# BATTELLE CERTIFICATE OF ANALYSIS

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|---|---|
| Name: | (±)-*cis*-3-Methyl fentanyl HCl |
| Supplier: | Cerilliant |
| Catalog No.: | M-222 |
| Lot #: | N/A |
| Chemical Formula: | $C_{23}H_{30}N_2O$ (free base)<br>$C_{23}H_{31}ClN_2O$ (salt) |
| Molecular Weight: | 350.50 (free base)<br>386.96 (salt) |

**Analytical Results**

| | |
|---|---|
| Monoisotopic Exact Mass: | 351.2431 (calc. for $[M+H]^+$)<br>351.2428 (obsd. ESI-MS) |
| Isotopic Purity: | N/A |
| Date of COA: | September 9, 2019 |

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**Figure 1: LC-QTOF Analysis of M-222**

**Table 1: LC-QTOF Conditions Used to Analyze M-222**

| | |
|---|---|
| Mass Spectrometer | Sciex TripleTOF 5600 |
| UPLC Hardware | Waters Acquity I-Class |
| Mass Spectrometer Source | TurboIonSpray, Positive |
| Scan Ranges | TOF-MS: 50-1000 m/z<br>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<br>0 – 0.5 / 5<br>0.5 – 5 / Ramp 5 to 100<br>5 – 5.5 / 100<br>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 M-222**

**Figure 3. Mass Spectrum (EI) of M-222 (Main Analyte)**
**Fragment Structure Assignments are Tentative and Unconfirmed**

**Table 2. GC-MS Conditions Used to Analyze M-222**

| | |
|---|---|
| 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<br>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 351) of M-222**
**Fragment Structure Assignments are Tentative and Unconfirmed**

**Table 3. LC-MS/MS-UV Conditions Used to Analyze M-222**

| | |
|---|---|
| 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<br>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 |