

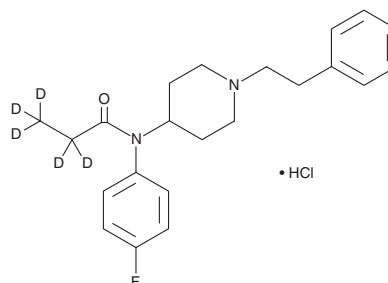
# CONFIRMATION of ANALYSIS



ACCREDITED  
ISO 17034 #AR-1774

## *para*-Fluorofentanyl-d<sub>5</sub> (hydrochloride)

Reference Material



|                       |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |
|-----------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Item No.:             | 19597                                                                                                                                                                                                                                                                                                                                                                                                                                                                          |
| Batch No.:            | 0484763                                                                                                                                                                                                                                                                                                                                                                                                                                                                        |
| CAS Registry No.:     | 2747915-22-4                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |
| Molecular Formula:    | C <sub>22</sub> H <sub>22</sub> D <sub>5</sub> FN <sub>2</sub> O • HCl                                                                                                                                                                                                                                                                                                                                                                                                         |
| Formula Weight:       | 396.0 amu                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
| UV λ <sub>max</sub> : | 202, 259 nm                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
| Expiry Date:          | 06JUL2031 (valid from date of certification)                                                                                                                                                                                                                                                                                                                                                                                                                                   |
| Supplied as:          | A neat solid                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |
| Storage:              | Unopened at -20°C ± 10°C                                                                                                                                                                                                                                                                                                                                                                                                                                                       |
| Safety:               | Refer to Safety Data Sheet                                                                                                                                                                                                                                                                                                                                                                                                                                                     |
| Intended Use:         | For analytical testing purposes only, not intended for human or animal use.                                                                                                                                                                                                                                                                                                                                                                                                    |
| Instructions for Use: | Store reference materials away from light, away from sources of heat, and in dry conditions. Once opened this material should be minimally exposed to ambient conditions and returned to recommended storage conditions immediately after use. Ongoing stability testing supports a negligible decrease in purity over a series of thaw-refreeze cycles. It is recommended that laboratories perform periodic testing to verify the material remains fit for the intended use. |

Approval:   
Roxanne Franckowski

Title: Senior Manager of ISO Quality Confirmation Date: 06JUL2016

Cayman Chemical certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration date when stored unopened as recommended.



# CONFIRMATION of ANALYSIS

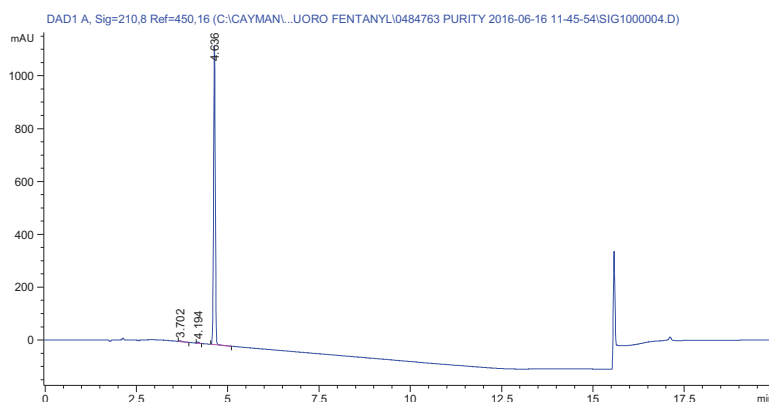


| Qualifier                    | Method                       | Result      |
|------------------------------|------------------------------|-------------|
| Appearance                   | Visual inspection            | White solid |
| Chromatographic Purity, HPLC | Cayman Method TST SD139      | 99.29%      |
| Identity, LC-MS              | Cayman Method TST SD13, +ESI | 360.4 amu   |
| Identity, GC-MS              | Cayman Method TST SD12       | Conforms    |
| Identity, FTIR               | Cayman Method TST SD03       | Conforms    |
| % LOD                        | Cayman Method TST SD24       | 0.25%       |
| % ROI                        | Cayman Method TST SD06       | <0.10%      |
| Identity, NMR                | <sup>1</sup> H NMR           | Conforms    |

Appearance, NMR and optical rotation (if applicable) are provided as supplemental information but are not within scope of ISO accreditation.

## Supplemental Data (Neat Material)

### HPLC-UV



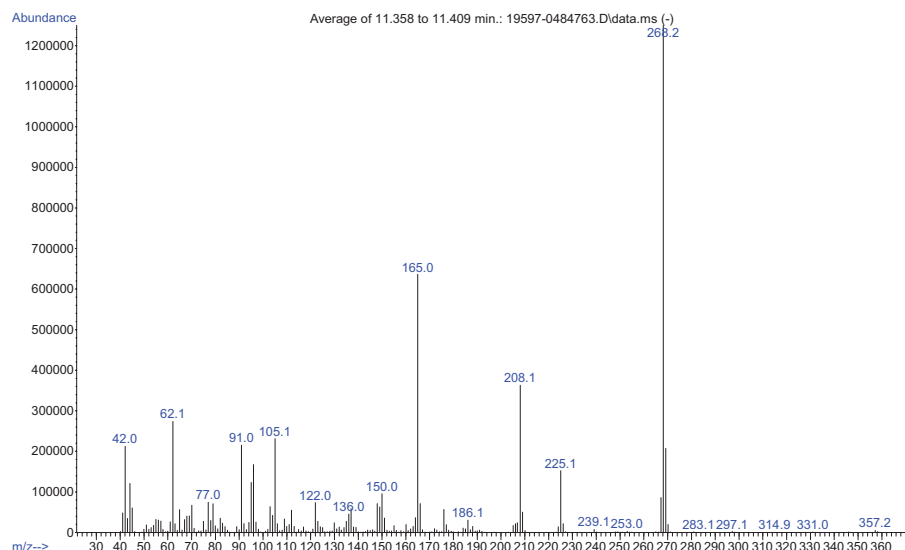
### Conditions

| Instrument   | Agilent 1100/1200 Series                                                                                                                                                                                                          |            |    |      |        |       |     |         |     |
|--------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------|----|------|--------|-------|-----|---------|-----|
| Column       | 4.6 x 150 mm, 5 µm Gemini C18                                                                                                                                                                                                     |            |    |      |        |       |     |         |     |
| Mobile Phase | A: 0.025% Trifluoroacetic acid in water<br>B: Acetonitrile                                                                                                                                                                        |            |    |      |        |       |     |         |     |
| Gradient     | <table border="1"> <thead> <tr> <th>Time (min)</th> <th>%B</th> </tr> </thead> <tbody> <tr> <td>0-10</td> <td>20-95%</td> </tr> <tr> <td>10-13</td> <td>95%</td> </tr> <tr> <td>13.1-20</td> <td>20%</td> </tr> </tbody> </table> | Time (min) | %B | 0-10 | 20-95% | 10-13 | 95% | 13.1-20 | 20% |
| Time (min)   | %B                                                                                                                                                                                                                                |            |    |      |        |       |     |         |     |
| 0-10         | 20-95%                                                                                                                                                                                                                            |            |    |      |        |       |     |         |     |
| 10-13        | 95%                                                                                                                                                                                                                               |            |    |      |        |       |     |         |     |
| 13.1-20      | 20%                                                                                                                                                                                                                               |            |    |      |        |       |     |         |     |
| Flow Rate    | 1 ml/min                                                                                                                                                                                                                          |            |    |      |        |       |     |         |     |
| Column Temp  | 30°C                                                                                                                                                                                                                              |            |    |      |        |       |     |         |     |
| Wavelength   | UV monitored at 210 nm                                                                                                                                                                                                            |            |    |      |        |       |     |         |     |

# CONFIRMATION of ANALYSIS



## GC-MS

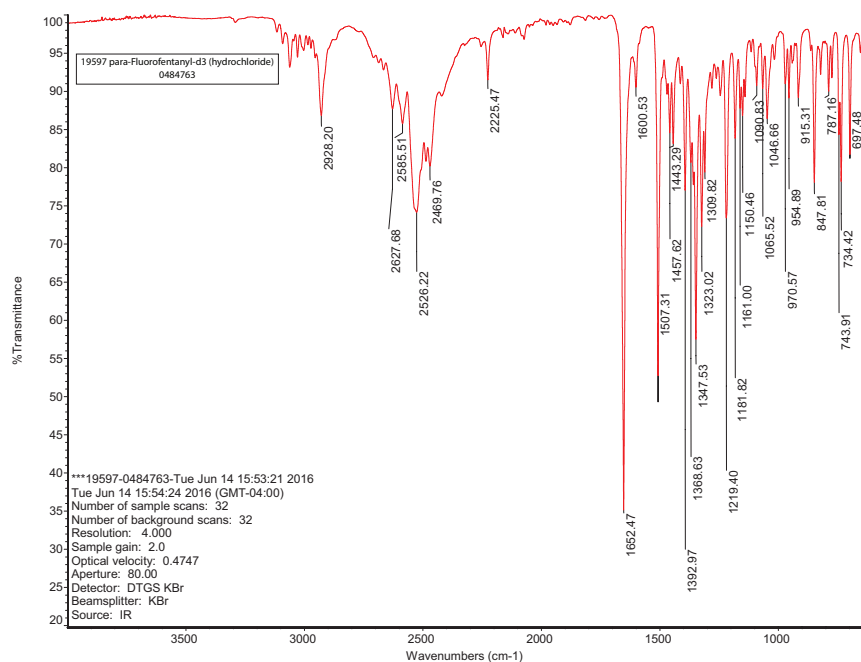


## Conditions

|                    |                                                                                   |
|--------------------|-----------------------------------------------------------------------------------|
| Instrument         | Agilent GC MSD                                                                    |
| Column             | 30 m x 0.32 mm,<br>0.5 µm Rtx-5MS                                                 |
| Carrier Gas        | He                                                                                |
| Flow Rate          | 2 ml/min                                                                          |
| Inlet Temp         | 300°C                                                                             |
| Split Ratio        | 15:1                                                                              |
| Oven Program       | 50°C hold for 1 min, ramp<br>to 300°C at 30°C per min,<br>hold at 300°C to 15 min |
| Transfer Line Temp | 300°C                                                                             |
| Voltage            | 70eV EI MS                                                                        |
| Scan Range         | 40-600 m/z                                                                        |
| Tune File          | stune                                                                             |

Apex spectrum – background (1 min window in front of peak)

## FTIR



## Conditions

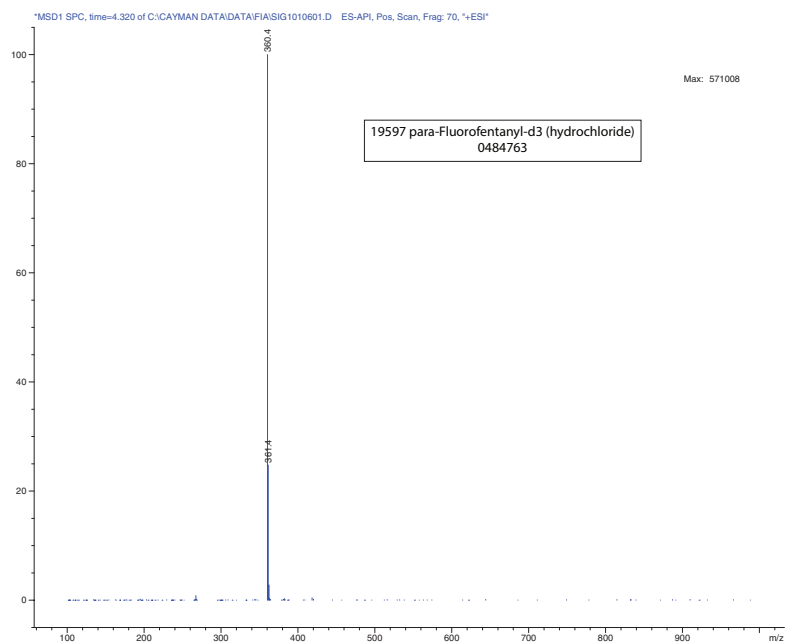
|            |                                                                   |
|------------|-------------------------------------------------------------------|
| Instrument | Thermo Nicolet iS10 FTIR /<br>Diamond SmartATR (single<br>bounce) |
| Scans      | 32 scans /<br>32 background scans                                 |
| Range      | 650-4,000 cm <sup>-1</sup>                                        |
| Resolution | 4.000                                                             |

ATR and background corrected

# CONFIRMATION of ANALYSIS



## ESI-MS



## Conditions

|                      |                                                |
|----------------------|------------------------------------------------|
| Instrument           | Agilent HPLC MSD                               |
| Mobile Phase         | 50:50:0.1<br>MeOH/H <sub>2</sub> O/Acetic acid |
| Flow Rate            | 0.5 ml/min                                     |
| Ionization Mode      | +ESI                                           |
| Mass Range           | 100-1,000 m/z                                  |
| Nebulizer            | 60 psi                                         |
| Desolvation Gas      | 13 L/min                                       |
| Desolvation Temp     | 350°C                                          |
| Electrospray Voltage | 4kV                                            |

MS collected across peak width at half height

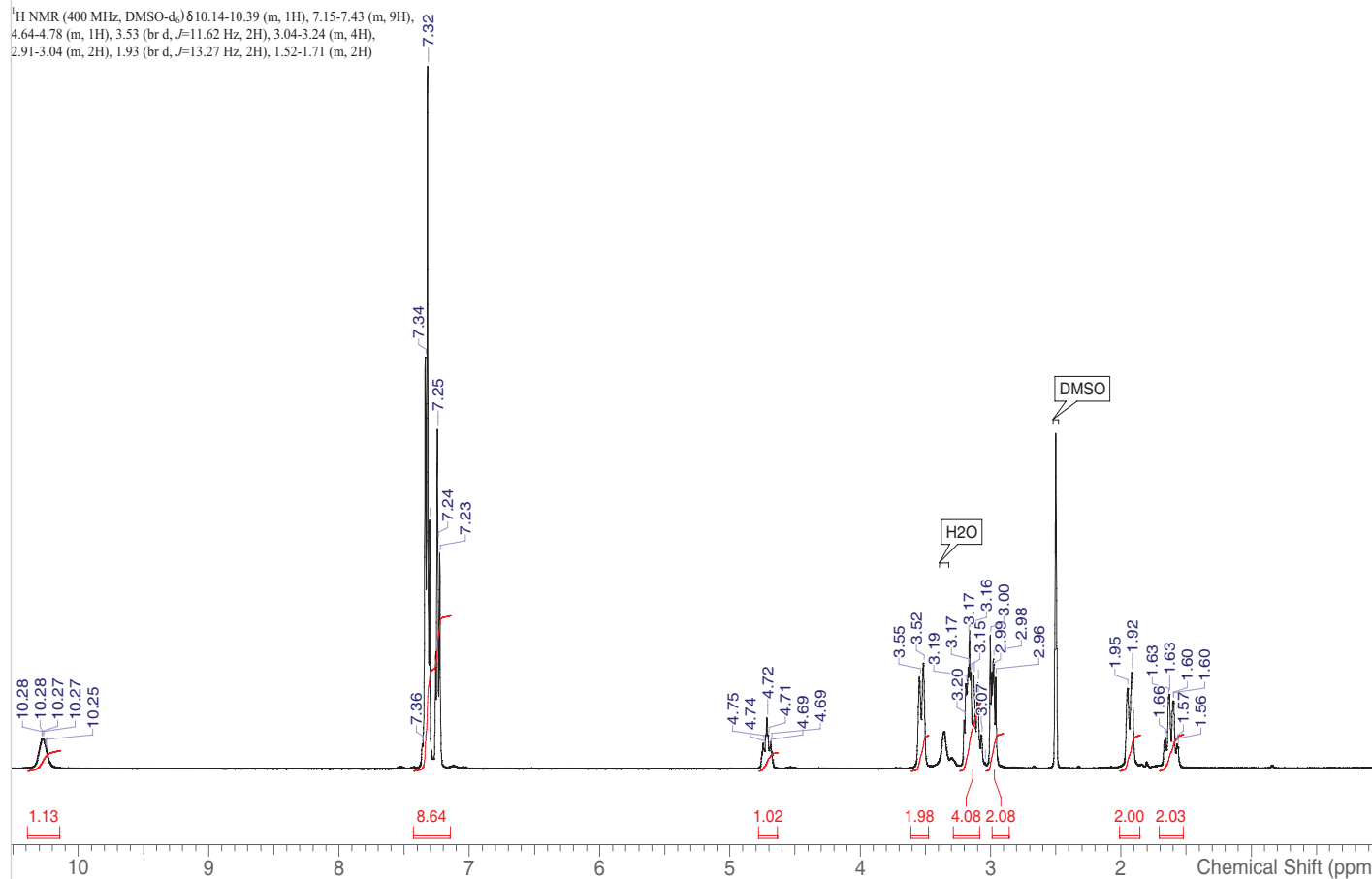
# CONFIRMATION of ANALYSIS



## NMR (not within scope of ISO accreditation)

|                  |                                                                                                           |                               |                |
|------------------|-----------------------------------------------------------------------------------------------------------|-------------------------------|----------------|
| <b>File Name</b> | \\sulfur\private\nmrdata\PD-1399-169A_20160606_PD-1399-169A_20160606_01\PD-1399-169A_20160606_001.fid.tif |                               |                |
| <b>Date</b>      | Jun 6 2016                                                                                                | <b>Nucleus</b>                | <sup>1</sup> H |
| <b>Solvent</b>   | DMSO-d <sub>6</sub>                                                                                       | <b>Number of Transients</b>   | 64             |
|                  |                                                                                                           | <b>Frequency (MHz)</b>        | 399.9682       |
|                  |                                                                                                           | <b>Temperature (degree C)</b> | 26.000         |

<sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>) δ 10.14-10.39 (m, 1H), 7.15-7.43 (m, 9H), 4.64-4.78 (m, 1H), 3.53 (br d, J=11.62 Hz, 2H), 3.04-3.24 (m, 4H), 2.91-3.04 (m, 2H), 1.93 (br d, J=13.27 Hz, 2H), 1.52-1.71 (m, 2H)



### Conditions

|                   |                         |
|-------------------|-------------------------|
| <b>Instrument</b> | Varian Inova 400MHZ NMR |
| <b>Scans</b>      | 64 scans                |

### Homogeneity

A minimum sample size of 1.5 µg was used to determine homogeneity of the bulk solid. The recommended minimum quantity for use is 1.5 µg. Quantities below this have not been evaluated.

### Short-Term Stability

No decrease in the purity was observed at ambient or 60°C after two weeks. This data supports shipping of this product at ambient temperature.

### Long-Term Stability

Long-term stability data predicts 15 years stability at the -20°C storage temperature. Long-term stability studies are ongoing and the Certificate of Analysis will be updated upon study completion.

# CONFIRMATION of ANALYSIS



## Quality Standard Documentation

The manufacturer of this Reference Material is accredited under ISO 17034:2016 accreditation issued by ANAB. Refer to ANAB certificate and scope of accreditation AR-1774.

The manufacturer of this Reference Material is accredited under ISO/IEC 17025:2017 accreditation issued by ANAB. Refer to the ANAB certificate and scope of accreditation AT-1773.

## Revision History

| Revision No. | Date      | Reason for Revision                                        |
|--------------|-----------|------------------------------------------------------------|
| 01           | 06JUL2016 | Initial version                                            |
| 02           | 10JUN2019 | Expiry date extension                                      |
| 03           | 23JUN2020 | Expiry date extension                                      |
| 04           | 20JUL2021 | Expiry date extension                                      |
| 05           | 26JUL2022 | Updated format, accreditation symbols, and expiry date     |
| 06           | 02AUG2023 | Updated format to version 5.1, CAS number, and expiry date |

## Disclaimers

### Material Safety Data

This material should be considered hazardous until information to the contrary becomes available. Do not ingest, swallow, or inhale. Do not get in eyes, on skin, or on clothing. Wash thoroughly after handling. This information contains some but not all of the information required for the safe and proper use of this material. Before use, review the complete Safety Data Sheet, which has been sent *via* email to your institution.

### Warranty and Limitation of Remedy

Cayman Chemical Company makes no warranty or guarantee of any kind, whether written or oral, expressed or implied, including without limitation, any warranty of fitness for a particular purpose, suitability and merchantability, which extends beyond the description of the chemicals hereof. Cayman warrants only to the original customer that the material will meet our specifications at the time of delivery.

Cayman will carry out its delivery obligations with due care and skill. Thus, in no event will Cayman have any obligation or liability, whether in tort (including negligence) or in contract, for any direct, indirect, incidental or consequential damages, even if Cayman is informed about their possible existence.

This limitation of liability does not apply in the case of intentional acts or negligence of Cayman, its directors or its employees.

Buyer's exclusive remedy and Cayman's sole liability hereunder shall be limited to a refund of the purchase price, or at Cayman's option, the replacement, at no cost to Buyer, of all material that does not meet our specification.

Said refund or replacement is conditioned on Buyer giving written notice to Cayman within thirty (30) days after arrival of the material at its destination. Failure of Buyer to give said notice within thirty (30) days shall constitute a waiver of Buyer of all claims hereunder with respect to said material.

For further details, please refer to our Warranty and Limitations of Remedy located on our website and in our catalog.

This Certificate shall not be reproduced except in full, without written approval from the Cayman Chemical ISO Quality Manager.

ISO CRT SD01 v 5.1

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