

Effective method development and purification using highly durable semi-preparative column

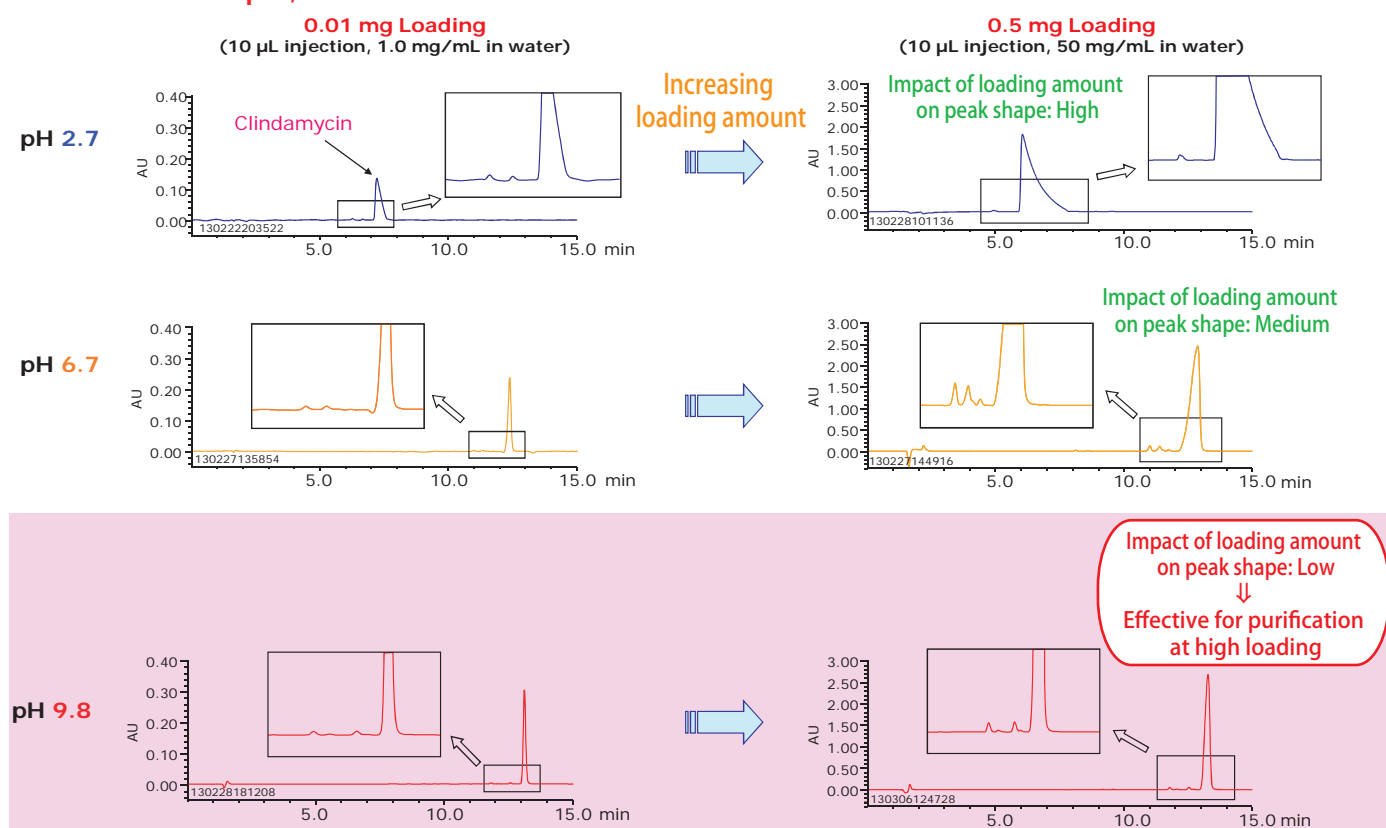
~Purification of basic drug: Clindamycin~

S130502AE

[Method development on analytical scale]

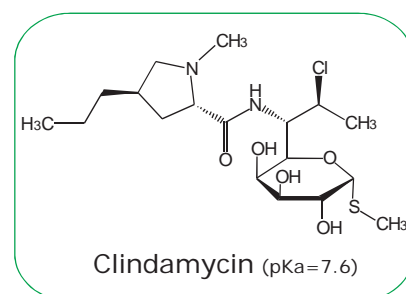
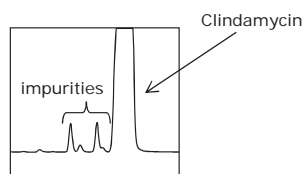
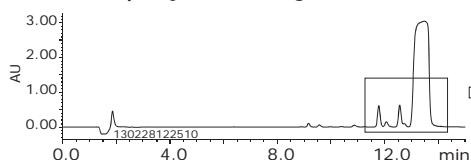
pH screening of purification condition

YMC-Triart C18 5 μ m, 150 X 4.6 mm I.D.



Optimization of loading amount

pH 9.8 **2.5 mg Loading**
(50 μ L injection, 50 mg/mL in water)



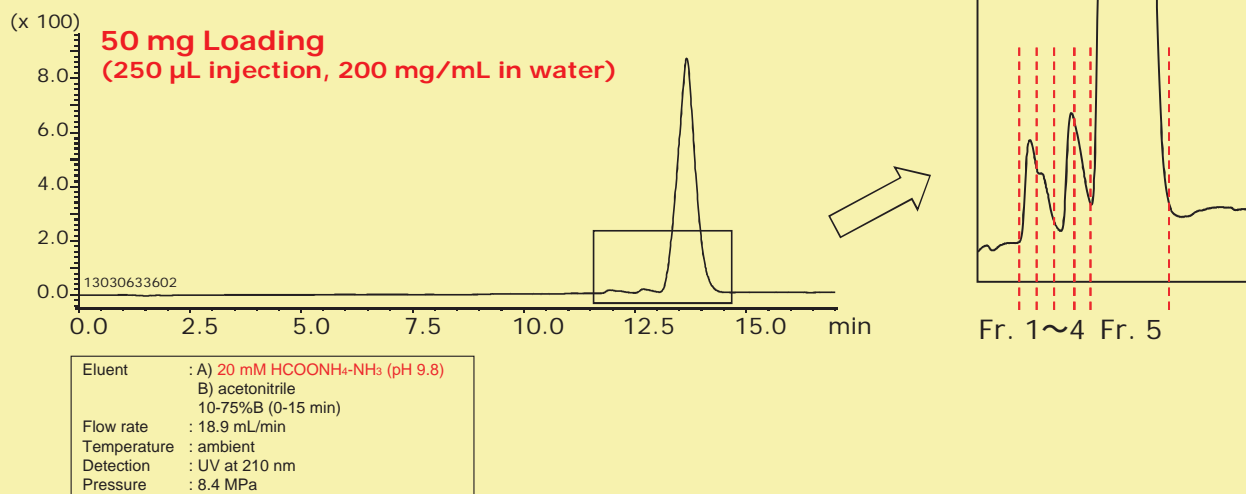
Scaling up to Semi-preparative scale

Eluent	: A) 20 mM HCOOH for pH 2.7 20 mM HCOONH ₄ for pH 6.7 20 mM HCOONH ₄ -NH ₃ for pH 9.8
	B) acetonitrile 10-75%B (0-15 min)
Flow rate	: 1.0 mL/min
Temperature	: 25°C
Detection	: UV at 210 nm
Pressure	: 7.0 MPa

Clindamycin is a basic antibiotic that is usually used to treat infections with anaerobic bacteria. Clindamycin and its impurities (related compounds) are more hydrophobic in their un-ionized form and are retained stronger at pH 9.8. At higher pH condition, the resolution between main peak and impurities is improved and the peak shape is less affected by increase of mass loading. Excellent chemical durability of YMC-Triart offers an option of purification at a high pH that is effective for basic compounds by increasing retention and mass loading.

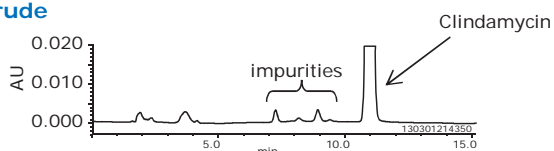
[Scaling up from analytical to semi-preparative]

YMC-Actus Triart C18 5 μ m, 150 X 20 mm I.D.

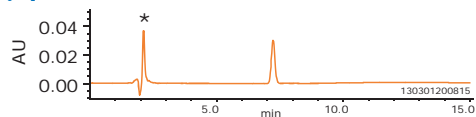


[Analyses of fractions]

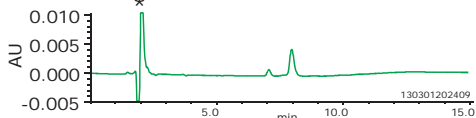
Crude



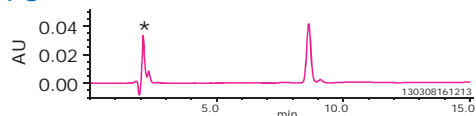
Fr. 1



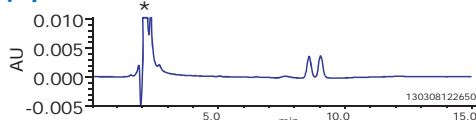
Fr. 2



Fr. 3

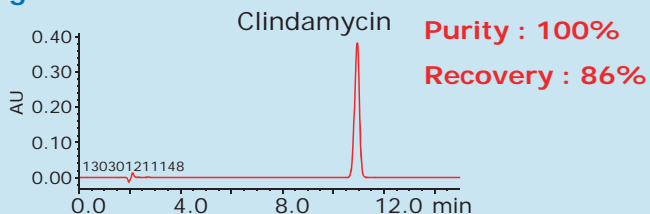


Fr. 4



*: solvent peak

Fr. 5



Column	: YMC-Triart C18 5 μ m
	150 X 4.6 mm I.D.
Eluent	: 50 mM KH ₂ PO ₄ (pH 7.5 adjusted by 8 M KOH)/acetonitrile (55/45)
Flow rate	: 1.0 mL/min
Temperature	: 25°C
Detection	: UV at 210 nm
Injection	: 20 μ L

The analytical method for Clindamycin is linearly scaled up to semi-preparative column by adjusting loading amount and flow rate in accordance with the ratio of cross sectional areas of both columns. YMC-Actus Triart C18 has excellent durability and separation ability provided by its high packing density. In addition, it has identical selectivity to analytical Triart C18. These features enable predictable and highly efficient scale up to semi-preparative purification. The combination of YMC-Triart and YMC-Actus offers highly efficient purification of various compounds.