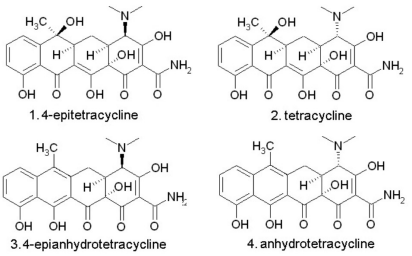
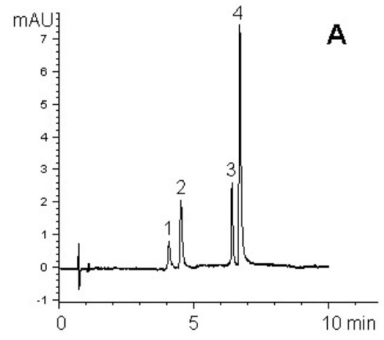
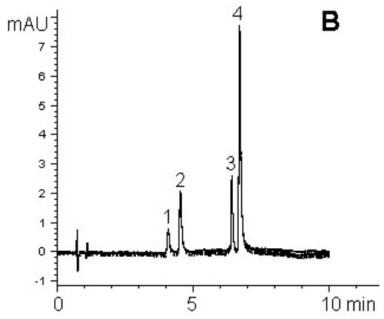
Stability Testing of Tetracycline HCl Capsules

Robust, high-throughput separation of API from its degradation products







Notes: Tetracycline is a broad-spectrum antibiotic widely used in both human and veterinary medicine. It is known to degrade primarily by two pathways: Dehydration and epimerization.

Method Conditions

Column: **Cogent Bidentate C18™**, 4µm, 100Å

Catalog No.: 40018-75P

Dimensions: 4.6 x 75 mm

Solvents: A: DI H_2O/ 0.1% formic acid
B: Acetonitrile/ 0.1% formic acid

Gradient:

time (min.)	%B
0	10
4	30
6	80
7	10

Post Time: 3 min

Flow rate: 1 mL/min

Detection: UV 360 nm (0–5 min) 430 nm (5–7 min)

Sample: Stock Solution: 10 mg capsule contents were diluted with 10 mL 1.0 HCl and sonicated for 10 minutes. 1 mL aliquot was diluted to 20 mL with DI H_2O. Solution was heated at 80°C for 30 minutes. Solution was then diluted to 100 mL with DI H_2O and filtered through a 0.45µm nylon membrane HPLC filter (MicroSolv Technology). Stock solution was diluted 10x using 0.01 N HCl diluent.

Peaks: 1. 4-epitetracycline
2. Tetracycline
3. 4-epianhydrotetracycline
4. Anhydrotetracycline

Discussion

Figure A shows the chromatogram obtained from a single injection of the degraded tetracycline capsule extract. Tetracycline is well-resolved from its three main degradation products, the identities of which were confirmed by individual standards under non-degrading conditions. Amine-containing analytes such as these often require the use of an ion-pairing agent in the mobile phase in order to reduce peak tailing from silanolic interactions. However, ion-pairing agents often lead to poor reproducibility and long equilibration times due to slow uptake and release of these agents from the column. TYPE-C Silica based columns have most of the surface silanols replaced, and therefore ion- pairing agents are not necessary to obtain good peak shapes. Figure B shows an overlay of five sequential injections of the degraded tetracycline solution, illustrating the good repeatability of the method. Retention time %RSDs for all of the analytes were < 0.1%. In addition, the post time was minimal (3 min).

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