BATTELLE CERTIFICATE OF ANALYSIS

* = ^{13}C label

Name:	Valeryl fentanyl-$^{13}C_6$ HCl
Supplier:	Cerilliant
Catalog No.:	V-075
Lot #:	N/A
Chemical Formula:	$^{13}C_6C_{18}H_{32}N_2O$ (free base) $^{13}C_6C_{18}H_{33}ClN_2O$ (salt)
Molecular Weight:	370.48 (free base) 406.94 (salt)

Analytical Results

Monoisotopic Exact Mass:	371.2789 (calc. for $[M+H]^+$) 371.2791 (obsd. ESI-MS)
Isotopic Purity:	<0.4% unlabeled
Date of COA:	September 12, 2019

COA Prepared by: E. A. Lucas

505 King Avenue | Columbus, Ohio 43201-2696 | 614.424.7719 | lucasea@battelle.org | battelle.org

Figure 1: LC-QTOF Analysis of V-075

Table 1: LC-QTOF Conditions Used to Analyze V-075

Mass Spectrometer	Sciex TripleTOF 5600
UPLC Hardware	Waters Acquity I-Class
Mass Spectrometer Source	TurboIonSpray, 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	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

Figure 2. GC-MS Chromatogram (EI, TIC) of V-075

Figure 3. Mass Spectrum (EI) of V-075 (Main Analyte)
Fragment Structure Assignments are Tentative and Unconfirmed

Table 2. GC-MS Conditions Used to Analyze V-075

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 371) of V-075
Fragment Structure Assignments are Tentative and Unconfirmed

Figure 6. Extracted Ion Chromatograms: Labeled (m/z 371) and Unlabeled (Inset, m/z 365) Vertical Scale of Inset is Expanded ca. 1000x

Table 3. LC-MS/MS-UV Conditions Used to Analyze V-075

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	

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