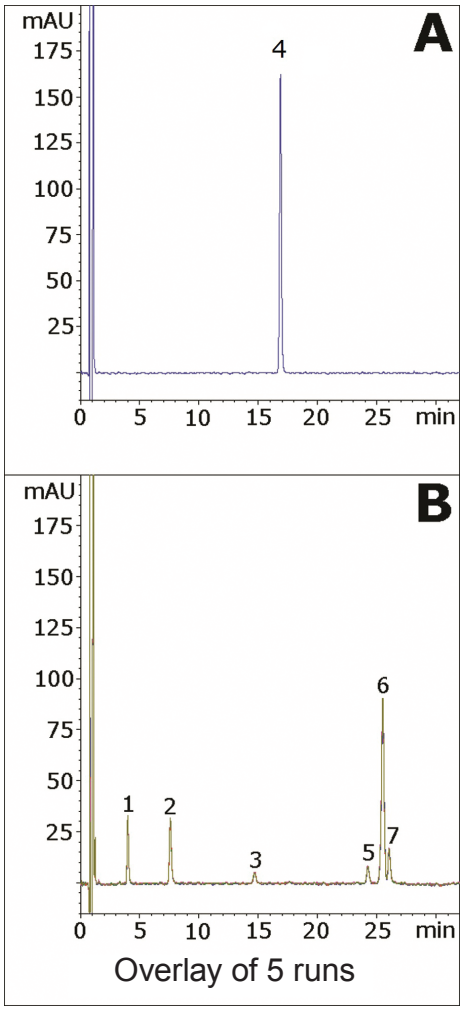
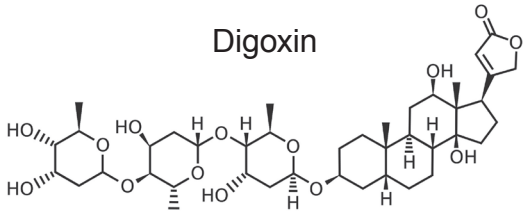
Forced Degradation of Digoxin

Separation of API from acid degradation products





Note: Digoxin is a cardiac glycoside used to treat a number of heart conditions. It is a natural product obtained from the foxglove plant (Digitalis Purpurea). Accurate quantitation of digoxin is crucial since it is very toxic at higher levels.

Method Conditions

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

Catalog No.: 40018-75P

Dimensions: 4.6 x 75 mm

Mobile Phase: A: DI H_2O / 0.1% formic acid (v/v)
B: Acetonitrile / 0.1% formic acid (v/v)

Gradient:

time (min.)	%B
0	15
2	15
26	37
28	80
29	15

Post Time: 3 min

Injection vol.: 10µL

Flow rate: 1.0 mL/min

Detection: UV 218 nm

Samples: **Fig. A** (Non-degraded): 0.1 mg/mL digoxin in methanol diluent.
Fig. B (Acid degradation): 0.1 mg/mL digoxin in 50/50 methanol / 1 N HCl diluent. Sample was heated at 85°C for 10 min.

Peaks: 1–3. Degradants
4. Digoxin
5–7. Degradants

t_0: 0.9 min

Discussion

Digoxin had several sugar residues that can be hydrolyzed in acidic conditions. In this forced degradation method, six degradants are observed. Some of these peaks are likely the various hydrolyzed forms, including the aglycone digoxigenin. The method illustrates how the Cogent Bidentate C18 column is capable of separating a wide variety of degradants. Figure A shows the chromatogram of the non-degraded sample while Figure B shows 5 consecutive runs of the acid-degraded extract.

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