

## Analysis of Fluoroquinolones by LC-MS-MS

Instrumentation: [Shimadzu LC System](#) with [AB Sciex LC/MS/MS System](#)

Column: [Waters XBridge C18 2.5µm 2.1 X 50mm Column](#) ([Ctrl + Click to follow link](#))

Elution Type: Gradient

Mobile Phase A: Water with 0.1% Formic Acid

Mobile Phase B: Methanol

Gradient Profile:

Step No.	Time (min)	Pct A	Pct B
1	0	85	15
3	6	10	90
4	6	10	90

Flow Rate: 0.4 mL/min

Col. Temp: 40 °C

Detection: [Tandem Mass Spec \(MS-MS\) @ amu \(550 °C\)](#) ([Ctrl + Click to follow link](#))

Detector Info: [AB Sciex API 4000](#) ([Ctrl + Click to follow link](#))

**MS Conditions**

Source temp: 550 °C

Dwell: 25 msec

Polarity: Positive

**Sample Preparation:**

**Solid Phase Extraction (SPE) procedure:** Note: The solvent volumes shown below are for a 200 mg bed mass. The solvent volumes will need to be adjusted for a smaller or larger bed mass.

**Condition:**

1. Methanol 3 mL @ 2 mL/min
2. Water 3 mL @ 2 mL/min

**Load:** Dissolve 125mg of propolis in 5mL of MTBE.

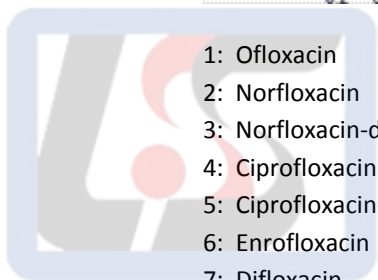
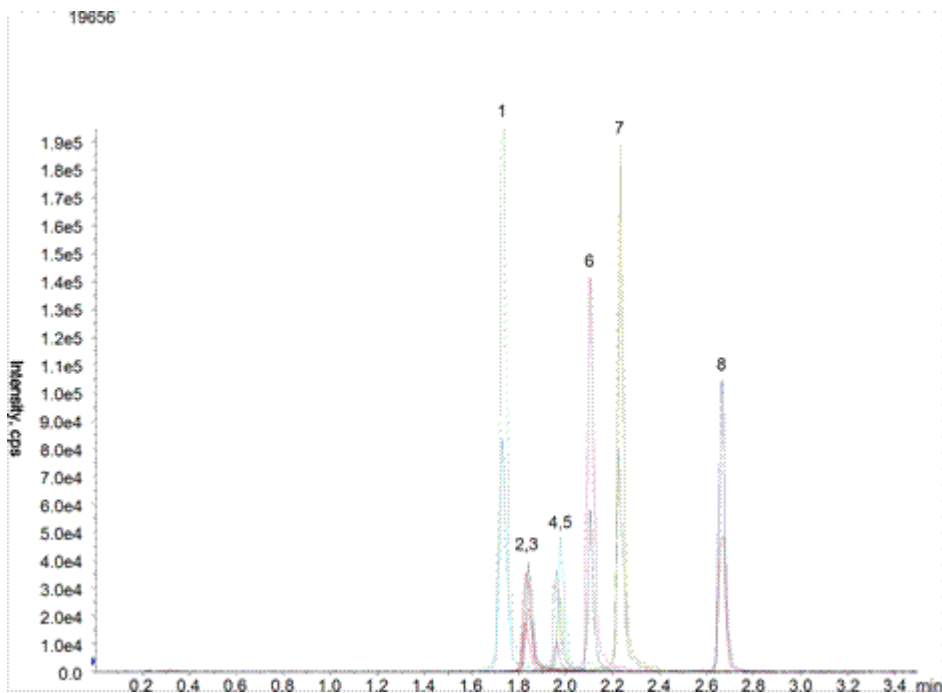
Using Phosphate buffer with 2% TCA, extract analytes from propolis by 2X liquid:liquid extraction.

Aqueous fraction is then ready for loading onto SPE cartridge.

**Dry:** 5min @ >10 inches Hg

**Elute:** Methanol/ACN/Acetic acid 49/49/2, 2 mL @ 1 mL/min

**Final Prep and Analysis:** Inject: 20 µL on HPLC / Mass Spectrometer (MS) @ amu (550 °C)



- 1: Ofloxacin
- 2: Norfloxacin
- 3: Norfloxacin-d5
- 4: Ciprofloxacin
- 5: Ciprofloxacin-d8
- 6: Enrofloxacin
- 7: Difloxacin
- 8: Sparfloxacin

# SpectralLab

Scientific Incorporation

**Reference:** <http://www.phenomenex.com/Application/Detail/19656>