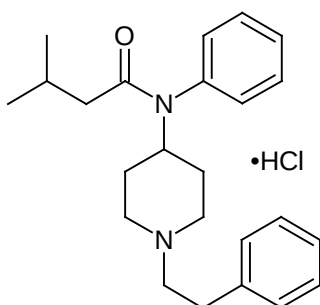


BATTELLE CERTIFICATE OF ANALYSIS



Name: Isovaleryl fentanyl (HCl)

Supplier: Cayman Chemical

Catalog No.: 22990

Lot #: N/A

Chemical Formula: $\text{C}_{24}\text{H}_{32}\text{N}_2\text{O}$ (free base)
 $\text{C}_{24}\text{H}_{33}\text{ClN}_2\text{O}$ (salt)

Molecular Weight: 364.52 (free base)
400.98 (salt)

Analytical Results

Monoisotopic Exact Mass: 365.2587 (calc. for $[\text{M}+\text{H}]^+$)
365.2590 (obsd. ESI-MS)

Isotopic Purity: N/A

Date of COA: April 4, 2019

COA Prepared by: E. A. Lucas

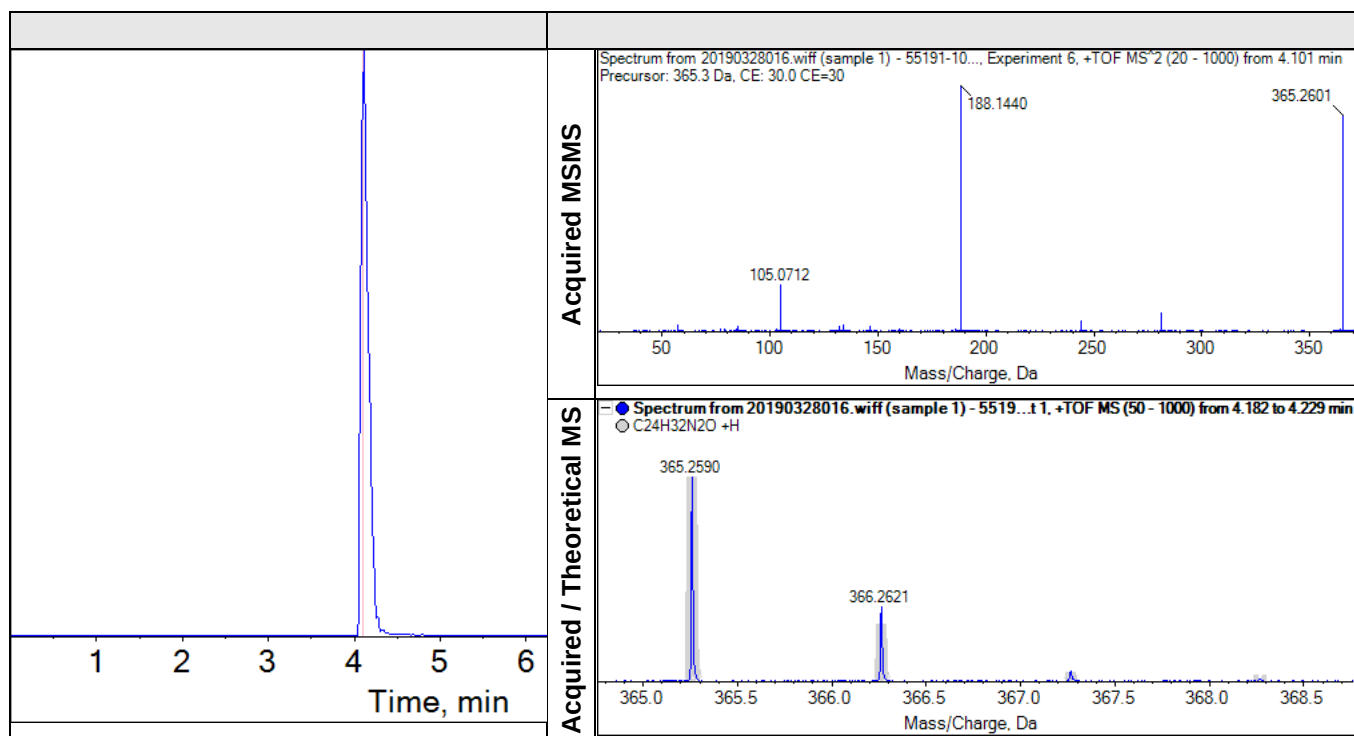


Figure 1: LC-QTOF Analysis of 22990

Table 1: LC-QTOF Conditions Used to Analyze 22990

Mass Spectrometer	Sciex TripleTOF 5600										
UPLC Hardware	Waters Acquity I-Class										
Mass Spectrometer Source	Turbolonspray, Positive										
Scan Ranges	TOF-MS: 50-1000 m/z MS/MS: 20-1000 m/z										
UPLC Column	Waters Acquity BEH C18, 2.1 × 50 mm, 1.7 μm										
Column Temperature	40°C										
Mobile Phase A	0.1% Formic Acid in Water										
Mobile Phase B	0.1% Formic Acid in Acetonitrile										
Gradient Table	<table> <tr> <th>Time, min</th><th>%B</th></tr> <tr> <td>0 – 0.5</td><td>5</td></tr> <tr> <td>0.5 – 5</td><td>Ramp 5 to 100</td></tr> <tr> <td>5 – 5.5</td><td>100</td></tr> <tr> <td>5.51 – 10</td><td>5</td></tr> </table>	Time, min	%B	0 – 0.5	5	0.5 – 5	Ramp 5 to 100	5 – 5.5	100	5.51 – 10	5
Time, min	%B										
0 – 0.5	5										
0.5 – 5	Ramp 5 to 100										
5 – 5.5	100										
5.51 – 10	5										
Flow Rate	0.3 mL/min										
Injection Volume	5 μL										
Run Time	10 minutes										

Abundance

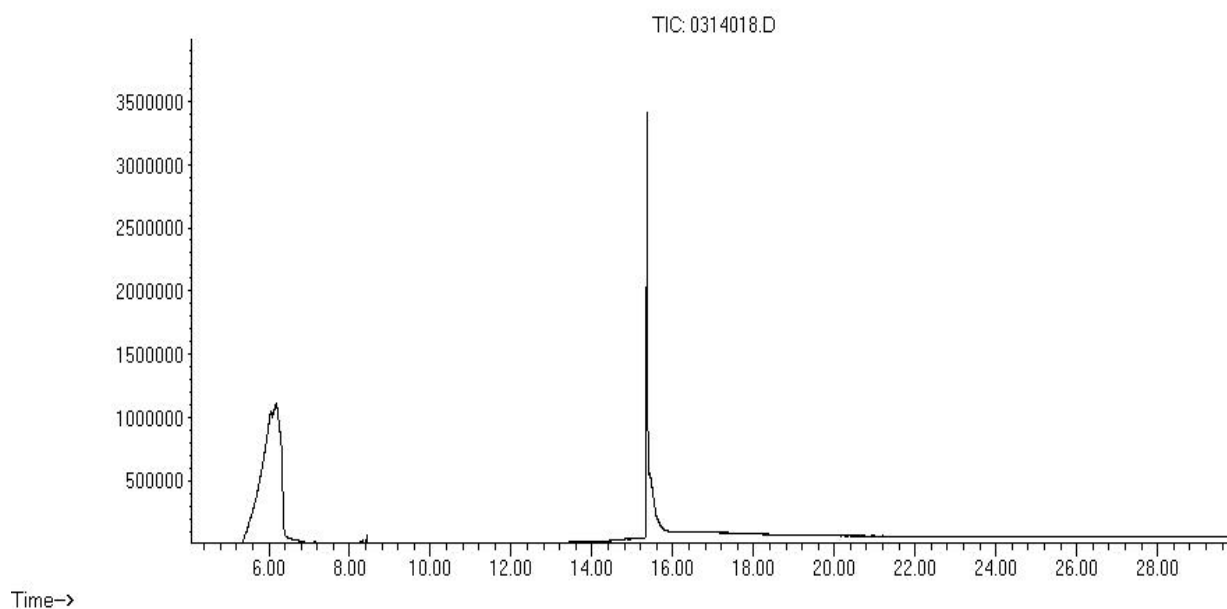
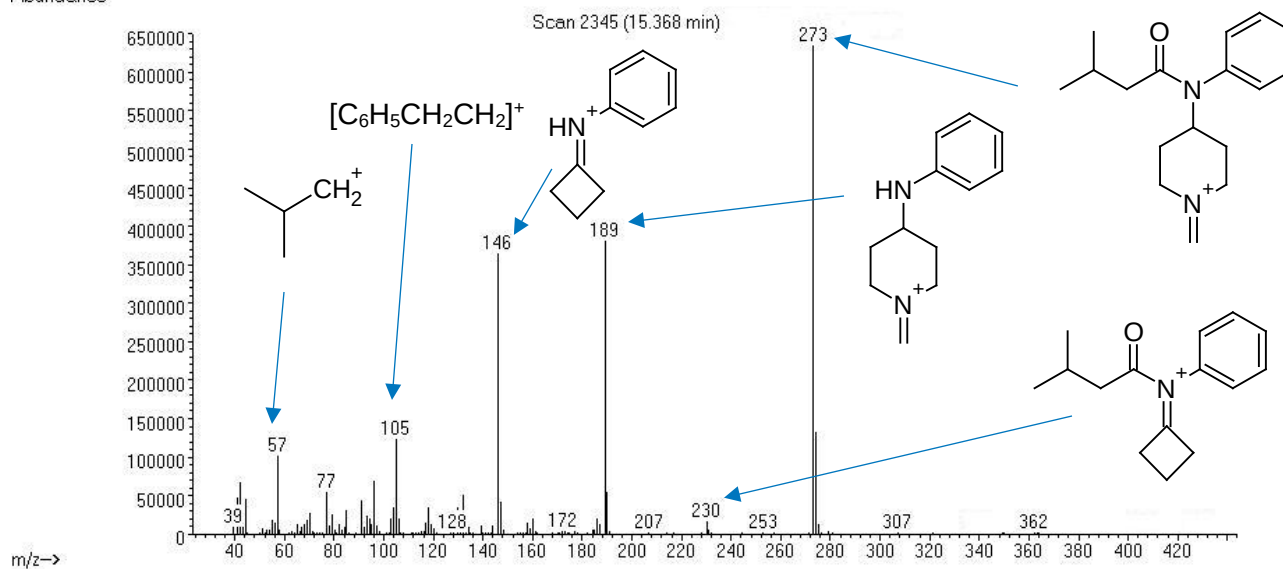


Figure 2. GC-MS Chromatogram (EI, TIC) of 22990

Abundance



**Figure 3. Mass Spectrum (EI) of 22990 (Main Analyte)
Fragment Structure Assignments are Tentative and Unconfirmed**

Table 2. GC-MS Conditions Used to Analyze 22990

Instrument	Agilent 6890 GC/5973 MSD
Carrier Gas:	Helium
Column:	Restek Rtx-5MS 30m x 0.25mm x 0.25um
Oven Program:	45°C hold for 1.0 min 20°C/min. to 320°C, hold for 15 min.
Total Run Time:	30 minutes
Transfer Line Temp.:	280°C
Injection:	Cool-on-column
Injection Port Temperature:	Tracking oven temperature
Carrier Flow Rate:	1.6 mL/min.
Injection Volume:	0.5 uL
Acquisition Mode:	EI
Scan Range:	35 to 500 amu
Electron Impact:	70 eV
MS Quad Temperature:	150°C
MS Source Temperature:	230°C

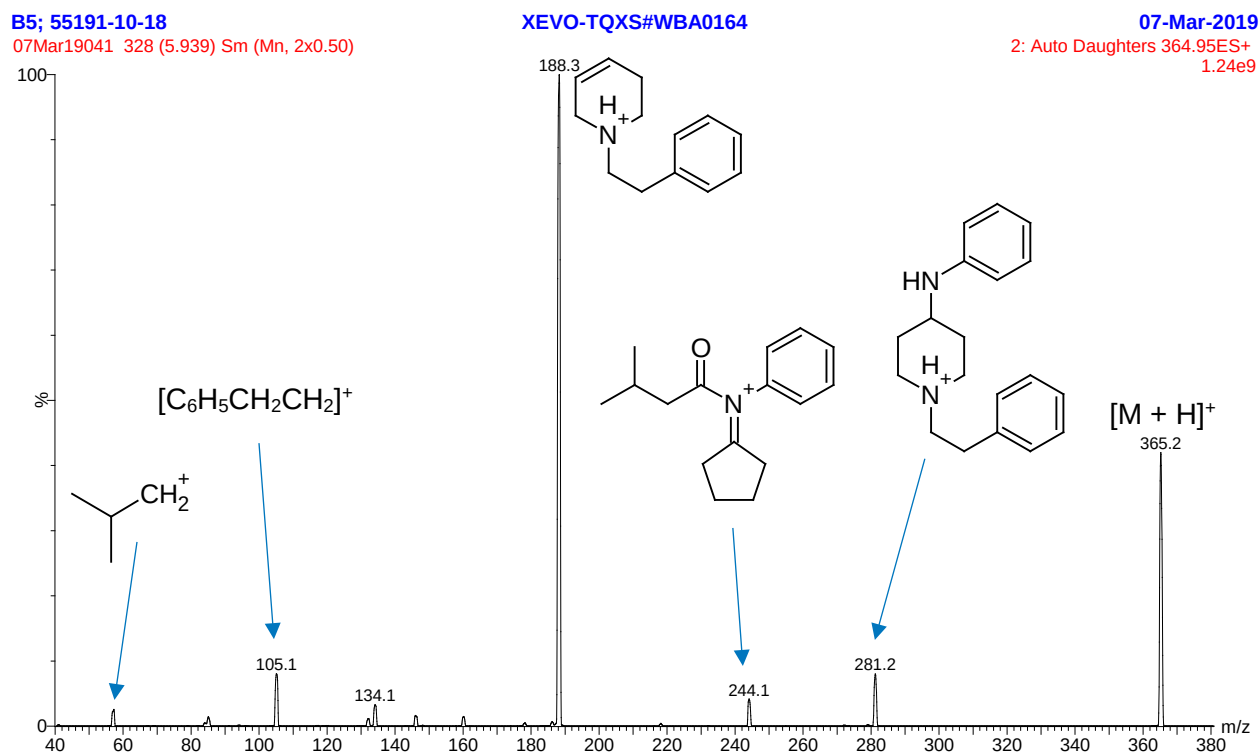


Figure 5. Low-resolution MS/MS (ESI, product ions of m/z 365) of 22990
Fragment Structure Assignments are Tentative and Unconfirmed

Table 3. LC-MS/MS-UV Conditions Used to Analyze 22990

Mass Spectrometer	Waters TQ-XS																	
UPLC Hardware	Waters Acquity I-Class																	
Mass Spectrometer Source	Electrospray, Positive Ionization																	
Photo Diode Array	Waters Acquity PDA																	
Scan Ranges	MS: 40-700 m/z PDA: 210-300 nm																	
Mass Selected for MS/MS fragmentation	Observed mass ± 0.25 Da																	
UPLC Column	Phenomenex Prodigy ODS (3), 100mm x 2.0mm, 3µm																	
Column Temperature	50°C																	
Mobile Phase A	1% Formic Acid in Water																	
Mobile Phase B	1% Formic Acid in Acetonitrile																	
Gradient Table	<table><tr><th>Time, min</th><th>%B</th><th>Flow, mL/min</th></tr><tr><td>0 – 1</td><td>2</td><td>0.3</td></tr><tr><td>1 – 11</td><td>Ramp 2 to 100</td><td>0.3</td></tr><tr><td>11.1 – 12</td><td>100</td><td>0.5</td></tr><tr><td>12.1 – 14</td><td>2</td><td>0.3</td></tr></table>			Time, min	%B	Flow, mL/min	0 – 1	2	0.3	1 – 11	Ramp 2 to 100	0.3	11.1 – 12	100	0.5	12.1 – 14	2	0.3
Time, min	%B	Flow, mL/min																
0 – 1	2	0.3																
1 – 11	Ramp 2 to 100	0.3																
11.1 – 12	100	0.5																
12.1 – 14	2	0.3																
Injection Volume	25 µL																	
Run Time	14 minutes																	