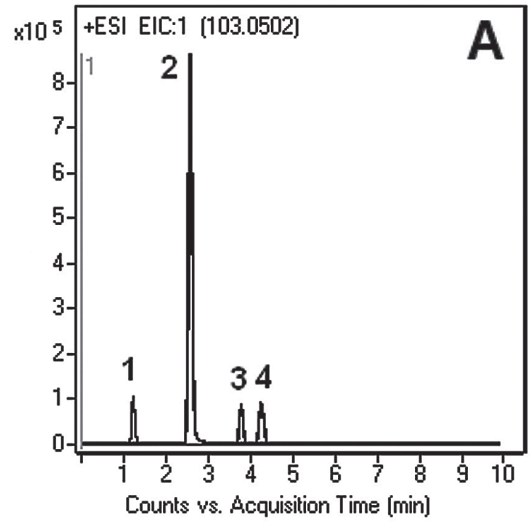
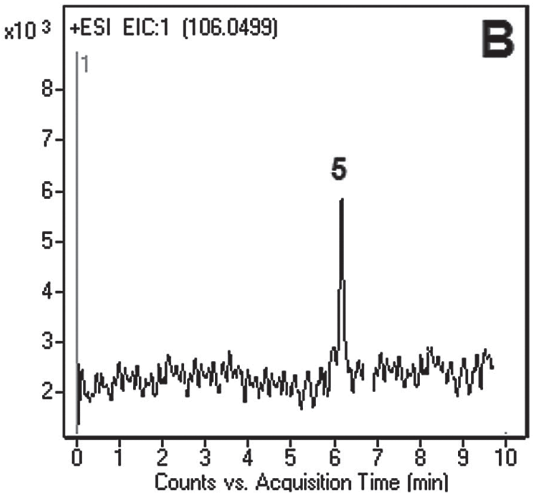
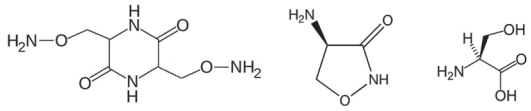
Cycloserine

LC-MS separation of impurities and degradants







3. Cycloserine dimer 4. Cycloserine 5. Serine

Note: Until recently, cycloserine has not been in wide-spread use for the treatment of tuberculosis due to its toxicity. With more drug-resistant strains of TB emerging, cycloserine treatment is becoming more common.

Method Conditions

Column: **Cogent Diamond Hydride™**, 4µm, 100Å

Catalog No.: 70000-15P-2

Dimensions: 2.1 x 150 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	70
2	20
6	20
7	70

Post Time: 2 min

Injection vol.: 1µL

Flow rate: 0.4 mL/min

Detection: ESI – POS - Agilent 6210 MSD TOF mass spectrometer

Sample: Stock solutions of the analytes were made in DI H_2O in the range of 0.2–0.7 mg/mL. All samples were filtered through a disposable 0.45µm filter (MicroSolv Tech Corp.). Samples for injection were diluted 1:10 with 50:50 solvent A:B mixture.

Peaks: 1. Unknown at m/z 285
2. Unknown at m/z 245
3. Cycloserine dimer at m/z 205.0931
4. Cycloserine at m/z 103.0502 (Fig. A)
5. Serine at m/z 106.0499 (Fig. B).

t_0: 0.9 min

Discussion

While two major impurities observed in this cycloserine method were not directly identified, several possibilities can be suggested based on their *m/z* values in the mass spectrum and their relationship to the serine structure. Serine was also identified but in very low abundance and only after the samples were several weeks old.

This study did not involve the development of a fully validated method; however a linear relationship following the equation 2E+6x - 280000 was obtained for the determination of cycloserine over the concentration range of 0.2–1.0µg/mL having an R^2 value of 0.993. The limit of detection is estimated to be 0.1µg/mL. The repeatablility of the method, both inter and intra-day, is excellent.

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