose tablets described in the JP 17th Edition using a Nexera series HPLC. The results of a system suitability test confirmed that the system meets all criteria of the JP 17th Edition for system performance and repeatability.

In addition, the repeated analyses of low concentration of 25 µg/L standard solution resulted in good repeatability. This means that Nexera series provides reliable results regardless of the concentration of the target compounds.

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