

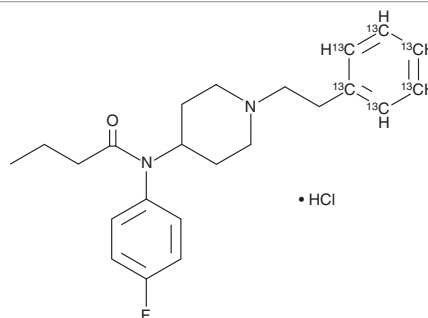
CONFIRMATION of ANALYSIS



ACCREDITED
ISO 17034 #AR-1774

para-Fluorobutyl fentanyl- $^{13}\text{C}_6$ (hydrochloride)

Reference Material



Item No.:	26216
Batch No.:	0545297
Molecular Formula:	$\text{C}_{17}[^{13}\text{C}]_6\text{H}_{29}\text{FN}_2\text{O} \cdot \text{HCl}$
Formula Weight:	410.90 amu
UV λ_{max} :	202, 259 nm
Expiry Date:	03DEC2033 (valid from date of certification)
Supplied as:	A neat solid
Storage:	Unopened at $-20^\circ\text{C} \pm 10^\circ\text{C}$
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: 03DEC2018

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.



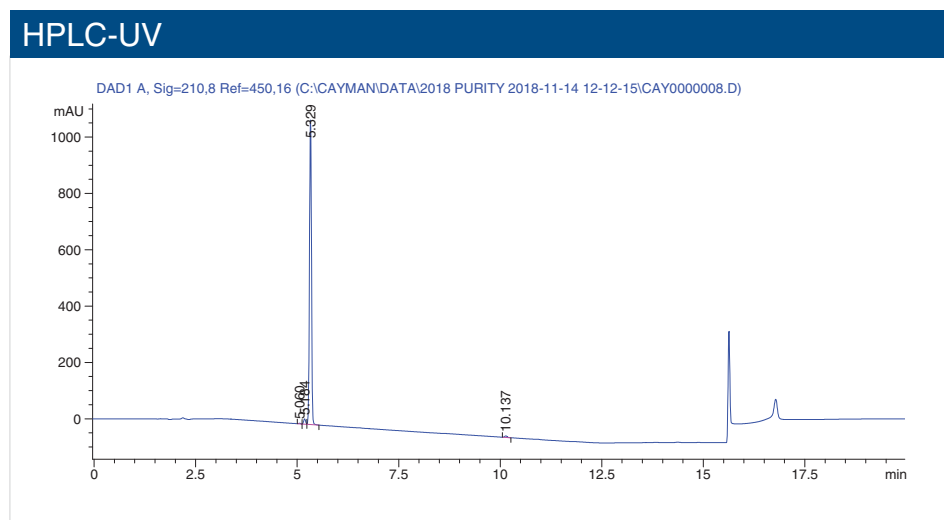
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Qualifier	Method	Result
Appearance	Visual inspection	White solid
Chromatographic Purity, HPLC	Cayman Method TST SD139	97.60%
Identity, LC-MS	Cayman Method TST SD13, +ESI	375.2 amu
Identity, GC-MS	Cayman Method TST SD12	Conforms
Identity, FTIR	Cayman Method TST SD03	Conforms
% LOD	Cayman Method TST SD24	0.16%
% ROI	Cayman Method TST SD06	<0.10%
Isotopic Purity, LC-MS	Cayman Method TST SD154	<0.10% $^{13}\text{C}_0$ vs $^{13}\text{C}_6$
Identity, NMR	^1H 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)

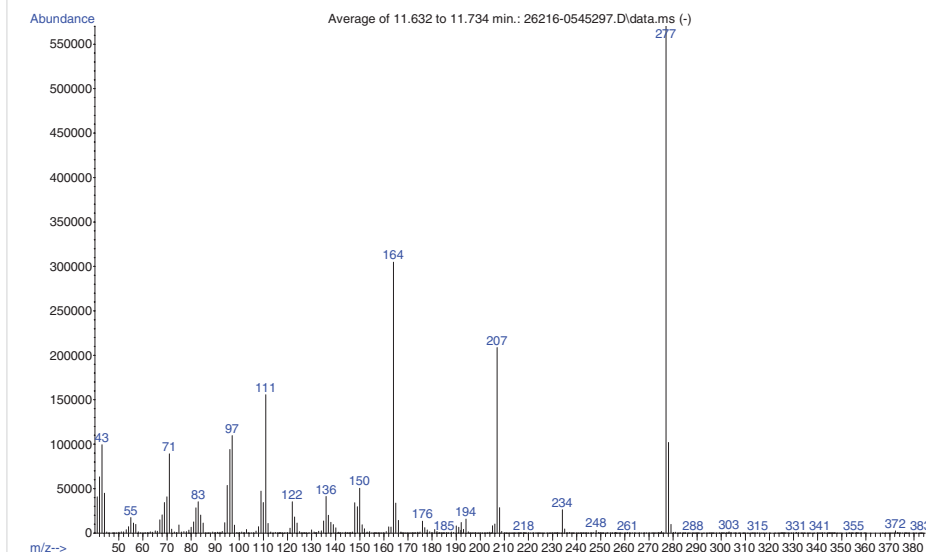


Conditions									
Instrument	Agilent 1100/1200 Series								
Column	4.6 x 150 mm, 5 μm Gemini C18								
Mobile Phase	A: 0.025% Trifluoroacetic Acid B: Acetonitrile								
Gradient	<table border="1"> <thead> <tr> <th>Time (min)</th> <th>%B</th> </tr> </thead> <tbody> <tr> <td>0-10</td> <td>20-95%</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								

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GC-MS

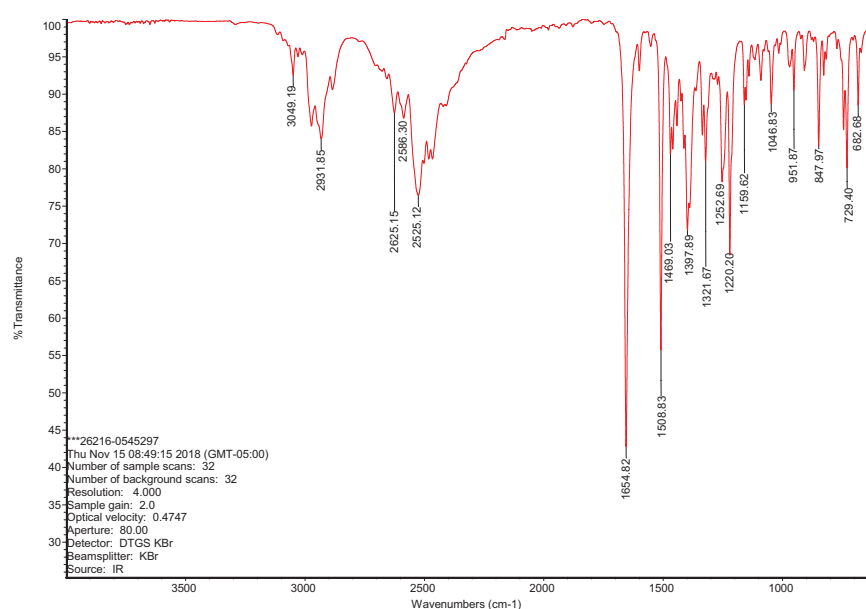


Conditions

Instrument	Agilent GC MSD
Column	30 m x 0.32 mm, 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 to 300°C at 30°C per min, 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

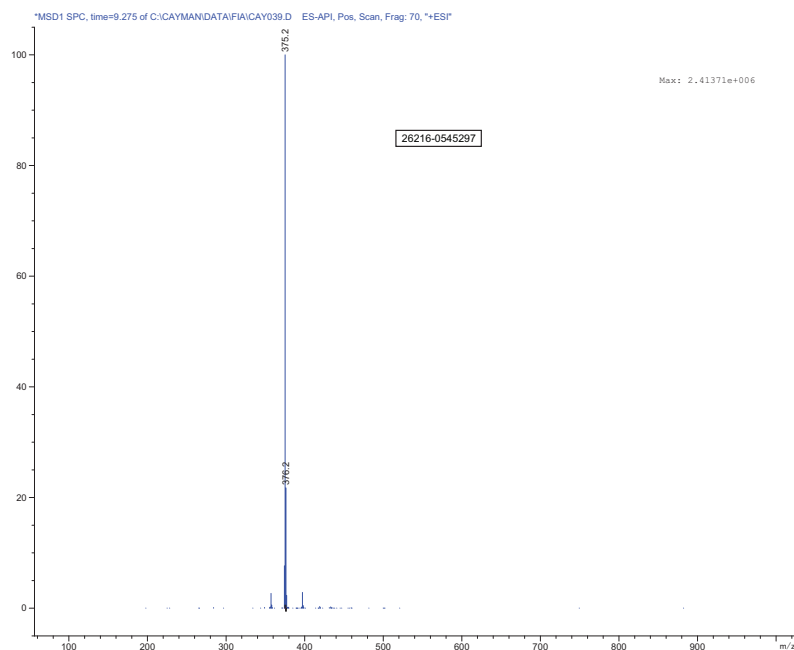
Instrument	Thermo Nicolet iS10 FTIR / Diamond SmartATR (single bounce)
Scans	32 scans / 32 background scans
Range	650-4,000 cm ⁻¹
Resolution	4.000

ATR and background corrected

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ESI-MS



Conditions

Instrument	Agilent HPLC MSD
Mobile Phase	50:50:0.1 Methanol/Water/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

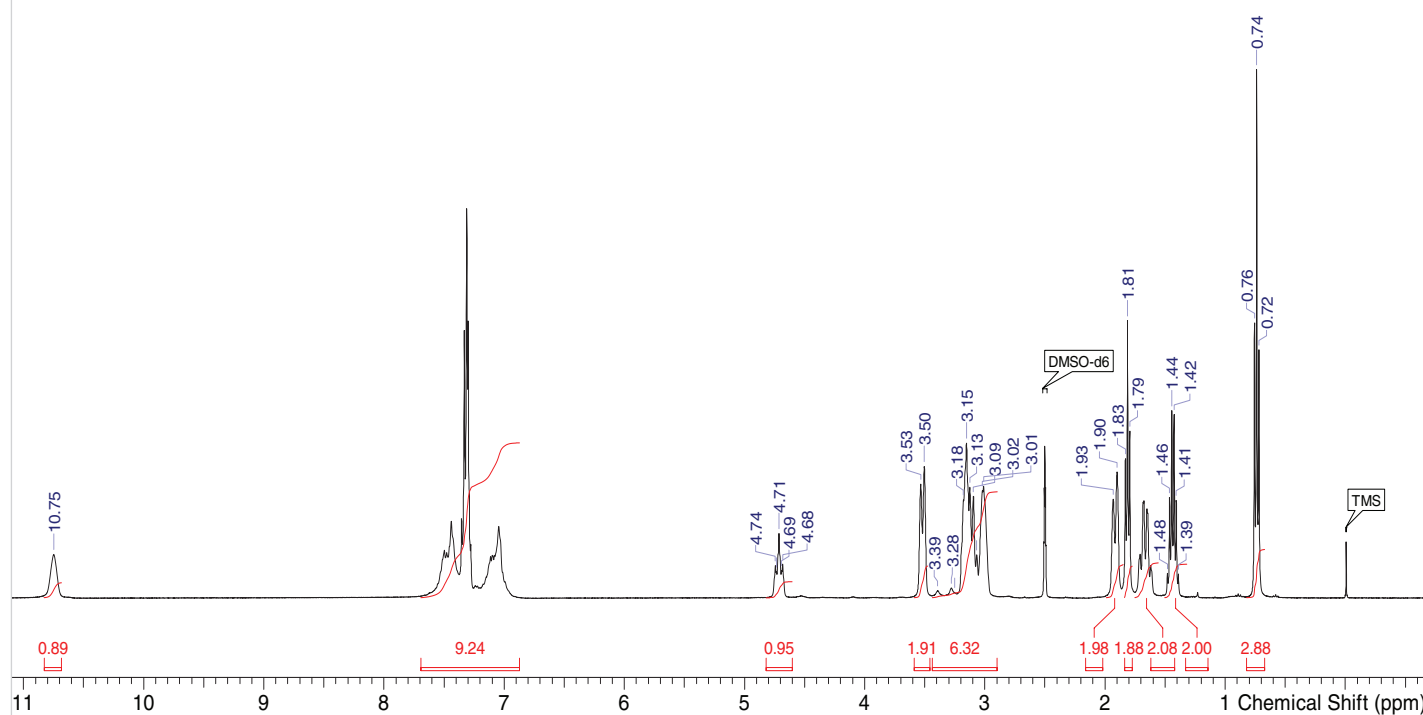
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NMR (not within scope of ISO accreditation)

Date	Nov 12 2018			
File Name	\\sulfur\private\nmrdata\ 2018\26216-0545297_ 20181112_ V26216-0545297_ 20181112_ 01\26216-0545297_ 20181112_ 001.fid\fid			
Frequency (MHz)	399.9680	Nucleus	1H	Number of Transients 32
Solvent	DMSO-d6	Temperature (degree C)	27.000	

¹H NMR (DMSO-d₆, 400 MHz) δ 10.75 (br s, 1H), 6.9-7.7 (m, 9H), 4.6-4.8 (m, 1H), 3.52 (br d, 2H, J=11.7 Hz), 2.9-3.4 (m, 6H), 1.92 (br d, 2H, J=12.5 Hz), 1.81 (t, 2H, J=7.2 Hz), 1.6-1.8 (m, 2H), 1.43 (sxt, 2H, J=7.3 Hz), 0.74 (t, 3H, J=7.4 Hz)



Conditions

Instrument	Varian Inova 400MHZ NMR
Scans	32 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	03DEC2018	Initial version
02	24JUN2020	Expiry date extension
03	10SEP2021	Updated format, accreditation symbols, and expiry date
04	09AUG2022	Expiry date extension
05	24JUL2023	Updated format to version 5.0, expiry date, and HPLC image

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 5.0

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