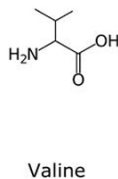
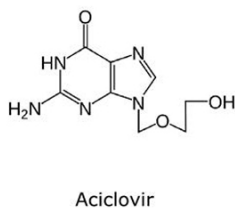
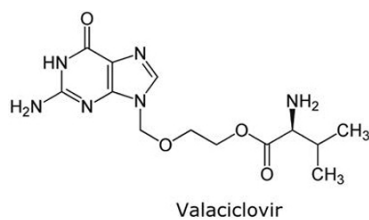
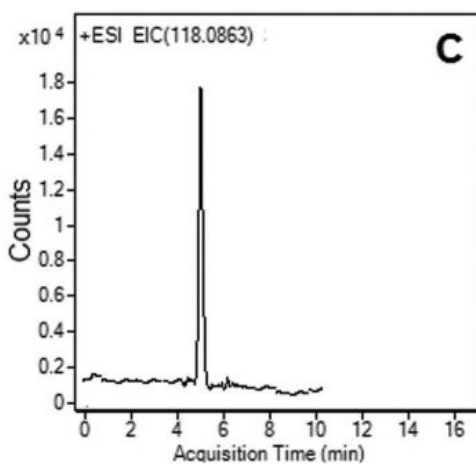
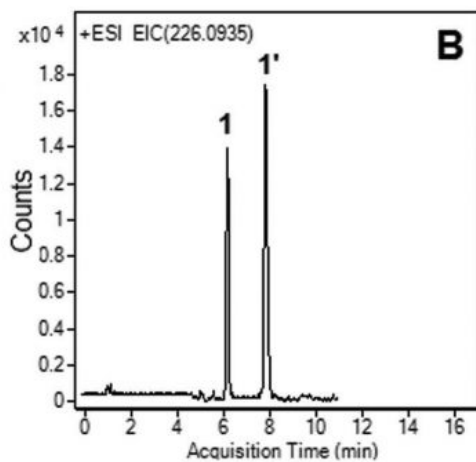
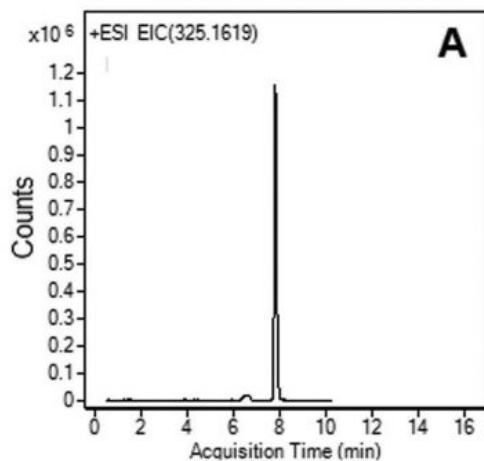


Valaciclovir & Degradants Analyzed with LCMS – AppNote

Separation of Valaciclovir from two Degradants

The anti-viral Herpes drug Valaciclovir and its degradation products are analyzed using Aqueous Normal Phase HPLC with MS detection. The Method development was completed using UV detection, where excellent Separation between the Prodrug Valaciclovir and its active converted degradant, Acyclovir, was observed.

The Method transfer from UV to MS detection was accomplished using only a change of the flow rate. When MS detection was used, a second Degradant (D,L-Valine, formed from hydrolysis of the prodrug) was also detected without derivatization.



Peaks:

Figure A: Valaciclovir 325.1619 m/z

Figure B: Acyclovir 226.0935 [M+H]⁺ (degradant / active form)

Peaks 1 and 1' have the same mass. 1' is the Peak that corresponds to the Acyclovir formed in the source

Figure C: D,L-Valine 118.0863 m/z [M+H]⁺

Method Conditions

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

Catalog No.: 70000-15P-2

Dimensions: 2.1 x 150mm

Mobile Phase:

A: DI Water with 0.1% Formic Acid (v/v)

B: Acetonitrile with 0.1% Formic Acid (v/v)

Gradient:

| Time (minutes) | %B |
|----------------|----|
| 0 | 95 |
| 1 | 95 |
| 6 | 40 |
| 7 | 40 |
| 8 | 95 |

Post Time: 3 minutes

Injection vol.: 1µL

Flow rate: 0.4mL / minute

Detection: ESI - POS - Agilent 6210 MSD TOF Mass Spectrometer

Sample Preparation: Stock Solution: 1000mg strength Valtrex® tablet was ground and added to 100mL volumetric flask containing 50mL 50:50 DI Water / Acetonitrile diluent. The solution was sonicated 10 minutes, diluted to mark, and mixed. A portion was filtered through a 0.45µm Nylon Syringe Filter (MicroSolv Tech Corp.). The stock solution was diluted 1:100 with 50:50 Acetonitrile / DI Water (*Figure A*). Another aliquot was diluted 1:100 with 50:50 1N HCL / Acetonitrile mixture and heated at 85°C for 30 minutes (*Figures B and C*).

t₀: 0.9 minutes



Attachment

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