

Application News

Liquid Chromatography Mass Spectrometry

No.C73

Impurity Analysis of Drugs Using Shim-pack XR-Phenyl Column

Selection of the appropriate column and setting the separation conditions are extremely important in impurity analysis. Although an ODS column is used most of the time, use of phenyl columns is increasing recently with the aim of improving separation. In particular, it is believed that the effectiveness of many pharmaceutical substances is linked to utilization of the aromatic ring π - π interaction. Here we introduce examples of impurity analysis of nitrendipine and nifedipine using the Prominence UFLC and LCMS-2020.

■ Impurity Analysis of Nitrendipine

The structures of nitrendipine and some impurities are shown in Fig. 1. The TIC chromatogram and mass chromatogram obtained following an injection of nitrendipine (1 mg/mL methanol solution, 24 hours after preparation) are shown in Fig. 2. While nitrendipine and Compound A have a dihydropyridine structure, Compound B has a pyridine structure. Therefore, when a phenyl column is used, Compound B interacts with the solid phases stronger than nitrendipine and Compound A, resulting in delayed elution.

Reference: European Pharmacopoeia 6.0

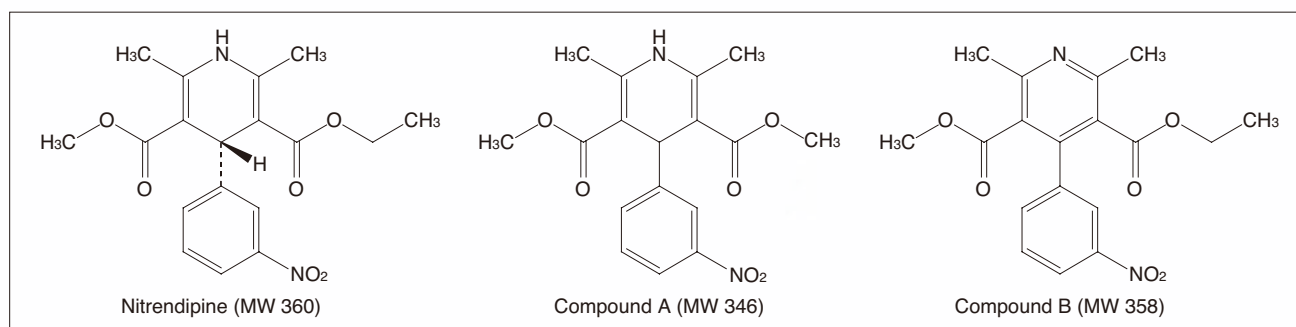


Fig. 1 Structures of Nitrendipine and Impurities

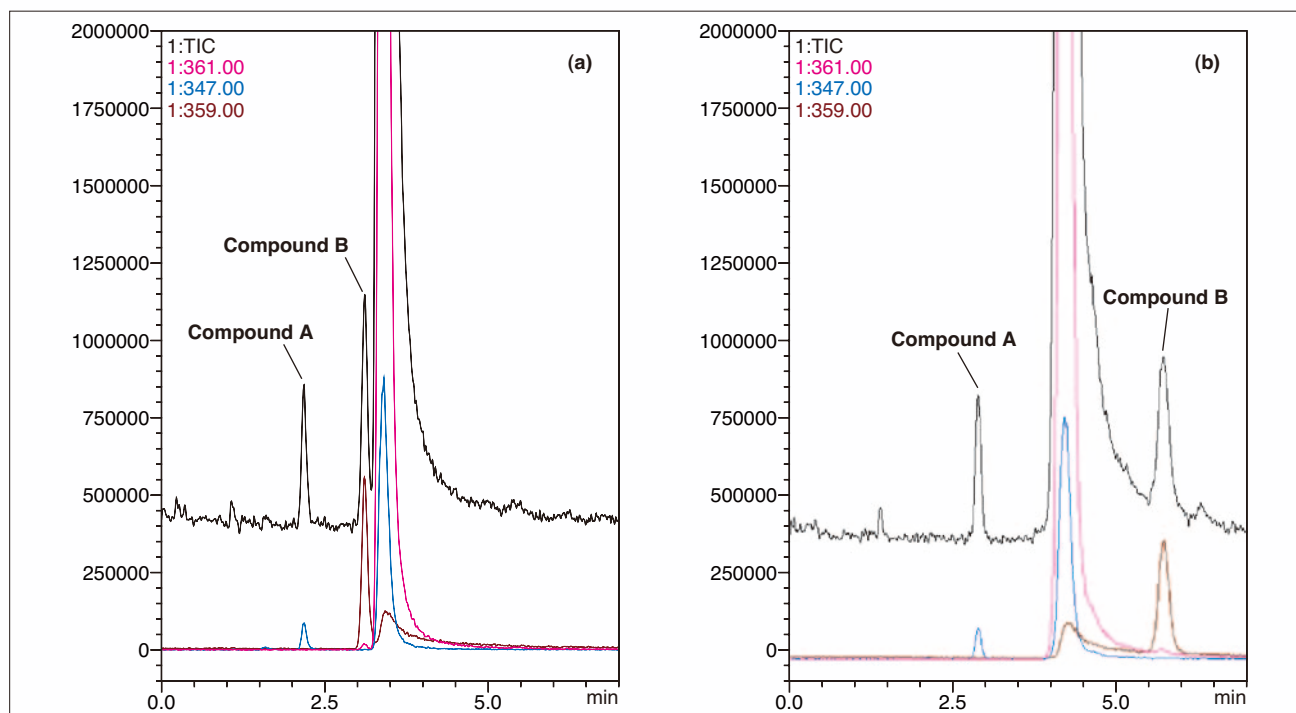


Fig. 2 Chromatograms of Nitrendipine ((a) ODS Column, (b) Phenyl Column)

■ Impurity Analysis of Nifedipine

The structures of nifedipine and some of its impurities are shown in Fig. 3. The TIC chromatogram and mass chromatogram obtained following injection of nifedipine (1 mg/mL methanol solution, right after preparation and 24 hours after preparation) are shown in Fig. 4.

Nifedipine had almost completely disappeared 24 hours after preparation of the solution. Nifedipine and

Compound C were not separated when the phenyl column was used, but the Compound E peak was prominently observed. Using the ODS column, Compound E probably eluted at the same time as Compound C, so unless separation improvement is obtained through adjustment of the mobile phase composition, etc., use of the phenyl column would be more effective.

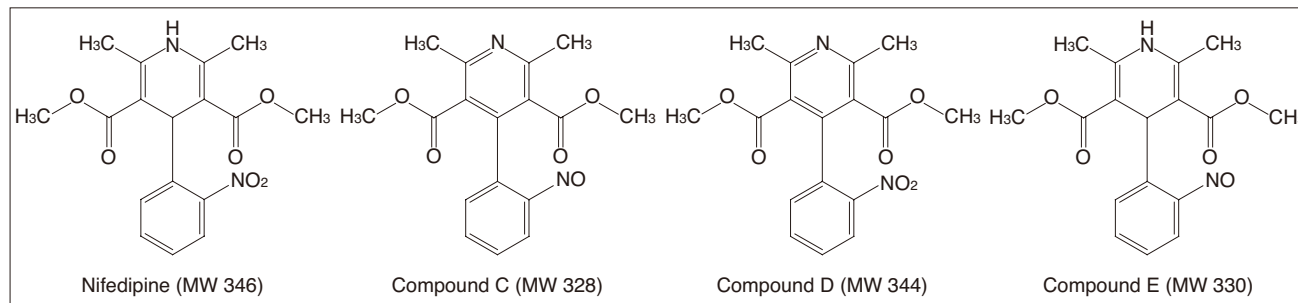


Fig. 3 Structures of Nifedipine and Impurities

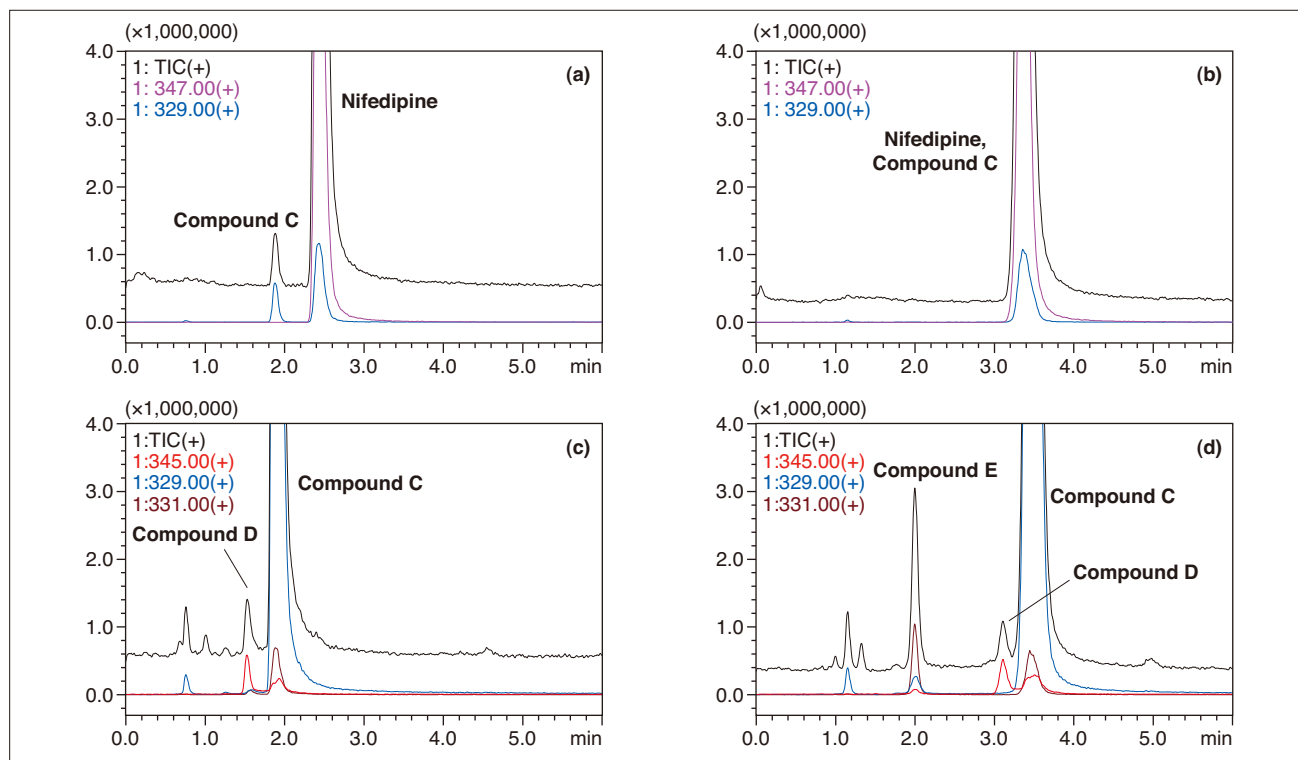


Fig. 4 Chromatograms of Nifedipine ((a) 0 h, ODS, (b) 0 h, Phenyl, (c) 24 h, ODS, (d) 24 h, Phenyl)

Table 1 Analytical Conditions

Column	: Shim-pack XR-ODS (50 mmL. × 2.0 mmI.D., 2.2 μm) Shim-pack XR-Phenyl (50 mmL. × 2.0 mmI.D., 2.2 μm)	MS	: LCMS-2020
Mobile Phase	: A: 0.1 % formic acid in water B: methanol A/B = 45/55 (Nitrendipine), 50/50 (Nifedipine)	Probe Voltage	: + 4.5 kV(ESI-Positive mode)
Flow Rate	: 0.4 mL/min	Nebulizing Gas Flow	: 1.5 L/min
Column Temperature	: 40 °C	Drying Gas Flow	: 15.0 L/min
Injection Volume	: 0.5 μL	DL Temperature	: 250 °C
		Block Heater Temperature	: 450 °C
		DL, Q-Array Voltages	: default values
		Event Time	: 0.1 sec
		Scan Range	: m/z 120-800



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