

Analysis of Kasugamycin and Validamycin in Tea Leaf by LC-MS

Kasugamycin and validamycin are aminoglycoside antibiotics that are widely used as agricultural chemicals for their effects on sheath blight disease and damping-off in rice and vegetables. Both are highly polar substances, and when analyzed by HPLC, retention is difficult in the reversed phase mode using a C18 column. The Ministry of Health, Labor and Welfare in Japan requires that certain food products are monitored for the presence of validamycin, suggesting a method in which the packing material is a silica gel to which a triacontyl group (C30) has been introduced.

This application describes a HILIC method for the analysis of kasugamycin and validamycin in tea leaves. The detection limits under these analytical conditions were 0.5µg/L and 0.6µg/L for kasugamycin and validamycin, respectively. In addition, when tea leaves spiked with reference standards at concentrations of 10ng/g were analyzed, the results showed recovery rates of 85% to 93%.

Figure 1. Structures of Kasugamycin and Validamycin

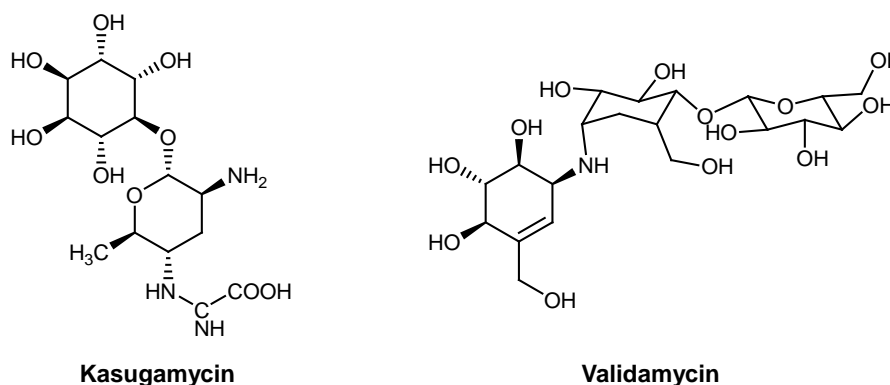


Figure 2. Chromatograms of kasugamycin and validamycin (50µg/L)

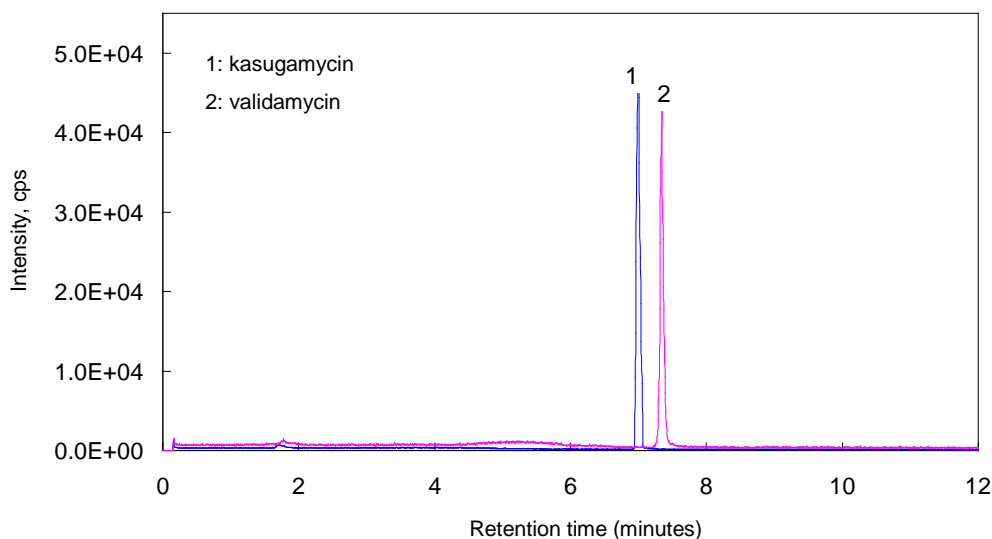


Table 1. Analytical conditions

Column:	TSKgel NH ₂ -100, 3μm, 2.0mm ID x 15cm
Mobile phase:	A: 10mmol/L ammonium formate, pH 3.75 B: acetonitrile
Gradient:	0min (90%B) → 10min (20%B) → 12min (20%B) → 14min (90%B)
Flow rate:	0.2mL/min
Temperature:	40°C
Injection vol.:	2μL
Instrument:	Agilent 1200SL series QTRAP® (AB SCIEX)
Ion source:	ESI (Positive) <i>m/z</i> : 380.0 (kasugamycin), 498.0 (validamycin)

Figure 3. Pretreatment of tea leaf sample

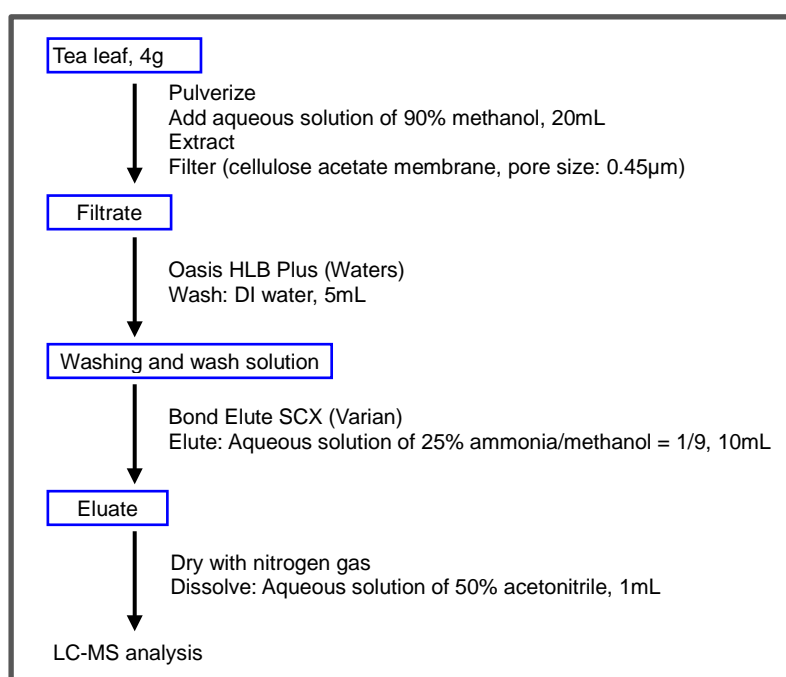
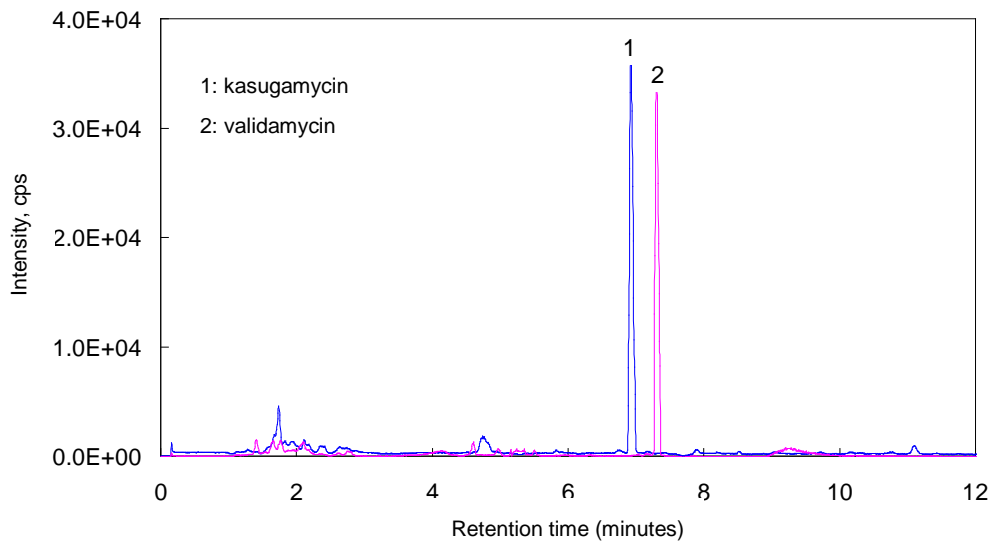


Figure 4. Chromatogram of tea leaf (spiked with 10ng/g each) extract



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