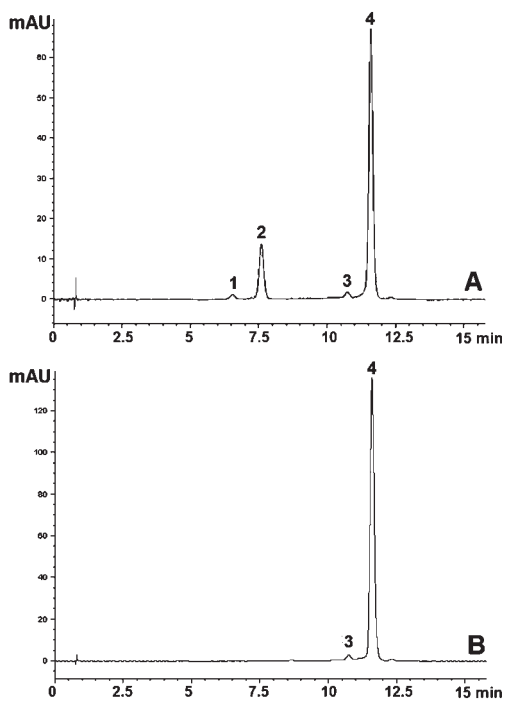
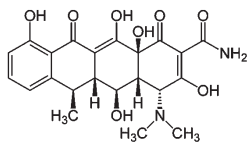
Doxycycline Analysis

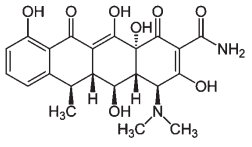
Forced degradation method using shape selectivity



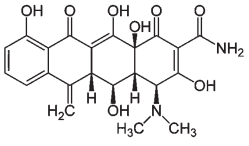
Overlay of 5 runs



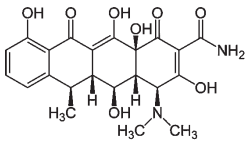
4-epidoxycycline



6-epidoxycycline



methacycline



doxycycline

Note: Doxycycline is a member of the tetracycline class of antibiotics. It can be used to treat various kinds of bacterial infections.

Method Conditions

Column: **Cogent UDC-Cholesterol™**, 4µm, 100Å

Catalog No.: 69069-7.5P

Dimensions: 4.6 x 75 mm

Solvents: A: DI H_2O / 0.1% TFA
B: Acetonitrile / 0.1% TFA

Gradient:

time (min.)	%B
0	5
12	30
13	5

Temperature: 25˚C

Post time: 3 min

Injection vol.: 20µL

Flow rate: 1.5 mL/min

Detection: UV 350 nm

Figures: A.Doxycycline forced degradation extract
B.Doxycycline non-degraded extract

Samples: 100 mg strength doxycycline hyclate tablets were ground and added to 50 mL volumetric flasks. Portions of diluent were added and the flasks were sonicated 10 min. After diluting to mark, portions were filtered (0.45µm, nylon) and each diluted 1:200. The diluents used were 50% 1N HCl / 50% solvent B for Fig. A and 50% solvent A / 50% solvent B for Fig. B. The extract used in Fig. A was also heated at 80°C for 30 min.

Peaks: 1. Epimer degradant
2. Epimer degradant
3. Methacycline
4. Doxycycline

t_0: 0.8 min

Discussion

Doxycycline is known to epimerize at the C4 and C6 positions in acid conditions. Epimers can be difficult to separate in reverse phase due to subtle differences in hydrophobicity. However, the Cogent UDC-Cholesterol column can differentiate based on shape as well as hydrophobicity and is therefore well suited to this isomer separation.

The method not only meets the USP system suitability requirements for resolution between the API and both epimer degradants, but also demonstrates resolution from the methacycline impurity.

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