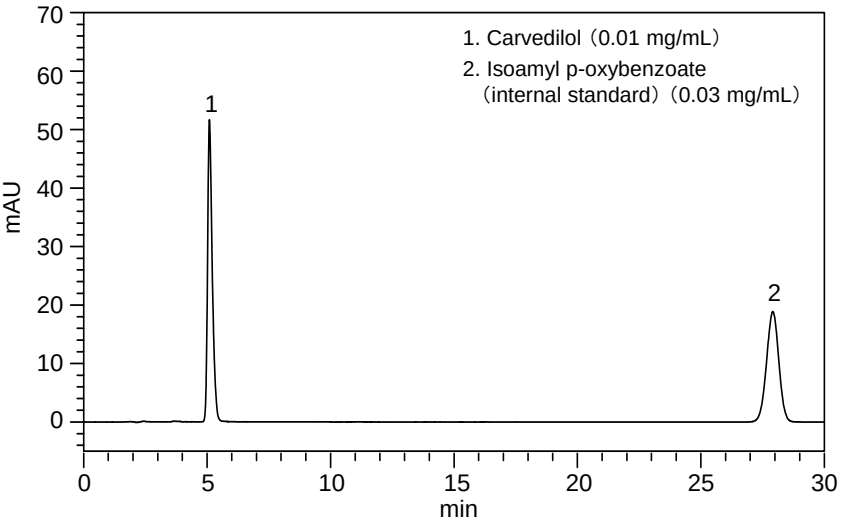
■ Analysis of Two New Drugs in the Japanese Pharmacopoeia Sixteenth Edition

AS/LC-003

More than 100 drugs and their analytical test methods were added to the Japanese Pharmacopoeia Sixteenth Edition, which went into effect in April 2011. Presented here is the system performance and repeatability for the methods used to analyze tablet forms of two of the added drugs, carvedilol and glimepiride.
Carvedilol is used to treat high blood pressure and angina pectoris. It suppresses the heart rate and acts as a vasodilator by blocking α and β-receptors in the sympathetic nervous system. Glimepiride, a drug used to treat diabetes, reduces blood glucose levels by acting on the pancreas and promoting insulin secretion.

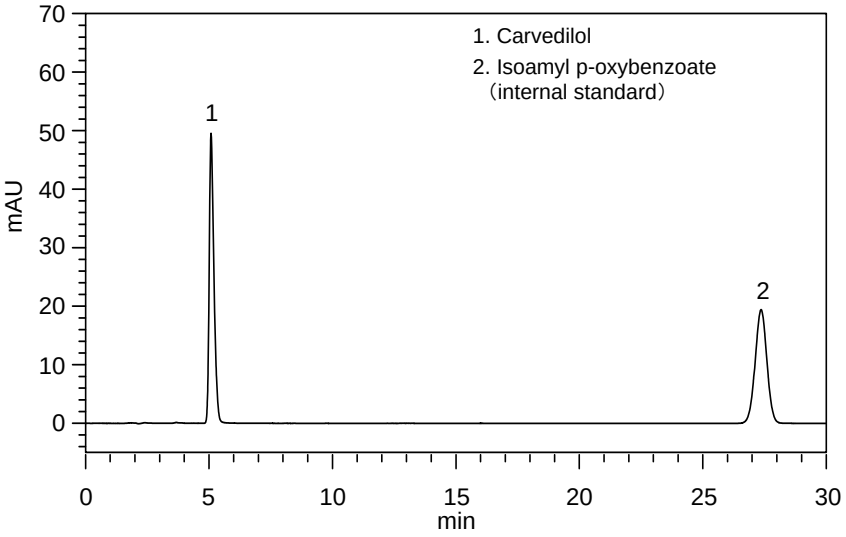
■ Analysis of Carvedilol Standard and Carvedilol Tablet



[Analysis of Standard]

<Analysis Conditions>

Column	:	HITACHI LaChrom C18 (5 μm) 4.6 mm I.D. × 150 mm
Eluent	:	Phosphate buffer (pH 5.0)/methanol = 450 / 550 (v/v)
Flow rate	:	1.0 mL/min
Column temperature	:	40°C
Detection wavelength	:	UV 240 nm
Injection volume	:	10 μL



[Analysis of Carvedilol Tablet]

[Sample Preparation Method for Carvedilol Tablet]

Sample weighing (equivalent to 2.5 mg of carvedilol)
← Add 0.5 mL of internal standard (14.3 mg/mL)
← Add 0.1 mol/L hydrochloric acid/methanol = 1 / 1 (v/v) and make up the volume to 25 mL
Stir for 30 min
Collect 2 mL
← Add eluent and make up the volume to 20 mL
Filter through a 0.45 μm filter
After discarding the first 10 mL, collect as the sample solution

[System Suitability for Carvedilol]
According to the system performance described in the Japanese Pharmacopoeia Sixteenth Edition, the resolution between carvedilol and the internal standard in the standard solution must be 20 or greater. The system repeatability, measured as the standard deviation of the area ratio for the carvedilol peak relative to the internal standard peak in the standard solution, should not be more than 1.0% (n = 6).
The analytical results indicate that these requirements are met.

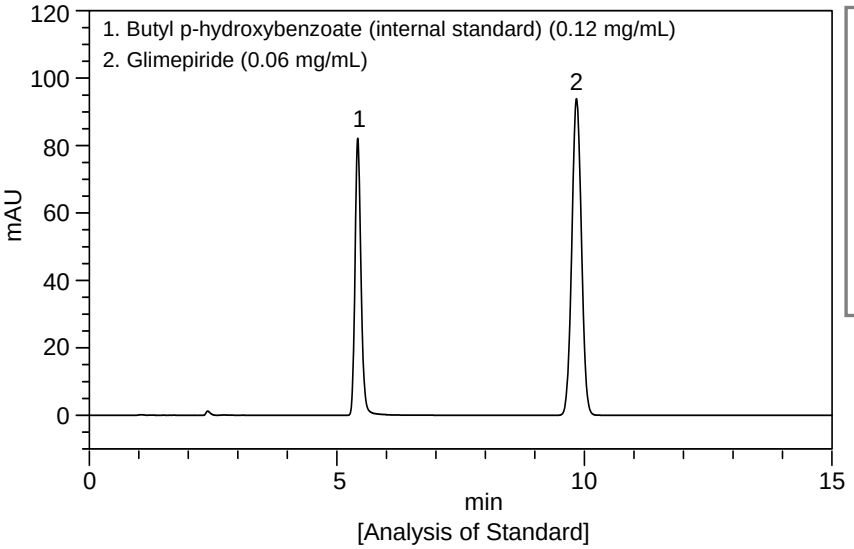
	Requirement for system suitability	Measurement result
Resolution	≥ 20	36.7
Relative standard deviation of peak area ratio (n = 6)	≤ 1.0%	0.075%

*This analysis sample was provided by Division of Physical Pharmaceutical Chemistry, Faculty of Pharmacy, Keio University.

■ Analysis Example for Drugs in the Japanese Pharmacopoeia Sixteenth Edition

AS/LC-003

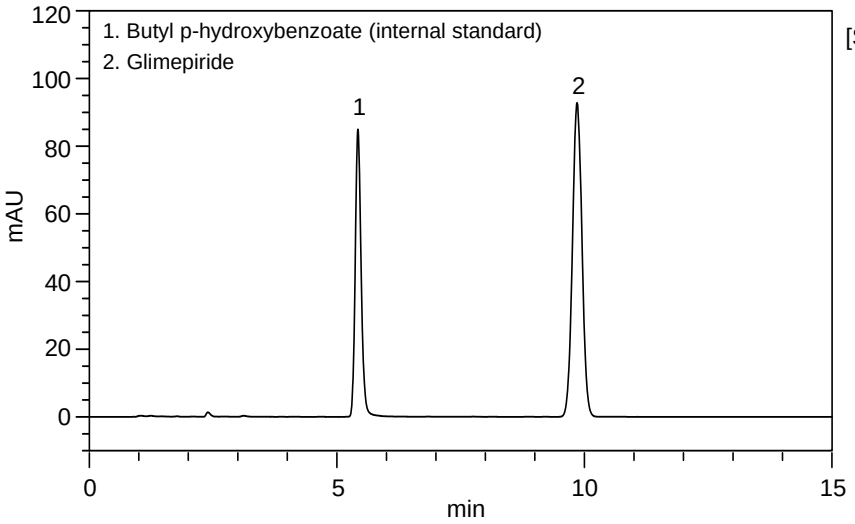
■ Analysis of Glimepiride Standard and Glimepiride Tablet



[Analysis of Standard]

<Analysis Conditions>

Column : HITACHI LaChrom C18 (5 μm) 4.6 mm I.D. × 150 mm
Eluent : Phosphate buffer/acetonitrile = 500 / 500 (pH 3.5) (v/v)
Flow rate : 1.4 mL/min
Column temperature : 25°C
Detection wavelength : UV 228 nm
Injection volume : 10 μL



[Analysis Example of Glimepiride Tablet]

[Sample Preparation Method for Glimepiride Tablet]

Sample weighing (equivalent to glimepiride 0.6 mg)
← Add 0.6 mL of water
← Add 6 mL of acetonitrile/water = 4 / 1 (v/v)
Dissolution
← Add 1.2 mL of internal standard solution (1 mg/mL)
← Add acetonitrile/water = 4/1 (v/v) to make up the volume to 10 mL
Centrifuge 3,000 rpm, 10 min
Filter the supernatant through a 0.45 μm filter

[System Suitability for Glimepiride]
According to the system performance described in the Japanese Pharmacopoeia Sixteenth Edition, the resolution between the glimepiride and internal standard in the standard solution must be 6 or greater. The system repeatability, measured as the standard deviation of the area ratio for the glimepiride peak relative to the internal standard peak in the standard solution, should not be more than 1.0% (n = 6).
The analytical results indicate that these requirements are met.

	Requirement for system suitability	Measurement result
Resolution	NLT 6	15.2
Relative standard deviation of peak area ratio (n = 6)	NMT 1.0 %	0.077 %

* The pharmacopoeia, the column size of 4 mm I.D. × 125 mm is specified.
* This analysis sample was provided by Division of Physical Pharmaceutical Chemistry, Faculty of Pharmacy, Keio University.

Main instrument configuration : Chromaster
5110 pump, 5210 autosampler, 5310 column oven, and 5410 UV detector

Note: The data here is shown as an example of the analysis and does not warrant the performance of the instrument.