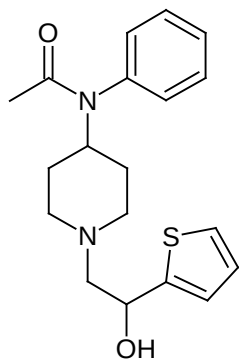


## BATTELLE CERTIFICATE OF ANALYSIS

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Name:  $\beta$ -hydroxy thio  
acetyl fentanyl

Supplier: Cayman Chemical

Catalog No.: 24280

Lot #: N/A

Chemical Formula:  $C_{19}H_{24}N_2O_2S$

Molecular Weight: 344.47

### Analytical Results

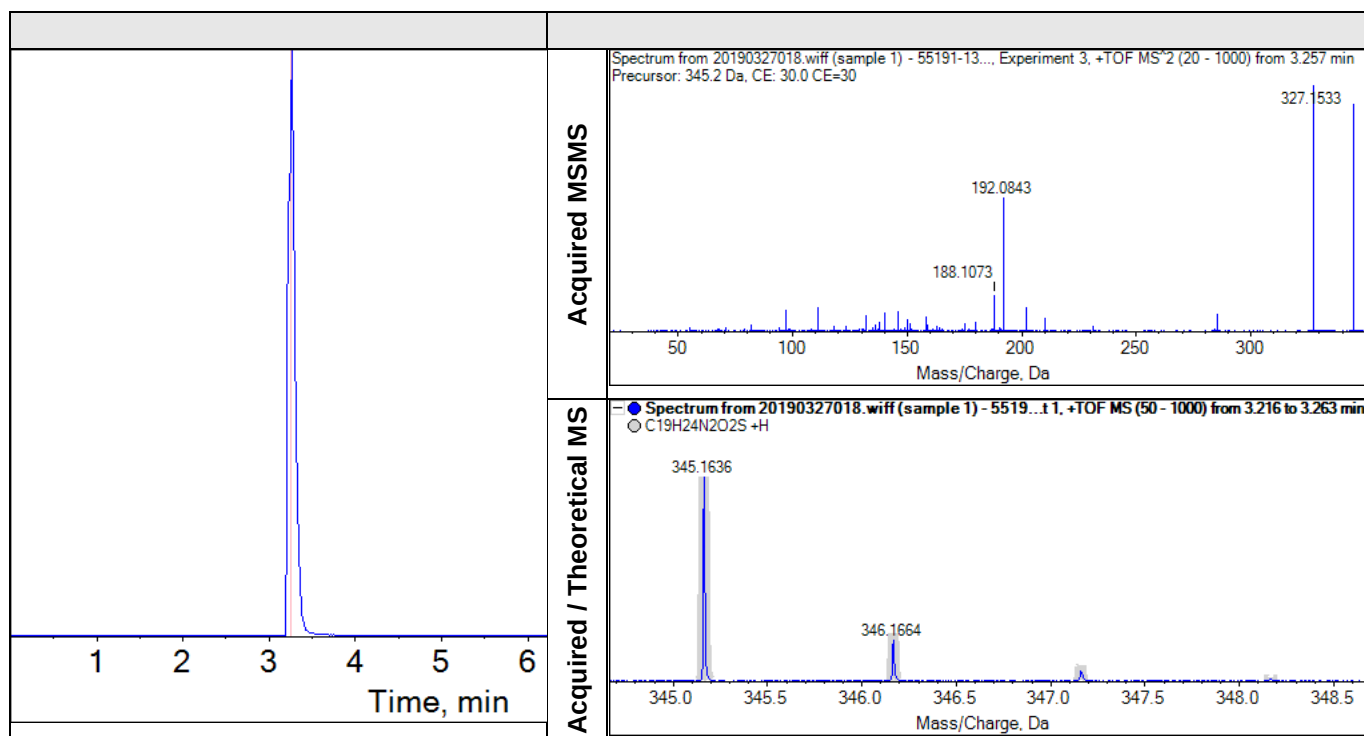
Monoisotopic Exact Mass: 345.1631 (calc. for  $[M+H]^+$ )  
345.1636 (obsd. ESI-MS)

Isotopic Purity: N/A

Date of COA: April 10, 2019

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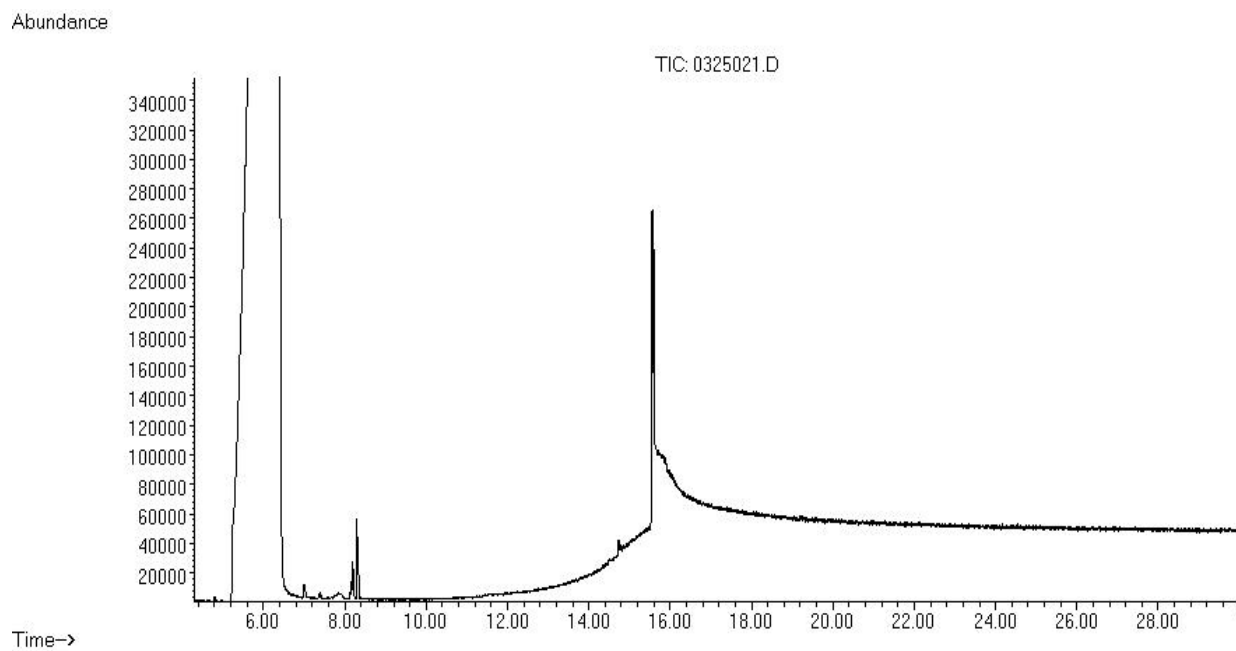
COA Prepared by: E. A. Lucas



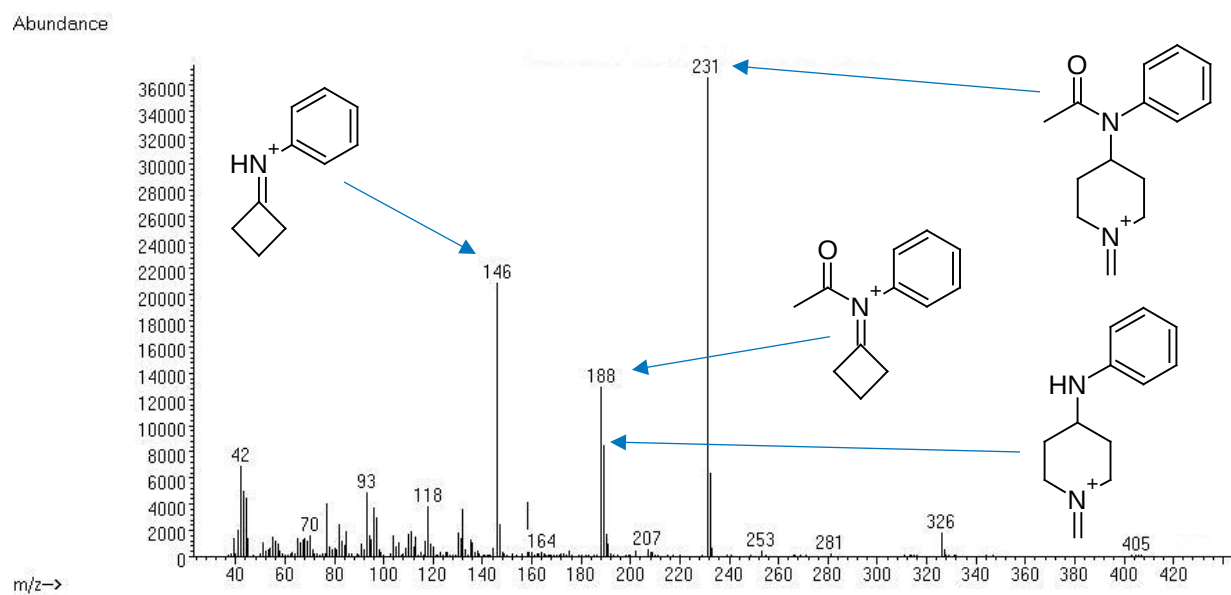
**Figure 1: LC-QTOF Analysis of 24280**

**Table 1: LC-QTOF Conditions Used to Analyze 24280**

| Mass Spectrometer        | Sciex TripleTOF 5600                                                                                                                                                                                                                                                      |           |    |         |   |         |               |         |     |           |   |
|--------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|----|---------|---|---------|---------------|---------|-----|-----------|---|
| UPLC Hardware            | Waters Acquity I-Class                                                                                                                                                                                                                                                    |           |    |         |   |         |               |         |     |           |   |
| Mass Spectrometer Source | TurbolonSpray, 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           | <table border="1"> <thead> <tr> <th>Time, min</th><th>%B</th></tr> </thead> <tbody> <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> </tbody> </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                                                                                                                                                                                                                                                                |           |    |         |   |         |               |         |     |           |   |



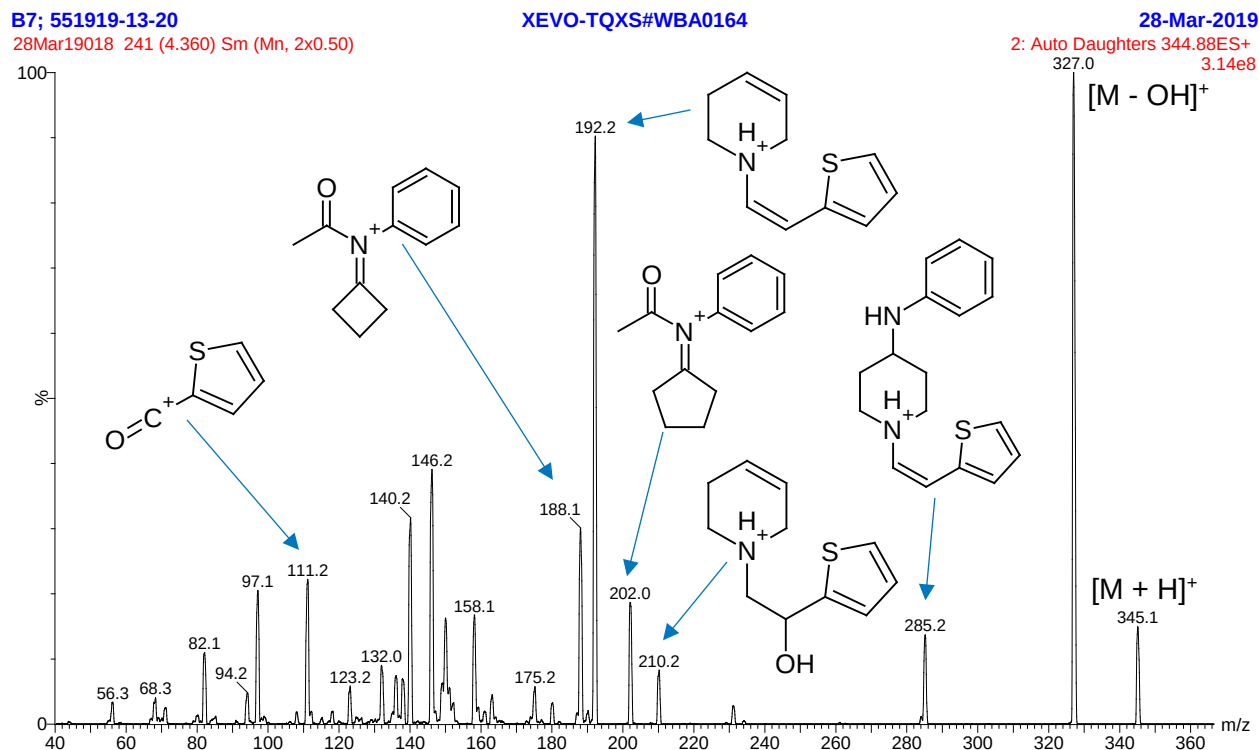
**Figure 2. GC-MS Chromatogram (EI, TIC) of 24280**



**Figure 3. Mass Spectrum (EI) of 24280 (Main Analyte)  
Fragment Structure Assignments are Tentative and Unconfirmed**

**Table 2. GC-MS Conditions Used to Analyze 24280**

|                             |                                                               |
|-----------------------------|---------------------------------------------------------------|
| 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 345) of 24280  
Fragment Structure Assignments are Tentative and Unconfirmed**

**Table 3. LC-MS/MS-UV Conditions Used to Analyze 24280**

| 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                        | <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                                                                                                                                                                                                                                                                            |              |  |           |    |              |       |   |     |        |               |     |           |     |     |           |   |     |