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Application News

No. L550

High Performance Liquid Chromatography

Analysis of Meloxicam in Accordance with the United States Pharmacopoeia by Nexera™ XR

Meloxicam, inhibits the biosynthesis of prostaglandin, is utilized as an anti-inflammatory and analgesic for arthritis. In the United States Pharmacopoeia (USP), the HPLC method using a UV-Vis absorbance detector is adopted as the test method for meloxicam.

This article introduces an example of the analysis of meloxicam in accordance with the USP by Nexera XR, and compatibility with Prominence™ series using ACTO (Analytical Condition Transfer and Optimization) function.

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System Suitability Test

Table 1 shows the analytical conditions with the assay. Table 2 shows the method of preparing the solution used for system suitability test, and Fig. 1 shows the analysis results. The relative retention time for meloxicam related compound A[1] to meloxicam was 0.6, which is close to the reference value of 0.7. Table 3 shows the criteria and results for the system suitability test. It is evident that system suitability was satisfied for all items.

Table 1 Analytical Conditions (Assay)

Column	: Shim-pack™ GIST C18 (USP code:L1)
	: (150 mmL. × 4.6 mmI.D. , 5 µm)
Mode	: Isocratic
Mobile phase	: Methanol/solution A = 21/29
Flow rate	: 1 mL/min
Column temp.	: 45 °C
Injection volume	: 10 µL
Detection	: PDA detector (360 nm)

Table 2 Solution Preparation Method (Assay)

Solution A:
Mixture of a 0.1 % (w/v) solution of ammonium acetate adjusted with 10 % ammonia solution to a pH of 9.1

Diluent:
Methanol and 1 N sodium hydroxide (250:1)

System suitability solution:
0.08 mg/mL each of meloxicam and meloxicam related compound A. Prepared by dissolving in 50 % of the flask volume of diluent and diluting with water to volume.

Standard solution:
0.2 mg/mL of meloxicam. Prepared by dissolving in 50 % of the flask volume of diluent and diluting with water to volume.

Table 3 Results for System Suitability Test (Assay)

Test items	Criteria	Result	Judgement
Resolution between meloxicam related compound A and meloxicam	≥ 3.0	9.37	Passed
Tailing factor (meloxicam)	≤ 2.0	1.14	Passed
Relative standard deviation of peak area (n=6)	≤ 2.0 %	0.046 %	Passed

1) Isopropyl-4-hydroxyl-2-methyl-2*H*-1, 2-benzothiazune-3-carboxylate-1, 1-dioxide

2) 2-Amino-5-methyl-thiazole

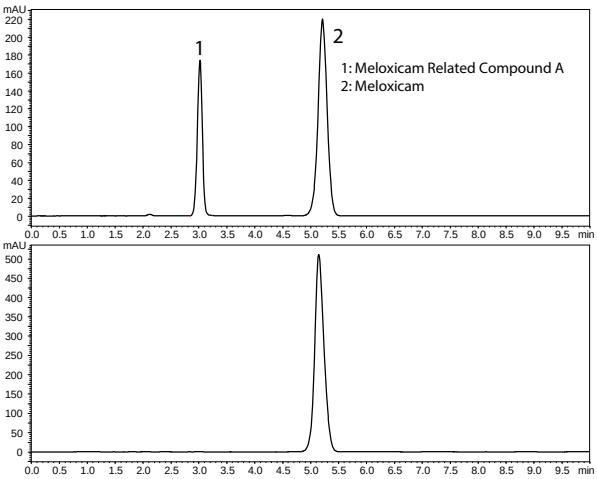

Fig. 1 Chromatograms from the Assay
Top: System Suitability Solution
Bottom: Standard Solution

Table 4 shows the analytical conditions for the impurity analysis. Table 5 shows the method of preparing the solution used for system suitability test, and Fig. 2 shows the analysis results. The relative retention times for meloxicam related compounds B[2] and A to Meloxicam were 0.5 and 1.3 respectively, which are close to the reference values of 0.4 and 1.4. Table 6 shows the criteria and results for the system suitability test. It is evident that system suitability was satisfied for all items.

Table 4 Analytical Conditions (Impurity Analysis)

Column	: Shim-pack GIST C18 (USP code:L1)
	: (150 mmL. × 4.6 mmI.D., 5 µm)
Mode	: High pressure gradient
Mobile phase	: A) SolutionA B) Methanol
Time program	: 40 %B (0 min) => 40 %B (2 min) => 70 %B (10 min) =>70 %B (15 min) => 40 %B (15.01 min) => 40 %B (18 min)
Flow rate	: 1 mL/min
Column temp.	: 45 °C
Injection volume	: 5 µL
Detection	: PDA detector (350 nm , 260 nm)

Table 5 Solution Preparation Method (Impurity Analysis)

Solution A:
0.1 % (w/v) solution of monobasic potassium phosphate adjusted with 1 N sodium hydroxide to a pH of 6.0

Diluent:
Methanol and 1 N sodium hydroxide (50:3)

System suitability solution:
0.08 mg/mL each of meloxicam, meloxicam related compound A, and meloxicam B. Prepared by dissolving in 10 % of the flask volume of diluent and diluting with methanol to volume.

Standard stock solution:
0.6 mg/mL of meloxicam. Prepared by dissolving in 25 % of the flask volume of diluent and diluting with methanol to volume.

Standard solution:
0.012 mg/mL of meloxicam in methanol from standard stock solution.

Table 6 Results for System Suitability Test (Impurity Analysis)

Test items	Criteria	Result	Judgement
Resolution between meloxicam and meloxicam related compound A at 350 nm (system suitability solution)	≥ 3.0	7.18	Passed
Resolution between meloxicam and meloxicam related compound B at 260 nm (system suitability solution)	≥ 3.0	17.6	Passed
Relative standard deviation of peak area (n=6) (Standard solution)	≤ 10 %	0.30 % (350 nm)	Passed

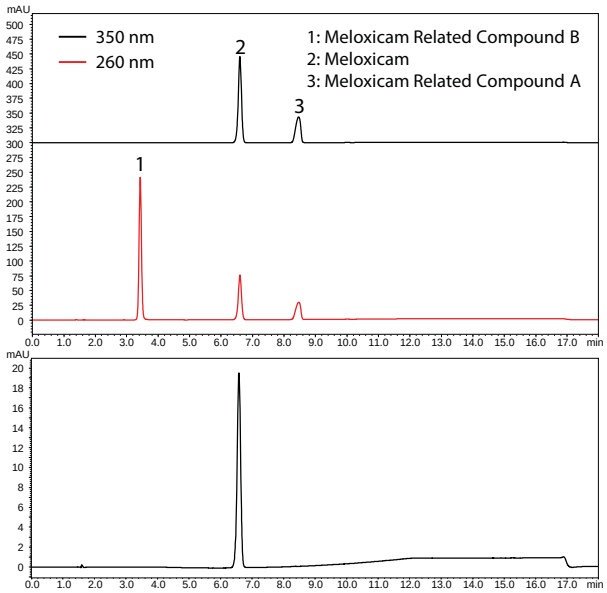

Fig. 2 Chromatograms from the Impurity Analysis
Top: System Suitability Solution
Bottom: Standard Solution

Compatibility with Prominence Series Using ACTO Function

In a gradient analysis, differences in the gradient delay volume between HPLC systems result different chromatograms. Fig. 3 shows a comparison of the chromatograms of system suitability solution (impurity analysis) using Nexera XR (mixer volume 180 µL) and Prominence (mixer volume 0.5, 1.6, and 2.7 mL). It is evident that the retention time changes depending on system differences. In such cases, the gradient start timing can be adjusted using ACTO function equipped in Labsolutions™. This enables a smooth method transfer without replacing piping, mixers, or other parts. Fig. 4 shows the results of a confirmation of compatibility with Nexera XR and Prominence using ACTO function. From this, it is evident that using ACTO function, comparable chromatograms can be obtained despite differing HPLC systems.

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References

United States Pharmacopeia 40-NF 35, 2017

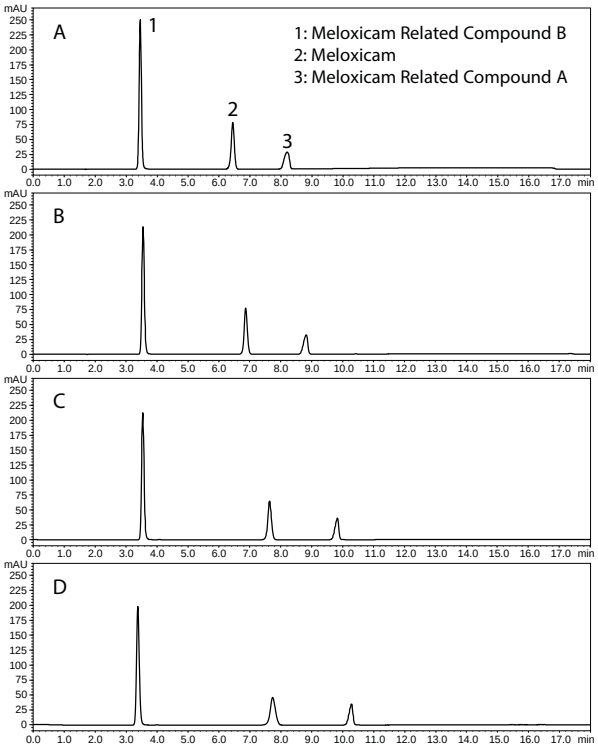

Fig. 3 Comparison of Chromatograms from Nexera XR and Prominence
A: Nexera XR (Mixer volume 180 µL)
B: Prominence (Mixer volume 0.5 mL)
C: Prominence (Mixer volume 1.7 mL)
D: Prominence (Mixer volume 2.5 mL)

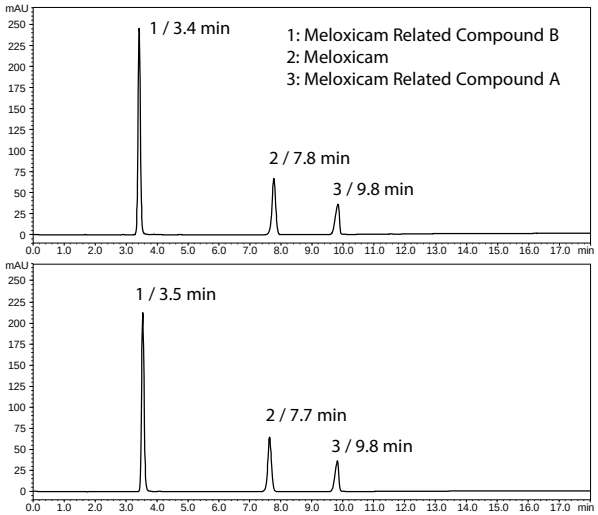

Fig. 4 Adjustment of Gradient Delay Volume by ACTO function
Top: Nexera XR (After adjustment of the gradient start timing)
Bottom: Prominence (Mixer volume 1.7 mL)

Summary

In this article, an analysis of meloxicam was performed in accordance with the USP using the Nexera XR. It was confirmed that system suitability was satisfied. Additionally, even when meloxicam is analyzed with an system with big gradient delay volume, comparable analysis can be performed by adjusting the gradient start timing using ACTO function.

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